Prepared for
**Nevada Environmental Response Trust**
**Henderson, Nevada**

Prepared by
**Ramboll Environ US Corporation**
**Emeryville, California**

Project Number
**21-41400C**

Date
**October 26, 2017**

# QUALITY ASSURANCE PROJECT PLAN, REVISION 2

## NEVADA ENVIRONMENTAL RESPONSE TRUST SITE HENDERSON, NEVADA

RAMBOLL ENVIRON

# Quality Assurance Project Plan, Revision 2

**Nevada Environmental Response Trust**
**(Former Tronox LLC Site)**
**Henderson, Nevada**

**Nevada Environmental Response Trust (Trust) Representative Certification**
I certify that this document and all attachments submitted to the Division were prepared at the request of, or under the direction or supervision of the Trust. Based on my own involvement and/or my inquiry of the person or persons who manage the system(s) or those directly responsible for gathering the information or preparing the document, or the immediate supervisor of such person(s), the information submitted and provided herein is, to the best of my knowledge and belief, true, accurate, and complete in all material respects.

Office of the Nevada Environmental Response Trust

Le Petomane XXVII, Inc., not individually, but solely in its representative capacity as the Nevada Environmental Response Trust Trustee

**Signature:** Jay A Steinberg, not individually, but solely in his representative capacity as President of the Nevada Environmental Response Trust Trustee

**Name:** Jay A. Steinberg, not individually, but solely in his representative capacity as President of the Nevada Environmental Response Trust Trustee

**Title:** Solely as President and not individually

**Company:** Le Petomane XXVII, Inc., not individually, but solely in its representative capacity as the Nevada Environmental Response Trust Trustee

**Date:** 10/24/17

# Quality Assurance Project Plan, Revision 2

**Nevada Environmental Response Trust**
**(Former Tronox LLC Site)**
**Henderson, Nevada**

**Responsible Certified Environmental Manager (CEM) for this project**

I hereby certify that I am responsible for the services described in this document and for the preparation of this document. The services described in this document have been provided in a manner consistent with the current standards of the profession and, to the best of my knowledge, comply with all applicable federal, state and local statutes, regulations and ordinances.

_____ 10/26/2017
_____

**John M. Pekala, PG** Date
**Senior Manager**

Certified Environmental Manager
Ramboll Environ US Corporation
CEM Certificate Number: 2347
CEM Expiration Date: September 20, 2018

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|---|---|
| **Project Title** | Quality Assurance Project Plan (QAPP) |
| **Site Name** | Nevada Environmental Response Trust Site |
| **Site Location** | Henderson, Nevada |
| **Company** | Ramboll Environ US Corporation |
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QAPP Reviewed and Approved:

_____ Date: October 26, 2017

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## APPENDICES

## ACRONYMS AND ABBREVIATIONS

| | |
|---|---|
| BMI | Black Mountain Industrial |
| CD | Compact Disc |
| CEM | Certified Environmental Manager |
| CFR | Code of Federal Regulations |
| CSM | Conceptual Site Model |
| °C | Degrees Celsius |
| DI | Deionized |
| DO | Dissolved Oxygen |
| DQI | Data Quality Indicator |
| DQO | Data Quality Objective |
| DVSR | Data Validation Summary Report |
| EMSL | EMSL Analytical, Inc. |
| EB | Equipment Blank |
| EDD | Electronic Data Deliverable |
| FB | Field Blank |
| FD | Field Duplicate |
| FSP | Field Sampling Plan |
| GES | Geotechnical & Environmental Services, Inc. |
| GWETS | Groundwater Extraction and Treatment System |
| HASP | Health and Safety Plan |
| HRA | Health Risk Assessment |
| ICP | Inductively Coupled Plasma |
| ID | Identification |
| ISRACR | Interim Soil Removal Action Completion Report |
| LCS | laboratory control sample |
| LCSD | laboratory control sample duplicates |
| LDC | Laboratory Data Consultants, Inc. |
| MDA | Minimum Detectable Activity |
| MDL | Method Detection Limit |
| Microbial Insights | Microbial Insights, Inc. |
| µm | Micrometer |
| mL | Milliliter |
| MPA | Masters of Public Affairs |

| | |
|---|---|
| MPH | Masters of Public Health |
| MS/MSD | Matrix Spike/Matrix Spike Duplicate |
| NDEP | Nevada Division of Environmental Protection |
| NELAC | National Environmental Laboratory Accreditation Conference |
| NERT | Nevada Environmental Response Trust |
| ORP | Oxygen Reduction Potential |
| OSHA | Occupational Safety and Health Administration |
| OVM | Organic Vapor Meter |
| PDF | Portable Data Format |
| PE | Professional Engineer |
| PG | Professional Geologist |
| PID | photoionization detector |
| PM | Project Manager |
| PTS | PTS Laboratories, Inc. |
| PQL | Practical Quantitation Limit |
| QA | Quality Assurance |
| QAM | Quality Assurance Manual |
| QAPP | Quality Assurance Project Plan |
| QC | Quality Control |
| %R | Percent Recovery |
| Ramboll Environ | Ramboll Environ US Corporation |
| RI/FS Work Plan | Remedial Investigation and Feasibility Study Work Plan |
| RISB | Remedial Investigation Soil Boring |
| RISG | Remedial Investigation Soil Gas Samples |
| RIT | Trench Samples |
| RL | Reporting Limit |
| RPD | Relative Percent Difference |
| RPM | Remedial Project Manager |
| %RSD | Percent Relative Standard Deviation |
| SDG | Sample Delivery Group |
| Silver State | Silver State Analytical Laboratories, Inc. |
| Site | Nevada Environmental Response Trust (NERT) Site |
| SMP | Site Management Plan |
| SOP | Standard Operating Procedure |

| | |
|---|---|
| TB | Trip Blank |
| Trust | Nevada Environmental Response Trust |
| UMCf | Upper Muddy Creek Formation |
| USEPA | U.S. Environmental Protection Agency |
| VOC | Volatile Organic Compounds |
| ZVI | Zero Valent Iron |

## DISTRIBUTION LIST

This QAPP will be distributed to the entities listed below. The QAPP may also be distributed to other project personnel including, but not limited to, client representatives and consultants, analytical laboratories, remediation contractors, and subcontractors, as needed.

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Project personnel from the organizations listed above are responsible for having the most recent version of this QAPP. The parties should contact Ramboll Environ's Project Manager or Project Quality Assurance/Quality Control Officer for the most recent version. Individual pages include a revision number; any revised pages will be clearly marked with a new revision number and a list of revised pages will be distributed with any revisions.

# 1. PROJECT MANAGEMENT/DATA QUALITY OBJECTIVES

## 1.1 Introduction

On behalf of the Nevada Environmental Response Trust (the Trust) and under the direction of the Nevada Division of Environmental Protection (NDEP), Ramboll Environ US Corporation (Ramboll Environ) prepared this Quality Assurance Project Plan (QAPP) to describe the quality assurance/quality control (QA/QC) procedures and performance criteria applicable to data collection tasks associated with the Remedial Investigation and Feasibility Study (RI/FS), including, but not limited to, field investigations, laboratory treatability studies, and field treatability/pilot studies for the Nevada Environmental Response Trust (NERT) Site located in Clark County, Nevada (the Site).

The purpose of this QAPP is to (1) describe the QA/QC procedures that the project team will follow during sampling and analysis; and (2) specify methods, performance criteria, and protocols to produce data that are representative of field conditions, meet the established data quality objectives (DQOs), and are of acceptable quality to meet industry standards. As stated above, the QAPP is intended to apply to tasks related to the RI/FS. This revised QAPP is not intended to be applicable to the remedial performance groundwater monitoring program, data collection activities associated with permit compliance, data collection associated with operation of the Groundwater Extraction and Treatment System (GWETS), or any other non-RI/FS data collection activity. Groundwater remedial performance monitoring is performed in accordance with the Remedial Performance Groundwater Sampling and Analysis Plan (Ramboll Environ 2017b).

This revision to the QAPP replaces the prior version of the QAPP, which was submitted along with the Field Sampling Plan (FSP) and the Health and Safety Plan (HASP) as part of a combined *Sampling and Analysis Plan (SAP), Revision 1* dated July 18, 2014 (approved by NDEP on August 1, 2014). A revision to the QAPP is necessary at this time for three primary reasons:

1. The prior version of the QAPP was written to cover only those activities described in the RI/FS Work Plan, Revision 2 (ENVIRON 2014a) and the FSP, Revision 1 (ENVIRON 2014b). Additional phases of investigation and treatability studies that have been planned since the development of the prior QAPP need to be incorporated.
2. Since the submittal of the prior QAPP, the Site has been expanded through the incorporation of the Downgradient Study Area, the Eastside Area, and the Northeast Area. The incorporation of these areas into the RI/FS require changes to procedures, methods, performance criteria, and/or protocols specified in the QAPP.
3. The QAPP needs to be structured in a manner that is easy to update in order to incorporate future RI/FS data collection tasks. This will be accomplished by preparing QAPP Addenda, the structure of which are defined in this QAPP revision.

The QAPP will be *implemented* in conjunction with the following RI/FS project-specific documents:

- The RI/FS Work Plan, Revision 2 (ENVIRON 2014a);
- The RI Data Evaluation Technical Memorandum (Ramboll Environ 2016a)
- The RI/FS Work Plan Addendum: Phase 3 Remedial Investigation (Ramboll Environ 2017a);

- Groundwater Sampling Plan Downgradient Study Area (AECOM 2016a);
- Surface Water Sampling Plan Downgradient Study Area (AECOM 2016b);
- The Surface Water Investigation Plan Downgradient Study Area (AECOM 2016c); and
- The Unit 4 and 5 Buildings Investigation Work Plan (Tetra Tech 2015).

The project-specific documents contain a description of the investigation activities to be performed at the Site and specify the methods and procedures to be used to collect representative samples. Collectively, these documents will be referred to as the "RI/FS Work Plans" throughout this QAPP.

In addition, this QAPP will be implemented in conjunction with current treatability studies that are on-going at the Site and are related to RI/FS data collection tasks. Project-specific details regarding current treatability studies are specified in the following documents:

- In-Situ Chromium Treatability Study Work Plan (Tetra Tech 2016a); and
- Final Seep Well Field Area Bioremediation Treatability Study Work Plan (Tetra Tech 2016b).

Sampling details necessary to complete future RI/FS tasks that are not currently addressed in project-specific documents will be specified in task-specific work plans and the QAPP will be modified through the use of task-specific addenda.

Certain other documents are referenced herein as necessary to describe activities performed pursuant to the Interim Consent Agreement (Agreement) for the Site, effective February 14, 2011. These include the Interim Soil Removal Action Completion Report (ISRACR) (ENVIRON 2012), Annual Groundwater Monitoring Reports (Annual Reports; e.g. Ramboll Environ 2016b), and the Site Management Plan (SMP), Revision 3 (Ramboll Environ 2017c).

This QAPP has been prepared in general accordance with the applicable elements of several United States Environmental Protection Agency (USEPA) guidance documents, including *Guidance on Systematic Planning Using the Data Quality Objectives Process*, EPA QA/G-4 (USEPA, 2006); *EPA Requirements for Quality Assurance Project Plans*, EPA QA/R-5 (USEPA, 2001); *Guidance for Quality Assurance Project Plans*, EPA QA/G-5 (USEPA, 2002) and *Region 9 Guidance for Quality Assurance Program Plans*, EPA R9QA/03.2 (USEPA, 2012).

## 1.2 QAPP Organization

This plan is provided in both hard copy and electronic forms. Where electronic files are referenced or information is stated as provided on compact disc (CD), this information is contained on the CD attached to the hard copy document.

The main body of the QAPP (Sections 1 – 4) provide overall DQOs, general procedures and protocols, and baseline performance criteria applicable to all RI/FS collection tasks. Section 5 describes task-specific modules that will be employed to prepare QAPP Addenda and identify variances for future scopes of work (e.g. additional investigations, treatability studies, pilot studies, etc.).

This QAPP is organized as follows:

- **Section 1** presents the purpose, objectives, and organization of the QAPP.
- **Section 2** provides guidance for measurement and data acquisition.
- **Section 3** describes the requirements for assessment and oversight.

- **Section 4** describes the requirements for data validation and data usability.
- **Section 5** describes the procedure for preparing QAPP Addenda.
- **Section 6** lists citations for key documents referenced in the QAPP.

## 1.3 QAPP Objectives and Use

The overall goal of the QAPP is to outline the procedures, methods, and other specifications a site investigation/monitoring project will use to ensure that the samples are collected and analyzed, the data are stored and managed, and the reporting of data are of high enough quality to meet project needs. Quality Assurance (QA) and Quality Control (QC) are activities undertaken to achieve this goal. QA is generally understood to be more comprehensive than QC. QA can be defined as the integrated system of activities that ensures that a project meets defined standards.

QC is the basic building block of data quality. It starts with activities whose purpose is to control quality at the source by finding problems and defects. At its simplest, QC is inspecting, testing or checking data to make sure it is correct, valid, or otherwise in accordance with established specifications. The intent is to identify data that is not correct, and either correct or eliminate it, to make sure all data conforms to the specifications, and/or functions as required. QC does not ensure quality, it only finds instances where quality is absent or below established criteria.

QA asserts that data quality can be improved by looking 'further up the line'. It is aimed at preventing nonconforming or invalid data. QA can be defined as the integrated system of activities that ensures that a project meets defined standards. QA still has QC at its core to control data quality, but it goes beyond testing or inspection to also consider related activities or processes (such as training, document control and audits) that may be resulting in systemic and recurring data quality issues. The overall goal of the QA/QC procedures and specifications established in this QAPP is to ensure that comparable and representative data are produced during the implementation of the RI/FS data collection tasks and that data quality is consistently assessed and documented with respect to its precision, accuracy, sensitivity, and completeness. The specific QAPP objectives are to:

- Provide standardized methods and quality specifications for all anticipated field sampling, analysis, and data review procedures;
- Provide guidance and criteria for selected field and analytical procedures; and
- Establish procedures for reviewing and documenting compliance with field and analytical procedures.

This QAPP documents the planning, implementation, and assessment procedures for the QA/QC program to be followed for current RI/FS data collection tasks; including the following:

- Collecting soil, soil gas, surface water and groundwater samples,
- Conducting field analysis of water quality parameters
- Labeling and shipping samples to laboratories
- Documenting field activities
- Coordinating laboratory services

- Reviewing and validating laboratory data
- Preparing data validation summary reports
- Submitting finalized, validated data

The QAPP will be expanded if further sampling work activities or analyses are identified. Similarly, should the list of chemicals of interest change, this QAPP will be modified to reflect those changes.

## 1.4 Project Organization/Roles and Responsibilities

Implementation of the approved QAPP requires the involvement of a wide range of individuals and organizations working together as a team. The project organization, and roles and responsibilities of the individuals involved, are defined in the QAPP to promote a clear understanding of the role that each party plays and to provide the lines of authority and reporting for the project. Personnel assigned to the project will be required to familiarize themselves with pertinent protocols and procedures presented in this QAPP. Key project positions relate to project oversight, project management, sampling and analytical data acquisition management, data validation management, and database management.

Ramboll Environ, AECOM, and Tetra Tech, on behalf of the Trust, will be responsible for implementing RI/FS tasks. Ramboll Environ is responsible for the direction of the Phase 1, 2, and 3 RI/FS Work Plan implementation as well as specific RI/FS treatability and pilot studies. AECOM is responsible for the Downgradient Study Area investigation. Tetra Tech is responsible for implementing the Unit 4/5 Investigation as well as specific RI/FS treatability studies, pilot studies, and surface water sampling activities. The consultants are all responsible for performing the scope of work as directed by the Trust to the satisfaction of NDEP and US EPA. The project organization/roles and responsibilities are summarized in the sections below. Appendix A contains a table of the current individuals participating in the project and their specific roles and responsibilities. Members of the project team are subject to change. A change in team members alone will not necessitate a revision to the QAPP; however Appendix A will be update as necessary.

### 1.4.1 Nevada Environmental Response Trust

#### *NERT Remediation Director*

The Trust will provide overall project coordination and will be responsible for communications with NDEP and neighboring property owners. The NERT Remediation Director directs all RI/FS activities performed by the Trust, communicates with the consultants and the NDEP Remedial Project Manager.

### 1.4.2 Nevada Division of Environmental Protection

#### *NDEP Remedial Project Manager*

The NDEP Remedial Project Manager (NDEP RPM) has overall responsibility for regulatory oversight of all phases of the project and will be responsible for reviewing and approving the QAPP.

### 1.4.3 Consultant Roles

#### *Project Manager*

The Project Manager (PM) is responsible for technical and policy decisions involving the project, including interaction and coordination with project staff, and NDEP. The PM is also responsible for reviewing the sampling program(s) and associated field activities for

compliance with the QAPP, including QA/QC, strategies, and review of all documents. The PM will have primary responsibility for project QA/QC and will evaluate and, if necessary, implement any corrective actions regarding data quality issues.

#### *Project Quality Assurance/Quality Control Officer*

The QA/QC Officer will enforce implementation of QA/QC procedures during the field sampling program and is responsible for reviewing the project QA/QC program as it relates to the collection and completeness of data from field and laboratory operations. During the contracting process the QA/QC Officer will ensure that method control limits are sufficient to meet this QAPP and are adequate for the use of the data. After receiving analytical results, the QA/QC Officer will evaluate the field and laboratory data against the requirements of the QAPP.

#### *Task Leaders*

Task Leaders are responsible for scope, cost, and technical considerations of the project; staff and project coordination; and implementation and review of overall project quality of the collection, completeness, and presentation of the data. If field conditions require modifications to protocol outlined in the QAPP, or if questions arise, the Task Leaders will be the primary contact for direction of field personnel. The Task Leaders will also be responsible for overseeing review of the QA/QC programs related to the compilation of data.

Field Task Leader

The Field Task Leader is responsible for overall implementation of the approved work plan, including work conducted by the Site contractor and is responsible for general oversight of field activities.

Health Risk Assessment (HRA) Task Leader

The HRA Task Leader will work with the other Task Leaders and QA Officer to ensure that work is conducted in compliance with health risk assessment objectives and applicable QA procedures.

Analytical Task Leader

The Analytical Task Leader is responsible for coordination with the analytical laboratories, review of analytical data, and tracking data through the data validation and reporting processes and will work with the other Task Leaders to ensure that work is conducted in compliance with project-specific objectives and applicable QA/QC procedures.

#### Database Administrator

The Database Administrator is responsible for working with the Analytical Task Leader to assist with review of analytical data, and tracking data through the data validation and reporting processes. The Database Administrator is responsible for preparing the data for electronic submission to the database and submitting finalized, validated data to NERT databases.

### 1.4.4 Analytical Laboratories

#### *Laboratory PMs*

Each Laboratory PM is the primary point-of-contact at the analytical laboratory for the project, and is responsible for ensuring project data meet the QA/QC objectives established herein. The Laboratory PM is also responsible for tracking the progress of testing in the laboratory and ensuring the timely delivery of data or other laboratory deliverables to the

project team. The laboratories used for chemical and radiochemical soil and groundwater testing will be certified by the State of Nevada for the analysis of interest. In the absence of Nevada certification for a particular analysis, as is the case for soil gas and asbestos, National Environmental Laboratory Accreditation Conference (NELAC) certification will be considered an acceptable substitute.

- **Laboratory Project Manager at TestAmerica Laboratories, Inc. (TestAmerica)**
  The primary subcontracted laboratory for soil, surface water, and groundwater analysis (with the exception of asbestos and organic acid analysis) for this project is TestAmerica's Irvine, California location. Because of the variety of specialized analyses required for this project, several additional Nevada-certified TestAmerica laboratories will be used during this project including the following TestAmerica laboratories: Sacramento, California; Denver, Colorado; Buffalo, New York; and St. Louis, Missouri. TestAmerica will also subcontract with ALS Environmental (Kelso, Washington) for arsenic speciation analysis. The Laboratory PM will coordinate with individual laboratory managers for this project. The primary laboratory may also subcontract analyses to other certified laboratories that can meet the requirements of this QAPP upon written approval of the PM or appropriate Analytical Task Leader and following consultation with NDEP.
- **Laboratory Project Manager at EMSL Analytical, Inc. (EMSL)**
  The primary subcontracted laboratory for asbestos analysis for this project is EMSL, which is a NELAC certified laboratory. Analysis for asbestos will take place at EMSL's laboratory in Cinnaminson, New Jersey.
- **Laboratory Project Manager at Silver State Analytical Laboratories, Inc. (Silver State)**
  Silver State will be subcontracted to analyze water samples for hexavalent chromium. The samples will be analyzed at Silver State's laboratory in Las Vegas, Nevada.
- **Laboratory Project Manager at Geotechnical & Environmental Services, Inc. (GES)**
  GES will be subcontracted for geotechnical analyses. The analyses will be performed at the GES laboratory located in Las Vegas, Nevada.
- **Laboratory Project Manager at PTS Laboratories, Inc. (PTS)**
  PTS will be subcontracted for geotechnical analyses. The analyses will be performed at the PTS laboratory located in Houston, Texas.
- **Laboratory Project Manager at Microbial Insights, Inc. (Microbial Insights)**
  Microbial Insights will be subcontracted for the analysis of phospholipid fatty acid analysis and perchlorate reductase by quantitative polymerase chain reaction. The analyses will be performed by the Microbial Insights laboratory in Knoxville, Tennessee.

### 1.4.5 Data Validation Subcontractors

***Data Validation Project Managers***

A Data Validation PM is responsible for validating and managing the data, including review of data from the laboratory at the appropriate level, adding any qualifiers to call-out differences between guidelines and the reported data, and preparing the data for electronic submission to the database. Consultants or their designee perform data validation. The following data validation subcontractors may perform data validation for the projects included in this QAPP:

- **Laboratory Data Consultants, Inc. (LDC), Data Validation Project Manager**
  LDC of Carlsbad, California will be providing data validation for soil, groundwater and soil gas samples collected for this project, with the exception of samples analyzed for asbestos.
- **Neptune and Company, Inc. (Neptune), Data Validation Project Manager**
  Neptune of Lakewood, Colorado will provide data validation for all samples analyzed for asbestos during this project.

## 1.5 Problem Definition and Background

The problem definition and Site background are presented in the Ramboll Environ RI/FS Work Plans (ENVIRON 2014a, Ramboll Environ 2016a, and Ramboll Environ 2017a), Downgradient Study Area Investigation Work Plans (AECOM 2016a, 2016b, and 2016c), and the Unit 4 and 5 Buildings Investigation Work Plan (Tetra Tech 2015). Problem definitions for treatability studies are presented in task-specific work plans (Tetra Tech 2014, 2016a, and 2016b). Additional details regarding Site history, historical and future land use, and potential contaminant releases at the Site are presented in the ISRACR (ENVIRON 2012) and the Annual Reports (Ramboll Environ 2016).

## 1.6 Project Description

The work to be completed as described in the RI/FS Work Plans include soil, surface water, groundwater, and soil gas sampling and chemical analyses to fill data gaps remaining from previous investigations, thereby providing additional information, including data regarding the magnitude and extent of selected chemicals in soil and groundwater at the Site. This information will be used to support the overall purpose of the RI/FS process, which is "to gather information sufficient to support an informed risk management decision regarding which remedy appears to be most appropriate for a given site" (USEPA 1988).

Treatability studies are conducted to support further development of preliminary remedial action alternatives for evaluation during the RI/FS process. Treatability studies can provide data important to an adequate evaluation of certain technologies for a given response action including information performance, operating parameters, and cost in sufficient detail to support the remedy selection process and subsequent design activities. Treatability and pilot studies can involve both field data collection tasks and bench-scale tests.

Current tasks that are being implemented or planned for implementation at the Site, their purposes, and the current status, include the following:

| Task | Purpose | Current Status | Planned Activities |
|---|---|---|---|
| **Remedial Investigation** | | | |
| **Phase 1 RI** | Collect data to address identified data gaps in the On and Off-Site NERT RI Study Areas. | NDEP approved the RI/FS Work Plan, Revision 2 in July 2014. The majority of the field investigations were performed in 2014-2015. | Soil investigation beneath the former AP-5 pond is pending decommissioning of the pond; Groundwater sampling before, during, and after weir construction on the Las Vegas Wash is pending initiation of the weir construction project by the Southern Nevada Water Authority. |
| **Phase 2 RI** | Collect data to address identified data gaps in the On and Off-Site NERT RI Study Areas. | NDEP approved the Phase 2 RI work plan in August 2016. Ramboll Environ began on-site sampling in February 2017. | Off-site field work is expected to begin in June 2017. All field work is expected to be completed by October 2017. |
| **Phase 3 RI** | Investigate extent of contamination related to Henderson Legacy Conditions in the NERT Eastside Study Area. | Ramboll Environ submitted the RI/FS Work Plan Addendum for the Phase 3 RI in April 2017. | Ramboll Environ will carry out the Phase 3 RI field investigation, pending work plan approval, beginning in the second half of 2017. |
| **Unit Building Investigation** | Investigate the perchlorate distribution near Unit Buildings 4 and 5. | Tetra Tech completed first and second mobilizations in October-December 2015 and June 2016-January 2017, respectively. A technical memorandum was submitted in May 2017 summarizing the second mobilization. | The third mobilization was initiated in August 2017. |

| Task | Purpose | Current Status | Planned Activities |
|---|---|---|---|
| **Downgradient Study Area Investigation** | Identify areas of perchlorate mass flux from groundwater to the Las Vegas Wash. | AECOM submitted memoranda to NDEP in October and November 2016 detailing initial groundwater and surface water sampling.<br>A surface water investigation plan was submitted in December 2016 and the field work related to the surface water investigation was completed in February 2017. A Preliminary Draft Surface Water Investigation Technical Memorandum was submitted in June 2017.<br>A transducer installation plan was submitted in December 2016. AECOM installed transducers in 20 wells in April/May 2017. Monthly groundwater level measurements and quarterly downloading of transducer data has been completed since installation.<br>A geophysical pilot test plan was submitted in July 2016. Field work for the geophysical pilot test was conducted in March 2017. A Preliminary Draft Geophysical Pilot Test Technical Memorandum was submitted in June 2017. The full-scale geophysical investigation will no longer be conducted, as previously planned.<br>A work plan for Phase I Groundwater Monitoring Well Installation was finalized in May 2017. | A summary report related to the surface water investigation will be prepared in second quarter 2017.<br>AECOM will perform a two-phase groundwater investigation beginning with monitoring well installation in December 2017.<br>AECOM will install transducers in April 2017.<br>AECOM will initiate a full-scale geophysical investigation in December 2017. |

| Task | Purpose | Current Status | Planned Activities |
|---|---|---|---|
| Treatability and Pilot Studies | | | |
| **Groundwater Bioremediation Treatability Study** | Evaluate bioremediation using a slow-release carbon substrate near the City of Henderson Bird Viewing Ponds. | Tetra Tech submitted the Groundwater Bioremediation Treatability Study Results Report to NDEP on November 25, 2016. | Results will be incorporated into the Feasibility Study and the Seep Well Field area bioremediation treatability study. |
| **Soil Flushing Treatability Study** | Evaluate the remediation of perchlorate using soil flushing in vadose zone soils in the Central Retention Basin. | The study was conducted from March 2015 to August 2016. Tetra Tech submitted the Soil Flushing Treatability Study Report to NDEP on March 2, 2017. | Results will be incorporated into the Feasibility Study and future treatability studies, as required. |
| **Seep Well Field Area Bioremediation Treatability Study** | Evaluate effectiveness of bioremediation to reduce perchlorate mass flux to the Las Vegas Wash. | NDEP approved the final work plan on September 22, 2016. Geophysical surveys, soil borings, and monitoring well installation is complete. Soil and groundwater sampling, aquifer tests, and bench-scale studies are ongoing. | Tetra Tech will complete the first injections in third quarter of 2017, with field activities (injections and monitoring) continuing into 2018. Tetra Tech will report the results in 2018. |
| **In-Situ Chromium Treatability Study** | Evaluate in-situ reduction of hexavalent chromium near the Interceptor Well Field. | NDEP approved the work plan on August 19, 2016. Preliminary bench-scale tests were conducted in late 2016 and early 2017. | Tetra Tech began implementing field testing in second quarter 2017. Tetra Tech will report the results in fourth quarter 2017. |
| **Ammonium Perchlorate Area Soil Flushing Treatability Study** | Build on the 2015 soil flushing study by conducting study in the Ammonium Perchlorate Area. | Down flushing and operations of Plot 1 extraction wells began in late October 2016. | Plot 2 extraction wells will begin operating in July 2017 and study will continue through 2017. Results will be reported in early 2018. |
| **Unit Building 4 Source Areas In-situ Bioremediation Treatability Study** | Evaluate soil flushing and bioremediation for source reduction. | Tetra Tech is developing a work plan that will be finalized in fourth quarter 2017. | It is anticipated the study will be implemented in 2018. |
| **Vacuum Enhanced Recovery Treatability Study** | Evaluate effectiveness of vacuum enhanced capture below the IWF and barrier wall. | Tetra Tech is developing a work plan that will be finalized in third quarter 2017. | It is anticipated the study will be implemented in late 2017-2018. |

| Task | Purpose | Current Status | Planned Activities |
|---|---|---|---|
| **Galleria Road Biobarrier Treatability Study** | Evaluate in-situ perchlorate bioremediation using an organic reagent in the NERT Eastside Study Area. | Tetra Tech is developing a work plan that will be finalized in fourth quarter 2017. | It is anticipated the study will be implemented in 2018. |
| **Zero Valent Iron (ZVI) Enhanced In-Situ Treatability Study** | Evaluate in-situ perchlorate treatment using granular ZVI in the NERT Eastside Study Area. | Ramboll Environ is developing a work plan that will be finalized in fourth quarter 2017. | It is anticipated the study will be implemented in 2018. |
| **Las Vegas Wash Groundwater Bioremediation Pilot Study** | Evaluate potential implementation of bioremediation along the Las Vegas Wash. | Tetra Tech is developing a work plan that will be finalized in fourth quarter 2017. | It is anticipated the study will be implemented in 2018. |
| **Henderson Legacy Conditions Mass Estimates and Performance Metrics** | Develop a strategy for expanding the performance metrics for the entire NERT RI Study Area. | Ramboll Environ is currently evaluating data in support of the strategy and methodology development for the expanded mass estimates and performance metrics. | The proposed strategy will be presented to NDEP and EPA during a meeting in third quarter 2017. A technical memorandum outlining the strategy is anticipated for submittal in third quarter 2017. |
| **Barrier Wall Geophysical Integrity Evaluation** | Evaluate the integrity of the barrier wall. | Ramboll Environ is developing a work plan to evaluate potential flow through, around, and underneath the barrier wall. | The study is anticipated to be implemented 2017-2018, pending NDEP approval of the work plan. |
| **Athens Road Well Field Capture Evaluation and Upper Muddy Creek formation (UMCf) Matrix Diffusion Study** | Address NDEP concerns regarding model-predicted capture efficiencies. | Ramboll Environ is developing a work plan to evaluate modeled capture efficiencies and back diffusion from the UMCf into the alluvium. | It is anticipated the work plan will be submitted to NDEP in third quarter 2017. The study will be implemented in 2017-2018, pending approval of the work plan. |

## 1.7 Data Quality Objectives

The overall goal of the QA/QC procedures and specifications established in this QAPP is to ensure that comparable and representative data are produced and that data quality is consistently assessed and documented in order to accomplish the objectives of the RI/FS Work Plan. To achieve this goal, a systematic approach is followed in the planning of this project equivalent to the USEPA Data Quality Objective (DQO) Process, as described in

*Guidance on Systematic Planning Using the Data Quality Objectives Process*, EPA QA/G-4 (USEPA 2006).

The DQO Process is a series of logical steps that guides users to a plan for the resource-effective acquisition of environmental data. It is used to establish performance and acceptance criteria, which serve as the basis for designing a plan for generating data of sufficient quality and quantity to support the goals of the study. The DQO Process consists of seven iterative steps; the iterative nature of the DQO Process allows one or more of these steps to be revisited as more information on the problem is obtained. The seven steps are as follows:

1. State the Problem
2. Identify the Goal of the Study
3. Identify the Information Inputs
4. Define the Boundaries of the Study
5. Develop the Analytical Approach
6. Specify Performance of Acceptance Criteria
7. Develop the Detailed Plan for Obtaining Data

The approach to the DQO process is described in Section 2 the FSP (ENVIRON 2014b). Following the DQO Process has driven the development of the RI/FS Work Plan, the choice of analytical methods, the establishment of relevant data validation procedures, and related aspects of the collection of environmental measurement data. The DQOs specify the data type, quality, quantity, and uses needed to make decisions and are the basis for designing data collection activities. The QA/QC procedures for this project require that the data meet minimum requirements for precision, accuracy, completeness, representativeness, comparability, and sensitivity. The procedures and minimum requirements are presented in the subsequent sections of this QAPP.

The primary and all other subcontracted laboratories will perform analytical work in accordance with this QAPP as well as with their internal Standard Operating Procedures (SOPs) and QA Manuals, which comply with NELAC standards and USEPA protocols established in Test Methods for Evaluating Solid Waste, SW-846, Update III, dated June 1997, (SW-846) (USEPA 1997). The QA Manuals include names of the responsible oversight individuals, QA/QC manual review and update procedures, organization and responsibilities of various individuals, QA/QC objectives and reports, QA/QC policies and procedures including sampling and receiving policies, equipment calibrations and maintenance information, necessary reagents and standards, extraction and analysis methods, data review and reporting processes, QA/QC procedures, system audits and corrective actions, certifications, recordkeeping and sample retention, sample disposal procedures, recent method detection limit (MDL) studies, and other QA/QC criteria relevant to the specific analytical methods.

The QA/QC Officer will evaluate the field and laboratory data against the requirements of the QAPP. Each analytical laboratory will provide the most current QA/QC information, SOPs, and QA Manuals to the QA/QC Officer(s) that specify laboratory QA/QC samples and acceptance levels for each method. Laboratories contracted to perform analyses for this

project are summarized on Table 1. The project specific MDLs, reporting limits (RLs), and QC limits for the analytes to be tested are provided in Tables 2 through 5.

Project laboratories will either use the limits specified in this QAPP or propose equally or more stringent statistically calculated QC limits. Specific QA/QC samples will be analyzed to satisfy the DQOs. The QA/QC samples to be used and the minimum frequency of their analysis for this project are summarized in Table 6. The data obtained will conform to the quality control requirements specified in this QAPP. The project QA/QC Officer will be responsible for performing the data quality evaluations, the results of which will be included in the QA/QC sections of reports. A discussion of the measurement parameters and how they will be used to evaluate project analytical data follows.

This QAPP, and any QAPP addendum, collectively, will specify explicitly the data that are needed to meet the objectives of the project and how that data will be used. In addition, this QAPP discusses implementation of control mechanisms and standards that are used to obtain data of sufficient quality to meet all project DQOs. The project DQOs provide an internal means for control and review so the environmentally related measurements and data collected by the project team are valid, scientifically sound, and of known, acceptable, and documented quality.

### 1.7.1 Characteristics of Data Quality

The term “data quality” refers to the level of uncertainty associated with a particular data set. Data quality associated with environmental measurement is a function of the sampling plan rationale and procedures used to collect the samples, as well as of the analytical methods and instrumentation used in making the measurements. Uncertainty cannot be eliminated entirely from environmental data. However, QA programs effective in measuring uncertainty in data are employed to monitor and control deviations from the desired DQOs. Sources of uncertainty that can be traced to the sampling component include poor sampling plan design, incorrect sample handling, faulty sample transportation, and inconsistent use of SOPs. The most common sources of uncertainty that can be traced to the analytical component of the total measurement system are problems associated with calibration and contamination.

The purpose of this QAPP is to ensure that the data collected are of known and documented quality and useful for the purposes for which they are intended. The procedures described are designed to obtain data quality indicators for each field procedure and analytical method. To ensure that quality data continues to be produced, systematic checks must show that test results and field procedures remain reproducible and that the analytical methodology is actually measuring the quantity of analytes in each sample.

All laboratory analytical data will be generated by a Nevada-certified (NELAC-certified for soil gas and asbestos) laboratory and validated by the data validation consultant. This applies to the primary laboratory and any laboratory subcontracted by the primary laboratory. Laboratories must have an in-place program for data reduction, validation, and reporting as discussed in this QAPP. The reliability and credibility of analytical laboratory results can be corroborated by the inclusion of a program of scheduled replicate analyses, analyses of standard or spiked samples, and analysis of split samples with QA laboratories for some projects. Regularly scheduled analyses of known duplicates, standards, and spiked samples are a routine aspect of data reduction, validation, and reporting procedures.

### 1.7.2 Measurement Performance Criteria

Performance and acceptance criteria are often expressed in terms of data quality indicators (DQIs). The principal data quality indicators are sensitivity, accuracy, precision, completeness, representativeness, and comparability. These DQIs are discussed below.

**Sensitivity** refers to the amount of analyte necessary to produce a detector response that can be reliably detected (the "Method Detection Limit" or "MDL") or quantified (the "Reporting Limit" or "RL," which is also known as the "Practical Quantitation Limit" or "PQL"). Where practicable, to reduce the possibility of false negatives, the RL of each contaminant of concern should be lower than corresponding screening value. In cases where screening values are below RLs, the MDLs can be used to evaluate the presence or absence of the analyte from environmental samples. Furthermore, to be considered valid for project use under normal conditions, the concentrations of contaminants of concern in any blank, e.g., equipment blank, field blank, and/or method blank, should not exceed the laboratory RLs, unless a higher number is considered valid to reflect actual field and laboratory conditions. Ideally, and to reduce the possibility of false positives, all blanks associated with project samples should be free of detectable contamination. The project specific MDLs, PQLs, and screening values for the analytes to be tested are summarized in Tables 2 through 5.

In the case of radionuclides, the actual result of the analysis is reported regardless of the minimum detectable activity (MDA) metric (NDEP 2008). The MDA is a sample-specific value defined as the lowest level of activity in a sample that is statistically distinguishable from a sample with no activity. For radiochemical analysis the MDA is functionally equivalent to the MDL and no PQL is reported.

Asbestos data will be reported as a raw asbestos fiber counts per sample (NDEP 2008). While there are no RLs with this method, sensitivity is calculated by the concentration of protocol structures per volume of PM10.

**Accuracy** of the data is the measure of the overall agreement of a measured value to the true value. It includes a combination of systematic error (bias) and random error (precision) components of sampling and analytical operations. It reflects the total error associated with a measurement. A measurement is considered accurate when the value reported does not differ from the true value or known concentration of a spike sample or standard beyond an acceptable margin. Field and laboratory activities are subject to accuracy checks.

To estimate the accuracy of the data, a selected sample is spiked with a known amount of a standard and is analyzed; the results of which are used to calculate percent recovery. Accuracy of laboratory analyses will be assessed by comparing results for a surrogate standard, matrix spike (MS) or laboratory control sample (LCS), and initial and continuing calibration of instruments to control limits. Laboratory accuracy is expressed as the percent recovery (%R). If the %R is determined to be outside of acceptance criteria, the data will be flagged for reporting purposes. Accuracy goals vary for analytical data by the type of analysis employed. Laboratory goals are established as part of the laboratory QA/QC program as described in the QA Manual and SOPs.

Accuracy of field measured data will be maintained by keeping the field instruments in proper working condition and calibrating as specified by operation manuals. The specific maintenance and calibration procedures in the operation manuals will be followed. The results of calibrations will be evaluated against the limits established in operation manuals specific to each instrument and recorded in field logbooks. Field accuracy will also be

assessed in part through adherence to all sample handling, preservation, and holding time requirements as described in this QAPP.

**Precision** of the data is the measure of reproducibility or agreement among repeated measurements of the same sample under identical or substantially similar conditions. It is represented as either a range of values or as a standard deviation about the mean value. Precision goals vary for analytical data by the type of quality control samples measured. Both laboratory and field quality control samples are utilized to measure precision. Precision may be expressed as a percentage of the mean of measurements, such as relative range or relative standard deviation.

Analytical precision is a measurement of the variability associated with duplicate or replicate analyses of the same sample in the laboratory. Analytical precision is determined by analysis of laboratory quality control samples, such as matrix spike duplicates (MSD) or laboratory control sample duplicates (LCSD), or sample duplicates. These samples should contain concentrations of an analyte above the RL. The most commonly used estimates of precision are percent relative standard deviation (%RSD) and the relative percent difference (RPD) when only two samples are used. RPDs for laboratory control samples are listed in Tables 2 through 5 under matrix spike RPD and blank spike/LCS RPD. %RSD values are calculated when there are more than two replicates, and the values are comparable to RPD values. The objectives for field sample RPDs are ≤30% for aqueous samples and ≤50% for solids and soil gas samples. Field sample RPDs are listed in Tables 2 through 5 under Duplicate RPDs. Samples outside the limits will be noted and reported with qualifiers.

Total precision is a measurement of the variability associated with the entire sampling and analytical process. It is determined by analysis of duplicate samples, which measure variability introduced by the laboratory and field operations. Field duplicate samples are analyzed to assess field and analytical precision.

Table 6 sets forth the frequency with which laboratory duplicate samples (i.e., LCSD and MSD) will be analyzed as well as the allowable difference in results for laboratory QA/QC samples. If the precision goals indicated in this QAPP are not met, the data will be qualified for reporting purposes.

**Completeness** is defined as the percentage of measurements judged to be valid based on the number of planned analyses. The completeness goal is to generate a sufficient amount of valid data to meet project needs and is calculated and reported for each method, matrix, and analyte combination. Completeness describes the content of the data set once errors, if any, have been identified and qualified and rejected data have been removed from the data set. Completeness may also be impacted when planned samples are not collected (e.g., caliche makes borehole advancement impossible) or collected samples are not analyzed (e.g., sample bottle broken in transit). The number of valid results divided by the number of planned results, expressed as a percentage, determines the completeness of the data set. The target completeness objective for this project is 90% for all types of samples; however, the actual completeness may be different, depending on the intrinsic nature of the samples. The data set will be considered complete if at least 90% of the data planned for collection is usable without meaningful qualifiers or errors. If the goal is not achieved, the rationale for the incompleteness will be assessed and reported. The data completeness will be evaluated during the data validation review process.

**Representativeness** is a qualitative term used to express the degree to which data accurately and precisely represent a characteristic of a population. It is mostly concerned

with the proper design of the sampling program. Sample collection and handling methods, sample preparation, analytical procedures, holding times, and QA protocols developed for this project, and discussed in the subsequent sections of this document, have been established to ensure that the collected data are representative.

**Comparability** is a qualitative term used to express the confidence with which one data set can be compared to another data set. The objective for the QA/QC program is to produce data with the greatest possible degree of comparability. The number of matrices that are samples and the range of field conditions as encountered are considered in determining comparability. Data comparability will be sustained in this project through the use of defined procedures for sampling and analysis (sample collection and handling, sample preparation, and analytical procedures), reporting in standard units, normalizing results to standard conditions, and using standard and comprehensive reporting formats.

The data set will be considered comparable when USEPA or other standard methods have been used for analyses, the data set is representative and the field investigation is conducted in accordance with accepted industry standards. Laboratory analyses for soil and groundwater will be performed in accordance with prescribed USEPA protocols established in the document *Test Methods for Evaluating Solid Waste, SW-846, Update III*, dated June 1997 (USEPA 1997), or other appropriate methods as required.

## 1.8 Specific Training Requirements/Certification

Personnel conducting field activities will be required to have completed Occupational Safety and Health Administration (OSHA) Hazardous Waste Operations and Emergency Response 40-hour training with current refresher training as detailed in Title 29 Code of Federal Regulations (CFR) Part 1910.120 for general site workers. Staff records documenting compliance with OSHA requirements are kept on file by the consultant.

The HASP (ENVIRON 2014c) and task-specific HASPs have been developed for the RI/FS. These HASPs address accident prevention, personnel protection, and emergency response procedures. The HASPs establish in detail the protocols necessary for protecting workers from the hazards associated with the contaminants at the Site, and other physical hazards (such as slips, trips, and falls, electrical hazards, poisonous insects and plants, temperature hazards, etc.). All field staff working at the Site must comply with the appropriate HASP for each RI/FS activity.

The primary laboratory and all subcontracted laboratories will maintain current Nevada certification (NELAC-certification for soil gas and asbestos). The PM will be responsible for ensuring necessary training and certification requirements are met for field operations. The Laboratory PM will be responsible for ensuring certification is maintained for the analytical laboratory.

## 1.9 Documents and Records

This section includes information about the requirements for laboratory data packages. Requirements for field documentation are also outlined in Section 5 (field sheets, data sheets, photographs) and Section 6 (sample labels and sample custody) of the FSP (ENVIRON 2014b).

Records that may be generated during field work include field logs and data sheets, photographic logs, sample chain-of-custody records, sample labels, equipment inspection/calibration records, and others as necessary. Units of measure for any field measurements and/or analyses will be clearly identified on the field forms and in notes and

logs as necessary. The QA/QC Officer, or other appropriate person designated by the PM, will review the field data to evaluate the completeness of the field records.

Analytical data will contain the necessary sample results and quality control data to assure compliance with the DQOs defined for the project. Laboratory data will be provided in hard copy or Portable Data Format (PDF), and in Electronic Data Deliverable (EDD) format in accordance with this QAPP.

### 1.9.1 Field Notes

Field logbooks or a digital data collection device (such as a tablet) will provide the means of recording data collection activities at the time they take place. The logbooks/tablets will be bound field survey notebooks assigned to field personnel, but they will be stored with the project files in a centralized document repository at an office location when not in use. Activities will be described in as much detail as possible such that the activity being described can be reconstructed without reliance on memory. Entries will be made in language that is objective, factual, and free of personal opinions or terminology that might later prove unclear or ambiguous.

The cover of each logbook will be identified by the project name, project-specific document number, and the time period which the logbook describes (beginning and end dates). The title page of each logbook will have contact information for the consultant Principal in Charge and PM. Entries into the logbook will contain a variety of project-specific information. At the beginning of each entry, the date, start time, weather, names of all team members present, level of personal protection being used, and the signature of the person making the entry will be entered. Names and affiliations of visitors to the site and the purpose of their visit will be recorded.

All logbook entries will be made in ink signed and dated and no erasures will be made. If an incorrect entry is made, the information will be crossed out with a single strike mark, initialed, and dated by the user. Whenever a sample is collected or a measurement is made it shall be recorded. Any photographs taken will be identified by number and a description of the photograph will be provided. All equipment used to conduct measurements will be identified including serial number and any calibration conducted will be recorded. Entries made on electronic devices will contain the same information as recorded in hard copy logbooks.

### 1.9.2 Field Data Sheets

Field data sheets will be completed by field personnel during sample collection activities. The types of field data sheets used include groundwater sampling logs, soil boring logs, well construction logs, well development logs, and soil gas sampling logs. If deemed necessary by the PM, electronic copies of the data sheets may be produced after sampling has been completed and these can be provided in the RI report or other reports as required, describing sampling conducted. Example field data sheets are provided in Appendix B of the FSP.

### 1.9.3 Photographs

Digital photographs will be taken if necessary to supplement and verify information entered into field logbooks. For each photograph taken, the following will be recorded in the field logbook:

- Date, time, and location,

- Number and brief description of the photograph, and
- Direction in which the photograph was taken, if relevant.

If a number of photographs are taken during a task, general notes will be sufficient on the group of photographs taken, so long as the information outlined above can be inferred from the information provided for each photograph.

### 1.9.4 Sample Labels

Sample labels will be provided with sample containers for laboratory analysis. Each sample collected will be assigned a unique identification number. All samples will be labeled in a clear and precise way for proper identification in the field, laboratory, and progress reports. Section 2.3 provides additional detail on the sample labeling requirements for this project.

### 1.9.5 Chain-of-Custody Forms and Custody Seals

Completed original chain-of-custody forms will be sent with each sample shipment to document collection and shipment of samples for off-site laboratory analysis with copies to be maintained with the Site's project files. The chain-of-custody form will identify the contents of each shipment and maintain the custodial integrity of the samples. A custody seal signed by the sampler will be used to maintain custodial integrity of the samples during collection and shipment to the laboratory. Section 2.3 provides additional detail on chain-of-custody and custody seal requirements for this project.

### 1.9.6 Verification of Electronic Data

Electronic data are generally derived from automated data acquisition systems in an analytical laboratory setting. Analytical instruments are equipped with software that performs various manipulations, identifications, and calculations of data. Software calculations are verified manually during the data validation process. Other data generated by the analytical laboratories may consist of manually recorded results. This data may be documented in a logbook and may subsequently be entered in the form of electronic files. As a part of their periodic reviews of logbooks and deliverables, the analytical laboratories will review transcriptions to ensure accuracy. Any errors encountered will trigger further auditing until no transcription errors are encountered in the audit set, up to and including 100 percent review.

Data can be reported in either hard copy form or electronic form. Screening level data are generally reported in summary form including sample identification (ID) information, results for the sample analyses, and a summary of the QC data including calibrations and verifications of precision, accuracy, and representativeness, where appropriate.

If data manipulation or reduction is performed electronically, outside of the raw data produced by purchased instrumentation, the formulae or macros employed for these purposes will be validated by comparing the results of a sample manual calculation to the result produced electronically. This validation will be documented and maintained in central files.

### 1.9.7 Electronic Data Deliverables (EDDs)

In addition to hard copy or PDF data reports provided by the contract laboratory, analytical data will be submitted to the consultant QA/QC Officer as Electronic Data Deliverables (EDDs). The names of analytical and preparation methods should be consistent with NDEP guidance (NDEP 2013). It is the responsibility of the analytical laboratory to ensure that the hard copy data and electronic data are identical. The data reported in EDDs and in the hard

copy reports must correspond exactly, including significant digits and units. It is preferable that the hard copy and EDD are generated at approximately the same time from the same data source.

The laboratory will provide an EDD for each Sample Delivery Group (SDG). The EDD should conform to the appropriate consultant's EDD format. Ramboll Environ's Laboratory Electronic Data Deliverable Format Specification, EQuIS Edition is provided as Appendix D. At the discretion of the PM and the database administrator, an exception may be made to accept an alternative EDD format, which must contain the following information at a minimum:

- Sample ID
- Sample Date
- Sample Time
- Laboratory Sample ID
- Analytical Method
- Analyte Name
- CAS#
- Result
- Detect Flag (y/n)
- Laboratory Qualifier
- Units
- Reporting Limit or PQL
- MDL
- Sample Adjusted MDL
- Spike Levels
- Percent Recovery
- RPD
- Control limits for %R and RPD
- Extraction Method
- Cleanup Method
- Sample Receipt Date
- Extraction Date
- Analysis Date
- Analysis Time
- Dilution Factor
- Result Reportable (y/n)
- Batch Number

- SDG

The Data Validation Contractor or consultant designee will compare 10% of electronic entries with hardcopy results to check for consistency.

### 1.9.8 Laboratory Documentation

The following section discusses general laboratory requirements for preparing data packages. Data packages provided by contract analytical laboratories will be at USEPA Level II, Level III, or Level IV, depending on the level of data validation required.

The Level II data package includes the following information:

- Sample and client information
- Sampling time and date
- Sample number
- Analytical method
- Environmental sample results or measurements
- Reporting limits and method detection limits
- Chain of custody
- Sample receipt checklist
- Summary of QA/QC results
- Method blank results
- Surrogate recoveries, if applicable
- LCS/Laboratory control spike duplicate (LCSD) results, recoveries, RPDs and control limits
- Matrix spike (MS)/Matrix spike duplicate (MSD) results, recoveries, RPDs, and control limits
- Duplicate results RPD
- Spike amount
- Dilution factors
- Initial sample aliquots (weights or volumes) and final sample volumes
- Percent solids (soil samples)
- Sample preparation and analytical batch association
- Case narrative

The Level III data package includes the same information as the Level II data package with this additional information:

- Instrument summary forms for initial calibration, tunes (mass spectrometry methods only), calibration verification, internal standards, interference check standards (metals only), serial dilutions (metals only), and post digestion spikes (metals only).

The Level IV data package includes the same information as the Level III data package with this additional information:

- Raw data for all samples including chromatograms and instrument outputs for internal standards (when applicable), tunes, calibrations, QA/QC samples, etc.
- Sample preparation logs, sample run logs or injection logs

The case narrative will be written and the release of data will be authorized by the laboratory director or his/her designee. Items to be included in the case narrative are the field sample ID with the corresponding laboratory ID, parameters analyzed for in each sample and the methodology used (USEPA method numbers or other citation), detailed description of all problems encountered and corrective actions taken, discussion of possible reasons for results exceeding the acceptable laboratory QA/QC results, and observations regarding any occurrences which may affect sample integrity or data quality.

Legible copies of the chain of custody forms for each sample will be maintained in the data package. Cooler log-in sheets will be associated with the corresponding chain of custody form/s. Any integral laboratory tracking document will also be included.

For each environmental sample analysis, this summary shall include field ID and corresponding laboratory ID, sample matrix, collection date/time, laboratory receipt date/time, date of sample extraction (if applicable), date and time of analysis, identification of the instrument used for analysis, instrument specifications, weight or volume of sample used for analysis/extraction, dilution or concentration factor used for the sample extract, method detection limit or sample quantitation limit, definitions of any data qualifiers used, and analytical results.

The following QA/QC results will be presented in summary form. Acceptance limits for all categories of QC criteria will be provided with the data. The summary of QA/QC results for analyses will include, but will not be limited to the following:

- Method Blank Analyses – The concentrations of any analytes found in blanks will be reported, even if the detected amounts are less than the PQL. The samples and QA/QC analyses associated with each method blank will be stated.
- Surrogate Standard Recovery (organic analyses only) – The name and concentration of each surrogate compound added will be detailed. The percent recovery of each surrogate compound in the samples, method blanks, MS/MSD, and other QA/QC analyses will be summarized with sample IDs such that the information can be linked to sample and QA/QC analyses.
- Matrix Spike/Matrix Spike Duplicate – For MS/MSD analyses the sample results, spiked sample results, percent recovery, and associated recovery and RPD control limits will be detailed. Parent sample results will also be included on the summary form.
- Laboratory Control Sample/ Laboratory Control Sample Duplicate – For LCS/LCSD analyses the spiked sample results, percent recovery, and associated recovery and RPD control limits will be detailed. LCS/LCSD analyses will also include: source of the sample(s), true value concentrations, found concentrations, percent recovery for each element analyzed, and the date and time of analysis.
- Laboratory Duplicates – For laboratory duplicate analyses the sample results, RPD between duplicate analyses, and control limits will be reported, as applicable. For

laboratory QC check and/or LCS analyses, the %R and acceptable control limits for each analyte will be reported. All batch QC information will be linked to the corresponding sample groups.

All data packages will be reviewed by the individual laboratory QA Officer or designated data review specialists to ensure accurate documentation of any deviations from sample preparation, analysis, and/or QA/QC procedures and descriptions. Any problems identified by the laboratory QA Officer or designated data review specialists will be documented in the narrative of the report.

### 1.9.9 Laboratory Record Retention

Raw data will be available for further inspection, if required, and maintained in each laboratory's central job file. Records related to the analytical effort (i.e., cost information, scheduling, custody) are maintained at the laboratories in a secured location. Moreover, analytical laboratories will have the ability to archive data and quality records in a secured area protected from fire and environmental deterioration. Electronic data should be protected against exposure to magnetic or electronic sources.

All records necessary to reproduce the analytical calculations and support the reported results must be maintained for a minimum of five years. Types of records to be maintained for the project include, but are not limited to the following:

- Chain of custody forms, including: information regarding the sampler's name, date of sampling, type of sampling, sampling location and depth, number and type of sampling containers, signatures of sample custodians with transfer date and times noted, and sample receipt information including temperature and conditions upon arrival at the laboratory;
- Cooler receipt form documenting sample conditions upon arrival at the laboratory;
- Any discrepancy/deficiency report forms due to problems encountered during sampling, transportation, or analysis;
- Sample destruction authorization forms containing information on the manner of final disposal of samples upon completion of analysis;
- All laboratory notebooks including raw data readings, calibration details, QC checks, etc.;
- Hard copies of data system printouts (chromatograms, mass spectra, inductively coupled plasma [ICP] data files, etc.);
- Tabulation of analytical results with supporting QC information; and
- Sample preparation documents/records.

### 1.9.10 Field Document Retention

All field documentation generated during data collection for RI/FS tasks, including any electronic files produced, will be kept on file in a secured central repository in accordance with an established document retention policy.

# 2. DATA GENERATION AND ACQUISITION

This section discusses sampling process design; sampling methods; sample handling and custody; analytical methods; quality control; instrument/equipment testing, inspection, maintenance, and calibration; inspection/acceptance of supplies; non-direct measurements, and data management.

## 2.1 Sampling Process Design

This QAPP is intended to cover soil, soil vapor, surface water, and groundwater sampling. In the event that a task requires additional media to be sampled, a task-specific QAPP addendum will be prepared. Samples will be collected according to applicable NDEP guidelines and following the procedures described in project-specific work plans. The collected data will be used to fill data gaps identified in previous investigations, thereby completing delineation of the lateral and vertical extent of selected chemicals in soil, soil gas, surface water and groundwater at the Site, as described in the RI/FS Work Plans.

## 2.2 Sampling Methods

Sampling will be conducted in accordance with the procedures described in the RI/FS Work Plans.

## 2.3 Sample Handling and Custody Requirements

In general, the samples and subcontracted analytical laboratories will handle samples in a manner to maximize data quality. Samples will be collected, handled, and stored in such a manner that they are representative of their original condition and chemical composition. Identification of samples and maintenance of custody are important elements that must also be utilized to ensure samples characterize Site conditions. All samples will be properly identified and maintained under chain-of-custody protocol to protect sample integrity. The following sections discuss the sample handling and custody requirements in detail.

### 2.3.1 Sample Identification

To maintain consistency, a sample identification convention has been developed and will be followed throughout data collection. The sample identification numbers (IDs) will be entered onto the sample labels, field forms, chain-of-custody forms, logbooks, and other records documenting sampling activities.

Unless specified in an approved task-specific work plan, the identification system for primary field samples collected for RI activities will include the soil boring (RISB), trench (RIT), groundwater well (M for on-Site, PC for off-Site) or soil gas (RISG) well ID, trench sampling node if applicable (alpha numeric), a sample start depth if applicable (for discrete depth samples only), and the date in YYYYMMDD format. Grab groundwater samples collected from soil borings will be identified similarly to a soil sample but with “GW” in place of the depth. For example,

- A soil sample collected from a depth of 10 to 10.5 feet bgs at borehole RISB-1 on July 1, 2014 will be identified as RISB-1-10.0-20140701.
- A soil sample collected from a depth of 10 to 10.5 feet bgs at monitoring well borehole M-189 on July 1, 2014 will be identified as M-189-10.0-20140701.
- A grab groundwater sample collected from borehole RISB-1 on July 1, 2014 will be identified as RISB-1-GW-20140701.

- A trench soil sample collected from trench RIT-1, node A, at a depth of 2 to 2.5 feet bgs will be identified as RIT-1-A-2.0-20140701.
- A soil gas sample collected from a depth of 5 feet bgs in soil gas sample point RISG-1 on July 1, 2014 will be identified as RISG-1-5.0-20140701.
- A groundwater sample collected from monitoring well M-161D on July 1, 2014 will be identified as M-161D-20140701.

Sample identifications for treatability and pilot studies may adopt a specific identification system appropriate for the work performed as specified in task-specific work plans.

### 2.3.2 Field QA/QC Sample IDs

Field QA/QC samples and procedures are discussed in Section 2.5.1. The field QC sample codes that may be applied to RI activities include:

- EB for Equipment Blanks
- FB for Field Blanks
- TB for Trip Blanks
- FD for Field Duplicates

Field QA/QC sample codes will be appended to the end of the primary sample ID that is represented by the field QA/QC sample.

An Equipment Blank (EB) should be named for the sample collected immediately prior to the collection of the EB.

The Field Blank (FB) and Trip Blank (TB) each represent a group of samples: a batch of twenty for the FB, and all samples within one sample cooler or other shipping container for the TB. Thus the FB and the TB should be named after the first sample of the batch (for FB) or the first sample placed in the cooler or shipping container (for TB).

The Field Duplicate (FD) represents the primary sample that is being duplicated, thus the FD should be named after the corresponding primary sample.

For example, the first soil sample to be placed in a cooler is RISB-1-10.0-20140701. The sample is to be analyzed for volatile organic compounds (VOCs), and a duplicate sample is collected. A TB is placed in the cooler with the sample, and an EB is collected immediately following the collection of the soil sample (after decontamination of sampling equipment). The associated field QA/QC samples will be identified as:

- RISB-1-10.0-20140701-EB (Equipment Blank)
- RISB-1-10.0-20140701-FB (Field Blank)
- RISB-1-10.0-20140701-TB (Trip Blank)
- RISB-1-10.0-20140701-FD (Field Duplicate)
- Field QA/QC samples and the frequencies of collection are summarized in Table 6.

Field QA/QC sample IDs for treatability and pilot studies may adopt a specific identification system appropriate for the work performed as specified in task-specific work plans.

### 2.3.3 Sample Labels

A sample label will be affixed to all sample containers sent to the analytical laboratory. Field personnel will complete an identification label for each sample with the following information written in waterproof, permanent ink:

- Client or Site name ("NERT") and project number
- Sample location and depth, if relevant
- Unique sample identifier
- Date and time sample collected
- Filtering performed, if any
- Preservative used, if any
- Name or initials of sampler
- Analyses or analysis code requested

The use of pre-printed sample labels is preferred in order to reduce sample misidentification problems due to transcription errors. Sample labels must be completed and affixed to the sample container in the field at the time of sample collection.

If errors are made on a sample label, corrections will be made by drawing a single line through the error and recording the correct information. All corrections will be dated and initialed.

### 2.3.4 Containers, Preservation, and Hold Time

The analytical methods, type of sample containers to be used for each sample type and analysis, preservation requirements for all samples, and holding times are provided in Table 7.

Each lot of preservative and sampling containers will be certified as contaminant-free by the provider and/or the laboratory. The laboratories will maintain certification documentation in their files. All preserved samples will be clearly identified on the sample label and chain-of-custody form. If samples requiring preservation are not preserved, field records will clearly specify the reason for the discrepancy.

Soil and groundwater sample containers will be placed in airtight plastic bags, if possible, and refrigerated or placed in a cooler with ice to chill and maintain a sample temperature of ≤ 6 degrees Celsius (°C). Aqueous samples should not be frozen.

Chemical activity continues in the sample until it is either analyzed or preserved. Once the sample has been preserved, the sample may be held for a period of time before analysis. The time from the collection of the sample to the analysis is defined as the holding time.

Certain soil samples will be submitted on hold ("contingent samples") with instructions for extraction at a later date, or pending analytical results of a corresponding sample submitted for initial analysis.

The laboratory will immediately notify the PM and QA/QC Officer in the event that the analysis or reporting of results for initial soil samples may be delayed beyond the acceptable hold time of corresponding contingent sample(s). In such a scenario, the affected contingent sample(s) will be extracted in order to extend the acceptable hold time. Once

the results of the initial soil samples are available, the PM and/or QA/QC Officer will decide whether the extractions of the corresponding contingent samples should be analyzed.

### 2.3.5 Sample Handling and Transport

Proper sample handling techniques are used to ensure the integrity and security of the samples. Samples for field measured parameters will be analyzed immediately in the field by the sampling crew and recorded in the field logbook and field data sheets. Field guidance documents within Appendix A of the FSP (ENVIRON 2014b) provide detailed information on groundwater and soil sampling and handling procedures. Samples for laboratory analysis will be transferred immediately to appropriate laboratory supplied containers in accordance with the following sample handling protocols:

Proper sample handling techniques are used to ensure the integrity and security of the samples. Samples for field measured parameters will be analyzed immediately in the field by the sampling crew and recorded in the field logbook and field data sheets. Samples for laboratory analysis will be transferred immediately to appropriate laboratory supplied containers in accordance with the following sample handling protocols:

- Don clean gloves before touching any sample containers, and take care to avoid direct contact with the sample.
- Samples will be quickly observed for color, appearance, and composition and recorded as necessary.
- The sample container will be labeled before or immediately after sampling in accordance with Section 2.3.2.
- Groundwater and soil sample containers and liners will be capped with Teflon™-lined caps before being placed in Ziploc™-type plastic bags. The samples will be placed in an ice chest and cooled to 4 °C or lower for transport to the laboratory.
- Summa canisters used for soil gas collection do not require cooling or additional bagging.
- All sample lids will stay with the original containers, and will not be mixed.
- Sample bottles or canisters will be wrapped in bubble wrap as necessary to minimize the potential for breakage or damage during shipment.
- The chain-of-custody form will be placed in a separate plastic bag and taped to the cooler lid or placed inside the cooler. A custody seal will be affixed to the cooler.

The samplers are responsible for proper handling practices until receipt at the laboratory, or by the courier, at which time the Laboratory Project Manager assumes responsibility of the samples through analysis and ultimately to the appropriate disposal of samples. Sample handling procedures specific to the laboratory are described in the individual laboratory QA Manuals.

### 2.3.6 Sample Custody

Standard sample custody procedures will be used to maintain and document sample integrity during collection, transportation, storage, and analysis. Custody documents must be written in waterproof, permanent ink. Documents will be corrected by drawing one line through the incorrect entry, entering the correct information, and initialing and dating the correction. The PM is responsible for proper custody practices so that possession and handling of individual samples can be traced from the time of collection until receipt at the

laboratory, or by the courier. The Laboratory PM is responsible for establishing and implementing a control system for the samples in their possession that allows tracing from receipt of samples to disposal.

The chain-of-custody form provides an accurate written record that traces the possession of individual samples from the time of collection in the field until they are accepted at the analytical laboratory. The chain-of-custody form also documents the samples collected and the analyses requested. The sampler will record the following information on the chain-of-custody forms:

- Client and project number
- Name or initials and signature of sampler
- Name of destination analytical laboratory
- Name and phone number of Project Manager in case of questions
- Unique sample identifier for each sample
- Data and time of collection for each sample
- Number and type of containers included for each sample
- Analysis or analyses requested for each sample
- Preservatives used, if any, for each sample
- Sample matrix for each sample
- Any filtering performed, if applicable, for each sample
- Signatures of all persons having custody of the samples
- Dates and times of transfers of custody
- Shipping company identification number, if applicable
- Any other pertinent notes, comments, or remarks

Unused lines on the form will be crossed out and initialed.

A sample is considered to be under the control of, and in the custody of, the responsible person if the samples are in their physical possession, locked or sealed in a tamper-proof container, or stored in a secure area.

The person who collects the sample is the initial custodian of the sample. Any transfers are documented on the chain-of-custody by the individuals relinquishing and receiving the sample, along with their signature, and the date and time of transfer. This transfer must continue until the custody is released to a commercial carrier (i.e. FedEx), or the laboratory (either at the laboratory or to a laboratory employed courier). If relinquished to a commercial carrier, the carrier assumes custody through their shipping receipt. A copy of the shipping receipt should be attached to the chain-of-custody form as a permanent part of the custody control. If the sample is relinquished to a laboratory courier, the courier will then need to relinquish the sample to the stationary laboratory upon arrival. Once the sample has arrived at the stationary laboratory, it must be entered into the sample custody control system of the laboratory. If the sample is further transported to a subcontracted laboratory, the laboratory will produce an internal chain-of-custody form that will be

available upon request. Chain-of-custody forms will be maintained in the consultant's project file and at the analytical laboratory.

To discourage tampering during transport, a custody seal will be placed on each cooler after the samples are packed. These consist of a security tape or label with the date and initial of the sampler or person currently in possession of the sample. Receiving personnel at the laboratory will note on the cooler receipt form whether or not the custody seals are intact.

### 2.3.7 Shipping Procedures

If shipping samples using a commercial courier is necessary, each container sent will have a separate chain-of-custody form. Samples collected during the investigation will be identified as environmental samples. Samples will be packed in the same manner as when being transported from the sampler to the laboratory, with the following changes:

- Dry ice is not allowed to be used to chill samples requiring commercial shipment.
- Extra packing material will be used to fill the coolers in order to limit movement within the container.
- Ice should be contained in zip-closure bags and the cooler should be lined with plastic as described below.
- Coolers containing ice and/or liquid samples should be lined with a plastic bag (such as a contractor garbage bag) to limit the potential for leaks in the event of ice bags leaking or sample container breakage. All necessary precautions must be taken to prevent any liquids leaking from sample coolers while in transit.
- Coolers will be closed and taped shut. If the cooler has a drain, it too will be closed and taped shut to prevent leaks.
- A minimum of two custody seals will be affixed to the front and side openings of the cooler so that the cooler cannot be opened without breaking a seal. The seals will be covered with wide clear tape so that the seals do not accidentally break in transit.
- Non-perishable samples collected on the weekend may be held for more than three days if there is no threat of exceeding hold times. If the samples require being chilled and maintained at a cool temperature, they will be stored under refrigeration and shipped the following work day.

#### 2.3.7.1 Transport Container Receipt

Upon receipt of the transport container, the analytical laboratories will review the contents and sign and date the chain-of-custody forms. Additional information will also be added to the chain-of-custody form including: the status of the custody seals; the temperature of the cooler, how it was evaluated, and whether or not the samples were on ice; the conditions of samples and identification of any broken sample containers; description of any discrepancies on the chain-of-custody forms; sample labels and/or requested analyses; and the pH of any preserved water samples.

The analytical laboratory will contact the appropriate Analytical Task Leader or other designated person regarding any discrepancies in paperwork and/or chemical or thermal sample preservation. Nonconformance and corrective actions will be documented in accordance with the laboratories QA/QC documents. After samples have been accepted, checked, and logged in, the laboratories will maintain them in a manner consistent with the custody and security requirements specified in the laboratory QA/QC documents.

## 2.4 Analytical Methods

Both field measurement methods and stationary analytical laboratory methods will be utilized to analyze samples during implementation of this QAPP. Analytical methods including MDLs and PQLs to be used are listed on Tables 2 through 5. Laboratory SOPs for the listed methods have been developed and approved by the laboratories performing the analyses. The dates of the current SOPs are summarized for each laboratory on Table 1.

### 2.4.1 Field Measurement Methods

Samplers may conduct in-field measurement for depth to water; pH, conductivity, ferrous iron, sulfide, dissolved oxygen (DO), oxygen reduction potential (ORP), turbidity and temperature of groundwater samples; field screening of organic vapors in soil samples; and field screening for leak detection compounds in soil vapor samples. An appropriate pH meter and standardization buffers as recommended by the instrument manufacturer will be used. All meter standardizations, QC, and sample results will be recorded on the appropriate field forms.

### 2.4.2 Laboratory Analytical Methods

The project will involve, at a minimum, the analysis of soil, soil vapor, and groundwater samples. The primary methods that will be used to analyze samples are summarized in Table 2 through 5.

Each analytical laboratory used during implementation of this QAPP will be expected to provide a current statement of Qualifications and laboratory QA/QC documents (including Quality Assurance Manual [QAM] and SOPs) for review by the QA/QC Manager. In addition, analytical laboratories may be requested to provide current MDL studies, proposed RLs and other sources that contain QC procedures, QC acceptance criteria, and corresponding corrective actions for the analytical methods to be used during implementation of the QAPP.

The laboratory will use analytical methods and QA/QC procedures in conformance with approved methods for all samples. Copies of the laboratory QA Manuals and SOPs for all laboratories will be retained on file with Ramboll Environ. Table 1 provides the specific analytical method to be used for each analyte and matrix. In the event that the listed procedures cannot be performed, the laboratory will notify the appropriate (i.e. Ramboll Environ, AECOM, or Tetra Tech) Analytical Task Leader of the conflict. The appropriate Task Leader or PM will notify the NDEP RPM for resolution. Unless specifically directed otherwise by the NDEP RPM, the standard or superseding test methods will govern. No changes in prescribed analytical methods will be made unless approved by the NDEP RPM.

PQLs compiled in Tables 2 through 5 are from a review of RLs generally achieved by the laboratories used for implementation of this QAPP. It should be noted that the limits listed in Tables 2 through 5 are laboratory and sample dependent and may not always be achievable due to matrix effects, necessary dilution of the sample, and/or interferences.

## 2.5 Quality Control Requirements

There is potential variability in any sample collection, analysis, or measurement activity. QC activities are those technical activities routinely performed, not to eliminate or minimize errors, but to assess/demonstrate reliability and confidence in the measurement data generated. This section identifies quality control checks for sample collection, field measurements, and laboratory analyses for RI/FS data collected.

### 2.5.1 Field QC Procedures

Field QA/QC samples that will be collected during the proposed investigation include field duplicate samples, field blanks, and equipment blanks. The description and purpose of these samples is discussed in this section. The frequency of analysis of field QA/QC samples is summarized in Table 6.

#### 2.5.1.1 Field Duplicates

The FD is a replicate sample collected as close as possible to the same time that the primary sample is collected and from the same location, depth, or source, and is used to document analytical precision. FD samples will be labeled and packaged in the same manner as primary samples but with "FD" appended to the sample ID. FDs will be collected at a frequency of one in every 10 primary samples and will be analyzed for the same suite of parameters as the primary sample. The relative percent difference (RPD) between the field duplicate sample and the primary sample is evaluated to assess the homogeneity of the sample matrix and to assess the reproducibility of laboratory and field sample collection techniques.

#### 2.5.1.2 Field Blanks

FB samples are used to assess the presence of contaminants arising from field sampling procedures. FB samples are obtained by filling a clean sampling container with analyte-free deionized (DI) water, in the field at a sample location. The sample then is analyzed in the same manner as the primary sample. FB samples will be collected at a frequency of one in every 20 samples and will be analyzed for the same suite of parameters as the primary sample to assess potential background contamination, contamination due to bottles and preservatives, or errors in the sampling process.

#### 2.5.1.3 Equipment Blanks

EB samples are used to assess the effectiveness of decontamination procedures. EB samples are obtained by filling decontaminated sampling equipment with analyte-free DI water, sampling this water, and submitting the sample for analysis. Alternatively, DI water can be poured over or through the decontaminated sampling equipment and then collected and submitted for analysis. EBs will be collected at a frequency of one in every 20 samples and will be analyzed for the same suite of parameters as the primary sample to assess the effectiveness of decontamination procedures.

#### 2.5.1.4 Trip Blanks

TB samples are used to assess the potential for cross-contamination of VOCs between samples during storage and shipment. TB samples are only necessary when VOCs are being analyzed in soil, groundwater, and/or soil gas samples. A TB sample consists of one or more sample containers that are prepared at the analytical laboratory by filling with reagent-grade DI water (or, for soil gas sampling, VOC-free air). The TB sample is added to the sample cooler or other shipping container as soon as the first primary sample is collected. The TB sample accompanies the primary samples to the laboratory and is analyzed using the same analytical method as the primary samples.

### 2.5.2 Laboratory QC Procedures

The laboratory QA/QC program includes (i) performing analytical methods according to prescribed protocols and (ii) analyzing laboratory QA/QC samples to measure precision and accuracy of laboratory methods and equipment, instrument calibration and preventive maintenance. Laboratory QA/QC samples and parameters that will be analyzed include

method blanks, laboratory control samples, matrix spikes, laboratory duplicates, and surrogates. The acceptable limits of the laboratory QA/QC samples are provided in Tables 2 through 5. The frequency of analysis of laboratory QA/QC samples is summarized in Table 6.

#### 2.5.2.1 Method Blanks

A method blank is a sample of a matrix similar to the batch of associated samples. It is used to assess potential contamination in the laboratory process (e.g., contaminated reagents, improperly cleaned or calibrated equipment). For each analytical method, the laboratory will analyze one method blank sample per 20 primary field samples, or one per preparation batch, whichever is more frequent.

#### 2.5.2.2 Laboratory Control Samples

A laboratory control sample is a known matrix (e.g., washed sea sand, reagent water, zero air) that has been spiked with a known concentration of specific target analytes. It is used to demonstrate the accuracy of the analytical process. For each analytical method a laboratory control sample will be analyzed once per 20 primary field samples, or one per preparation batch, whichever is more frequent.

#### 2.5.2.3 Matrix Spikes

Matrix spikes are performed by the analytical laboratory in order to evaluate the efficiency of the sample extraction and analysis procedures. Matrix spike samples are necessary because matrix interference (i.e., interference from the sample matrix -water or soil) may have a widely varying impact on the accuracy and precision of the extraction analysis. The matrix spike is prepared by the addition of known quantities of specific target compounds to a sample. The sample then is extracted and analyzed. The results of the analysis are compared with the known additions and a matrix spike recovery is calculated giving an evaluation of the accuracy of the extraction and analysis procedures. Typically, matrix spikes are performed in duplicate in order to evaluate the precision of the procedures as well as the accuracy. Matrix spike recoveries (%R) are reviewed to check that they are within acceptable range. For applicable analytical methods matrix spikes and matrix spike duplicates will be analyzed by the laboratory at a frequency of at least 1 per 20 primary field samples, or one per preparation batch, whichever is more frequent.

#### 2.5.2.4 Laboratory Duplicates

Duplicate samples are used to assess precision in the analytical method. An additional aliquot is extracted from the primary sample and analyzed using the identical procedures as the primary sample. Then the results are compared to assess the precision. There are three types of duplicates: sample duplicates, laboratory control sample duplicates and matrix spike duplicates. For applicable analytical methods duplicates will be collected and analyzed at a frequency of at least 1 per 20 primary field samples, or one per preparation batch, whichever is more frequent.

#### 2.5.2.5 Surrogates

A surrogate is a chemically similar compound spiked into each sample analyzed. Surrogates assess the precision and accuracy of each individual analysis based on the surrogate recoveries. A surrogate (typically more than one) will be analyzed for each primary sample when applicable to the specified method. Surrogate recovery should fall within the limits set by the laboratory in accordance with procedures specified by the method.

### 2.5.3 Corrective Actions

Corrective actions may be initiated if precision or accuracy goals are not achieved. The initial step in corrective action will be to instruct the laboratory to examine its procedures to assess whether analytical or computational errors caused the anomalous results. At the same time, sample collection and handling procedures will be reviewed to assess whether they could have contributed to the anomalous results. Based on this evaluation, the appropriate PM or Analytical Task Leader, together with the appropriate Project QA Officer, will assess whether re-analysis or re-sampling is required or whether any protocol should be modified for future sampling events. Any changes in laboratory methods, or quality assurance parameters or limits, require written approval prior to implementation by the laboratory.

## 2.6 Instrument/Equipment Testing, Inspection, and Maintenance

### 2.6.1 Field Instrumentation

Equipment used in the collection of field measurements will be maintained according to the manufacturer's specifications, and will be inspected and calibrated prior to use. Field equipment requiring testing, inspection, and maintenance are:

- Organic Vapor Meter (OVM) utilized for measuring total organic vapors in soil and breathing zones;
- Particulate Meter utilized for measuring particulate matter in breathing zones and air column
- Water quality meter utilized to measure pH, temperature, and conductivity;
- A flow through cell to measure DO and ORP of certain water samples
- Turbidity meter utilized to measure turbidity of water samples;
- Electric water level meter utilized to measure depth to groundwater;
- Low flow adjustable sampling pump utilized for collection of groundwater, and
- Pressure transducers for water level/temperature monitoring and data logging.

The operating manuals for each piece of field equipment used describe the procedures required for testing, inspecting, and maintaining this equipment. The types and frequencies of testing, calibration, and maintenance for field instruments are presented in Table 8. The results of testing, inspections, or maintenance conducted will be summarized in the field logbook. Testing, inspection, and maintenance of field equipment and documentation of completion of these activities will be the responsibility of field personnel under the direction of the Field Task Leader.

### 2.6.2 Laboratory Equipment

Instrument maintenance logbooks are maintained in the laboratory. In general, the logbooks contain a schedule of maintenance, as well as a complete history of past maintenance, both routine and non-routine, for that particular instrument.

Preventive maintenance is performed according to the procedures specified in the manufacturer's instrument manuals, including lubrication, source cleaning, and detector cleaning, and the frequency of such maintenance. Chromatographic carrier gas purification traps, injector liners, and injector septa are cleaned or replaced on a regular basis. Precision and accuracy data are examined for trends and excursion beyond control limits to

determine evidence of instrument malfunction. Maintenance will be performed when an instrument begins to degrade as evidenced by the degradation of peak resolution, shift in calibration curves, decrease in sensitivity, or failure to meet one or another of the pre-determined QC criteria.

## 2.7 Instrument Calibration and Frequency

### 2.7.1 Field Calibration Procedures

Instruments requiring calibration include air monitoring equipment (e.g., photoionization detectors (PIDs), gas multimeters, and dust monitoring meters) and water quality meters (e.g., pH, dissolved oxygen, specific conductivity, and turbidity meters). Equipment that can be field calibrated will be calibrated at least once per day prior to beginning sampling activities, with calibration results documented on an Instrument Calibration Log or in the field logbook. Equipment that must be calibrated in a laboratory setting should be used only if a current calibration certificate is available (for example, a calibration certificate is provided with a piece of rental monitoring equipment). Calibration procedures should be consistent with manufacturer instruction manuals for each instrument. Calibration and maintenance procedures for field equipment are detailed in Table 8.

### 2.7.2 Laboratory Calibration Procedures

The laboratory SOPs and QAMs address the calibration and frequency of calibration required for laboratory instruments as well as a description of documentation that will be completed. Laboratory QAMs are located in Appendix B. Laboratory SOPs are located in Appendix C. Table 9 summarizes the minimum frequency and scope of laboratory checks and calibrations to be performed during this project. Laboratories may have more stringent requirements as part of their SOPs, but must meet these minimum requirements as well as satisfying specific requirements of the standard methods specified for this project.

The Laboratory PM will be responsible for ensuring proper calibration and recordkeeping are conducted and will inform the appropriate Analytical Task Leader of any issues that may impact analytical results.

## 2.8 Inspection/Acceptance of Supplies and Consumables

Inspection will be conducted of field and laboratory supplies and consumables that may directly or indirectly affect the quality of results. Only supplies and consumables that have been determined to be acceptable will be utilized for the project.

Containers and individually certified SummaTM canisters will be provided by the laboratory or their approved supplier for samples to be analyzed by the laboratory. The analytical sample containers will be considered critical field supplies and consumables and the laboratory will provide an inventory describing the number and types of containers and/or canisters that have been provided. An inventory of containers received for each sampling event will be conducted by the field personnel and only new undamaged containers or canister will be utilized. If any container is found to have a defect or damage it will be properly discarded and replacements will be requested as necessary. Canister gauges will be checked to ensure that vacuum conditions exist within the canister.

Other field supplies and consumables to be used include items such as bailer cord, calibration standards, disposable bladders for pumping, sample tubing, and distilled water. These supplies will be inspected upon receipt in part to verify they are new and in their original packaging. If any defects are noted or suspected they will be properly discarded

and replaced prior to use. At the direction of NDEP, water samples collected for non-compliance perchlorate analysis by Method 314.0 do not require sterile filtration.

The supplies and consumables for this project will be handled and stored in such a manner such that they will not compromise sampling results. This will involve keeping items in their original containers before use, sealing containers properly between uses, or storing items in new or dedicated plastic bags.

The Field Task Leader with assistance from field personnel will be responsible for inspecting and accepting field supplies and consumables and providing replacements as necessary. Field personnel will inventory critical supplies on a regular basis and report to the Field Task Leader to ensure that work will not be delayed unnecessarily. The Field Task Leader will in turn provide updates on a regular basis to the PM.

### 2.8.1 Laboratory Supplies and Consumables

A detailed description of the laboratory inspection and acceptance policy for supplies and consumables is provided in the laboratory QA Manual. A list of primary supplies and consumables necessary for each laboratory analysis are provided in the individual SOPs.

Laboratory analytical group supervisors are responsible for ensuring that supplies and consumables are appropriate and adhere to laboratory policy as described in their QA Manual. Any issues regarding supplies that could have a negative effect on data quality will be communicated to the Laboratory PM who will inform the appropriate Analytical Task Leader in a timely manner.

## 2.9 Non-Direct Measurements

The historic data were generated as part of previous investigations performed at and around the Site. This data was evaluated during development of the RI/FS Work Plans, ISRACR, and Annual Groundwater Monitoring Reports.

The sampling and analysis as described in the RI/FS Work Plans and in this QAPP has been designed to generate data that will be comparable to the historic data and add to the Conceptual Site Model (CSM) developed for the Site.

Non-direct data such as historical reports, maps, literature searches, and previously collected analytical data will be reviewed prior to use to determine its acceptability based on the end use of the data.

## 2.10 Data Management

Data for this project will be generated in one of two ways; on-site from sampling and measurement activities and at the laboratory via analytical testing of soil, soil vapor, surface water, and groundwater samples. An overview of the management and reporting of this data is described in the following sections. Detailed requirements for the recording of field data and reporting of analytical data are included in Section 1.8 of this QAPP.

### 2.10.1 Field Data

Data that may be collected in the field primarily consist of; field-measured water quality parameters (pH, conductance, temperature), depth to groundwater measurements, sample depth measurements, and information and measurements of the location of borings.

Upon generation all field data will be immediately recorded in site-dedicated field logbooks. Calibration results will also be included in field logbooks and/or appropriate field forms. As necessary, field data from logbooks and field forms will be tabulated in spreadsheets to be

included in reports. The QA/QC Officer, or other appropriate person designated by the Field Task Leader will review the field data to evaluate the completeness and accuracy of the field records.

### 2.10.2 Laboratory Data

A detailed description of laboratory data management procedures is provided in the laboratory QA Manuals. The Laboratory PM will be responsible for ensuring the established data management procedures are followed.

### 2.10.3 Data Management

The data will be entered into an EQuIS® database system maintained by Ramboll Environ. The database will be maintained on a secure, enterprise-level database server that is backed-up regularly. Access to the database will be restricted to authorized users. Data management will be further discussed in the NERT Data Management Plan which is under development and scheduled to be completed in late 2017.

EDDs provided by the laboratories should be in the EQuIS 4-File EDD format as defined by the Ramboll Environ Laboratory Electronic Data Deliverable Format Specification, EQuIS Edition and/or the Automated Data Review Software (ADR) EDD specifications. The EQuIS and ADR EDD format specifications are defined in Appendix C. The laboratories will check that their EDD submittals are consistent with lists of valid values provided in this QAPP. Prior to loading into the database, EDDs will be reviewed for consistency with the file format and valid values. Data collected in the field will also be entered into the database and integrated with laboratory data.

The data validator will provide an EDD with data qualifiers, reason codes, and validation level columns appended to the data results. The validation data will be applied to the results records in the EQuIS® database.

Upon completion of data validation, an Access database consistent with NDEP specifications provided in Guidance on Unified Chemical Electronic Data Deliverable Format (NDEP 2013) will be created. The Access databases will be created as often as required by individual work plans.

# 3. ASSESSMENT AND OVERSIGHT

Assessment and oversight are designed to determine whether the QAPP is being implemented as approved, to increase confidence in the information obtained, and ultimately, to determine whether the information may be used for its intended purpose(s).

## 3.1 Assessment and Response Actions

### 3.1.1 Field Assessments and Response Actions

Consultants are responsible for conducting field assessments for the task-specific work they are implementing. During the collection of RI/FS data, the Project QA/QC Officer, or other person designated by the PM, will perform periodic assessments of compliance with the QAPP. When problems or issues are identified, the field personnel will be notified of the issue and instructed as to how to proceed going forward. If a subsequent assessment reveals that the problem has not been corrected, a field audit will be conducted. In addition, periodic unannounced audits may be conducted of field operations. Such audits may include evaluation of the following actions: field procedures, sampling activities, field forms and logbooks, chain-of-custody procedures, field measurements, field equipment calibration procedures, and sample packaging and shipment. Additional routine audits may be conducted during the course of collecting RI/FS data as deemed necessary by the QA/QC Officer to verify conformance with corrective actions identified in a previous audit and/or to provide additional qualitative assessment of field procedures. The Field Task Leader, in consultation with the PM; will be responsible for ensuring corrective actions identified by the audit are completed.

### 3.1.2 Laboratory Assessments and Response Actions

The laboratory will be responsible for its own compliance with the QAPP. If an internal audit identifies a nonconformance that affects analytical results for this project then the Laboratory PM will notify the appropriate Analytical Task Leader in writing describing the nonconformance, the impact to analytical results, and corrective actions implemented to respond to the nonconformance.

During the data validation process, the consultant will review selected elements of the laboratory performance as it relates to the QAPP. If non-compliance issues are identified, the laboratory will be notified as to what issue(s) has been identified and will be required to prepare a written response to the consultant regarding what corrective action will be taken to address the issue. If non-compliance problems persist, audits and/or further performance evaluation may be implemented.

## 3.2 Descriptions of Audits

Internal audits will be performed to review and evaluate the adequacy of the QAPP and to ascertain that it is being implemented.

A systems audit will include an evaluation of field and laboratory QA/QC procedures. If the systems audit shows a significant discrepancy from the RI/FS Work Plan or the QAPP, the responsible party will remedy the situation before work continues. Each major system change will require a written summary to document the change made.

A performance audit will include a careful evaluation of field, laboratory, and data documentation and management procedures to determine accuracy. Upon discovery of significant deviation from the QAPP, the nature and extent of the deviation will be recorded. Corrective action will be taken to remedy the deviation as necessary.

The Project QA/QC Officer has the responsibility of performing audits as deemed necessary and upon learning of any nonconformance. The PM may request an audit at any time. The PM and Task Leader(s) have ultimate responsibility for implementing corrective actions.

## 3.3 Reports to Management

Upon completion of any audit, the Project QA/QC Officer will document and report the QA/QC results and the identified issues (i.e., laboratory and/or field) to the Task Leader(s). The Task Leader(s) will evaluate the impact of the QA/QC issues and determine if the deviations will result in an adverse effect on the project conclusions. If it is determined that corrective actions are necessary, procedures outlined in Section 2.5.3 will be implemented.

# 4. DATA VALIDATION AND USABILITY

## 4.1 Data Review, Validation, and Verification Requirements

Data generated during performance of the RI/FS will undergo two levels of review. The laboratories and consultant will provide data verification. Data validation will be performed by consultant, and/or independent contractors, LDC and Neptune.

## 4.2 Validation and Verification Methods

### 4.2.1 Procedures Used for Verification of Field Data

Procedures to verify field data include checking for transcription errors and review of field logbooks at the time of data collection. Field sampling efforts as described in the field logbooks will be reviewed at the conclusion of each sampling event to confirm sampling procedures followed established procedures. If any significant nonconformance issues are noted they will be reported with a description of the potential effect of the nonconformance to the data. This task will be the responsibility of the Field Task Leader, or designee.

### 4.2.2 Procedures Used for Laboratory Data Verification

Initial data reduction, verification, and reporting will be performed by the laboratory as described in laboratory QAMs (Appendix B) and SOPs (Appendix C).

The laboratory will perform in-house analytical data verification under the direction of their own QA Officer and the Laboratory PM. The laboratory will be responsible for assessing data quality and advising of any data rated “preliminary”, “unacceptable”, or other notations that would caution the data user of possible nonconformance.

The Laboratory QA Officer will routinely audit or provide a secondary review of reports to assess data quality. This data assessment will be based on the assumption that the sample was properly collected and handled. Per NDEP guidance (2007), cation-anion balance calculations must be performed on groundwater samples prior to submission to clients in order to ensure the anion-cation balance is within the limits of Standard Methods Section 1030E.

The Laboratory QA Officer will conduct a systematic review of the data for compliance with the established quality control criteria based on spike, duplicate and blank results and an evaluation of data precision, accuracy, and completeness will be performed.

## 4.3 Procedures Used for Laboratory Data Validation

Data validation evaluates the analytical quality of a data set and occurs after data verification. The company that receives the laboratory deliverables is responsible for ensuring that the data are validated per NDEP requirements. The most current versions of USEPA’s National Functional Guidelines (USEPA 2017 and 2016) and NDEP’s data validation guidance will be used to conduct data validation. A summary of NDEP and Trust validation guidance follows and are included in Appendix E; the specific guidance documents should be reviewed for further detail by those responsible for planning analytical data collection efforts.

1. NDEP’s 2009 letter (NDEP 2009) combines all prior guidance on data validation in the BMI Plant Sites and Common Areas into a single document and defines validation stages 1, 2A, 2B, 3, and 4. According to this document, all data should be validated to at least Stage 2B (as defined in this document) and at least 10% of all data within a single Data Validation Summary Report (DVSR) should be validated to Stage 4 (as defined in this document). Note that the Stage 2B and Stage 4 requirements have been superseded as

described in items 4 and 5 below. This document also outlines the requirements for DVSRs.

2. NDEP's January 2012 letter (NDEP 2012a) provides guidance on how to qualify samples when contamination is found in blank samples.
3. NDEP's July 2012 letter (NDEP 2012b) explains how to validate asbestos data.
4. NDEP's 2017 email (NDEP 2017a) updates the previous validation requirements so that NERT treatability studies only need to be validated to Stage 2A (and not to Stage 2B or Stage 4). The email indicates that this change will be included in a forthcoming formal NDEP-issued guidance document, and the email serves as authorization to deviate from previously issued NDEP guidance until such a guidance document is issued.
5. NDEP's 2017 email (NDEP 2017b) updates the previous validation requirements so that all groundwater and surface water samples collected on or after March 1, 2017 only need to be validated to Stage 2A (and not to Stage 2B or Stage 4). Note that this update only applies to surface water and groundwater samples. The email indicates that this change will be included in a forthcoming formal NDEP-issued guidance document, and the email serves as authorization to deviate from previously issued NDEP guidance until such a guidance document is issued.

The Trust has adopted guidelines for validation based on the above NDEP guidance and correspondence combined with the end-use of the data, as summarized below:

<u>10% Stage 4 and 90% Stage 2B</u>

- Soil samples to support the RI/FS – Intended use of data is to support risk assessment/risk decision making
- Soil samples collected to characterize backfill material – Intended use of date is to evaluate if soil can be used on-site for backfill
- Samples taken to determine post remedial investigation, feasibility study, or treatability study characterization

<u>Stage 2A</u>

- Soil and groundwater samples for field-scale treatability studies – Intended use of data is to support feasibility study technology selection
- Groundwater samples to support the RI/FS – Intended use of data is to support risk assessment/risk decision making
- Site-wide groundwater monitoring program samples – Intended use of data is to support risk assessment/risk decision making
- Surface water samples – Intended use of data is to support risk assessment/risk decision making
- Samples for Interim Remedial Measures – Intended use of data is to support IRM performance monitoring; may also be used to support feasibility study technology selection

Stage 1

- Waste characterization samples to support disposal decisions – Validation not required by receiving entity
- Soil and groundwater samples for bench-scale treatability studies – Intended use of data is to support field studies; non-routine analytical generally performed by non-certified research laboratory
- Groundwater Extraction and Treatment System (GWETS) performance monitoring samples – Intended use of data is to support day-to-day GWETS operations
- GWETS compliance samples – Intended use of data is to document permit compliance; validation not required by receiving entity
- Geotechnical and microbial samples – Analyses are generally for physical properties rather than contaminant concentrations
- Samples to support H&S decisions – Intended use of data is to support internal decision making; validation not required by receiving entity

Data validation will be consistent with NDEP Supplemental Guidance on Data Validation for the BMI Plant Sites and Common Areas Projects (2009b and 2009c) as well as EPA Functional Guidelines (USEPA 2016 and 2017).

Stage 1 data validation checks include:

- Completeness Check
- Chain of Custody Review
- Evaluate sample results by comparing sample conditions upon receipt at the laboratory (e.g., preservation checks) and sample characteristics (e.g., percent moisture to the requirements and guidelines present in national or regional data validation documents, analytical methods(s) or contract.

Stage 2A data validation checks include:

- All parameters reviewed for Stage 1
- Review of Holding Times
- Review of Quality Control Summaries, including negative controls (blanks), positive controls (LCS), and Sample Specific Controls (replicates, matrix spikes, surrogates, tracers/yields)
- Frequency of QC samples checked for appropriateness (e.g., one LCS per twenty samples in a preparation batch)

Stage 2B data validation checks include:

- All parameters reviewed for Stage 1 and Stage 2B
- Initial and Continuing Calibration
- Review of Internal Standards
- Interference Check Sample, ICP Serial Dilution, GC/MS instrument performance check, and Reporting Limits
- Project or sampling specific items that have been identified for review

- Overall Assessment

Stage 4 data validation checks include:

- All parameters reviewed for Stage 1, Stage 2A, and Stage 2B
- Random recalculation (10-20%) of reported results versus raw data
- Review of Compound Identification, and TICs (where appropriate)
- Random check (10-20%) of integration and mass spectrum matches (where appropriate)

## 4.4 Reconciliation with Data Quality Objectives

Analytical results obtained from the project will be reconciled with the requirements specified in this QAPP. Data validation and usability include the final project checks to evaluate if the data obtained conforms to the project's objectives, and to estimate the effect of any deviations. Assessment of data for precision, accuracy, and completeness will be performed according to the following quantitative definitions.

### 4.4.1 Precision

If calculated from duplicate measurements:

$$RPD = \frac{(C_1 - C_2) * 100}{(C_1 + C_2)/2}$$

where:

RPD = relative percent difference
$C_1$ = larger of the two observed values
$C_2$ = smaller of the two observed values

If calculated from three or more replicates, use percent relative standard deviation (%RSD) rather than RPD:

$$\%RSD = \left(s/\bar{y}\right)100$$

%RSD = percent relative standard deviation
$s$ = standard deviation of replicates
$\bar{y}$ = mean of replicate analyses

Standard deviation is defined as follows:

$$s = \sqrt{\sum_{i=1}^{n} \frac{\left(y_i/\bar{y}\right)^{-2}}{n-1}}$$

$s$ = standard deviation
$y_i$ = measured value of the $i^{th}$ replicate
$\bar{y}$ = mean of replicate analyses
$n$ = number of replicates

### 4.4.2 Accuracy

For measurements where matrix spikes are used:

$$\%R = 100\left[\frac{S - U}{C_{sa}}\right]$$

$\%R$ = percent recovery
$S$ = measured concentration in spiked aliquot
$U$ = measured concentration in unspiked aliquot
$C_{sa}$ = actual concentration of spike added

### 4.4.3 Completeness (Statistical)

Defined as follows for all measurements:

$$\%C = 100\left[\frac{V}{T}\right]$$

$\%C$ = percent completeness
$V$ = number of measurements judged valid
$T$ = total number of planned measurements

## 4.5 Data Submittals to NDEP

### 4.5.1 Data Validation Summary Report

After the data validation process is complete, a DVSR will be prepared. The DVSR will summarize the data reviewed, any nonconformances, and validation actions. Data qualifiers and reason codes will be added based on this evaluation. The data qualifiers will be based on EPA guidance. A standard set of reason codes have been established and are listed on Table 10. The DVSR will include tables of all qualified data, the reason for qualification, any DQOs not met, the value of the exceedance, and the criteria exceeded will be provided, per NDEP specifications (NDEP 2013; NDEP 2009).

### 4.5.2 Electronic Data Deliverable

Following data validation, the EQuIS database will be used to create an Access database consistent with current NDEP guidance (2013).

## 4.6 Reconciliation With Data User Requirements

Each of the Trust's consultants will review the laboratory data for which they are responsible, as wells as the data's validation results to determine if the data meet the DQOs. Project results that do not meet DQOs will be reviewed by the appropriate consultant's Project QA Officer. Raw analytical data, laboratory notebooks, or other laboratory data may be obtained and examined as necessary. Corrective actions will begin with identifying the source of the problem. Potential problem sources may include failure to adhere to method procedures, improper data reduction, equipment malfunctions, or systemic contamination.

The first level of responsibility for identifying problems and initiating corrective action will be with the sampler or field personnel under the supervision of the appropriate Field Task Leader. The second level of responsibility will be with any person reviewing the data including the appropriate Project QA Officer and/or Analytical Task Leader.

If critical data are found to not meet quality control objectives the appropriate Analytical Task Leader will take appropriate action to obtain acceptable data as determined necessary. This may include re-analyzing existing samples, collecting new investigative samples, or other actions that will result in obtaining acceptable data. The specific course of action will be determined on a case-by-case basis based in part on the effect the nonconformance may have on the RI/FS objectives.

Data that provide useful information but are not critical for achieving RI/FS objectives will be appropriately documented if they do not meet quality control objectives. However, resampling or re-analysis to address such data will typically not be necessary.

Other corrective actions may include more intensive training, equipment repair followed by a more intensive preventive maintenance program, or removal of the source of systemic problems. Any and all corrective actions will be reviewed by the Task Leader(s) for certainty that resolution was achieved. Once resolved, the corrective action procedure will be fully documented.

# 5. QAPP ADDENDA

## 5.1 Procedures for Updating QAPP

Consultants are required to evaluate the existing QAPP requirements during the planning phase of a new project. Modifications to this QAPP to incorporate additional RI/FS data collection tasks will be addressed in QAPP Addenda. Appendix F presents the structure and the minimum task-specific elements that will be required to prepare a QAPP Addendum. The QAPP Addendum will be included as an appendix to any work plans for new RI/FS data collection tasks. The Addendum will be approved by NERT and NDEP at the time of work plan approval and will then become a part of this program QAPP. The following elements are required information for the preparation of the QAPP Addenda:

- Title, Version and Approval/Sign-off
- New Data Collection Task Information (includes DQOs, project organization, sampling design, sampling methods, analytical methods, field QC procedures)
- Laboratory Requirements (includes laboratory contact information, analytical methods, QC requirements, parameter lists, RLs, screening criteria, QAMs, and SOPs)
- Data Validation and Usability (identified stage of validation needed, validation subcontractor if necessary, validation criteria, guidance required, validation qualifiers, and reason codes)

## 5.2 Variance Submittal Procedure

Variances to the program QAPP must be documented in QAPP Addenda. For example if a new laboratory or analytical method is required to complete an on-going task, the associated information must be documented in a QAPP Addendum.

# 6. REFERENCES

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AECOM. 2016c. Surface Water Investigation Plan, NERT Remedial Investigation – Downgradient Study Area, Nevada Environmental Response Trust Site, Henderson, Nevada. December.

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ENVIRON. 2014a. Remedial Investigation and Feasibility Study Work Plan, Revision 2, Nevada Environmental Response Trust Site, Henderson, Nevada. June 19. NDEP Approved on July 2, 2014.

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NDEP. 2010. Email from Sara Rairick, Lab Certification Officer, Nevada Division of Environmental Protection, re: Sterile Filtration Required for Perchlorate Sampling. July 9.

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# TABLES

**TABLE 1. ANALYTICAL METHODS AND LABORATORIES**
**QUALITY ASSURANCE PROJECT PLAN**
**Nevada Environmental Response Trust Site; Henderson, Nevada**

| ANALYTES | MATRIX | ANALYTICAL METHOD | ANALYTICAL LABORATORY | SOPs REVIEW DATE[1] |
|---|---|---|---|---|
| Volatile Organic Compounds (VOCs) [2] | Water | EPA Method 8260B | TestAmerica (Irvine, CA) | March 2, 2015 |
| | Soil | EPA Method 8260B | | |
| | Water | EPA Method 8260B SIM | TestAmerica (Irvine, CA) | August 3, 2015 |
| | Soil | EPA Method 8260B SIM | | |
| | Soil Gas | EPA Method TO-15 | TestAmerica (Sacramento, CA) | July 31, 2015 |
| Semivolatile Organic Compounds (SVOCs) | Water | EPA Method 8270C | TestAmerica (Irvine, CA) | March 7, 2016 |
| | Soil | EPA Method 8270C | | |
| Phthalic Acid | Water | EPA Method 8270C | TestAmerica (Denver, CO) | January 31, 2014 |
| | Soil | EPA Method 8270C | | |
| Polyaromatic Hydrocarbons (PAHs) | Water | EPA Method 8270 SIM | TestAmerica (Irvine, CA) | September 22, 2015 |
| | Soil | EPA Method 8270 SIM | | |
| 4-chlorobenzenesulfonic acid (p-CBSA) | Water | EPA Method 8321A | TestAmerica (Sacramento, CA) | October 5, 2012 |
| Volatile Fatty Acids | Water | Lab SOP by Ion Chromatography SOP No. BF-MB-009, Rev 3 | TestAmerica (Buffalo, NY) | March 29, 2016 |
| Organochlorine Pesticides | Water | EPA Method 8081A | TestAmerica (Irvine, CA) | October 7, 2016 |
| | Soil | EPA Method 8081A | | |
| Organophosphorus Pesticides | Water | EPA Method 8141A | TestAmerica (Denver, CO) | September 30, 2016 |
| | Soil | EPA Method 8141A | | |
| PCBs as Aroclors | Water | EPA Method 8082 | TestAmerica (Irvine, CA) | November 4, 2016 |
| | Soil | EPA Method 8082 | | |
| PCBs as Congeners | Water | EPA Method 1668A | TestAmerica (Sacramento, CA) | September 2, 2016 |
| | Soil | EPA Method 1668A | | |
| Dioxins/Furans | Water | EPA Method 8290 or 8280[7] | TestAmerica (Sacramento, CA) | November 4, 2016 |
| | Soil | EPA Method 8290 or 8280[7] | | |
| Gasoline Range Organics (GROs) | Water | EPA Method 8015B | TestAmerica (Irvine, CA) | November 6, 2015 |
| | Soil | EPA Method 8015B | | |
| Diesel/Oil Range Organics (DROs/OROs) | Water | EPA Method 8015B | TestAmerica (Irvine, CA) | November 11, 2016 |
| | Soil | EPA Method 8015B | | |

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| ANALYTES | MATRIX | ANALYTICAL METHOD | ANALYTICAL LABORATORY | SOPs REVIEW DATE[1] |
|---|---|---|---|---|
| Methane | Water | Method RSK 175 | TestAmerica (Irvine, CA) | October 18, 2016 |
| Metals[3] | Water | EPA Method 200.7 / 6010 | TestAmerica (Irvine, CA) | March 2, 2015 |
| | Soil | EPA Method 6010 | | |
| Metals[4] | Water | EPA Method 200.8 / 6020 | TestAmerica (Irvine, CA) | July 12, 2017 |
| | Soil | EPA Method 6020 | | |
| Rare Earth Metals[5] | Water | EPA Method 6020A | TestAmerica (St. Louis, MO) | August 27, 2013 |
| | Soil | EPA Method 6020A | | |
| Arsenic III/V | Water | EPA Method 1632 | ALS (Kelso, Washington) | Febraury 15, 2014 |
| Mercury | Water | EPA Method 7470A | TestAmerica (Irvine, CA) | November 11, 2016 |
| | Soil | EPA Method 7471A | | |
| Hexavalent Chromium | Water | EPA Method 218.6 | TestAmerica (Irvine, CA) | December 14, 2015 |
| | Soil | EPA Method 7199 | | |
| Alkalinity and Carbonate | Water | SM 2320B | TestAmerica (Irvine, CA) | September 30, 2016 |
| | Soil | SM 2320B | | |
| Hardness | Water | SM 2340C | TestAmerica (Irvine, CA) | June 6, 2016 |
| Ammonia | Water | SM 4500-$NH_3$ D | TestAmerica (Irvine, CA) | August 30, 2013 |
| | Soil | SM 4500-$NH_3$ D | | |
| Total Kjedahl Nitrogen (TKN) | Water | EPA Method 351.2 | TestAmerica (Irvine, CA) | March 2, 2015 |
| Inorganic Anions[6] | Water | EPA Method 300.0 | TestAmerican (Irvine, CA) | June 30, 2017 |
| | Soil | EPA Method 300.0 | | |
| Chlorate | Water | EPA Method 300.1 | TestAmerican (Irvine, CA) | August 1, 2016 |
| | Soil | EPA Method 300.1 | | |
| Cyanide | Water | EPA Method 9014B | TestAmerica (Irvine, CA) | October 5, 2015 |
| | Soil | EPA Method 9014B | | |
| Formaldehyde | Water | EPA Method 8315A | TestAmerica (Irvine, CA) | March 1, 2017 |
| | Soil | EPA Method 8315A | | |

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| ANALYTES | MATRIX | ANALYTICAL METHOD | ANALYTICAL LABORATORY | SOPs REVIEW DATE[(1)] |
|---|---|---|---|---|
| Phosphorus | Water | EPA Method 365.3 | TestAmerica (Irvine, CA) | October 5, 2015 |
| Sulfide | Water | EPA Method 4500S-2 D | TestAmerica (Irvine, CA) | December 19, 2016 |
| Perchlorate | Water | EPA Method 314.0 | TestAmerica (Irvine, CA) | November 30, 2015 |
| | Soil | EPA Method 314.0 | | |
| pH | Soil | EPA Method 9045C | TestAmerica (Irvine, CA) | March 7, 2016 |
| Specific Conductance | Water | EPA Method 120.1 / SM 2510B | TestAmerica (Irvine, CA) | August 1, 2016 |
| | Soil | EPA Method 120.1 / SM 2510B | | |
| Total Dissolved Solids (TDS) | Water | SM 2540C | TestAmerica (Irvine, CA) | December 19, 2016 |
| Total and/or Dissolved Organic Carbon | Water | SM 5310B | TestAmerica (Irvine, CA) | March 22, 2016 |
| | Soil | SM 5310B | | |
| Surfactants | Soil | SM 5540C | TestAmerica (Irvine, CA) | June 6, 2016 |
| Radium 226 | Water | EPA Method 903.0 | TestAmerica (St. Louis, MO) | May 2, 2017 |
| | Soil | EPA Method 903.0 | | |
| Radium 228 | Water | EPA Method 904.0 | TestAmerica (St. Louis, MO) | August 21, 2013 |
| | Soil | EPA Method 904.0 | | |
| Thorium 228, 230, 232 and Uranium 234, 235, and 238 | Water | DOE EML HASL 300 A-01-R (alpha spectroscopy) | TestAmerica (St. Louis, MO) | May 30, 2017 |
| | Soil | DOE EML HASL 300 A-01-R (alpha spectroscopy) | | |
| Asbestos | Soil | EPA Method 540-R-97-028 modified per Berman & Kolk (2000) | EMSL Analytical (Cinnaminson, NJ) | June 2, 2017 |
| Helium | Soil Gas | ASTM D1946 | TestAmerica (Sacramento, CA) | July 1, 2011 |

**Notes:**

ASTM = American Society for Testing and Materials
DOE = Department of Energy
GS = gas chromatography
EPA = United States Environmental Protection Agency
KPA = Kinetic Phosphorescense Analyzer
SIM = Single Ion Monitoring

## TABLE 1. ANALYTICAL METHODS AND LABORATORIES
## QUALITY ASSURANCE PROJECT PLAN
## Nevada Environmental Response Trust Site; Henderson, Nevada

| ANALYTES | MATRIX | ANALYTICAL METHOD | ANALYTICAL LABORATORY | SOPs REVIEW DATE(1) |
|---|---|---|---|---|

GC/MS = gas chromatography-mass spectrometry

HASL = Health and Safety Laboratory

HPLC = High-performance liquid chromatography

EML = Environmental Measurements Laboratory

SM = Standard Method

(1) The Standard Operating Procedures (SOPs) Review Date is the date of the laboratory's current approved SOPs that will be implemented for this project. Laboratories are responsible for notifying ENVIRON of any revisions to the SOPs referenced above. The use of revised SOPs are subject to approval.

(2) 1,4 dioxane and 1,2,3-Trichloropropane will be run by EPA Method 8260B SIM.

(3) Silicon and phosphorus can also analyzed by this method.

(4) Certain metals will be analyzed by EPA Method 200.8 / 6020 to overcome matrix interference from saltine groundwater and/or to achieve lower PQLs and MDLs.

(5) Niobium, palladium, sulfur and/or uranium

(6) Fluoride, chloride, bromide, sulfate, ortho-phosphate as $PO_4$, nitrate, and/or nitrate.

(7) EPA Method 8280 may be used to analyze dioxin samples with concentrations that are too high to be accurately measured by EPA Method 8290. An initial screening will be performed by the laboratory to determine which dioxin analysis method should be used.

**Sources:**

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**TABLE 2. SOIL ANALYTES AND ANALYTICAL PERFORMANCE CRITERIA**
**QUALITY ASSURANCE PROJECT PLAN**
**Nevada Environmental Response Trust Site; Henderson, Nevada**

| ANALYTES | CAS Number | Screening Level | Screening Level Source[1] | Practical Quantitation Limit (PQL) | Method Detection Limit (MDL) | QUALITY CONTROL LIMITS[2] Surrogate %R | | Duplicate RPD | Matrix Spike %R | Matrix Spike RPD | Blank Spike/LCS %R | Blank Spike/LCS RPD |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| **Metals (mg/kg)** | | | | | | | | | | | | |
| *EPA Method 6010* | | | | | | | | | | | | |
| Aluminum | 7429-90-5 | 100,000 | NDEP 2017 | 10 | 7.7 | -- | -- | 50 | 75 - 125 | 20 | 80 - 120 | 20 |
| Barium | 7440-39-3 | 100,000 | NDEP 2017 | 1.5 | 0.75 | -- | -- | 50 | 75 - 125 | 20 | 80 - 120 | 20 |
| Beryllium | 7440-41-7 | 2,540 | NDEP 2017 | 0.50 | 0.25 | -- | -- | 50 | 75 - 125 | 20 | 80 - 120 | 20 |
| Boron | 7440-42-8 | 100,000 | NDEP 2017 | 5 | 2.5 | -- | -- | 50 | 75 - 125 | 20 | 80 - 120 | 20 |
| Cadmium | 7440-43-9 | 1,114 | NDEP 2017 | 0.5 | 0.25 | -- | -- | 50 | 75 - 125 | 20 | 80 - 120 | 20 |
| Calcium | 7440-70-2 | -- | NDEP 2017 | 25 | 13.5 | -- | -- | 50 | 75 - 125 | 20 | 80 - 120 | 20 |
| Chromium (total) | 7440-47-3 | 100,000 | NDEP 2017 | 1 | 0.5 | -- | -- | 50 | 75 - 125 | 20 | 80 - 120 | 20 |
| Cobalt | 7440-48-4 | 385 | NDEP 2017 | 1 | 0.5 | -- | -- | 50 | 75 - 125 | 20 | 80 - 120 | 20 |
| Copper | 7440-50-8 | 3,670 | NDEP 2017 | 2 | 1.1 | -- | -- | 50 | 75 - 125 | 20 | 80 - 120 | 20 |
| Iron | 7439-89-6 | 100,000 | NDEP 2017 | 10 | 6.9 | -- | -- | 50 | 75 - 125 | 20 | 80 - 120 | 20 |
| Lead | 7439-92-1 | 800 | NDEP 2015 | 2 | 1 | -- | -- | 50 | 75 - 125 | 20 | 80 - 120 | 20 |
| Magnesium | 7439-95-4 | 100,000 | NDEP 2017 | 10 | 5 | -- | -- | 50 | 75 - 125 | 20 | 80 - 120 | 20 |
| Manganese | 7439-96-5 | 28,100 | NDEP 2017 | 2 | 1 | -- | -- | 50 | 75 - 125 | 20 | 80 - 120 | 20 |
| Molybdenum | 7439-98-7 | 6,490 | NDEP 2017 | 2 | 1 | -- | -- | 50 | 75 - 125 | 20 | 80 - 120 | 20 |
| Nickel | 7440-02-0 | 24,700 | NDEP 2017 | 2 | 1 | -- | -- | 50 | 75 - 125 | 20 | 80 - 120 | 20 |
| Phosphorus | 7723-14-0 | -- | NDEP 2017 | 5 | 2.5 | -- | -- | 50 | 75 - 125 | 20 | 80 - 120 | 20 |
| Potassium | 7440-09-7 | -- | NDEP 2017 | 62.5 | 32.5 | -- | -- | 50 | 75 - 125 | 20 | 80 - 120 | 20 |
| Silver | 7440-22-4 | 6,490 | NDEP 2017 | 1.5 | 0.89 | -- | -- | 50 | 75 - 125 | 20 | 80 - 120 | 20 |
| Sodium | 7440-23-5 | -- | NDEP 2017 | 62.5 | 32 | -- | -- | 50 | 75 - 125 | 20 | 80 - 120 | 20 |
| Strontium | 7440-24-6 | 100,000 | NDEP 2017 | 5 | 2.5 | -- | -- | 50 | 75 - 125 | 20 | 80 - 120 | 20 |
| Tin | 7440-31-5 | 100,000 | NDEP 2017 | 10 | 5 | -- | -- | 50 | 75 - 125 | 20 | 80 - 120 | 20 |
| Titanium | 7440-32-6 | 100,000 | NDEP 2017 | 2 | 1 | -- | -- | 50 | 75 - 125 | 20 | 80 - 120 | 20 |
| Tungsten | 7440-33-7 | 1,040 | NDEP 2017 | 5 | 2.5 | -- | -- | 50 | 75 - 125 | 20 | 80 - 120 | 20 |
| Vanadium | 7440-62-2 | 6,420 | NDEP 2017 | 1 | 0.5 | -- | -- | 50 | 75 - 125 | 20 | 80 - 120 | 20 |
| Zinc | 7440-66-6 | 100,000 | NDEP 2017 | 5 | 2.5 | -- | -- | 50 | 75 - 125 | 20 | 80 - 120 | 20 |
| Zirconium | 7440-67-7 | 104 | NDEP 2017 | 10 | 5 | -- | -- | 50 | 75 - 125 | 20 | 80 - 120 | 20 |
| *EPA Method 6020* | | | | | | | | | | | | |
| Antimony | 7440-36-0 | 519 | NDEP 2017 | 1 | 0.27 | -- | -- | 50 | 75 - 125 | 20 | 80 - 120 | 20 |

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| ANALYTES | CAS Number | Screening Level | Screening Level Source[1] | Practical Quantitation Limit (PQL) | Method Detection Limit (MDL) | QUALITY CONTROL LIMITS[2] Surrogate %R | | Duplicate RPD | Matrix Spike %R | Matrix Spike RPD | Blank Spike/LCS %R | Blank Spike/LCS RPD |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| Arsenic | 7440-38-2 | 2.15 | NDEP 2017 | 0.5 | 0.25 | -- | -- | 50 | 80 - 120 | 20 | 80 - 120 | 20 |
| Selenium | 7782-49-2 | 6,490 | NDEP 2017 | 1 | 0.2 | -- | -- | 50 | 80 - 120 | 20 | 80 - 120 | 20 |
| Thallium | 7440-28-0 | 13 | NDEP 2017 | 0.5 | 0.25 | -- | -- | 50 | 75 - 125 | 20 | 80 - 120 | 20 |
| *EPA Method 6020A* | | | | | | | | | | | | |
| Niobium | 7440-03-1 | 130 | NDEP 2017 | 2.5 | 0.38 | -- | -- | 50 | 75 - 125 | 30 | 80 - 120 | 20 |
| Palladium | 7440-05-3 | -- | NDEP 2017 | 0.1 | 0.011 | -- | -- | 50 | 75 - 125 | 30 | 80 - 120 | 20 |
| Sulfur | 7704-34-9 | -- | NDEP 2017 | 500 | 81.1 | -- | -- | 50 | 75 - 125 | 30 | 80 - 120 | 20 |
| Total Uranium | 7440-61-1 | 3,830 | NDEP 2017 | 0.1 | 0.0199 | -- | -- | 50 | 75 - 125 | 30 | 80 - 120 | 20 |
| *EPA Method 7199* | | | | | | | | | | | | |
| Chromium (hexavalent) | 18540-29-9 | 7.01 | NDEP 2017 | 0.3 | 0.15 | -- | -- | 50 | 55 - 110 | 20 | 65 - 110 | 20 |
| *EPA Method 7471A* | | | | | | | | | | | | |
| Mercury | 7439-97-6 | 3.13 | NDEP 2017 | 0.02 | 0.012 | -- | -- | 50 | 70 - 130 | 20 | 80 - 120 | 20 |
| **Volatile Organic Compounds (mg/kg)** | | | | | | | | | | | | |
| *EPA Method 8260B* | | | | | | | | | | | | |
| 1,1,1,2-Tetrachloroethane | 630-20-6 | 9.95 | NDEP 2017 | 2.00 | 0.001 | -- | -- | 50 | 65 - 145 | 20 | 70 - 130 | 20 |
| 1,1,1-Trichloroethane | 71-55-6 | 638 | NDEP 2017 | 0.001 | 0.0005 | -- | -- | 50 | 65 - 145 | 20 | 65 - 135 | 20 |
| 1,1,2,2-Tetrachloroethane | 79-34-5 | 3.18 | NDEP 2017 | 0.002 | 0.001 | -- | -- | 50 | 40 - 160 | 30 | 55 - 140 | 30 |
| 1,1,2-Trichloroethane | 79-00-5 | 5.79 | NDEP 2017 | 0.001 | 0.0005 | -- | -- | 50 | 65 - 140 | 30 | 65 - 135 | 20 |
| 1,1-Dichloroethane | 75-34-3 | 17.3 | NDEP 2017 | 0.001 | 0.0005 | -- | -- | 50 | 65 - 135 | 25 | 70 - 130 | 20 |
| 1,1-Dichloroethene | 75-35-4 | 1,100 | NDEP 2017 | 0.002 | 0.0005 | -- | -- | 50 | 65 - 135 | 25 | 70 - 125 | 20 |
| 1,1-Dichloropropene | 563-58-6 | -- | NDEP 2017 | 0.001 | 0.0005 | -- | -- | 50 | 65 - 135 | 20 | 70 - 130 | 20 |
| 1,2,3-Trichlorobenzene | 87-61-6 | 151 | NDEP 2017 | 0.002 | 0.001 | -- | -- | 50 | 45 - 145 | 30 | 60 - 130 | 20 |
| 1,2,3-Trichloropropane | 96-18-4 | 0.121 | NDEP 2017 | 0.01 | 0.001 | -- | -- | 50 | 50 - 150 | 30 | 60 - 135 | 25 |
| 1,2,4-Trichlorobenzene | 120-82-1 | 125 | NDEP 2017 | 0.01 | 0.001 | -- | -- | 50 | 50 - 140 | 30 | 70 - 135 | 20 |
| 1,2,4-Trimethylbenzene | 95-63-6 | 218 | NDEP 2017 | 0.002 | 0.001 | -- | -- | 50 | 65 - 140 | 25 | 70 - 125 | 20 |
| 1,2-Dibromo-3-Chloropropane | 96-12-8 | 0.0714 | NDEP 2017 | 0.005 | 0.002 | -- | -- | 50 | 40 - 150 | 30 | 50 - 135 | 30 |
| 1,2-Dibromoethane (EDB) | 106-93-4 | 0.184 | NDEP 2017 | 0.001 | 0.0005 | -- | -- | 50 | 65 - 140 | 25 | 70 - 130 | 20 |
| 1,2-Dichlorobenzene | 95-50-1 | 376 | NDEP 2017 | 0.001 | 0.0005 | -- | -- | 50 | 70 - 130 | 25 | 75 - 120 | 20 |
| 1,2-Dichloroethane | 107-06-2 | 2.3 | NDEP 2017 | 0.001 | 0.0005 | -- | -- | 50 | 60 - 150 | 25 | 60 - 140 | 20 |

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| ANALYTES | CAS Number | Screening Level | Screening Level Source[1] | Practical Quantitation Limit (PQL) | Method Detection Limit (MDL) | QUALITY CONTROL LIMITS[2] Surrogate %R | | Duplicate RPD | Matrix Spike %R | Matrix Spike RPD | Blank Spike/LCS %R | Blank Spike/LCS RPD |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| 1,2-Dichloropropane | 78-87-5 | 4.98 | NDEP 2017 | 0.001 | 0.0005 | -- | -- | 50 | 65 - 130 | 20 | 70 - 130 | 20 |
| 1,3,5-Trimethylbenzene | 108-67-8 | 182 | NDEP 2017 | 0.002 | 0.001 | -- | -- | 50 | 65 - 135 | 25 | 70 - 125 | 20 |
| 1,3-Dichlorobenzene | 541-73-1 | 373 | NDEP 2017 | 0.001 | 0.0005 | -- | -- | 50 | 70 - 130 | 25 | 75 - 125 | 20 |
| 1,3-Dichloropropane | 142-28-9 | 18,300 | NDEP 2017 | 0.001 | 0.0005 | -- | -- | 50 | 65 - 140 | 25 | 70 - 125 | 20 |
| 1,4-Dichlorobenzene | 106-46-7 | 47.2 | NDEP 2017 | 0.001 | 0.0005 | -- | -- | 50 | 70 - 130 | 25 | 75 - 120 | 20 |
| 2,2-Dichloropropane | 594-20-7 | -- | NDEP 2017 | 0.002 | 0.001 | -- | -- | 50 | 65 - 150 | 25 | 60 - 145 | 20 |
| 2-Butanone | 78-93-3 | 28,400 | NDEP 2017 | 0.01 | 0.005 | -- | -- | 50 | 25 - 170 | 40 | 40 - 145 | 35 |
| 2-Chlorotoluene | 95-49-8 | 907 | NDEP 2017 | 0.002 | 0.001 | -- | -- | 50 | 60 - 135 | 25 | 70 - 125 | 20 |
| 2-Hexanone | 591-78-6 | 1,650 | NDEP 2017 | 0.010 | 0.005 | -- | -- | 50 | 35 - 160 | 40 | 40 - 150 | 35 |
| 4-Chlorotoluene | 106-43-4 | 18,300 | NDEP 2017 | 0.002 | 0.001 | -- | -- | 50 | 65 - 135 | 25 | 75 - 125 | 20 |
| 4-Methyl-2-pentanone | 108-10-1 | 3,360 | NDEP 2017 | 0.01 | 0.0025 | -- | -- | 50 | 40 - 155 | 40 | 40 - 145 | 35 |
| Acetone | 67-64-1 | 100,000 | NDEP 2017 | 0.020 | 0.008 | -- | -- | 50 | 20 - 145 | 40 | 25 - 145 | 30 |
| Benzene | 71-43-2 | 5.82 | NDEP 2017 | 0.001 | 0.0005 | -- | -- | 50 | 65 - 130 | 20 | 65 - 120 | 20 |
| Bromobenzene | 108-86-1 | 679 | NDEP 2017 | 0.002 | 0.001 | -- | -- | 50 | 65 - 140 | 25 | 75 - 120 | 20 |
| Bromochloromethane | 74-97-5 | 692 | NDEP 2017 | 0.002 | 0.001 | -- | -- | 50 | 65 - 145 | 25 | 70 - 135 | 20 |
| Bromodichloromethane | 75-27-4 | 1.43 | NDEP 2017 | 0.001 | 0.0005 | -- | -- | 50 | 65 - 145 | 20 | 70 - 135 | 20 |
| Bromoform | 75-25-2 | 104 | NDEP 2017 | 0.002 | 0.001 | -- | -- | 50 | 50 - 145 | 30 | 55 - 135 | 25 |
| Bromomethane | 74-83-9 | 33.3 | NDEP 2017 | 0.002 | 0.001 | -- | -- | 50 | 60 - 155 | 25 | 60 - 145 | 20 |
| Carbon Tetrachloride | 56-23-5 | 3.24 | NDEP 2017 | 0.002 | 0.0005 | -- | -- | 50 | 60 - 145 | 25 | 65 - 140 | 20 |
| Chlorobenzene | 108-90-7 | 18,300 | NDEP 2017 | 0.001 | 0.0005 | -- | -- | 50 | 70 - 130 | 25 | 75 - 120 | 20 |
| Chloroethane | 75-00-3 | 2,110 | NDEP 2017 | 0.002 | 0.001 | -- | -- | 50 | 60 - 150 | 25 | 60 - 140 | 25 |
| Chloroform | 67-66-3 | 1.53 | NDEP 2017 | 0.001 | 0.0005 | -- | -- | 50 | 65 - 135 | 20 | 70 - 130 | 20 |
| Chloromethane | 74-87-3 | 510 | NDEP 2017 | 0.002 | 0.001 | -- | -- | 50 | 40 - 145 | 25 | 45 - 145 | 25 |
| cis-1,2-Dichloroethene | 156-59-2 | 2,360 | NDEP 2017 | 0.001 | 0.0005 | -- | -- | 50 | 65 - 135 | 25 | 70 - 125 | 20 |
| cis-1,3-Dichloropropene | 10061-01-5 | 25.7 | NDEP 2017 | 0.001 | 0.0005 | -- | -- | 50 | 70 - 135 | 25 | 75 - 125 | 20 |
| Dibromochloromethane | 124-48-1 | 43.3 | NDEP 2017 | 0.001 | 0.0005 | -- | -- | 50 | 60 - 145 | 25 | 65 - 140 | 20 |
| Dibromomethane | 74-95-3 | 10,000 | NDEP 2017 | 0.001 | 0.0005 | -- | -- | 50 | 65 - 140 | 25 | 70 - 130 | 20 |
| Dichlorodifluoromethane | 75-71-8 | 403 | NDEP 2017 | 0.002 | 0.001 | -- | -- | 50 | 30 - 160 | 35 | 35 - 160 | 30 |
| Diisopropyl ether (DIPE) | 108-20-3 | 2,260 | NDEP 2017 | 0.002 | 0.001 | -- | -- | 50 | 60 - 150 | 25 | 60 - 140 | 20 |
| Ethylbenzene | 100-41-4 | 233 | NDEP 2017 | 0.001 | 0.0005 | -- | -- | 50 | 70 - 135 | 25 | 70 - 125 | 20 |

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| ANALYTES | CAS Number | Screening Level | Screening Level Source[1] | Practical Quantitation Limit (PQL) | Method Detection Limit (MDL) | QUALITY CONTROL LIMITS[2] Surrogate %R | Duplicate RPD | Matrix Spike %R | Matrix Spike RPD | Blank Spike/LCS %R | Blank Spike/LCS RPD |
|---|---|---|---|---|---|---|---|---|---|---|---|
| Ethyl-tert-butyl ether (ETBE) | 637-92-3 | -- | NDEP 2017 | 0.002 | 0.001 | -- -- | 50 | 60 - 145 | 30 | 60 - 140 | 20 |
| Hexachlorobutadiene | 87-68-3 | 6.14 | NDEP 2017 | 0.002 | 0.001 | -- -- | 50 | 50 - 145 | 35 | 60 - 135 | 20 |
| Isopropyl benzene | 98-82-8 | 91,600 | NDEP 2017 | 0.001 | 0.0005 | -- -- | 50 | 70 - 145 | 25 | 75 - 130 | 20 |
| m,p-Xylene[5] | 179601-23-1 | 387 | NDEP 2017 | 0.002 | 0.001 | -- -- | 50 | 70 - 130 | 25 | 70 - 125 | 20 |
| Methylene Chloride | 75-09-2 | 1,550 | NDEP 2017 | 0.01 | 0.005 | -- -- | 50 | 55 - 145 | 25 | 55 - 135 | 20 |
| Methyl-tert-butyl ether (MTBE) | 1634-04-4 | 238 | NDEP 2017 | 0.00 | 0.001 | -- -- | 50 | 55 - 155 | 35 | 60 - 140 | 25 |
| Naphthalene | 91-20-3 | 18.4 | NDEP 2017 | 0.002 | 0.001 | -- -- | 50 | 40 - 150 | 40 | 55 - 135 | 25 |
| n-Butylbenzene | 104-51-8 | 108 | NDEP 2017 | 0.002 | 0.001 | -- -- | 50 | 55 - 145 | 30 | 70 - 130 | 20 |
| n-Propylbenzene | 103-65-1 | 264 | NDEP 2017 | 0.001 | 0.0005 | -- -- | 50 | 65 - 140 | 25 | 70 - 130 | 20 |
| o-Xylene | 95-47-6 | 434 | NDEP 2017 | 0.001 | 0.0005 | -- -- | 50 | 65 - 130 | 25 | 70 - 125 | 20 |
| p-Isopropyltoluene | 99-87-6 | 647 | NDEP 2017 | 0.001 | 0.0005 | -- -- | 50 | 60 - 140 | 25 | 75 - 125 | 20 |
| sec-Butylbenzene | 135-98-8 | 145 | NDEP 2017 | 0.002 | 0.001 | -- -- | 50 | 60 - 135 | 25 | 70 - 125 | 20 |
| Styrene | 100-42-5 | 867 | NDEP 2017 | 0.001 | 0.0005 | -- -- | 50 | 70 - 140 | 25 | 75 - 130 | 20 |
| tert-Amyl-methyl ether (TAME) | 994-05-8 | -- | NDEP 2017 | 0.002 | 0.001 | -- -- | 50 | 60 - 150 | 25 | 60 - 145 | 20 |
| tert-Butyl alcohol (TBA) | 75-65-0 | 21,300 | NDEP 2017 | 0.050 | 0.01 | -- -- | 50 | 65 - 145 | 30 | 70 - 135 | 20 |
| tert-Butylbenzene | 98-06-6 | 183 | NDEP 2017 | 0.002 | 0.001 | -- -- | 50 | 60 - 140 | 25 | 70 - 125 | 20 |
| Tetrachloroethene | 127-18-4 | 117 | NDEP 2017 | 0.001 | 0.0005 | -- -- | 50 | 65 - 135 | 25 | 70 - 125 | 20 |
| Toluene | 108-88-3 | 817 | NDEP 2017 | 0.001 | 0.0005 | -- -- | 50 | 70 - 130 | 20 | 70 - 125 | 20 |
| trans-1,2-Dichloroethene | 156-60-5 | 183,000 | NDEP 2017 | 0.001 | 0.0005 | -- -- | 50 | 70 - 135 | 25 | 70 - 125 | 20 |
| trans-1,3-Dichloropropene | 10061-02-6 | -- | NDEP 2017 | 0.001 | 0.0005 | -- -- | 50 | 60 - 145 | 25 | 70 - 135 | 20 |
| Trichloroethene | 79-01-6 | 6.92 | NDEP 2017 | 0.00 | 0.0005 | -- -- | 50 | 65 - 140 | 25 | 70 - 125 | 20 |
| Trichlorofluoromethane | 75-69-4 | 1,210 | NDEP 2017 | 0.002 | 0.001 | -- -- | 50 | 55 - 155 | 25 | 60 - 145 | 25 |
| Vinyl chloride | 75-01-4 | 2.21 | NDEP 2017 | 0.002 | 0.001 | -- -- | 50 | 55 - 140 | 30 | 55 - 135 | 25 |
| 4-Bromofluorobenzene (Surr) | 460-00-4 | -- | -- | -- | -- | 79 - 120 | -- | -- -- | -- | -- -- | -- |
| Dibromofluoromethane (Surr) | 1868-53-7 | -- | -- | -- | -- | 60 - 120 | -- | -- -- | -- | -- -- | -- |
| Toluene-d8 (Surr) | 2037-26-5 | -- | -- | -- | -- | 79 - 123 | -- | -- -- | -- | -- -- | -- |
| *EPA Method 8260B SIM* | | | | | | | | | | | |
| 1,2,3-Trichloropropane | 96-18-4 | 0.121 | NDEP 2013 | 0.01 | 0.004 | -- -- | 50 | 50 - 150 | 30 | #### - 135 | 25 |

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**Nevada Environmental Response Trust Site; Henderson, Nevada**

| | | | | | | QUALITY CONTROL LIMITS[2] | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | | | | Practical Quantitation Limit | Method Detection Limit | Surrogate | Duplicate | Matrix Spike | | Blank Spike/LCS | |
| ANALYTES | CAS Number | Screening Level | Screening Level Source[1] | (PQL) | (MDL) | %R | RPD | %R | RPD | %R | RPD |
| 1,4-dioxane | 123-91-1 | 36.3 | NDEP 2013 | 0.01 | 0.0011 | -- -- | 50 | 70 - 130 | 30 | 70 - 130 | 30 |
| Dibromofluoromethane (Surr) | 1868-53-7 | -- | -- | -- | -- | 80 - 125 | -- | -- -- | -- | -- -- | -- |
| **Semi-Volatile Organic Compounds (mg/kg)** | | | | | | | | | | | |
| *EPA Method 8270C* | | | | | | | | | | | |
| 1-Methylnaphthalene | 90-12-0 | 81.3 | NDEP 2017 | 0.35 | 0.15 | -- -- | 50 | 60 - 140 | 30 | 60 - 140 | 30 |
| 2,4,5-Trichlorophenol | 95-95-4 | 91,600 | NDEP 2017 | 0.33 | 0.13 | -- -- | 50 | 45 - 120 | 20 | 50 - 120 | 20 |
| 2,4,6-Trichlorophenol | 88-06-2 | 233 | NDEP 2017 | 0.33 | 0.075 | -- -- | 50 | 45 - 120 | 25 | 50 - 120 | 20 |
| 2,4-Dichlorophenol | 120-83-2 | 3,220 | NDEP 2017 | 0.33 | 0.067 | -- -- | 50 | 45 - 120 | 25 | 45 - 120 | 20 |
| 2,4-Dimethylphenol | 105-67-9 | 18,300 | NDEP 2017 | 0.33 | 0.13 | -- -- | 50 | 30 - 120 | 25 | 40 - 120 | 20 |
| 2,4-Dinitrophenol | 51-28-5 | 1,830 | NDEP 2017 | 0.66 | 0.33 | -- -- | 50 | 20 - 120 | 25 | 25 - 120 | 25 |
| 2,4-Dinitrotoluene | 121-14-2 | 8.30 | NDEP 2017 | 0.33 | 0.08 | -- -- | 50 | 50 - 125 | 25 | 55 - 125 | 20 |
| 2,6-Dinitrotoluene | 606-20-2 | 2.36 | NDEP 2017 | 0.33 | 0.095 | -- -- | 50 | 50 - 125 | 20 | 55 - 125 | 20 |
| 2-Chloronaphthalene | 91-58-7 | 175 | NDEP 2017 | 0.33 | 0.067 | -- -- | 50 | 45 - 120 | 20 | 45 - 120 | 20 |
| 2-Chlorophenol | 95-57-8 | 6,490 | NDEP 2017 | 0.33 | 0.07 | -- -- | 50 | 40 - 120 | 20 | 40 - 120 | 20 |
| 2-Methylnaphthalene | 91-57-6 | 368 | NDEP 2017 | 0.33 | 0.07 | -- -- | 50 | 40 - 120 | 20 | 45 - 120 | 20 |
| 2-Methylphenol | 95-48-7 | 45,800 | NDEP 2017 | 0.33 | 0.08 | -- -- | 50 | 40 - 120 | 25 | 40 - 120 | 20 |
| 2-Nitroaniline | 88-74-4 | 8,880 | NDEP 2017 | 0.33 | 0.067 | -- -- | 50 | 45 - 120 | 25 | 50 - 125 | 20 |
| 2-Nitrophenol | 88-75-5 | -- | NDEP 2017 | 0.33 | 0.133 | -- -- | 50 | 40 - 120 | 25 | 45 - 120 | 20 |
| 3,3'-Dichlorobenzidine | 91-94-1 | 5.70 | NDEP 2017 | 0.83 | 0.15 | -- -- | 50 | 20 - 130 | 25 | 20 - 130 | 25 |
| 3-Methylphenol + 4-Methylphenol | 106-44-5 | 45,800 | NDEP 2017 | 0.33 | 0.133 | -- -- | 50 | 50 - 120 | 25 | 50 - 120 | 20 |
| 3-Nitroaniline | 99-09-2 | -- | NDEP 2017 | 0.33 | 0.133 | -- -- | 50 | 30 - 120 | 25 | 35 - 120 | 25 |
| 4-Bromophenyl phenyl ether | 101-55-3 | -- | NDEP 2017 | 0.33 | 0.075 | -- -- | 50 | 45 - 120 | 20 | 45 - 120 | 20 |
| 4-Chloro-3-methylphenol | 59-50-7 | 91,600 | NDEP 2017 | 0.33 | 0.07 | -- -- | 50 | 50 - 125 | 25 | 50 - 125 | 20 |
| 4-Chloroaniline | 106-47-8 | 18.2 | NDEP 2017 | 0.33 | 0.133 | -- -- | 50 | 20 - 120 | 30 | 20 - 120 | 30 |
| 4-Chlorophenyl phenyl ether | 7005-72-3 | -- | NDEP 2017 | 0.33 | 0.085 | -- -- | 50 | 50 - 120 | 25 | 55 - 120 | 20 |
| 4-Nitroaniline | 100-01-6 | 128 | NDEP 2017 | 0.83 | 0.133 | -- -- | 50 | 40 - 125 | 30 | 45 - 125 | 20 |

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| ANALYTES | CAS Number | Screening Level | Screening Level Source[1] | Practical Quantitation Limit (PQL) | Method Detection Limit (MDL) | QUALITY CONTROL LIMITS[2] Surrogate %R | | Duplicate RPD | Matrix Spike %R | Matrix Spike RPD | Blank Spike/LCS %R | Blank Spike/LCS RPD |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| 4-Nitrophenol | 100-02-7 | 7,330 | NDEP 2017 | 0.83 | 0.14 | -- | -- | 50 | 35 - 125 | 30 | 40 - 125 | 20 |
| Acenaphthene | 83-32-9 | 118 | NDEP 2017 | 0.33 | 0.067 | -- | -- | 50 | 45 - 120 | 25 | 50 - 120 | 20 |
| Acenaphthylene | 208-96-8 | -- | NDEP 2017 | 0.33 | 0.07 | -- | -- | 50 | 45 - 120 | 20 | 50 - 120 | 20 |
| Aniline | 62-53-3 | 450 | NDEP 2017 | 0.42 | 0.085 | -- | -- | 50 | 25 - 120 | 30 | 25 - 120 | 20 |
| Anthracene | 120-12-7 | 4.26 | NDEP 2017 | 0.33 | 0.08 | -- | -- | 50 | 55 - 120 | 25 | 55 - 120 | 20 |
| Benzidine | 92-87-5 | 0.0112 | NDEP 2017 | 0.66 | 0.66 | -- | -- | 50 | 20 - 120 | 30 | 20 - 120 | 30 |
| Benzo[a]anthracene | 56-55-3 | 3.23 | NDEP 2017 | 0.33 | 0.07 | -- | -- | 50 | 50 - 120 | 25 | 55 - 120 | 20 |
| Benzo[a]pyrene | 50-32-8 | 0.323 | NDEP 2017 | 0.33 | 0.067 | -- | -- | 50 | 45 - 125 | 25 | 50 - 125 | 20 |
| Benzo[b]fluoranthene | 205-99-2 | 3.23 | NDEP 2017 | 0.33 | 0.067 | -- | -- | 50 | 45 - 125 | 30 | 45 - 125 | 25 |
| Benzo[g,h,i]perylene | 191-24-2 | 25,300 | NDEP 2017 | 0.33 | 0.11 | -- | -- | 50 | 25 - 130 | 30 | 35 - 130 | 25 |
| Benzo[k]fluoranthene | 207-08-9 | 32.3 | NDEP 2017 | 0.33 | 0.07 | -- | -- | 50 | 45 - 125 | 30 | 45 - 125 | 25 |
| Benzoic acid | 65-85-0 | 100,000 | NDEP 2017 | 0.83 | 0.15 | -- | -- | 50 | 20 - 120 | 30 | 20 - 120 | 30 |
| Benzyl alcohol | 100-51-6 | 91,600 | NDEP 2017 | 0.33 | 0.2 | -- | -- | 50 | 20 - 120 | 30 | 35 - 120 | 25 |
| Bis(2-chloroethoxy)methane | 111-91-1 | 2,750 | NDEP 2017 | 0.33 | 0.133 | -- | -- | 50 | 45 - 120 | 25 | 45 - 120 | 20 |
| Bis(2-chloroethyl)ether | 111-44-4 | 1.35 | NDEP 2017 | 0.33 | 0.06 | -- | -- | 50 | 35 - 110 | 25 | 35 - 120 | 25 |
| Bis(2-ethylhexyl) phthalate | 117-81-7 | 183 | NDEP 2017 | 0.33 | 0.09 | -- | -- | 50 | 45 - 130 | 25 | 50 - 130 | 20 |
| Butyl benzyl phthalate | 85-68-7 | 1,350 | NDEP 2017 | 0.33 | 0.08 | -- | -- | 50 | 45 - 125 | 25 | 50 - 125 | 20 |
| Chrysene | 218-01-9 | 323 | NDEP 2017 | 0.33 | 0.075 | -- | -- | 50 | 55 - 120 | 25 | 55 - 120 | 20 |
| Dibenz(a,h)anthracene | 53-70-3 | 0.323 | NDEP 2017 | 0.42 | 0.10 | -- | -- | 50 | 25 - 135 | 30 | 40 - 135 | 25 |
| Dibenzofuran | 132-64-9 | 171 | NDEP 2017 | 0.33 | 0.067 | -- | -- | 50 | 50 - 120 | 25 | 55 - 120 | 20 |
| Diethyl phthalate | 84-66-2 | 100,000 | NDEP 2017 | 0.33 | 0.095 | -- | -- | 50 | 50 - 125 | 25 | 50 - 125 | 20 |
| Dimethylphthalate | 131-11-3 | 100,000 | NDEP 2017 | 0.33 | 0.067 | -- | -- | 50 | 45 - 125 | 25 | 50 - 125 | 20 |
| Di-n-butyl phthalate | 84-74-2 | 91,600 | NDEP 2017 | 0.33 | 0.09 | -- | -- | 50 | 50 - 125 | 25 | 50 - 125 | 20 |
| Di-n-octyl phthalate | 117-84-0 | 9,160 | NDEP 2017 | 0.33 | 0.09 | -- | -- | 50 | 50 - 135 | 25 | 50 - 135 | 20 |
| Fluoranthene | 206-44-0 | 33,700 | NDEP 2017 | 0.33 | 0.07 | -- | -- | 50 | 45 - 120 | 25 | 55 - 120 | 20 |
| Fluorene | 86-73-7 | 93.1 | NDEP 2017 | 0.33 | 0.07 | -- | -- | 50 | 50 - 120 | 25 | 55 - 120 | 20 |
| Hexachlorobenzene | 118-74-1 | 0.231 | NDEP 2017 | 0.33 | 0.07 | -- | -- | 50 | 50 - 120 | 25 | 50 - 120 | 20 |
| Hexachlorocyclopentadiene | 77-47-4 | 15.7 | NDEP 2017 | 0.83 | 0.133 | -- | -- | 50 | 20 - 125 | 30 | 30 - 125 | 25 |

**TABLE 2. SOIL ANALYTES AND ANALYTICAL PERFORMANCE CRITERIA**
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| ANALYTES | CAS Number | Screening Level | Screening Level Source[1] | Practical Quantitation Limit (PQL) | Method Detection Limit (MDL) | QUALITY CONTROL LIMITS[2] Surrogate %R | Duplicate RPD | Matrix Spike %R | Matrix Spike RPD | Blank Spike/LCS %R | Blank Spike/LCS RPD |
|---|---|---|---|---|---|---|---|---|---|---|---|
| Hexachloroethane | 67-72-1 | 65.5 | NDEP 2017 | 0.33 | 0.133 | -- -- | 50 | 35 - 120 | 30 | 40 - 120 | 20 |
| Indeno[1,2,3-cd]pyrene | 193-39-5 | 3.23 | NDEP 2017 | 0.33 | 0.13 | -- -- | 50 | 20 - 130 | 30 | 30 - 135 | 25 |
| Isophorone | 78-59-1 | 2700 | NDEP 2017 | 0.33 | 0.067 | -- -- | 50 | 40 - 120 | 25 | 40 - 120 | 20 |
| Naphthalene | 91-20-3 | 290 | NDEP 2017 | 0.33 | 0.067 | -- -- | 50 | 40 - 120 | 25 | 45 - 120 | 20 |
| Nitrobenzene | 98-95-3 | 24.7 | NDEP 2017 | 0.33 | 0.07 | -- -- | 50 | 40 - 120 | 25 | 45 - 120 | 20 |
| N-Nitrosodi-n-propylamine | 621-64-7 | 0.366 | NDEP 2017 | 0.25 | 0.07 | -- -- | 50 | 35 - 120 | 25 | 40 - 120 | 20 |
| N-Nitrosodiphenylamine | 86-30-6 | 524 | NDEP 2017 | 0.33 | 0.08 | -- -- | 50 | 45 - 125 | 25 | 50 - 120 | 20 |
| Octachlorostyrene | 29082-74-4 | -- | NDEP 2017 | 3.30 | 2.3 | -- -- | 50 | 60 - 140 | 30 | 60 - 140 | 30 |
| Pentachlorophenol | 87-86-5 | 4 | NDEP 2017 | 0.83 | 0.15 | -- -- | 50 | 30 - 120 | 25 | 40 - 120 | 20 |
| Phenanthrene | 85-01-8 | 24.5 | NDEP 2017 | 0.33 | 0.067 | -- -- | 50 | 50 - 120 | 25 | 50 - 120 | 20 |
| Phenol | 108-95-2 | 100,000 | NDEP 2017 | 0.33 | 0.09 | -- -- | 50 | 40 - 120 | 25 | 40 - 120 | 20 |
| Pyrene | 129-00-0 | 44 | NDEP 2017 | 0.33 | 0.08 | -- -- | 50 | 40 - 125 | 30 | 45 - 125 | 25 |
| Pyridine | 110-86-1 | 1,300 | NDEP 2017 | 0.20 | 0.07 | -- -- | 50 | 25 - 130 | 30 | 25 - 130 | 30 |
| 2-Fluorophenol (Surr) | 367-12-4 | -- | -- | -- | -- | 35 - 120 | -- | -- -- | -- | -- -- | -- |
| 2,4,6-Tribromophenol (Surr) | 118-79-6 | -- | -- | -- | -- | 35 - 120 | -- | -- -- | -- | -- -- | -- |
| Nitrobenzene-d5 (Surr) | 4165-60-0 | -- | -- | -- | -- | 35 - 120 | -- | -- -- | -- | -- -- | -- |
| Terphenyl-d14 (Surr) | 1718-51-0 | -- | -- | -- | -- | 35 - 120 | -- | -- -- | -- | -- -- | -- |
| Phenol-d6 (Surr) | 13127-88-3 | -- | -- | -- | -- | 35 - 120 | -- | -- -- | -- | -- -- | -- |
| *EPA Method 8315A* | | | | | | | | | | | |
| Formaldehyde | 50-00-0 | 79.9 | NDEP 2017 | 1 | 0.5 | -- -- | 50 | 50 - 150 | 20 | 50 - 150 | 20 |
| **Polycyclic Aromatic Hydrocarbons (mg/kg)** | | | | | | | | | | | |
| *EPA Method 8270 SIM* | | | | | | | | | | | |
| Acenaphthene | 83-32-9 | 118 | NDEP 2017 | 0.03 | 0.004 | -- -- | 50 | 45 - 120 | 25 | 50 - 120 | 20 |
| Acenaphthylene | 208-96-8 | -- | NDEP 2017 | 0.03 | 0.004 | -- -- | 50 | 45 - 120 | 20 | 50 - 120 | 20 |
| Anthracene | 120-12-7 | 4.26 | NDEP 2017 | 0.03 | 0.004 | -- -- | 50 | 55 - 120 | 25 | 55 - 120 | 20 |
| Benzo(a)anthracene | 56-55-3 | 3.23 | NDEP 2017 | 0.03 | 0.004 | -- -- | 50 | 50 - 120 | 25 | 55 - 120 | 20 |
| Benzo(a)pyrene | 50-32-8 | 0.323 | NDEP 2017 | 0.03 | 0.004 | -- -- | 50 | 45 - 125 | 25 | 50 - 125 | 20 |
| Benzo(b)fluoranthene | 205-99-2 | 3.23 | NDEP 2017 | 0.03 | 0.004 | -- -- | 50 | 45 - 125 | 30 | 45 - 125 | 25 |
| Benzo(g,h,i)perylene | 191-24-2 | 25,300 | NDEP 2017 | 0.03 | 0.004 | -- -- | 50 | 25 - 130 | 30 | 35 - 130 | 25 |

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|---|---|---|---|---|---|---|---|---|---|---|---|
| Benzo(k)fluoranthene | 207-08-9 | 32.3 | NDEP 2017 | 0.03 | 0.004 | -- -- | 50 | 45 - 125 | 30 | 45 - 125 | 25 |
| Chrysene | 218-01-9 | 323 | NDEP 2017 | 0.03 | 0.004 | -- -- | 50 | 55 - 120 | 25 | 55 - 120 | 20 |
| Dibenz(a,h)anthracene | 53-70-3 | 0.323 | NDEP 2017 | 0.03 | 0.004 | -- -- | 50 | 25 - 135 | 30 | 40 - 135 | 25 |
| Fluoranthene | 206-44-0 | 33,700 | NDEP 2017 | 0.03 | 0.004 | -- -- | 50 | 45 - 120 | 25 | 55 - 120 | 20 |
| Fluorene | 86-73-7 | 93.1 | NDEP 2017 | 0.03 | 0.004 | -- -- | 50 | 50 - 120 | 25 | 55 - 120 | 20 |
| Indeno(1,2,3-cd)pyrene | 193-39-5 | 3.23 | NDEP 2017 | 0.03 | 0.004 | -- -- | 50 | 20 - 130 | 30 | 30 - 135 | 25 |
| Naphthalene | 91-20-3 | 18.4 | NDEP 2017 | 0.03 | 0.004 | -- -- | 50 | 40 - 120 | 25 | 45 - 120 | 20 |
| Phenanthrene | 85-01-8 | 24.5 | NDEP 2017 | 0.03 | 0.004 | -- -- | 50 | 50 - 120 | 25 | 50 - 120 | 20 |
| Pyrene | 129-00-0 | 44 | NDEP 2017 | 0.03 | 0.004 | -- -- | 50 | 40 - 125 | 30 | 45 - 125 | 25 |
| 2-Fluorobiphenyl (Surr) | 321-60-8 | -- | -- | -- | -- | 35 - 120 | -- | -- -- | -- | -- -- | -- |
| Nitrobenzene-d5 (Surr) | 4165-60-0 | -- | -- | -- | -- | 30 - 120 | -- | -- -- | -- | -- -- | -- |
| Terphenyl-d14 (Surr) | 1718-51-0 | -- | -- | -- | -- | 13 - 100 | -- | -- -- | -- | -- -- | -- |
| **Organophosphorous Pesticides (mg/kg)** | | | | | | | | | | | |
| *EPA Method 8141A* | | | | | | | | | | | |
| Atrazine | 1912-24-9 | 11.2 | NDEP 2017 | 0.07 | 0.0121 | -- -- | 50 | 49 - 115 | 50 | 49 - 115 | 50 |
| Azinphos-methyl | 86-50-0 | 2,750 | NDEP 2017 | 0.01 | 0.0035 | -- -- | 50 | 51 - 122 | 43 | 51 - 122 | 43 |
| Bolstar (Sulprofos) | 35400-43-2 | -- | NDEP 2017 | 0.01 | 0.00424 | -- -- | 50 | | | | |
| Chlorpyrifos | 2921-88-2 | 916 | NDEP 2017 | 0.02 | 0.00646 | -- -- | 50 | 38 - 130 | 37 | 38 - 130 | 37 |
| Coumaphos | 56-72-4 | -- | NDEP 2017 | 0.01 | 0.0028 | -- -- | 50 | 50 - 119 | 27 | 50 - 119 | 27 |
| Demeton, Total | 8065-48-3 | 36.7 | NDEP 2017 | 0.04 | 0.00752 | -- -- | 50 | 36 - 115 | 47 | 36 - 115 | 47 |
| Demeton-O | 298-03-3 | -- | NDEP 2017 | 0.04 | 0.00529 | -- -- | 50 | | | | |
| Demeton-S | 126-75-0 | -- | NDEP 2017 | 0.02 | 0.00486 | -- -- | 50 | | | | |
| Diazinon | 333-41-5 | 732 | NDEP 2017 | 0.02 | 0.00727 | -- -- | 50 | 53 - 115 | 40 | 53 - 115 | 40 |
| Dichlorvos | 62-73-7 | 8.85 | NDEP 2017 | 0.02 | 0.0074 | -- -- | 50 | 43 - 139 | 77 | 43 - 139 | 77 |
| Dimethoate | 60-51-5 | 183 | NDEP 2017 | 0.02 | 0.00708 | -- -- | 50 | 25 - 138 | 98 | 25 - 138 | 98 |
| Disulfoton | 298-04-4 | 51.9 | NDEP 2017 | 0.05 | 0.00773 | -- -- | 50 | 29 - 115 | 40 | 29 - 115 | 40 |
| EPN (Ethyl P-Nitrophenyl Benzenethiophosphate) | 2104-64-5 | 13 | NDEP 2017 | 0.01 | 0.00368 | -- -- | 50 | 58 - 131 | 50 | 58 - 131 | 50 |
| Ethoprop | 13194-48-4 | -- | NDEP 2017 | 0.02 | 0.00493 | -- -- | 50 | 53 - 115 | 54 | 53 - 115 | 54 |

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|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| Famphur | 52-85-7 | -- | NDEP 2017 | 0.01 | 0.00322 | -- | -- | 50 | 49 | 140 | 31 | 49 | 140 | 31 |
| Fensulfothion | 115-90-2 | -- | NDEP 2017 | 0.03 | 0.00815 | -- | -- | 50 | 52 | 121 | 49 | 52 | 121 | 49 |
| Fenthion | 55-38-9 | -- | NDEP 2017 | 0.03 | 0.00874 | -- | -- | 50 | 45 | 115 | 43 | 45 | 115 | 43 |
| Malathion | 121-75-5 | 18,300 | NDEP 2017 | 0.02 | 0.00464 | -- | -- | 50 | 50 | 122 | 53 | 50 | 122 | 53 |
| Merphos | 150-50-5 | 1.03 | NDEP 2017 | 0.03 | 0.00514 | -- | -- | 50 | 19 | 115 | 50 | 19 | 115 | 50 |
| Mevinphos | 7786-34-7 | -- | NDEP 2017 | 0.02 | 0.00462 | -- | -- | 50 | 10 | 226 | 78 | 10 | 226 | 78 |
| Naled | 300-76-5 | 1.29 | NDEP 2017 | 0.07 | 0.0226 | -- | -- | 50 | 10 | 115 | | 10 | 115 | |
| Parathion-ethyl | 56-38-2 | 5,500 | NDEP 2017 | 0.02 | 0.00529 | -- | -- | 50 | 24 | 163 | 47 | 24 | 163 | 47 |
| Parathion-methyl | 298-00-0 | 229 | NDEP 2017 | 0.02 | 0.00637 | -- | -- | 50 | 46 | 119 | 53 | 46 | 119 | 53 |
| Phorate | 298-02-2 | 183 | NDEP 2017 | 0.02 | 0.0057 | -- | -- | 50 | 40 | 115 | 40 | 40 | 115 | 40 |
| Ronnel | 299-84-3 | 26.8 | NDEP 2017 | 0.05 | 0.0152 | -- | -- | 50 | 43 | 118 | 41 | 43 | 118 | 41 |
| Simazine | 122-34-9 | -- | NDEP 2017 | 0.07 | 0.0221 | -- | -- | 50 | 11 | 179 | 58 | 11 | 179 | 58 |
| Stirphos (Tetrachlorovinphos) | 22248-79-9 | 107 | NDEP 2017 | 0.02 | 0.00436 | -- | -- | 50 | 44 | 118 | 24 | 44 | 118 | 24 |
| Sulfotepp | 3689-24-5 | 458 | NDEP 2017 | 0.02 | 0.00626 | -- | -- | 50 | 55 | 115 | 40 | 55 | 115 | |
| Thionazin | 297-97-2 | -- | NDEP 2017 | 0.02 | 0.00557 | -- | -- | 50 | 46 | 115 | 40 | 46 | 115 | 40 |
| Tokuthion | 34643-46-4 | -- | NDEP 2017 | 0.02 | 0.00391 | -- | -- | 50 | | | | | | |
| Trichloronate | 327-98-0 | -- | NDEP 2017 | 0.02 | 0.00625 | -- | -- | 50 | 27 | 115 | 43 | 27 | 115 | 43 |
| Chlormefos (Surr) | 24934-91-6 | -- | -- | -- | -- | 42 | 132 | -- | -- | -- | -- | -- | -- | -- |
| Triphenylphosphate (Surr) | 115-86-6 | -- | -- | -- | -- | 47 | 161 | -- | -- | -- | -- | -- | -- | -- |
| **Organochlorine Pesticides (mg/kg)** | | | | | | | | | | | | | | |
| *EPA Method 8081A* | | | | | | | | | | | | | | |
| 2,4'-DDE | 3424-82-6 | -- | NDEP 2017 | 0.01 | 0.0015 | -- | -- | 50 | 35 | 130 | 30 | 60 | 120 | 30 |
| 4,4'-DDD | 72-54-8 | 15.1 | NDEP 2017 | 0.01 | 0.0015 | -- | -- | 50 | 40 | 130 | 30 | 60 | 120 | 30 |
| 4,4'-DDE | 72-55-9 | 9.5 | NDEP 2017 | 0.01 | 0.0015 | -- | -- | 50 | 35 | 130 | 30 | 60 | 120 | 30 |
| 4,4'-DDT | 50-29-3 | 7.55 | NDEP 2017 | 0.01 | 0.0015 | -- | -- | 50 | 35 | 130 | 30 | 65 | 120 | 30 |
| Aldrin | 309-00-2 | 0.214 | NDEP 2017 | 0.01 | 0.0015 | -- | -- | 50 | 40 | 115 | 30 | 50 | 115 | 30 |
| alpha-BHC | 319-84-6 | 0.494 | NDEP 2017 | 0.01 | 0.0015 | -- | -- | 50 | 40 | 115 | 30 | 60 | 115 | 30 |
| alpha-Chlordane | 57-74-9 | 7.33 | NDEP 2017 | 0.05 | 0.01 | -- | -- | 50 | 60 | 140 | 30 | 60 | 140 | 30 |
| beta-BHC | 319-85-7 | 1.73 | NDEP 2017 | 0.01 | 0.0015 | -- | -- | 50 | 40 | 120 | 30 | 60 | 115 | 30 |

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|---|---|---|---|---|---|---|---|---|---|---|---|
| delta-BHC | 319-86-8 | 334 | NDEP 2017 | 0.01 | 0.0015 | -- -- | 50 | 45 - 120 | 30 | 60 - 115 | 30 |
| Dieldrin | 60-57-1 | 0.16 | NDEP 2017 | 0.01 | 0.0015 | -- -- | 50 | 40 - 125 | 30 | 65 - 115 | 30 |
| Endosulfan I | 959-98-8 | 5,500 | NDEP 2017 | 0.01 | 0.0015 | -- -- | 50 | 40 - 120 | 30 | 40 - 120 | 30 |
| Endosulfan II | 33213-65-9 | 5,500 | NDEP 2017 | 0.01 | 0.0015 | -- -- | 50 | 40 - 125 | 30 | 55 - 120 | 30 |
| Endosulfan sulfate | 1031-07-8 | 5,500 | NDEP 2017 | 0.01 | 0.002 | -- -- | 50 | 45 - 120 | 30 | 65 - 115 | 30 |
| Endrin | 72-20-8 | 30.2 | NDEP 2017 | 0.01 | 0.0015 | -- -- | 50 | 45 - 125 | 30 | 55 - 120 | 30 |
| Endrin aldehyde | 7421-93-4 | 30.2 | NDEP 2017 | 0.01 | 0.0015 | -- -- | 50 | 30 - 120 | 30 | 55 - 115 | 30 |
| Endrin Ketone | 53494-70-5 | 30.2 | NDEP 2017 | 0.01 | 0.002 | -- -- | 50 | 40 - 120 | 30 | 65 - 115 | 30 |
| gamma-BHC (Lindane) | 58-89-9 | 2.83 | NDEP 2017 | 0.01 | 0.0015 | -- -- | 50 | 40 - 120 | 30 | 55 - 115 | 30 |
| gamma-Chlordane | 57-74-9 | 7.33 | NDEP 2017 | 0.05 | 0.01 | -- -- | 50 | 60 - 140 | 30 | 60 - 140 | 30 |
| Heptachlor | 76-44-8 | 0.807 | NDEP 2017 | 0.01 | 0.002 | -- -- | 50 | 40 - 115 | 30 | 55 - 115 | 30 |
| Heptachlor epoxide | 1024-57-3 | 0.399 | NDEP 2017 | 0.01 | 0.002 | -- -- | 50 | 45 - 115 | 30 | 55 - 115 | 30 |
| Methoxychlor | 72-43-5 | 4,580 | NDEP 2017 | 0.01 | 0.0015 | -- -- | 50 | 40 - 135 | 30 | 65 - 120 | 30 |
| Toxaphene | 8001-35-2 | 2.33 | NDEP 2017 | 0.2 | 0.05 | -- -- | 50 | 60 - 140 | 30 | 60 - 140 | 30 |
| Decachlorobiphenyl (Surr) | 2051-24-3 | -- | -- | -- | -- | 45 - 120 | -- | -- -- | -- | -- -- | -- |
| **Dioxins/Furans (pg/g)[4]** | | | | | | | | | | | |
| *EPA Method 8290 or 8280(7)* | | | | | | | | | | | |
| 2,3,7,8- TCDD | 1746-01-6 | 19.7 | NDEP 2017 | 1 | EDL[3] | -- -- | 50 | 60 - 138 | 20 | 60 - 138 | 20 |
| OCDF | 39001-02-0 | -- | NDEP 2017 | 10 | EDL[3] | -- -- | 50 | 63 - 141 | 20 | 63 - 141 | 20 |
| OCDD | 3268-87-9 | -- | NDEP 2017 | 10 | EDL[3] | -- -- | 50 | 70 - 128 | 20 | 70 - 128 | 20 |
| 1,2,3,4,6,7,8-HpCDF | 67562-39-4 | -- | NDEP 2017 | 5 | EDL[3] | -- -- | 50 | 71 - 134 | 20 | 71 - 134 | 20 |
| 1,2,3,4,6,7,8-HpCDD | 35822-46-9 | -- | NDEP 2017 | 5 | EDL[3] | -- -- | 50 | 71 - 128 | 20 | 71 - 128 | 20 |
| 1,2,3,4,7,8,9-HpCDF | 55673-89-7 | -- | NDEP 2017 | 5 | EDL[3] | -- -- | 50 | 68 - 129 | 20 | 68 - 129 | 20 |
| 1,2,3,4,7,8-HxCDF | 70648-26-9 | -- | NDEP 2017 | 5 | EDL[3] | -- -- | 50 | 74 - 128 | 20 | 74 - 128 | 20 |
| 1,2,3,4,7,8-HxCDD | 39227-28-6 | -- | NDEP 2017 | 5 | EDL[3] | -- -- | 50 | 60 - 138 | 20 | 60 - 138 | 20 |
| 1,2,3,6,7,8-HxCDF | 57117-44-9 | -- | NDEP 2017 | 5 | EDL[3] | -- -- | 50 | 67 - 140 | 20 | 67 - 140 | 20 |
| 1,2,3,6,7,8-HxCDD | 57653-85-7 | -- | NDEP 2017 | 5 | EDL[3] | -- -- | 50 | 68 - 136 | 20 | 68 - 136 | 20 |
| 1,2,3,7,8,9-HxCDF | 72918-21-9 | -- | NDEP 2017 | 5 | EDL[3] | -- -- | 50 | 72 - 134 | 20 | 72 - 134 | 20 |
| 1,2,3,7,8,9-HxCDD | 19408-74-3 | -- | NDEP 2017 | 5 | EDL[3] | -- -- | 50 | 68 - 138 | 20 | 68 - 138 | 20 |

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|---|---|---|---|---|---|---|---|---|---|---|---|---|
| 1,2,3,7,8-PeCDF | 57117-41-6 | -- | NDEP 2017 | 5 | EDL[3] | -- | -- | 50 | 69 - 134 | 20 | 69 - 134 | 20 |
| 1,2,3,7,8-PeCDD | 40321-76-4 | -- | NDEP 2017 | 5 | EDL[3] | -- | -- | 50 | 70 - 122 | 20 | 70 - 122 | 20 |
| 2,3,4,6,7,8-HxCDF | 60851-34-5 | -- | NDEP 2017 | 5 | EDL[3] | -- | -- | 50 | 71 - 137 | 20 | 71 - 137 | 20 |
| 1,2,3,6,7,8-HxCDF | 57117-44-9 | -- | NDEP 2017 | 5 | EDL[3] | -- | -- | 50 | 67 - 140 | 20 | 67 - 140 | 20 |
| 2,3,7,8-TCDF | 51207-31-9 | -- | NDEP 2017 | 1 | EDL[3] | -- | -- | 50 | 56 - 158 | 20 | 56 - 158 | 20 |
| **PCBs as Congeners (mg/kg) [4]** | | | | | | | | | | | | |
| *EPA Method 1668A* | | | | | | | | | | | | |
| Total PCBs | 1336-36-3 | 1.15 | NDEP 2017 | 0.0002 | EDL[3] | -- | -- | 50 | 50 - 150 | 50 | 50 - 150 | 50 |
| 2-MoCB (PCB-1) | 2051-60-7 | 1.15 | NDEP 2017 | 0.00002 | EDL[3] | -- | -- | 50 | 50 - 150 | 50 | 50 - 150 | 50 |
| 3-MoCB (PCB-2) | 2051-61-8 | 1.15 | NDEP 2017 | 0.00002 | EDL[3] | -- | -- | 50 | 50 - 150 | 50 | 50 - 150 | 50 |
| 4-MoCB (PCB-3) | 2051-62-9 | 1.15 | NDEP 2017 | 0.00002 | EDL[3] | -- | -- | 50 | 50 - 150 | 50 | 50 - 150 | 50 |
| 2,2'-DiCB (PCB-4) | 13029-08-8 | 1.15 | NDEP 2017 | 0.00002 | EDL[3] | -- | -- | 50 | 50 - 150 | 50 | 50 - 150 | 50 |
| 2,3-DiCB (PCB-5) | 16605-91-7 | 1.15 | NDEP 2017 | 0.00002 | EDL[3] | -- | -- | 50 | 50 - 150 | 50 | 50 - 150 | 50 |
| 2,3'-DiCB (PCB-6) | 25569-80-6 | 1.15 | NDEP 2017 | 0.00002 | EDL[3] | -- | -- | 50 | 50 - 150 | 50 | 50 - 150 | 50 |
| 2,4-DiCB (PCB-7) | 33284-50-3 | 1.15 | NDEP 2017 | 0.00002 | EDL[3] | -- | -- | 50 | 50 - 150 | 50 | 50 - 150 | 50 |
| 2,4'-DiCB (PCB-8) | 34883-43-7 | 1.15 | NDEP 2017 | 0.00002 | EDL[3] | -- | -- | 50 | 50 - 150 | 50 | 50 - 150 | 50 |
| 2,5-DiCB (PCB-9) | 34883-39-1 | 1.15 | NDEP 2017 | 0.00002 | EDL[3] | -- | -- | 50 | 50 - 150 | 50 | 50 - 150 | 50 |
| 2,6-DiCB (PCB-10) | 33146-45-1 | 1.15 | NDEP 2017 | 0.00002 | EDL[3] | -- | -- | 50 | 50 - 150 | 50 | 50 - 150 | 50 |
| 3,3'-DiCB (PCB-11) | 2050-67-1 | 1.15 | NDEP 2017 | 0.00002 | EDL[3] | -- | -- | 50 | 50 - 150 | 50 | 50 - 150 | 50 |
| 3,4-DiCB (PCB-12) | 2974-92-7 | 1.15 | NDEP 2017 | 0.00004 | EDL[3] | -- | -- | 50 | 50 - 150 | 50 | 50 - 150 | 50 |
| 3,4'-DiCB (PCB-13) | 2974-90-5 | 1.15 | NDEP 2017 | 0.00004 | EDL[3] | -- | -- | 50 | 50 - 150 | 50 | 50 - 150 | 50 |
| 3,5-DiCB (PCB-14) | 34883-41-5 | 1.15 | NDEP 2017 | 0.00002 | EDL[3] | -- | -- | 50 | 50 - 150 | 50 | 50 - 150 | 50 |
| 4,4'-DiCB (PCB-15) | 2050-68-2 | 1.15 | NDEP 2017 | 0.00002 | EDL[3] | -- | -- | 50 | 50 - 150 | 50 | 50 - 150 | 50 |
| 2,2',3-TrCB (PCB-16) | 38444-78-9 | 1.15 | NDEP 2017 | 0.00002 | EDL[3] | -- | -- | 50 | 50 - 150 | 50 | 50 - 150 | 50 |
| 2,2',4-TrCB (PCB-17) | 37680-66-3 | 1.15 | NDEP 2017 | 0.00002 | EDL[3] | -- | -- | 50 | 50 - 150 | 50 | 50 - 150 | 50 |
| 2,2',5-TrCB (PCB-18) | 37680-65-2 | 1.15 | NDEP 2017 | 0.00004 | EDL[3] | -- | -- | 50 | 50 - 150 | 50 | 50 - 150 | 50 |
| 2,2',6-TrCB (PCB-19) | 38444-73-4 | 1.15 | NDEP 2017 | 0.00002 | EDL[3] | -- | -- | 50 | 50 - 150 | 50 | 50 - 150 | 50 |
| 2,3,3'-TrCB (PCB-20) | 38444-84-7 | 1.15 | NDEP 2017 | 0.00004 | EDL[3] | -- | -- | 50 | 50 - 150 | 50 | 50 - 150 | 50 |
| 2,3,4-TrCB (PCB-21) | 55702-46-0 | 1.15 | NDEP 2017 | 0.00004 | EDL[3] | -- | -- | 50 | 50 - 150 | 50 | 50 - 150 | 50 |

**TABLE 2. SOIL ANALYTES AND ANALYTICAL PERFORMANCE CRITERIA**
**QUALITY ASSURANCE PROJECT PLAN**
**Nevada Environmental Response Trust Site; Henderson, Nevada**

| ANALYTES | CAS Number | Screening Level | Screening Level Source[1] | Practical Quantitation Limit (PQL) | Method Detection Limit (MDL) | QUALITY CONTROL LIMITS[2] Surrogate %R | | Duplicate RPD | Matrix Spike %R | Matrix Spike RPD | Blank Spike/LCS %R | Blank Spike/LCS RPD |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| 2,3,4'-TrCB (PCB-22) | 38444-85-8 | 1.15 | NDEP 2017 | 0.00002 | EDL[3] | -- | -- | 50 | 50 - 150 | 50 | 50 - 150 | 50 |
| 2,3,5-TrCB (PCB-23) | 55720-44-0 | 1.15 | NDEP 2017 | 0.00002 | EDL[3] | -- | -- | 50 | 50 - 150 | 50 | 50 - 150 | 50 |
| 2,3,6-TrCB (PCB-24) | 55702-45-9 | 1.15 | NDEP 2017 | 0.00002 | EDL[3] | -- | -- | 50 | 50 - 150 | 50 | 50 - 150 | 50 |
| 2,3',4-TrCB (PCB-25) | 55712-37-3 | 1.15 | NDEP 2017 | 0.00002 | EDL[3] | -- | -- | 50 | 50 - 150 | 50 | 50 - 150 | 50 |
| 2,3',5-TrCB (PCB-26) | 38444-81-4 | 1.15 | NDEP 2017 | 0.00004 | EDL[3] | -- | -- | 50 | 50 - 150 | 50 | 50 - 150 | 50 |
| 2,3',6-TrCB (PCB-27) | 38444-76-7 | 1.15 | NDEP 2017 | 0.00002 | EDL[3] | -- | -- | 50 | 50 - 150 | 50 | 50 - 150 | 50 |
| 2,4,4'-TrCB (PCB-28) | 7012-37-5 | 1.15 | NDEP 2017 | 0.00004 | EDL[3] | -- | -- | 50 | 50 - 150 | 50 | 50 - 150 | 50 |
| 2,4,5-TrCB (PCB-29) | 15862-07-4 | 1.15 | NDEP 2017 | 0.00004 | EDL[3] | -- | -- | 50 | 50 - 150 | 50 | 50 - 150 | 50 |
| 2,4,6-TrCB (PCB-30) | 35693-92-6 | 1.15 | NDEP 2017 | 0.00004 | EDL[3] | -- | -- | 50 | 50 - 150 | 50 | 50 - 150 | 50 |
| 2,4',5-TrCB (PCB-31) | 16606-02-3 | 1.15 | NDEP 2017 | 0.00002 | EDL[3] | -- | -- | 50 | 50 - 150 | 50 | 50 - 150 | 50 |
| 2,4',6-TrCB (PCB-32) | 38444-77-8 | 1.15 | NDEP 2017 | 0.00002 | EDL[3] | -- | -- | 50 | 50 - 150 | 50 | 50 - 150 | 50 |
| 2',3,4-TrCB (PCB-33) | 38444-86-9 | 1.15 | NDEP 2017 | 0.00004 | EDL[3] | -- | -- | 50 | 50 - 150 | 50 | 50 - 150 | 50 |
| 2',3,5-TrCB (PCB-34) | 37680-68-5 | 1.15 | NDEP 2017 | 0.00002 | EDL[3] | -- | -- | 50 | 50 - 150 | 50 | 50 - 150 | 50 |
| 3,3',4-TrCB (PCB-35) | 37680-69-6 | 1.15 | NDEP 2017 | 0.00002 | EDL[3] | -- | -- | 50 | 50 - 150 | 50 | 50 - 150 | 50 |
| 3,3',5-TrCB (PCB-36) | 38444-87-0 | 1.15 | NDEP 2017 | 0.00002 | EDL[3] | -- | -- | 50 | 50 - 150 | 50 | 50 - 150 | 50 |
| 3,4,4'-TrCB (PCB-37) | 38444-90-5 | 1.15 | NDEP 2017 | 0.00002 | EDL[3] | -- | -- | 50 | 50 - 150 | 50 | 50 - 150 | 50 |
| 3,4,5-TrCB (PCB-38) | 53555-66-1 | 1.15 | NDEP 2017 | 0.00002 | EDL[3] | -- | -- | 50 | 50 - 150 | 50 | 50 - 150 | 50 |
| 3,4',5-TrCB (PCB-39) | 38444-88-1 | 1.15 | NDEP 2017 | 0.00002 | EDL[3] | -- | -- | 50 | 50 - 150 | 50 | 50 - 150 | 50 |
| 2,2',3,3'-TeCB (PCB-40) | 38444-93-8 | 1.15 | NDEP 2017 | 0.00004 | EDL[3] | -- | -- | 50 | 50 - 150 | 50 | 50 - 150 | 50 |
| 2,2',3,4-TeCB (PCB-41) | 52663-59-9 | 1.15 | NDEP 2017 | 0.00002 | EDL[3] | -- | -- | 50 | 50 - 150 | 50 | 50 - 150 | 50 |
| 2,2',3,4'-TeCB (PCB-42) | 36559-22-5 | 1.15 | NDEP 2017 | 0.00002 | EDL[3] | -- | -- | 50 | 50 - 150 | 50 | 50 - 150 | 50 |
| 2,2',3,5-TeCB (PCB-43) | 70362-46-8 | 1.15 | NDEP 2017 | 0.00002 | EDL[3] | -- | -- | 50 | 50 - 150 | 50 | 50 - 150 | 50 |
| 2,2',3,5'-TeCB (PCB-44) | 41464-39-5 | 1.15 | NDEP 2017 | 0.00006 | EDL[3] | -- | -- | 50 | 50 - 150 | 50 | 50 - 150 | 50 |
| 2,2',3,6-TeCB (PCB-45) | 70362-45-7 | 1.15 | NDEP 2017 | 0.00002 | EDL[3] | -- | -- | 50 | 50 - 150 | 50 | 50 - 150 | 50 |
| 2,2',3,6'-TeCB (PCB-46) | 41464-47-5 | 1.15 | NDEP 2017 | 0.00002 | EDL[3] | -- | -- | 50 | 50 - 150 | 50 | 50 - 150 | 50 |
| 2,2',4,4'-TeCB (PCB-47) | 2437-79-8 | 1.15 | NDEP 2017 | 0.00006 | EDL[3] | -- | -- | 50 | 50 - 150 | 50 | 50 - 150 | 50 |
| 2,2',4,5-TeCB (PCB-48) | 70362-47-9 | 1.15 | NDEP 2017 | 0.00002 | EDL[3] | -- | -- | 50 | 50 - 150 | 50 | 50 - 150 | 50 |
| 2,2',4,5'-TeCB (PCB-49) | 41464-40-8 | 1.15 | NDEP 2017 | 0.00004 | EDL[3] | -- | -- | 50 | 50 - 150 | 50 | 50 - 150 | 50 |
| 2,2',4,6-TeCB (PCB-50) | 62796-65-0 | 1.15 | NDEP 2017 | 0.00004 | EDL[3] | -- | -- | 50 | 50 - 150 | 50 | 50 - 150 | 50 |
| 2,2',4,6'-TeCB (PCB-51) | 68194-04-7 | 1.15 | NDEP 2017 | 0.00002 | EDL[3] | -- | -- | 50 | 50 - 150 | 50 | 50 - 150 | 50 |

**TABLE 2. SOIL ANALYTES AND ANALYTICAL PERFORMANCE CRITERIA**
**QUALITY ASSURANCE PROJECT PLAN**
**Nevada Environmental Response Trust Site; Henderson, Nevada**

| ANALYTES | CAS Number | Screening Level | Screening Level Source[1] | Practical Quantitation Limit (PQL) | Method Detection Limit (MDL) | QUALITY CONTROL LIMITS[2] Surrogate %R | | Duplicate RPD | Matrix Spike %R | Matrix Spike RPD | Blank Spike/LCS %R | Blank Spike/LCS RPD |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| 2,2',5,5'-TeCB (PCB-52) | 35693-99-3 | 1.15 | NDEP 2017 | 0.00002 | EDL[3] | -- | -- | 50 | 50 - 150 | 50 | 50 - 150 | 50 |
| 2,2',5,6'-TeCB (PCB-53) | 41464-41-9 | 1.15 | NDEP 2017 | 0.00004 | EDL[3] | -- | -- | 50 | 50 - 150 | 50 | 50 - 150 | 50 |
| 2,2',6,6'-TeCB (PCB-54) | 15968-05-5 | 1.15 | NDEP 2017 | 0.00002 | EDL[3] | -- | -- | 50 | 50 - 150 | 50 | 50 - 150 | 50 |
| 2,3,3',4'-TeCB (PCB-55) | 74338-24-2 | 1.15 | NDEP 2017 | 0.00002 | EDL[3] | -- | -- | 50 | 50 - 150 | 50 | 50 - 150 | 50 |
| 2,3,3',4'-TeCB (PCB-56) | 41464-43-1 | 1.15 | NDEP 2017 | 0.00002 | EDL[3] | -- | -- | 50 | 50 - 150 | 50 | 50 - 150 | 50 |
| 2,3,3',5-TeCB (PCB-57) | 70424-67-8 | 1.15 | NDEP 2017 | 0.00002 | EDL[3] | -- | -- | 50 | 50 - 150 | 50 | 50 - 150 | 50 |
| 2,3,3',5'-TeCB (PCB-58) | 41464-49-7 | 1.15 | NDEP 2017 | 0.00002 | EDL[3] | -- | -- | 50 | 50 - 150 | 50 | 50 - 150 | 50 |
| 2,3,3',6-TeCB (PCB-59) | 74472-33-6 | 1.15 | NDEP 2017 | 0.00006 | EDL[3] | -- | -- | 50 | 50 - 150 | 50 | 50 - 150 | 50 |
| 2,3,4,4'-TeCB (PCB-60) | 33025-41-1 | 1.15 | NDEP 2017 | 0.00002 | EDL[3] | -- | -- | 50 | 50 - 150 | 50 | 50 - 150 | 50 |
| 2,3,4,5-TeCB (PCB-61) | 33284-53-6 | 1.15 | NDEP 2017 | 0.00008 | EDL[3] | -- | -- | 50 | 50 - 150 | 50 | 50 - 150 | 50 |
| 2,3,4,6-TeCB (PCB-62) | 54230-22-7 | 1.15 | NDEP 2017 | 0.00006 | EDL[3] | -- | -- | 50 | 50 - 150 | 50 | 50 - 150 | 50 |
| 2,3,4',5-TeCB (PCB-63) | 74472-34-7 | 1.15 | NDEP 2017 | 0.00002 | EDL[3] | -- | -- | 50 | 50 - 150 | 50 | 50 - 150 | 50 |
| 2,3,4',6-TeCB (PCB-64) | 52663-58-8 | 1.15 | NDEP 2017 | 0.00002 | EDL[3] | -- | -- | 50 | 50 - 150 | 50 | 50 - 150 | 50 |
| 2,3,5,6-TeCB (PCB-65) | 33284-54-7 | 1.15 | NDEP 2017 | 0.00006 | EDL[3] | -- | -- | 50 | 50 - 150 | 50 | 50 - 150 | 50 |
| 2,3',4,4'-TeCB (PCB-66) | 32598-10-0 | 1.15 | NDEP 2017 | 0.00002 | EDL[3] | -- | -- | 50 | 50 - 150 | 50 | 50 - 150 | 50 |
| 2,3',4,5-TeCB (PCB-67) | 73575-53-8 | 1.15 | NDEP 2017 | 0.00002 | EDL[3] | -- | -- | 50 | 50 - 150 | 50 | 50 - 150 | 50 |
| 2,3',4,5'-TeCB (PCB-68) | 73575-52-7 | 1.15 | NDEP 2017 | 0.00002 | EDL[3] | -- | -- | 50 | 50 - 150 | 50 | 50 - 150 | 50 |
| 2,3',4,6-TeCB (PCB-69) | 60233-24-1 | 1.15 | NDEP 2017 | 0.00004 | EDL[3] | -- | -- | 50 | 50 - 150 | 50 | 50 - 150 | 50 |
| 2,3',4',5-TeCB (PCB-70) | 32598-11-1 | 1.15 | NDEP 2017 | 0.00008 | EDL[3] | -- | -- | 50 | 50 - 150 | 50 | 50 - 150 | 50 |
| 2,3',4',6-TeCB (PCB-71) | 41464-46-4 | 1.15 | NDEP 2017 | 0.00004 | EDL[3] | -- | -- | 50 | 50 - 150 | 50 | 50 - 150 | 50 |
| 2,3',5,5'-TeCB (PCB-72) | 41464-42-0 | 1.15 | NDEP 2017 | 0.00002 | EDL[3] | -- | -- | 50 | 50 - 150 | 50 | 50 - 150 | 50 |
| 2,3',5',6-TeCB (PCB-73) | 74338-23-1 | 1.15 | NDEP 2017 | 0.00002 | EDL[3] | -- | -- | 50 | 50 - 150 | 50 | 50 - 150 | 50 |
| 2,4,4',5-TeCB (PCB-74) | 32690-93-0 | 1.15 | NDEP 2017 | 0.00008 | EDL[3] | -- | -- | 50 | 50 - 150 | 50 | 50 - 150 | 50 |
| 2,4,4',6-TeCB (PCB-75) | 32598-12-2 | 1.15 | NDEP 2017 | 0.00006 | EDL[3] | -- | -- | 50 | 50 - 150 | 50 | 50 - 150 | 50 |
| 2',3,4,5-TeCB (PCB-76) | 70362-48-0 | 1.15 | NDEP 2017 | 0.00008 | EDL[3] | -- | -- | 50 | 50 - 150 | 50 | 50 - 150 | 50 |
| 3,3',4,4'-TeCB (PCB-77) | 32598-13-3 | 0.177 | NDEP 2017 | 0.00000 | EDL[3] | -- | -- | 50 | 50 - 150 | 50 | 50 - 150 | 50 |
| 3,3',4,5-TeCB (PCB-78) | 70362-49-1 | 1.15 | NDEP 2017 | 0.00002 | EDL[3] | -- | -- | 50 | 50 - 150 | 50 | 50 - 150 | 50 |
| 3,3',4,5'-TeCB (PCB-79) | 41464-48-6 | 1.15 | NDEP 2017 | 0.00002 | EDL[3] | -- | -- | 50 | 50 - 150 | 50 | 50 - 150 | 50 |
| 3,3',5,5'-TeCB (PCB-80) | 33284-52-5 | 1.15 | NDEP 2017 | 0.00002 | EDL[3] | -- | -- | 50 | 50 - 150 | 50 | 50 - 150 | 50 |
| 3,4,4',5-TeCB (PCB-81) | 70362-50-4 | 0.0589 | NDEP 2017 | 0.000002 | EDL[3] | -- | -- | 50 | 50 - 150 | 50 | 50 - 150 | 50 |

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| ANALYTES | CAS Number | Screening Level | Screening Level Source[1] | Practical Quantitation Limit (PQL) | Method Detection Limit (MDL) | QUALITY CONTROL LIMITS[2] Surrogate %R | | Duplicate RPD | Matrix Spike %R | Matrix Spike RPD | Blank Spike/LCS %R | Blank Spike/LCS RPD |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| 2,2',3,3',4-PeCB (PCB-82) | 52663-62-4 | 1.15 | NDEP 2017 | 0.000020 | EDL[3] | -- | -- | 50 | 50 - 150 | 50 | 50 - 150 | 50 |
| 2,2',3,3',5-PeCB (PCB-83) | 60145-20-2 | 1.15 | NDEP 2017 | 0.000020 | EDL[3] | -- | -- | 50 | 50 - 150 | 50 | 50 - 150 | 50 |
| 2,2',3,3',6-PeCB (PCB-84) | 52663-60-2 | 1.15 | NDEP 2017 | 0.000020 | EDL[3] | -- | -- | 50 | 50 - 150 | 50 | 50 - 150 | 50 |
| 2,2',3,4,4'-PeCB (PCB-85) | 65510-45-4 | 1.15 | NDEP 2017 | 0.000060 | EDL[3] | -- | -- | 50 | 50 - 150 | 50 | 50 - 150 | 50 |
| 2,2',3,4,5-PeCB (PCB-86) | 55312-69-1 | 1.15 | NDEP 2017 | 0.000120 | EDL[3] | -- | -- | 50 | 50 - 150 | 50 | 50 - 150 | 50 |
| 2,2',3,4,5'-PeCB (PCB-87) | 38380-02-8 | 1.15 | NDEP 2017 | 0.000120 | EDL[3] | -- | -- | 50 | 50 - 150 | 50 | 50 - 150 | 50 |
| 2,2',3,4,6-PeCB (PCB-88) | 55215-17-3 | 1.15 | NDEP 2017 | 0.000040 | EDL[3] | -- | -- | 50 | 50 - 150 | 50 | 50 - 150 | 50 |
| 2,2',3,4,6'-PeCB (PCB-89) | 73575-57-2 | 1.15 | NDEP 2017 | 0.000020 | EDL[3] | -- | -- | 50 | 50 - 150 | 50 | 50 - 150 | 50 |
| 2,2',3,4',5-PeCB (PCB-90) | 68194-07-0 | 1.15 | NDEP 2017 | 0.000060 | EDL[3] | -- | -- | 50 | 50 - 150 | 50 | 50 - 150 | 50 |
| 2,2',3,4',6-PeCB (PCB-91) | 68194-05-8 | 1.15 | NDEP 2017 | 0.000040 | EDL[3] | -- | -- | 50 | 50 - 150 | 50 | 50 - 150 | 50 |
| 2,2',3,5,5'-PeCB (PCB-92) | 52663-61-3 | 1.15 | NDEP 2017 | 0.000020 | EDL[3] | -- | -- | 50 | 50 - 150 | 50 | 50 - 150 | 50 |
| 2,2',3,5,6-PeCB (PCB-93) | 73575-56-1 | 1.15 | NDEP 2017 | 0.000040 | EDL[3] | -- | -- | 50 | 50 - 150 | 50 | 50 - 150 | 50 |
| 2,2',3,5,6'-PeCB (PCB-94) | 73575-55-0 | 1.15 | NDEP 2017 | 0.000020 | EDL[3] | -- | -- | 50 | 50 - 150 | 50 | 50 - 150 | 50 |
| 2,2',3,5',6-PeCB (PCB-95) | 38379-99-6 | 1.15 | NDEP 2017 | 0.000020 | EDL[3] | -- | -- | 50 | 50 - 150 | 50 | 50 - 150 | 50 |
| 2,2',3,6,6'-PeCB (PCB-96) | 73575-54-9 | 1.15 | NDEP 2017 | 0.000020 | EDL[3] | -- | -- | 50 | 50 - 150 | 50 | 50 - 150 | 50 |
| 2,2',3',4,5-PeCB (PCB-97) | 41464-51-1 | 1.15 | NDEP 2017 | 0.000120 | EDL[3] | -- | -- | 50 | 50 - 150 | 50 | 50 - 150 | 50 |
| 2,2',3',4,6-PeCB (PCB-98) | 60233-25-2 | 1.15 | NDEP 2017 | 0.000040 | EDL[3] | -- | -- | 50 | 50 - 150 | 50 | 50 - 150 | 50 |
| 2,2',4,4',5-PeCB (PCB-99) | 38380-01-7 | 1.15 | NDEP 2017 | 0.000020 | EDL[3] | -- | -- | 50 | 50 - 150 | 50 | 50 - 150 | 50 |
| 2,2',4,4',6-PeCB (PCB-100) | 39485-83-1 | 1.15 | NDEP 2017 | 0.000040 | EDL[3] | -- | -- | 50 | 50 - 150 | 50 | 50 - 150 | 50 |
| 2,2',4,5,5'-PeCB (PCB-101) | 37680-73-2 | 1.15 | NDEP 2017 | 0.000060 | EDL[3] | -- | -- | 50 | 50 - 150 | 50 | 50 - 150 | 50 |
| 2,2',4,5,6'-PeCB (PCB-102) | 68194-06-9 | 1.15 | NDEP 2017 | 0.000040 | EDL[3] | -- | -- | 50 | 50 - 150 | 50 | 50 - 150 | 50 |
| 2,2',4,5,'6-PeCB (PCB-103) | 60145-21-3 | 1.15 | NDEP 2017 | 0.000020 | EDL[3] | -- | -- | 50 | 50 - 150 | 50 | 50 - 150 | 50 |
| 2,2',4,6,6'-PeCB (PCB-104) | 56558-16-8 | 1.15 | NDEP 2017 | 0.000020 | EDL[3] | -- | -- | 50 | 50 - 150 | 50 | 50 - 150 | 50 |
| 2,3,3',4,4'-PeCB (PCB-105) | 32598-14-4 | 0.589 | NDEP 2017 | 0.000002 | EDL[3] | -- | -- | 50 | 50 - 150 | 50 | 50 - 150 | 50 |
| 2,3,3',4,5-PeCB (PCB-106) | 70424-69-0 | 1.15 | NDEP 2017 | 0.000020 | EDL[3] | -- | -- | 50 | 50 - 150 | 50 | 50 - 150 | 50 |

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| ANALYTES | CAS Number | Screening Level | Screening Level Source[1] | Practical Quantitation Limit (PQL) | Method Detection Limit (MDL) | QUALITY CONTROL LIMITS[2] Surrogate %R | | Duplicate RPD | Matrix Spike %R | Matrix Spike RPD | Blank Spike/LCS %R | Blank Spike/LCS RPD |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| 2,3,3',4',5-PeCB (pCB-107) | 70424-68-9 | 1.15 | NDEP 2017 | 0.000040 | EDL[3] | -- | -- | 50 | 50 - 150 | 50 | 50 - 150 | 50 |
| 2,3,3',4,5'-PeCB (PCB-108) | 70362-41-3 | 1.15 | NDEP 2017 | 0.000120 | EDL[3] | -- | -- | 50 | 50 - 150 | 50 | 50 - 150 | 50 |
| 2,3,3',4,6-PeCB (PCB-109) | 74472-35-8 | 1.15 | NDEP 2017 | 0.000020 | EDL[3] | -- | -- | 50 | 50 - 150 | 50 | 50 - 150 | 50 |
| 2,3,3',4',6-PeCB (PCB-110) | 38380-03-9 | 1.15 | NDEP 2017 | 0.000040 | EDL[3] | -- | -- | 50 | 50 - 150 | 50 | 50 - 150 | 50 |
| 2,3,3',5,5'-PeCB (PCB-111) | 39635-32-0 | 1.15 | NDEP 2017 | 0.000020 | EDL[3] | -- | -- | 50 | 50 - 150 | 50 | 50 - 150 | 50 |
| 2,3,3',5,6-PeCB (PCB-112) | 74472-36-9 | 1.15 | NDEP 2017 | 0.000020 | EDL[3] | -- | -- | 50 | 50 - 150 | 50 | 50 - 150 | 50 |
| 2,3,3',5',6-PeCB (PCB-113) | 68194-10-5 | 1.15 | NDEP 2017 | 0.000060 | EDL[3] | -- | -- | 50 | 50 - 150 | 50 | 50 - 150 | 50 |
| 2,3,4,4',5-PeCB (PCB-114) | 74472-37-0 | 0.589 | NDEP 2017 | 0.000002 | EDL[3] | -- | -- | 50 | 50 - 150 | 50 | 50 - 150 | 50 |
| 2,3,4,4',6-PeCB (PCB-115) | 74472-38-1 | 1.15 | NDEP 2017 | 0.000040 | EDL[3] | -- | -- | 50 | 50 - 150 | 50 | 50 - 150 | 50 |
| 2,3,4,5,6-PeCB (PCB-116) | 18259-05-7 | 1.15 | NDEP 2017 | 0.000060 | EDL[3] | -- | -- | 50 | 50 - 150 | 50 | 50 - 150 | 50 |
| 2,3,4',5,6-PeCB (PCB-117) | 68194-11-6 | 1.15 | NDEP 2017 | 0.000060 | EDL[3] | -- | -- | 50 | 50 - 150 | 50 | 50 - 150 | 50 |
| 2,3',4,4',5-PeCB (PCB-118) | 31508-00-6 | 0.589 | NDEP 2017 | 0.000002 | EDL[3] | -- | -- | 50 | 50 - 150 | 50 | 50 - 150 | 50 |
| 2,3',4,4',6-PeCB (PCB-119) | 56558-17-9 | 1.15 | NDEP 2017 | 0.000120 | EDL[3] | -- | -- | 50 | 50 - 150 | 50 | 50 - 150 | 50 |
| 2,3',4,5,5'-PeCB (PCB-120) | 68194-12-7 | 1.15 | NDEP 2017 | 0.000020 | EDL[3] | -- | -- | 50 | 50 - 150 | 50 | 50 - 150 | 50 |
| 2,3',4,5,'6-PeCB (PCB-121) | 56558-18-0 | 1.15 | NDEP 2017 | 0.000020 | EDL[3] | -- | -- | 50 | 50 - 150 | 50 | 50 - 150 | 50 |
| 2',3,3',4,5-PeCB (PCB-122) | 76842-07-4 | 1.15 | NDEP 2017 | 0.000020 | EDL[3] | -- | -- | 50 | 50 - 150 | 50 | 50 - 150 | 50 |
| 2',3,4,4',5-PeCB (PCB-123) | 65510-44-3 | 0.589 | NDEP 2017 | 0.000002 | EDL[3] | -- | -- | 50 | 50 - 150 | 50 | 50 - 150 | 50 |
| 2',3,4,5,5'-PeCB (PCB-124) | 70424-70-3 | 1.15 | NDEP 2017 | 0.000040 | EDL[3] | -- | -- | 50 | 50 - 150 | 50 | 50 - 150 | 50 |
| 2',3,4,5,6'-PeCB (PCB-125) | 74472-39-2 | 1.15 | NDEP 2017 | 0.000120 | EDL[3] | -- | -- | 50 | 50 - 150 | 50 | 50 - 150 | 50 |

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| ANALYTES | CAS Number | Screening Level | Screening Level Source[1] | Practical Quantitation Limit (PQL) | Method Detection Limit (MDL) | QUALITY CONTROL LIMITS[2] Surrogate %R | | Duplicate RPD | Matrix Spike %R | Matrix Spike RPD | Blank Spike/LCS %R | Blank Spike/LCS RPD |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| 3,3',4,4',5-PeCB (PCB-126) | 57465-28-8 | 0.000177 | NDEP 2017 | 0.000002 | EDL[3] | -- | -- | 50 | 50 - 150 | 50 | 50 - 150 | 50 |
| 3,3',4,5,5'-PeCB (PCB-127) | 39635-33-1 | 1.15 | NDEP 2017 | 0.000020 | EDL[3] | -- | -- | 50 | 50 - 150 | 50 | 50 - 150 | 50 |
| 2,2',3,3',4,4'-HxCB (PCB-128) | 38380-07-3 | 1.15 | NDEP 2017 | 0.000040 | EDL[3] | -- | -- | 50 | 50 - 150 | 50 | 50 - 150 | 50 |
| 2,2',3,3',4,5-HxCB (PCB-129) | 55215-18-4 | 1.15 | NDEP 2017 | 0.000060 | EDL[3] | -- | -- | 50 | 50 - 150 | 50 | 50 - 150 | 50 |
| 2,2',3,3',4,5'-HxCB (PCB-130) | 52663-66-8 | 1.15 | NDEP 2017 | 0.000020 | EDL[3] | -- | -- | 50 | 50 - 150 | 50 | 50 - 150 | 50 |
| 2,2',3,3',4,6-HxCB (PCB-131) | 61798-70-7 | 1.15 | NDEP 2017 | 0.000020 | EDL[3] | -- | -- | 50 | 50 - 150 | 50 | 50 - 150 | 50 |
| 2,2',3,3',4,6'-HxCB (PCB-132) | 38380-05-1 | 1.15 | NDEP 2017 | 0.000020 | EDL[3] | -- | -- | 50 | 50 - 150 | 50 | 50 - 150 | 50 |
| 2,2',3,3',5,5'-HxCB (PCB-133) | 35694-04-3 | 1.15 | NDEP 2017 | 0.000020 | EDL[3] | -- | -- | 50 | 50 - 150 | 50 | 50 - 150 | 50 |
| 2,2',3,3',5,6-HxCB (PCB-134) | 52704-70-8 | 1.15 | NDEP 2017 | 0.000040 | EDL[3] | -- | -- | 50 | 50 - 150 | 50 | 50 - 150 | 50 |
| 2,2',3,3',5,6'-HxCB (PCB-135) | 52744-13-5 | 1.15 | NDEP 2017 | 0.000040 | EDL[3] | -- | -- | 50 | 50 - 150 | 50 | 50 - 150 | 50 |
| 2,2',3,3',6,6'-HxCB (PCB-136) | 38411-22-2 | 1.15 | NDEP 2017 | 0.000020 | EDL[3] | -- | -- | 50 | 50 - 150 | 50 | 50 - 150 | 50 |
| 2,2',3,4,4',5-HxCB (PCB-137) | 35694-06-5 | 1.15 | NDEP 2017 | 0.000020 | EDL[3] | -- | -- | 50 | 50 - 150 | 50 | 50 - 150 | 50 |
| 2,2',3,4,4',5'-HxCB (PCB-138) | 35065-28-2 | 1.15 | NDEP 2017 | 0.000060 | EDL[3] | -- | -- | 50 | 50 - 150 | 50 | 50 - 150 | 50 |
| 2,2',3,4,4',6-HxCB (PCB-139) | 56030-56-9 | 1.15 | NDEP 2017 | 0.000040 | EDL[3] | -- | -- | 50 | 50 - 150 | 50 | 50 - 150 | 50 |
| 2,2',3,4,4',6'-HxCB (PCB-140) | 59291-64-4 | 1.15 | NDEP 2017 | 0.000040 | EDL[3] | -- | -- | 50 | 50 - 150 | 50 | 50 - 150 | 50 |
| 2,2',3,4,5,5'-HxCB (PCB-141) | 52712-04-6 | 1.15 | NDEP 2017 | 0.000020 | EDL[3] | -- | -- | 50 | 50 - 150 | 50 | 50 - 150 | 50 |
| 2,2',3,4,5,6-HxCB (PCB-142) | 41411-61-4 | 1.15 | NDEP 2017 | 0.000020 | EDL[3] | -- | -- | 50 | 50 - 150 | 50 | 50 - 150 | 50 |
| 2,2',3,4,5,6'-HxCB (PCB-143) | 68194-15-0 | 1.15 | NDEP 2017 | 0.000040 | EDL[3] | -- | -- | 50 | 50 - 150 | 50 | 50 - 150 | 50 |
| 2,2',3,4,5',6-HxCB (PCB-144) | 68194-14-9 | 1.15 | NDEP 2017 | 0.000020 | EDL[3] | -- | -- | 50 | 50 - 150 | 50 | 50 - 150 | 50 |

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|---|---|---|---|---|---|---|---|---|---|---|---|---|
| 2,2',3,4,6,6'-HxCB (PCB-145) | 74472-40-5 | 1.15 | NDEP 2017 | 0.000020 | EDL[3] | -- | -- | 50 | 50 - 150 | 50 | 50 - 150 | 50 |
| 2,2',3,4',5,5'-HxCB (PCB-146) | 51908-16-8 | 1.15 | NDEP 2017 | 0.000020 | EDL[3] | -- | -- | 50 | 50 - 150 | 50 | 50 - 150 | 50 |
| 2,2',3,4',5,6-HxCB (PCB-147) | 68194-13-8 | 1.15 | NDEP 2017 | 0.000040 | EDL[3] | -- | -- | 50 | 50 - 150 | 50 | 50 - 150 | 50 |
| 2,2',3,4',5,6'-HxCB (PCB-148) | 74472-41-6 | 1.15 | NDEP 2017 | 0.000020 | EDL[3] | -- | -- | 50 | 50 - 150 | 50 | 50 - 150 | 50 |
| 2,2',3,4',5',6-HxCB (PCB-149) | 38380-04-0 | 1.15 | NDEP 2017 | 0.000040 | EDL[3] | -- | -- | 50 | 50 - 150 | 50 | 50 - 150 | 50 |
| 2,2',3,4',6,6'-HxCB (PCB-150) | 68194-08-1 | 1.15 | NDEP 2017 | 0.000020 | EDL[3] | -- | -- | 50 | 50 - 150 | 50 | 50 - 150 | 50 |
| 2,2',3,5,5',6-HxCB (PCB-151) | 52663-63-5 | 1.15 | NDEP 2017 | 0.000040 | EDL[3] | -- | -- | 50 | 50 - 150 | 50 | 50 - 150 | 50 |
| 2,2',3,5,6,6'-HxCB (PCB-152) | 68194-09-2 | 1.15 | NDEP 2017 | 0.000020 | EDL[3] | -- | -- | 50 | 50 - 150 | 50 | 50 - 150 | 50 |
| 2,2',4,4',5,5'-HxCB (PCB-153) | 35065-27-1 | 1.15 | NDEP 2017 | 0.000040 | EDL[3] | -- | -- | 50 | 50 - 150 | 50 | 50 - 150 | 50 |
| 2,2',4,4',5',6-HxCB (PCB-154) | 60145-22-4 | 1.15 | NDEP 2017 | 0.000020 | EDL[3] | -- | -- | 50 | 50 - 150 | 50 | 50 - 150 | 50 |
| 2,2',4,4',6,6'-HxCB (PCB-155) | 33979-03-2 | 1.15 | NDEP 2017 | 0.000020 | EDL[3] | -- | -- | 50 | 50 - 150 | 50 | 50 - 150 | 50 |
| 2,3,3',4,4',5-HxCB (PCB-156) | 38380-08-4 | 0.589 | NDEP 2017 | 0.000004 | EDL[3] | -- | -- | 50 | 50 - 150 | 50 | 50 - 150 | 50 |
| 2,3,3',4,4',5'-HxCB (PCB-157) | 69782-90-7 | 0.589 | NDEP 2017 | 0.000004 | EDL[3] | -- | -- | 50 | 50 - 150 | 50 | 50 - 150 | 50 |
| 2,3,3',4,4',6-HxCB (PCB-158) | 74472-42-7 | 1.15 | NDEP 2017 | 0.000020 | EDL[3] | -- | -- | 50 | 50 - 150 | 50 | 50 - 150 | 50 |
| 2,3,3',4,5,5'-HxCB (PCB-159) | 39635-35-3 | 1.15 | NDEP 2017 | 0.000020 | EDL[3] | -- | -- | 50 | 50 - 150 | 50 | 50 - 150 | 50 |
| 2,3,3',4,5,6-HxCB (PCB-160) | 41411-62-5 | 1.15 | NDEP 2017 | 0.000020 | EDL[3] | -- | -- | 50 | 50 - 150 | 50 | 50 - 150 | 50 |
| 2,3,3',4,5',6-HxCB (PCB-161) | 74472-43-8 | 1.15 | NDEP 2017 | 0.000020 | EDL[3] | -- | -- | 50 | 50 - 150 | 50 | 50 - 150 | 50 |
| 2,3,3',4',5,5'-HxCB (PCB-162) | 39635-34-2 | 1.15 | NDEP 2017 | 0.000020 | EDL[3] | -- | -- | 50 | 50 - 150 | 50 | 50 - 150 | 50 |
| 2,3,3',4',5,6-HxCB (PCB-163) | 74472-44-9 | 1.15 | NDEP 2017 | 0.000060 | EDL[3] | -- | -- | 50 | 50 - 150 | 50 | 50 - 150 | 50 |

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|---|---|---|---|---|---|---|---|---|---|---|---|---|
| 2,3,3',4',5',6-HxCB (PCB-164) | 74472-45-0 | 1.15 | NDEP 2017 | 0.000020 | EDL[3] | -- | -- | 50 | 50 - 150 | 50 | 50 - 150 | 50 |
| 2,3,3',5,5',6-HxCB (PCB-165) | 74472-46-1 | 1.15 | NDEP 2017 | 0.000020 | EDL[3] | -- | -- | 50 | 50 - 150 | 50 | 50 - 150 | 50 |
| 2,3,4,4',5,6-HxCB (PCB-166) | 41411-63-6 | 1.15 | NDEP 2017 | 0.000040 | EDL[3] | -- | -- | 50 | 50 - 150 | 50 | 50 - 150 | 50 |
| 2,3',4,4',5,5'-HxCB (PCB-167) | 52663-72-6 | 0.589 | NDEP 2017 | 0.000002 | EDL[3] | -- | -- | 50 | 50 - 150 | 50 | 50 - 150 | 50 |
| 2,3',4,4',5',6-HxCB (PCB-168) | 59291-65-5 | 1.15 | NDEP 2017 | 0.000040 | EDL[3] | -- | -- | 50 | 50 - 150 | 50 | 50 - 150 | 50 |
| 3,3',4,4',5,5'-HxCB (PCB-169) | 32774-16-6 | 0.000589 | NDEP 2017 | 0.000002 | EDL[3] | -- | -- | 50 | 50 - 150 | 50 | 50 - 150 | 50 |
| 2,2',3,3',4,4',5-HpCB (PCB-170) | 35065-30-6 | 1.15 | NDEP 2017 | 0.000020 | EDL[3] | -- | -- | 50 | 50 - 150 | 50 | 50 - 150 | 50 |
| 2,2'3,3',4,4',6-HpCB (PCB-171) | 52663-71-5 | 1.15 | NDEP 2017 | 0.000040 | EDL[3] | -- | -- | 50 | 50 - 150 | 50 | 50 - 150 | 50 |
| 2,2',3,3',4,5,5'-HpCB (PCB-172) | 52663-74-8 | 1.15 | NDEP 2017 | 0.000020 | EDL[3] | -- | -- | 50 | 50 - 150 | 50 | 50 - 150 | 50 |
| 2,2',3,3',4,5,6-HpCB (PCB-173) | 68194-16-1 | 1.15 | NDEP 2017 | 0.000040 | EDL[3] | -- | -- | 50 | 50 - 150 | 50 | 50 - 150 | 50 |
| 2,2',3,3',4,5,6'-HpCB (PCB-174) | 38411-25-5 | 1.15 | NDEP 2017 | 0.000020 | EDL[3] | -- | -- | 50 | 50 - 150 | 50 | 50 - 150 | 50 |
| 2,2',3,3',4,5',6-HpCB (PCB-175) | 40186-70-7 | 1.15 | NDEP 2017 | 0.000020 | EDL[3] | -- | -- | 50 | 50 - 150 | 50 | 50 - 150 | 50 |
| 2,2',3,3',4,6,6'-HpCB (PCB-176) | 52663-65-7 | 1.15 | NDEP 2017 | 0.000020 | EDL[3] | -- | -- | 50 | 50 - 150 | 50 | 50 - 150 | 50 |
| 2,2',3,3',4',5,6-HpCB (PCB-177) | 52663-70-4 | 1.15 | NDEP 2017 | 0.000020 | EDL[3] | -- | -- | 50 | 50 - 150 | 50 | 50 - 150 | 50 |
| 2,2',3,3',5,5',6-HpCB (PCB-178) | 52663-67-9 | 1.15 | NDEP 2017 | 0.000020 | EDL[3] | -- | -- | 50 | 50 - 150 | 50 | 50 - 150 | 50 |
| 2,2',3,3',5,6,6'-HpCB (PCB-179) | 52663-64-6 | 1.15 | NDEP 2017 | 0.000020 | EDL[3] | -- | -- | 50 | 50 - 150 | 50 | 50 - 150 | 50 |
| 2,2',3,4,4',5,5'-HpCB (PCB-180) | 35065-29-3 | 1.15 | NDEP 2017 | 0.000040 | EDL[3] | -- | -- | 50 | 50 - 150 | 50 | 50 - 150 | 50 |
| 2,2',3,4,4',5,6-HpCB (PCB-181) | 74472-47-2 | 1.15 | NDEP 2017 | 0.000020 | EDL[3] | -- | -- | 50 | 50 - 150 | 50 | 50 - 150 | 50 |
| 2,2',3,4,4',5,6'-HpCB (PCB-182) | 60145-23-5 | 1.15 | NDEP 2017 | 0.000020 | EDL[3] | -- | -- | 50 | 50 - 150 | 50 | 50 - 150 | 50 |

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|---|---|---|---|---|---|---|---|---|---|---|---|---|
| 2,2',3,4,4',5',6-HpCB (PCB-183) | 52663-69-1 | 1.15 | NDEP 2017 | 0.000020 | EDL[3] | -- | -- | 50 | 50 - 150 | 50 | 50 - 150 | 50 |
| 2,2',3,4,4',6,6'-HpCB (PCB-184) | 74472-48-3 | 1.15 | NDEP 2017 | 0.000020 | EDL[3] | -- | -- | 50 | 50 - 150 | 50 | 50 - 150 | 50 |
| 2,2',3,4,5,5',6-HpCB (PCB-185) | 52712-05-7 | 1.15 | NDEP 2017 | 0.000020 | EDL[3] | -- | -- | 50 | 50 - 150 | 50 | 50 - 150 | 50 |
| 2,2',3,4,5,6,6'-HpCB (PCB-186) | 74472-49-4 | 1.15 | NDEP 2017 | 0.000020 | EDL[3] | -- | -- | 50 | 50 - 150 | 50 | 50 - 150 | 50 |
| 2,2',3,4',5,5',6-HpCB (PCB-187) | 52663-68-0 | 1.15 | NDEP 2017 | 0.000020 | EDL[3] | -- | -- | 50 | 50 - 150 | 50 | 50 - 150 | 50 |
| 2,2',3,4',5,6,6'-HpCB (PCB-188) | 74487-85-7 | 1.15 | NDEP 2017 | 0.000020 | EDL[3] | -- | -- | 50 | 50 - 150 | 50 | 50 - 150 | 50 |
| 2,3,3',4,4',5,5'-HpCB (PCB-189) | 39635-31-9 | 0.589 | NDEP 2017 | 0.000002 | EDL[3] | -- | -- | 50 | 50 - 150 | 50 | 50 - 150 | 50 |
| 2,3,3',4,4',5,6-HpCB (PCB-190) | 41411-64-7 | 1.15 | NDEP 2017 | 0.000020 | EDL[3] | -- | -- | 50 | 50 - 150 | 50 | 50 - 150 | 50 |
| 2,3,3',4,4',5',6-HpCB (PCB-191) | 74472-50-7 | 1.15 | NDEP 2017 | 0.000020 | EDL[3] | -- | -- | 50 | 50 - 150 | 50 | 50 - 150 | 50 |
| 2,3,3',4,5,5',6-HpCB (PCB-192) | 74472-51-8 | 1.15 | NDEP 2017 | 0.000020 | EDL[3] | -- | -- | 50 | 50 - 150 | 50 | 50 - 150 | 50 |
| 2,3,3',4',5,5',6-HpCB (PCB-193) | 69782-91-8 | 1.15 | NDEP 2017 | 0.000040 | EDL[3] | -- | -- | 50 | 50 - 150 | 50 | 50 - 150 | 50 |
| 2,2',3,3',4,4',5,5'-OcCB (PCB-194) | 35694-08-7 | 1.15 | NDEP 2017 | 0.000020 | EDL[3] | -- | -- | 50 | 50 - 150 | 50 | 50 - 150 | 50 |
| 2,2',3,3',4,4',5,6-OcCB (PCB-195) | 52663-78-2 | 1.15 | NDEP 2017 | 0.000020 | EDL[3] | -- | -- | 50 | 50 - 150 | 50 | 50 - 150 | 50 |
| 2,2',3,3',4,4',5,6'-OcCB (PCB-196) | 42740-50-1 | 1.15 | NDEP 2017 | 0.000020 | EDL[3] | -- | -- | 50 | 50 - 150 | 50 | 50 - 150 | 50 |
| 2,2',3,3',4,4',6,6'-OcCB (PCB-197) | 33091-17-7 | 1.15 | NDEP 2017 | 0.000020 | EDL[3] | -- | -- | 50 | 50 - 150 | 50 | 50 - 150 | 50 |
| 2,2',3,3',4,5,5',6-OcCB (PCB-198) | 68194-17-2 | 1.15 | NDEP 2017 | 0.000040 | EDL[3] | -- | -- | 50 | 50 - 150 | 50 | 50 - 150 | 50 |
| 2,2',3,3',4,5,5',6'-OcCB (PCB-199) | 52663-75-9 | 1.15 | NDEP 2017 | 0.000040 | EDL[3] | -- | -- | 50 | 50 - 150 | 50 | 50 - 150 | 50 |
| 2,2',3,3',4,5,6,6'-OcCB (PCB-200) | 52663-73-7 | 1.15 | NDEP 2017 | 0.000020 | EDL[3] | -- | -- | 50 | 50 - 150 | 50 | 50 - 150 | 50 |
| 2,2',3,3',4,5',6,6'-OcCB (PCB-201) | 40186-71-8 | 1.15 | NDEP 2017 | 0.000020 | EDL[3] | -- | -- | 50 | 50 - 150 | 50 | 50 - 150 | 50 |

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|---|---|---|---|---|---|---|---|---|---|---|---|
| 2,2',3,3',5,5',6,6'-OcCB (PCB-202) | 2136-99-4 | 1.15 | NDEP 2017 | 0.000020 | EDL[3] | -- -- | 50 | 50 - 150 | 50 | 50 - 150 | 50 |
| 2,2',3,4,4',5,5',6-OcCB (PCB-203) | 52663-76-0 | 1.15 | NDEP 2017 | 0.000020 | EDL[3] | -- -- | 50 | 50 - 150 | 50 | 50 - 150 | 50 |
| 2,2',3,4,4',5,6,6'-OcCB (PCB-204) | 74472-52-9 | 1.15 | NDEP 2017 | 0.000020 | EDL[3] | -- -- | 50 | 50 - 150 | 50 | 50 - 150 | 50 |
| 2,3,3',4,4',5,5',6-OcCB (PCB-205) | 74472-53-0 | 1.15 | NDEP 2017 | 0.000020 | EDL[3] | -- -- | 50 | 50 - 150 | 50 | 50 - 150 | 50 |
| 2,2',3,3',4,4',5,5',6-NoCB (PCB-206) | 40186-72-9 | 1.15 | NDEP 2017 | 0.000020 | EDL[3] | -- -- | 50 | 50 - 150 | 50 | 50 - 150 | 50 |
| 2,2',3,3',4,4',5,6,6'-NoCB (PCB-207) | 52663-79-3 | 1.15 | NDEP 2017 | 0.000020 | EDL[3] | -- -- | 50 | 50 - 150 | 50 | 50 - 150 | 50 |
| 2,2',3,3',4,5,5',6,6'-NoCB (PCB-208) | 52663-77-1 | 1.15 | NDEP 2017 | 0.000020 | EDL[3] | -- -- | 50 | 50 - 150 | 50 | 50 - 150 | 50 |
| DeCB (PCB-209) | 2051-24-3 | 1.15 | NDEP 2017 | 0.00002 | EDL[3] | -- -- | 50 | 50 - 150 | 50 | 50 - 150 | 50 |
| **PCBs as Aroclors (mg/kg)** | | | | | | | | | | | |
| *EPA Method 8082* | | | | | | | | | | | |
| Aroclor 1260 | 11096-82-5 | 1.15 | NDEP 2017 | 0.05 | 0.02 | -- -- | 50 | 50 - 125 | 30 | 65 - 115 | 30 |
| DCB Decachlorobiphenyl (Surr) | 2051-24-3 | -- | -- | -- | -- | 45 - 120 | -- | -- -- | -- | -- -- | -- |
| **Organic Acids (mg/kg)** | | | | | | | | | | | |
| *EPA Method 8270C* | | | | | | | | | | | |
| Phthalic acid[6] | 88-99-3 | -- | -- | 2.5 | 0.76 | -- -- | 50 | -- -- | -- | -- -- | -- |
| 2-fluorobiphenyl (Surr) | 321-60-8 | | | -- | -- | 29 - 120 | -- | -- -- | -- | -- -- | -- |
| **Total Petroleum Hydrocarbons (mg/kg)** | | | | | | | | | | | |
| *EPA Method 8015B* | | | | | | | | | | | |
| Gasoline Range Organics (C6-C10) | TPH-gasoline | 100 | ENVIRON 2012[7] | 0.40 | 0.15 | -- -- | 50 | 60 - 140 | 30 | 70 - 135 | 20 |
| 4-Bromofluorobenzene (Surr) | 460-00-4 | -- | -- | -- | -- | 65 - 140 | -- | -- -- | -- | -- -- | -- |
| Diesel Range Organics (C10-C28) | TPH-diesel | 100 | ENVIRON 2012[7] | 5 | 2.5 | -- -- | 50 | 40 - 120 | 30 | 45 - 115 | 25 |

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|---|---|---|---|---|---|---|---|---|---|---|---|
| Oil Range Organics (C29-C40) | TPH-oil | 100 | ENVIRON 2012[7] | 5 | 2.5 | -- -- | 50 | 40 - 120 | 30 | 45 - 115 | 25 |
| n-Octacosane (Surr) | 630-02-4 | -- | -- | -- | -- | 40 - 140 | -- | -- -- | -- | -- -- | -- |
| **Wet Chemistry and Miscellanous Analytes (mg/kg except as noted)** | | | | | | | | | | | |
| *SM 2320B* | | | | | | | | | | | |
| Alkalinity as $CaCO_3$ | -- | -- | NDEP 2017 | 500 | -- | -- -- | 50 | -- -- | -- | 80 - 120 | 20 |
| Bicarbonate as $HCO_3^-$ | -- | -- | NDEP 2017 | 610 | -- | -- -- | 50 | -- -- | -- | -- -- | -- |
| Carbonate as $CO_3^-$ | -- | -- | NDEP 2017 | 300 | -- | -- -- | 50 | -- -- | -- | -- -- | -- |
| Hydroxide as $OH^-$ | 14280-30-9 | -- | NDEP 2017 | 170 | -- | -- -- | 50 | -- -- | -- | -- -- | -- |
| *SM 4500-NH3 D* | | | | | | | | | | | |
| Ammonia as $NH_3$ | 7664-41-7 | 6,140 | NDEP 2017 | 12 | 2.4 | -- -- | 50 | 75 - 125 | 15 | 85 - 115 | 15 |
| *EPA Method 300.0* | | | | | | | | | | | |
| Bromide | 24959-67-9 | 100,000 | NDEP 2017 | 5.0 | 3.5 | -- -- | 50 | 80 - 120 | 20 | 90 - 110 | 20 |
| Chloride | 16887-00-6 | -- | NDEP 2017 | 5.0 | 4.0 | -- -- | 50 | 80 - 120 | 20 | 90 - 110 | 20 |
| Fluoride | 16984-48-8 | 51,900 | NDEP 2017 | 5.0 | 3.5 | -- -- | 50 | 80 - 120 | 20 | 90 - 110 | 20 |
| Nitrate | 14797-55-8 | 100,000 | NDEP 2017 | 1.1 | 0.8 | -- -- | 50 | 80 - 120 | 20 | 90 - 110 | 20 |
| Nitrite | 14797-65-0 | 100,000 | NDEP 2017 | 1.5 | 1.1 | -- -- | 50 | 80 - 120 | 20 | 90 - 110 | 20 |
| Orthophosphate as $PO_4$ | 14265-44-2 | -- | NDEP 2017 | 5.0 | 4.0 | -- -- | 50 | 80 - 120 | 20 | 90 - 110 | 20 |
| Sulfate | 14808-79-8 | -- | NDEP 2017 | 5.0 | 4.0 | -- -- | 50 | 80 - 120 | 20 | 90 - 110 | 20 |
| *EPA Method 300.1* | | | | | | | | | | | |
| Chlorate | 7790-93-4 | 38,900 | NDEP 2017 | 0.2 | 0.05 | -- -- | 50 | 75 - 125 | 25 | 75 - 125 | 25 |
| Dichloroacetic acid (Surr) | 79-43-6 | -- | -- | -- | -- | 90 - 115 | -- | -- -- | -- | -- -- | -- |
| *EPA Method 314.0* | | | | | | | | | | | |
| Perchlorate | 14797-73-0 | 908 | NDEP 2017 | 0.04 | 0.0095 | -- -- | 50 | 80 - 120 | 20 | 85 - 115 | 15 |
| *EPA Method 9014B* | | | | | | | | | | | |
| Cyanide (total) | 57-12-5 | 179 | NDEP 2017 | 0.5 | 0.43 | -- -- | 50 | 70 - 115 | 15 | 90 - 110 | 10 |
| *EPA Method 120.1 / SM 2510B* | | | | | | | | | | | |
| Conductivity (µmho/cm) | -- | -- | NDEP 2017 | 10.0 | -- | -- -- | 50 | -- -- | -- | 90 - 110 | 20 |
| *EPA Method 9045C (SU)* | | | | | | | | | | | |

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**QUALITY ASSURANCE PROJECT PLAN**
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| ANALYTES | CAS Number | Screening Level | Screening Level Source[1] | Practical Quantitation Limit (PQL) | Method Detection Limit (MDL) | QUALITY CONTROL LIMITS[2] Surrogate %R | Duplicate RPD | Matrix Spike %R | Matrix Spike RPD | Blank Spike/LCS %R | Blank Spike/LCS RPD |
|---|---|---|---|---|---|---|---|---|---|---|---|
| pH | -- | -- | NDEP 2017 | 0.1 | -- | -- -- | 50 | -- -- | -- | -- -- | -- |
| *SM 5540C* | | | | | | | | | | | |
| Surfactants (MBAS) | -- | -- | NDEP 2017 | 1 | 0.5 | -- -- | 50 | 50 - 125 | 20 | 90 - 110 | 20 |
| *SM 5310B* | | | | | | | | | | | |
| Total Organic Carbon | 7440-44-0 | -- | NDEP 2017 | 1 | 0.75 | -- -- | 50 | 80 - 120 | 20 | 90 - 110 | 20 |
| **Radionuclides (pCi/g)[8]** | | | | | | | | | | | |
| *See Table 1 for Individual Methods* | | | | | | | | | | | |
| Radium-226 | 13982-63-3 | 0.023 | NDEP 2017 | 1 | -- | -- -- | 50 | 72 - 140 | 40 | 65 - 140 | 40 |
| Radium-228 | 15262-20-1 | 0.041 | NDEP 2017 | 1 | -- | -- -- | 50 | 30 - 150 | 40 | 61 - 139 | 40 |
| Thorium-228 | 14274-82-9 | 0.025 | NDEP 2017 | 1 | -- | -- -- | 50 | 70 - 130 | 40 | 70 - 130 | 40 |
| Thorium-230 | 14269-63-7 | 8.3 | NDEP 2017 | 1 | -- | -- -- | 50 | 76 - 115 | 40 | 81 - 118 | 40 |
| Thorium-232 | 7440-29-1 | 7.4 | NDEP 2017 | 1 | -- | -- -- | 50 | 70 - 130 | 40 | 70 - 130 | 40 |
| Uranium-234 | 13966-29-5 | 11 | NDEP 2017 | 1 | -- | -- -- | 50 | 70 - 130 | 40 | 84 - 120 | 40 |
| Uranium-235 | 15117-96-1 | 0.35 | NDEP 2017 | 1 | -- | -- -- | 50 | -- -- | 40 | -- -- | 40 |
| Uranium-238 | 7440-61-1 | 1.4 | NDEP 2017 | 1 | -- | -- -- | 50 | 70 - 130 | 40 | 82 - 122 | 40 |
| **Asbestos (protocol structures)** | | | | | | | | | | | |
| *EPA Method 540-R-97-028 modified per Berman & Kolk (2000)* | | | | | | | | | | | |
| Total Amphibole Protocol Structures | 1332-21-4 | -- | -- | Fiber Count[9] | | -- -- | 50 | -- -- | -- | -- -- | -- |
| Long Amphibole Protocol Structures | 1332-21-4 | 1 or more | NDEP (2010) | Fiber Count[9] | | -- -- | 50 | -- -- | -- | -- -- | -- |
| Total Chrysotile Protocol Structures | 1332-21-4 | -- | -- | Fiber Count[9] | | -- -- | 50 | -- -- | -- | -- -- | -- |
| Long Chrysotile Protocol Structures | 1332-21-4 | More than 5 | NDEP (2010) | Fiber Count[9] | | -- -- | 50 | -- -- | -- | -- -- | -- |
| Total Asbestos Protocol Structures | 1332-21-4 | -- | -- | Fiber Count[9] | | -- -- | 50 | -- -- | -- | -- -- | -- |
| Long Asbestos Protocol Structures | 1332-21-4 | -- | -- | Fiber Count[9] | | -- -- | 50 | -- -- | -- | -- -- | -- |

**TABLE 2. SOIL ANALYTES AND ANALYTICAL PERFORMANCE CRITERIA**
**QUALITY ASSURANCE PROJECT PLAN**
**Nevada Environmental Response Trust Site; Henderson, Nevada**

| ANALYTES | CAS Number | Screening Level | Screening Level Source[1] | Practical Quantitation Limit (PQL) | Method Detection Limit (MDL) | QUALITY CONTROL LIMITS[2] Surrogate %R | Duplicate RPD | Matrix Spike %R | Matrix Spike RPD | Blank Spike/LCS %R | Blank Spike/LCS RPD |
|---|---|---|---|---|---|---|---|---|---|---|---|

**Notes:**

Shaded PQLs and MDLs exceed the lowest screening criteria.

-- = no value

µg/kg = milligram per kilogram

mg/kg = milligram per kilogram

pCi/g = picoCurie per gram

pg/g = picogram per gram

protocol structure = asbestos protocol structures greater than 10 micrometers (µm) in length and less than 0.4 µm in width that is most responsible for asbestos related disease (NDEP 2011).

Surr = Surrogate

TEQ = toxicity equivalence

EPA = United States Environmental Protection Agency

SM = Standard Method

(1) Screening values obtained from (a) NDEP (2017) and are the lower of the indoor and outdoor industrial/commercial worker soil Basic Comparison Levels (BCLs); and (b) NDEP (2010) and are site-specific levels for indoor and outdoor industrial/commercial workers or based on regional background concentrations.

(2) QC Limits = Quality Control Limits for %R (Percent Recovery) of spiked compounds in Laboratory Control Samples (LCS) and surrogate compounds and Relative Percent Difference (RPD) between Matrix Spike (MS) and MS Duplicate (MSD) samples and LCS and LCS duplicate (LCSD) samples. Laboratory historical control limits are subject to change as a result of periodic re-evaluation. Limits in use at the time of sample analysis are available from the laboratory. Duplicate RPDs apply to sample duplicates and field duplicates.

(3) EDL = Estimated Detection Limit. For each dioxin, furan, or PCB not detected, an EDL is calculated. The sample specific EDL is an estimate made by the laboratory of the concentration of a given chemical that would have to be present to produce a signal with a peak height of at least 2.5 times the background signal level. The estimate is specific to a particular analysis of the sample and will be affected by sample size, dilution, and so forth. Because of the toxicological significance of dioxins, the EDL value is reported for non-detected chemicals rather than reporting the MDL.

(4) Dioxins and PCB congeners shall be reported to the estimated detection limit (EDL). Dioxin toxicity equivalents (TEQ) will be calculated for the 16 dioxin and furan congeners and 12 PCB congeners with toxicity equivalent factors (TEFs) defined by the World Health Organization (Van den Berg et al. 2006) substituting half of the EDL for the congeners not detected.

(5) The screening level for m-xylene is used for m,p-xylene.

(6) Phthalic acid will be run with the SVOCs by EPA Method 8270C.

(7) A total TPH value of 100 mg/kg was used in the Interim Soil Removal Actions Report (ENVIRON 2012) and the Site Management Plan, Revision 1 (SMP) (2013).

(8) Radionuclide PQLs and MDLs are based on minimum detectable activity (MDA) values. The measured values are reported regardless of sample-specific MDA.

(9) Asbestos data will be reported as raw asbestos fiber counts per sample (NDEP 2008). There are no PQLs for this method, but sensitivity is calculated by the concentration of protocol structures per volume of PM10.

**TABLE 2. SOIL ANALYTES AND ANALYTICAL PERFORMANCE CRITERIA**
**QUALITY ASSURANCE PROJECT PLAN**
**Nevada Environmental Response Trust Site; Henderson, Nevada**

| ANALYTES | CAS Number | Screening Level | Screening Level Source[1] | Practical Quantitation Limit (PQL) | Method Detection Limit (MDL) | QUALITY CONTROL LIMITS[2] | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|
| | | | | | | Surrogate | Duplicate | Matrix Spike | | Blank Spike/LCS | |
| | | | | | | %R | RPD | %R | RPD | %R | RPD |

**Sources:**


ENVIRON. 2012. Interim Soil Removal Action , Nevada Environmental Response Trust Site, Henderson, Nevada, August 2010-November 2011. Revised September 2012. NDEP approved December 17, 2012.

ENVIRON. 2013. Site Management Plan, Revision 1, Nevada Environmental Response Trust Site, Henderson, Nevada. October 31.

NDEP. 2008. NDEP. 2008. NDEP Detection Limits and Data Reporting for the BMI Plant Sites and Common Areas Projects, Henderson, Nevada. December.

NDEP. 2010. Letter to Tronox LLC re: Response to: Results of Bioaccessibility Study for Dioxin/Furans in Soil, Tronox LLC, Henderson, Nevada (Revised), Dated May 24, 2010. May 25, 2010.

NDEP. 2011. Technical Guidance for the Calculation of Asbestos Related Risk in Soils for the Basic Management Incorporated (BMI) Complex and Common Areas. February.

NDEP. 2017. User's Guide and Background Technical Document for NDEP Basic Comparison Levels (BCLs) for Human Health for the BMI Complex and Common Areas. Revision 14, July.

Van den Berg et al., 2006. The 2005 World Health Organization Reevaluation of Human and Mammalian Toxic Equivalency Factors for Dioxins and Dioxin-Like Compounds. May 20.

**TABLE 3. SOIL GAS ANALYTES AND ANALYTICAL PERFORMANCE CRITERIA**
**QUALITY ASSURANCE PROJECT PLAN**
**Nevada Environmental Response Trust Site; Henderson, Nevada**

| ANALYTES | CAS Number | Screening Level | Screening Level Source[1] | Practical Quantitation Limit (PQL) | Method Detection Limit (MDL) | QUALITY CONTROL LIMITS[2] Surrogate %R | | Duplicate RPD | Blank Spike/LCS %R | Blank Spike/LCS RPD |
|---|---|---|---|---|---|---|---|---|---|---|
| **Soil Gas Analytes (µg/m$^3$)** | | | | | | | | | | |
| *EPA Method TO-15* | | | | | | | | | | |
| Acetone | 67-64-1 | 4.12E+08 | ENVIRON 2013 | 60 | 8.938 | -- | -- | 50 | 60 - 140 | 25 |
| Acrolein | 107-02-8 | -- | ENVIRON 2013 | 1.16 | 0.2331 | -- | -- | 50 | 60 - 140 | 25 |
| Acrylonitrile | 107-13-1 | 5.59E+02 | ENVIRON 2013 | 1.1 | 0.6105 | -- | -- | 50 | 60 - 140 | 25 |
| Benzene | 71-43-2 | 6.20E+03 | ENVIRON 2013 | 1.6 | 0.2597 | -- | -- | 50 | 60 - 140 | 25 |
| Benzyl chloride | 100-44-7 | 1.27E+03 | ENVIRON 2013 | 2.65 | 0.3783 | -- | -- | 50 | 60 - 140 | 25 |
| Bromodichloromethane | 75-27-4 | 3.19E+03 | ENVIRON 2013 | 3.5 | 0.01098 | -- | -- | 50 | 60 - 140 | 25 |
| Bromoform | 75-25-2 | 2.01E+05 | ENVIRON 2013 | 5.25 | 0.2214 | -- | -- | 50 | 60 - 140 | 25 |
| Bromomethane | 74-83-9 | 9.97E+04 | ENVIRON 2013 | 1.95 | 0.35814 | -- | -- | 50 | 60 - 140 | 25 |
| 1,3-Butadiene | 106-99-0 | 1.47E+03 | ENVIRON 2013 | 1.1 | 0.5535 | -- | -- | 50 | 60 - 140 | 25 |
| 2-Butanone (MEK) | 78-93-3 | 9.02E+07 | ENVIRON 2013 | 75 | 3.8542 | -- | -- | 50 | 60 - 140 | 25 |
| Carbon Disulfide | 75-15-0 | 1.07E+07 | ENVIRON 2013 | 1.6 | 0.41785 | -- | -- | 50 | 60 - 140 | 25 |
| Carbon Tetrachloride | 56-23-5 | 8.82E+03 | ENVIRON 2013 | 3.2 | 0.0115 | -- | -- | 50 | 60 - 140 | 25 |
| Chlorobenzene | 108-90-7 | 9.95E+05 | ENVIRON 2013 | 2.35 | 0.1584 | -- | -- | 50 | 60 - 140 | 25 |
| Chloroethane | 75-00-3 | 8.61E+07 | ENVIRON 2013 | 1.34 | 0.3462 | -- | -- | 50 | 60 - 140 | 25 |
| Chloroform | 67-66-3 | 1.86E+03 | ENVIRON 2013 | 2.45 | 0.01512 | -- | -- | 50 | 60 - 140 | 25 |
| Chloromethane | 74-87-3 | 2.08E+04 | ENVIRON 2013 | 1.05 | 0.1218 | -- | -- | 50 | 60 - 140 | 25 |
| Cyclohexane | 110-82-7 | 1.11E+08 | ENVIRON 2013 | 17.5 | 0.3815 | -- | -- | 50 | 60 - 140 | 25 |
| Dibromochloromethane | 124-48-1 | 6.38E+03 | ENVIRON 2013 | 4.35 | 0.0048 | -- | -- | 50 | 60 - 140 | 25 |
| 1,2-Dibromo-3-chloropropane | 96-12-8 | 1.83E+01 | ENVIRON 2013 | 0.123 | 0.0056 | -- | -- | 50 | 60 - 140 | 25 |
| 1,2-Dibromoethane (EDB) | 106-93-4 | 2.61E+02 | ENVIRON 2013 | 3.9 | 0.00374 | -- | -- | 50 | 60 - 140 | 25 |
| 1,2-Dichlorobenzene | 95-50-1 | 4.16E+06 | ENVIRON 2013 | 3.05 | 0.16448 | -- | -- | 50 | 60 - 140 | 25 |
| 1,3-Dichlorobenzene | 541-73-1 | 4.15E+06 | ENVIRON 2013 | 3.05 | 0.25811 | -- | -- | 50 | 60 - 140 | 25 |
| 1,4-Dichlorobenzene | 106-46-7 | 5.29E+03 | ENVIRON 2013 | 3.05 | 0.6161 | -- | -- | 50 | 60 - 140 | 25 |
| Dichlorodifluoromethane | 75-71-8 | 2.14E+06 | ENVIRON 2013 | 2.5 | 0.5586 | -- | -- | 50 | 60 - 140 | 25 |

**TABLE 3. SOIL GAS ANALYTES AND ANALYTICAL PERFORMANCE CRITERIA**
**QUALITY ASSURANCE PROJECT PLAN**
**Nevada Environmental Response Trust Site; Henderson, Nevada**

| ANALYTES | CAS Number | Screening Level | Screening Level Source[1] | Practical Quantitation Limit (PQL) | Method Detection Limit (MDL) | QUALITY CONTROL LIMITS[2] Surrogate %R | | Duplicate RPD | Blank Spike/LCS %R | Blank Spike/LCS RPD |
|---|---|---|---|---|---|---|---|---|---|---|
| **Soil Gas Analytes (µg/m$^3$)** | | | | | | | | | | |
| 1,1-Dichloroethane | 75-34-3 | 3.44E+04 | ENVIRON 2013 | 2.05 | 1.8354 | -- | -- | 50 | 60 - 140 | 25 |
| 1,2-Dichloroethane | 107-06-2 | 1.65E+03 | ENVIRON 2013 | 2.05 | 0.00624 | -- | -- | 50 | 60 - 140 | 25 |
| 1,1-Dichloroethene | 75-35-4 | 3.40E+06 | ENVIRON 2013 | 2 | 0.3104 | -- | -- | 50 | 60 - 140 | 25 |
| cis-1,2-Dichloroethene | 156-59-2 | 1.19E+06 | ENVIRON 2013 | 2 | 0.5626 | -- | -- | 50 | 60 - 140 | 25 |
| trans-1,2-Dichloroethene | 156-60-5 | 1.22E+06 | ENVIRON 2013 | 2 | 0.2073 | -- | -- | 50 | 60 - 140 | 25 |
| 1,2-Dichloropropane | 78-87-5 | 5.28E+03 | ENVIRON 2013 | 2.35 | 0.0103 | -- | -- | 50 | 60 - 140 | 25 |
| cis-1,3-Dichloropropene | 10061-01-5 | 1.57E+04 | ENVIRON 2013 | 2.3 | 0.0041 | -- | -- | 50 | 60 - 140 | 25 |
| trans-1,3-Dichloropropene | 10061-02-6 | 1.57E+04 | ENVIRON 2013 | 2.3 | 0.2622 | -- | -- | 50 | 60 - 140 | 25 |
| 1,2-Dichloro-1,1,2,2-tetrafluoroethane | 76-14-2 | 5.67E+08 | ENVIRON 2013 | 3.55 | 0.10668 | -- | -- | 50 | 60 - 140 | 25 |
| Diisopropyl ether (DIPE) | 108-20-3 | 1.53E+07 | ENVIRON 2013 | 2.1 | 0.3496 | -- | -- | 50 | 60 - 140 | 25 |
| 1,4-Dioxane | 123-91-1 | 5.49E+03 | ENVIRON 2013 | 1.85 | 0.1279 | -- | -- | 50 | 60 - 140 | 25 |
| Ethanol | 64-17-5 | 8.34E+08 | ENVIRON 2013 | 96 | 10.626 | -- | -- | 50 | 60 - 140 | 25 |
| Ethyl acetate | 141-78-6 | 1.27E+07 | ENVIRON 2013 | 1.85 | 0.3645 | -- | -- | 50 | 60 - 140 | 25 |
| Ethyl tert-butyl ether (ETBE) | 637-92-3 | 2.35E+05 | ENVIRON 2013 | 2.1 | 0.7985 | -- | -- | 50 | 60 - 140 | 25 |
| Ethylbenzene | 100-41-4 | 2.18E+04 | ENVIRON 2013 | 2.2 | 0.1793 | -- | -- | 50 | 60 - 140 | 25 |
| 4-Ethyltoluene | 622-96-8 | 8.72E+06 | ENVIRON 2013 | 2.5 | 0.0802 | -- | -- | 50 | 60 - 140 | 25 |
| Heptane | 142-82-5 | 7.06E+07 | ENVIRON 2013 | 21 | 5.683 | -- | -- | 50 | 60 - 140 | 25 |
| Hexachlorobutadiene | 87-68-3 | 3.12E+03 | ENVIRON 2013 | 5.4 | 0.1456 | -- | -- | 50 | 60 - 140 | 25 |
| Hexane | 110-54-3 | 7.06E+06 | ENVIRON 2013 | 18 | 0.4914 | -- | -- | 50 | 60 - 140 | 25 |
| 2-Hexanone | 591-78-6 | 5.24E+05 | ENVIRON 2013 | 2.1 | 0.1617 | -- | -- | 50 | 60 - 140 | 25 |
| Methylene chloride | 75-09-2 | 4.37E+06 | ENVIRON 2013 | 1.75 | 0.4445 | -- | -- | 50 | 60 - 140 | 25 |
| Methyl methacrylate | 80-62-6 | 1.33E+07 | ENVIRON 2013 | 2.08 | 0.4163 | -- | -- | 50 | 60 - 140 | 25 |
| 4-Methyl-2-pentanone (MIBK) | 108-10-1 | 5.80E+07 | ENVIRON 2013 | 2.1 | 0.1255 | -- | -- | 50 | 60 - 140 | 25 |
| Methyl-t-butyl ether (MTBE) | 1634-04-4 | 1.66E+05 | ENVIRON 2013 | 1.85 | 0.506 | -- | -- | 50 | 60 - 140 | 25 |
| Naphthalene | 91-20-3 | 1.93E+03 | ENVIRON 2013 | 5.3 | 0.028 | -- | -- | 50 | 60 - 140 | 25 |

**TABLE 3. SOIL GAS ANALYTES AND ANALYTICAL PERFORMANCE CRITERIA**
**QUALITY ASSURANCE PROJECT PLAN**
**Nevada Environmental Response Trust Site; Henderson, Nevada**

| ANALYTES | CAS Number | Screening Level | Screening Level Source[1] | Practical Quantitation Limit (PQL) | Method Detection Limit (MDL) | QUALITY CONTROL LIMITS[2] Surrogate %R | | Duplicate RPD | Blank Spike/LCS %R | Blank Spike/LCS RPD |
|---|---|---|---|---|---|---|---|---|---|---|
| **Soil Gas Analytes (µg/m$^3$)** | | | | | | | | | | |
| Propene | 115-07-1 | -- | ENVIRON 2013 | 88 | 18.519 | -- | -- | 50 | 60 - 140 | 25 |
| Styrene | 100-42-5 | 2.03E+07 | ENVIRON 2013 | 2.15 | 0.2025 | -- | -- | 50 | 60 - 140 | 25 |
| tert-Amyl methyl ether (TAME | 994-05-8 | 2.35E+05 | ENVIRON 2013 | 2.1 | 1.247 | -- | -- | 50 | 60 - 140 | 25 |
| t-Butyl alcohol (TBA) | 75-65-0 | 4.92E+08 | ENVIRON 2013 | 31 | 16.771 | -- | -- | 50 | 60 - 140 | 25 |
| 1,1,1,2-Tetrachloroethane | 630-20-6 | 7.69E+03 | ENVIRON 2013 | 3.5 | 0.00704 | -- | -- | 50 | 60 - 140 | 25 |
| 1,1,2,2-Tetrachloroethane | 79-34-5 | 9.78E+02 | ENVIRON 2013 | 3.5 | 0.00765 | -- | -- | 50 | 60 - 140 | 25 |
| Tetrachloroethene | 127-18-4 | 2.17E+05 | ENVIRON 2013 | 3.45 | 0.0077 | -- | -- | 50 | 60 - 140 | 25 |
| Tetrahydrofuran | 109-99-9 | 3.64E+07 | ENVIRON 2013 | 1.5 | 0.2068 | -- | -- | 50 | 60 - 140 | 25 |
| Toluene | 108-88-3 | 8.70E+07 | ENVIRON 2013 | 1.9 | 0.2566 | -- | -- | 50 | 60 - 140 | 25 |
| 1,2,4-Trichlorobenzene | 120-82-1 | 8.39E+04 | ENVIRON 2013 | 3.75 | 0.1731 | -- | -- | 50 | 60 - 140 | 25 |
| 1,1,1-Trichloroethane | 71-55-6 | 9.45E+07 | ENVIRON 2013 | 2.75 | 0.3454 | -- | -- | 50 | 60 - 140 | 25 |
| 1,1,2-Trichloroethane | 79-00-5 | 3.30E+03 | ENVIRON 2013 | 2.75 | 0.0121 | -- | -- | 50 | 60 - 140 | 25 |
| Trichloroethene | 79-01-6 | 1.28E+04 | ENVIRON 2013 | 2.75 | 0.0412 | -- | -- | 50 | 60 - 140 | 25 |
| Trichlorofluoromethane | 75-69-4 | 1.22E+07 | ENVIRON 2013 | 2.85 | 0.3579 | -- | -- | 50 | 60 - 140 | 25 |
| 1,1,2-Trichloro trifluoroethane (Freon 113) | 76-13-1 | 5.67E+08 | ENVIRON 2013 | 3.9 | 0.1558 | -- | -- | 50 | 60 - 140 | 25 |
| 1,2,4-Trimethylbenzene | 95-63-6 | 1.61E+05 | ENVIRON 2013 | 2.5 | 0.2508 | -- | -- | 50 | 60 - 140 | 25 |
| 1,3,5-Trimethylbenzene | 108-67-8 | 1.62E+05 | ENVIRON 2013 | 2.5 | 0.3499 | -- | -- | 50 | 60 - 140 | 25 |
| Vinyl Acetate | 108-05-4 | 3.54E+06 | ENVIRON 2013 | 1.8 | 0.6113 | -- | -- | 50 | 60 - 140 | 25 |
| Vinyl Chloride | 75-01-4 | 9.60E+03 | ENVIRON 2013 | 1.3 | 0.00754 | -- | -- | 50 | 60 - 140 | 25 |
| Xylenes, Total | 1330-20-7 | 1.91E+06 | ENVIRON 2013 | 6.6 | 0.8331 | -- | -- | 50 | 60 - 140 | 25 |
| 1,2-Dichloroethane-D4 (Surr) | 17060-07-0 | -- | ENVIRON 2013 | -- | -- | 60 | 140 | -- | -- -- | -- |
| Toluene-d8 (Surr) | 2037-26-5 | -- | ENVIRON 2013 | -- | -- | 60 | 140 | -- | -- -- | -- |
| 4-Bromofluorobenzene (Surr) | 460-00-4 | -- | ENVIRON 2013 | -- | -- | 60 | 140 | -- | -- -- | -- |
| ASTM D1946 | | | | | | | | | | |
| Helium | 7440-59-7 | -- | -- | 50 | -- | -- | -- | 50 | 70 - 130 | -- |

## TABLE 3. SOIL GAS ANALYTES AND ANALYTICAL PERFORMANCE CRITERIA
## QUALITY ASSURANCE PROJECT PLAN
## Nevada Environmental Response Trust Site; Henderson, Nevada

| ANALYTES | CAS Number | Screening Level | Screening Level Source[(1)] | Practical Quantitation Limit (PQL) | Method Detection Limit (MDL) | QUALITY CONTROL LIMITS[(2)] Surrogate %R | Duplicate RPD | Blank Spike/LCS %R | Blank Spike/LCS RPD |
|---|---|---|---|---|---|---|---|---|---|
| **Soil Gas Analytes (µg/m$^3$)** | | | | | | | | | |

**Notes:**

Shaded PQLs and MDLs exceed the lowest screening criteria.

-- = no value

µg/m$^3$ = micrograms per cubic meter

Surr = Surrogate

(1) ENVIRON derived risk-based concentrations (RBCs) using the inputs to the Johnson and Ettinger model and values for exposure assumptions and toxicity criteria presented in the NDEP-approved Soil Gas Investigation and Human Health Risk Assessment Work Plan for Parcels C, D, F, G, and H (ENVIRON 2013).

(2) QC Limits = Quality Control Limits for %R (Percent Recovery) of spiked compounds in Laboratory Control Samples (LCS) and surrogate compounds and Relative Percent Difference (RPD) between LCS and LCS Duplicate (LCSD) samples. Matrix spikes (MS) are not performed on soil gas samples. Laboratory historical control limits are subject to change as a result of periodic re-evaluation. Limits in use at the time of sample analysis are available from the laboratory. Duplicate RPDs apply to sample duplicates and field duplicates.

**Sources:**

ENVIRON. 2013. Soil Gas Investigation and Human Health Risk Assessment Work Plan for Parcels C, D, F, G, and H. Nevada Environmental Response Trust, Henderson, Nevada. March 18, 2013. Approved by NDEP April 9, 2013.

**TABLE 4. LEACHING-BASED SOIL ANALYTES AND ANALYTICAL PERFORMANCE CRITERIA**
**QUALITY ASSURANCE PROJECT PLAN**
**Nevada Environmental Response Trust Site; Henderson, Nevada**

| | | | | | | QUALITY CONTROL LIMITS[2] | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | | | | | | Surrogate | | Duplicate | Matrix Spike | | Blank Spike/LCS | |
| ANALYTES | CAS Number | Screening Level | Screening Level Source[1] | Practical Quantitation Limit (PQL) | Method Detection Limit (MDL) | %R | | RPD | %R | RPD | %R | RPD |
| **Metals (mg/kg)** | | | | | | | | | | | | |
| *EPA Method 6010* | | | | | | | | | | | | |
| Aluminum | 7429-90-5 | 75 | BCL | 10 | 5 | -- | -- | 50 | 75 - 125 | 20 | 80 - 120 | 20 |
| Barium | 7440-39-3 | 82 | BCL | 1.5 | 0.75 | -- | -- | 50 | 75 - 125 | 20 | 80 - 120 | 20 |
| Beryllium | 7440-41-7 | 3 | BCL | 0.5 | 0.25 | -- | -- | 50 | 75 - 125 | 20 | 80 - 120 | 20 |
| Boron | 7440-42-8 | 21.4 | BCL | 5 | 2.5 | -- | -- | 50 | 75 - 125 | 20 | 80 - 120 | 20 |
| Cadmium | 7440-43-9 | 0.40 | BCL | 0.5 | 0.25 | -- | -- | 50 | 75 - 125 | 20 | 80 - 120 | 20 |
| Calcium | 7440-70-2 | -- | -- | 25.0 | 12.5 | -- | -- | 50 | 75 - 125 | 20 | 80 - 120 | 20 |
| Chromium (total) | 7440-47-3 | 180,000 | RSL | 1 | 0.5 | -- | -- | 50 | 75 - 125 | 20 | 80 - 120 | 20 |
| Cobalt | 7440-48-4 | 0.453 | BCL | 1 | 0.5 | -- | -- | 50 | 75 - 125 | 20 | 80 - 120 | 20 |
| Copper | 7440-50-8 | 45.8 | BCL | 2 | 1 | -- | -- | 50 | 75 - 125 | 20 | 80 - 120 | 20 |
| Iron | 7439-89-6 | 7.56 | BCL | 10 | 5 | -- | -- | 50 | 75 - 125 | 20 | 80 - 120 | 20 |
| Lead | 7439-92-1 | 14 | RSL | 2 | 1 | -- | -- | 50 | 75 - 125 | 20 | 80 - 120 | 20 |
| Magnesium | 7439-95-4 | 889 | BCL | 10 | 5 | -- | -- | 50 | 75 - 125 | 20 | 80 - 120 | 20 |
| Manganese | 7439-96-5 | 1.3 | BCL | 2 | 1 | -- | -- | 50 | 75 - 125 | 20 | 80 - 120 | 20 |
| Molybdenum | 7439-98-7 | 3.37 | BCL | 2 | 1 | -- | -- | 50 | 75 - 125 | 20 | 80 - 120 | 20 |
| Nickel | 7440-02-0 | 7.0 | BCL | 2 | 1 | -- | -- | 50 | 75 - 125 | 20 | 80 - 120 | 20 |
| Phosphorus | 7723-14-0 | 0.0015 | RSL | 5 | 3 | -- | -- | 50 | 75 - 125 | 20 | 80 - 120 | 20 |
| Potassium | 7440-09-7 | -- | -- | 5 | 3 | -- | -- | 50 | 75 - 125 | 20 | 80 - 120 | 20 |
| Silicon | 7440-21-3 | -- | -- | 10 | 5 | -- | -- | 50 | 75 - 125 | 20 | 80 - 120 | 20 |
| Silver | 7440-22-4 | 0.85 | BCL | 1.5 | 0.75 | -- | -- | 50 | 75 - 125 | 20 | 80 - 120 | 20 |
| Sodium | 7440-23-5 | -- | -- | 62.5 | 30.00 | -- | -- | 50 | 75 - 125 | 20 | 80 - 120 | 20 |
| Strontium | 7440-24-6 | 420 | RSL | 5 | 2.5 | -- | -- | 50 | 75 - 125 | 20 | 80 - 120 | 20 |
| Tin | 7440-31-5 | 3,000 | RSL | 10 | 2.5 | -- | -- | 50 | 81 - 120 | 20 | 80 - 120 | 20 |
| Titanium | 7440-32-6 | 134,000 | BCL | 2 | 1.0 | -- | -- | 50 | 81 - 120 | 20 | 80 - 120 | 20 |
| Tungsten | 7440-33-7 | 37.6 | BCL | 10 | 5 | -- | -- | 50 | 75 - 125 | 20 | 80 - 120 | 20 |
| Vanadium | 7440-62-2 | 300 | BCL | 1 | 1 | -- | -- | 50 | 75 - 125 | 20 | 80 - 120 | 20 |
| Zinc | 7440-66-6 | 620 | BCL | 5 | 2.5 | -- | -- | 50 | 75 - 125 | 20 | 80 - 120 | 20 |
| Zirconium[3] | 7440-67-7 | 4.8 | RSL | 5 | 5 | -- | -- | 50 | 75 - 125 | 20 | 80 - 120 | 20 |

**TABLE 4. LEACHING-BASED SOIL ANALYTES AND ANALYTICAL PERFORMANCE CRITERIA**
**QUALITY ASSURANCE PROJECT PLAN**
**Nevada Environmental Response Trust Site; Henderson, Nevada**

| | | | | | | QUALITY CONTROL LIMITS[2] | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | | | | | | Surrogate | | Duplicate | Matrix Spike | | Blank Spike/LCS | |
| ANALYTES | CAS Number | Screening Level | Screening Level Source[1] | Practical Quantitation Limit (PQL) | Method Detection Limit (MDL) | %R | | RPD | %R | RPD | %R | RPD |
| *EPA Method 6020* | | | | | | | | | | | | |
| Antimony | 7440-36-0 | 0.30 | BCL | 10 | 5 | -- | -- | 50 | 75 - 125 | 20 | 80 - 120 | 20 |
| Arsenic | 7440-38-2 | 0.29 | RSL | 0.5 | 0.25 | -- | -- | 50 | 80 - 120 | 20 | 80 - 120 | 20 |
| Selenium | 7782-49-2 | 0.30 | BCL | 1 | 0.5 | -- | -- | 50 | 80 - 120 | 20 | 80 - 120 | 20 |
| Thallium | 7440-28-0 | 0.40 | BCL | 10 | 5.0 | -- | -- | 50 | 80 - 120 | 20 | 80 - 120 | 20 |
| *EPA Method 6020A* | | | | | | | | | | | | |
| Niobium | 7440-03-1 | 1.17 | BCL | 2.5 | 0.38 | -- | -- | 50 | 75 - 125 | 30 | 80 - 120 | 20 |
| Palladium | 7440-05-3 | -- | -- | 0.1 | 0.01 | -- | -- | 50 | 75 - 125 | 30 | 80 - 120 | 20 |
| Sulfur | 7704-34-9 | -- | -- | 500 | 21.7 | -- | -- | 50 | 75 - 125 | 30 | 80 - 120 | 20 |
| Uranium | 7440-61-1 | 13.5 | BCL | 0.1 | 0.0199 | -- | -- | 50 | 75 - 125 | 30 | 80 - 120 | 20 |
| *EPA Method 7199* | | | | | | | | | | | | |
| Chromium (hexavalent) | 18540-29-9 | 2.0 | BCL | 0.8 | 0.15 | -- | -- | 50 | 55 - 110 | 20 | 65 - 110 | 20 |
| *EPA Method 7471A* | | | | | | | | | | | | |
| Mercury | 7439-97-6 | 0.104 | BCL | 0.02 | 0.012 | -- | -- | 50 | 70 - 130 | 20 | 80 - 120 | 20 |
| **Volatile Organic Compounds (µg/kg)** | | | | | | | | | | | | |
| *EPA Method 8260B* | | | | | | | | | | | | |
| 1,1,1,2-Tetrachloroethane | 630-20-6 | 0.22 | RSL | 2000 | 1.0 | -- | -- | 50 | 65 - 145 | 20 | 70 - 130 | 20 |
| 1,1,1-Trichloroethane | 71-55-6 | 100 | BCL | 1 | 0.5 | -- | -- | 50 | 65 - 145 | 20 | 65 - 135 | 20 |
| 1,1,2,2-Tetrachloroethane | 79-34-5 | 0.2 | BCL | 2 | 1.0 | -- | -- | 50 | 40 - 160 | 30 | 55 - 140 | 30 |
| 1,1,2-Trichloroethane | 79-00-5 | 0.9 | BCL | 1 | 0.5 | -- | -- | 50 | 65 - 140 | 30 | 65 - 135 | 20 |
| 1,1-Dichloroethane | 75-34-3 | 1000 | BCL | 1 | 0.5 | -- | -- | 50 | 65 - 135 | 25 | 70 - 130 | 20 |
| 1,1-Dichloroethene | 75-35-4 | 3 | BCL | 2 | 0.5 | -- | -- | 50 | 65 - 135 | 25 | 70 - 125 | 20 |
| 1,1-Dichloropropene | 563-58-6 | -- | -- | 1 | 0.5 | -- | -- | 50 | 65 - 135 | 20 | 70 - 130 | 20 |
| 1,2,3-Trichlorobenzene | 87-61-6 | 21 | RSL | 2 | 1 | -- | -- | 50 | 45 - 145 | 30 | 60 - 130 | 20 |
| 1,2,3-Trichloropropane | 96-18-4 | 0.00032 | RSL | 10 | 1.0 | -- | -- | 50 | 50 - 150 | 30 | 60 - 135 | 25 |
| 1,2,4-Trichlorobenzene | 120-82-1 | 300 | BCL | 5 | 1.0 | -- | -- | 50 | 50 - 140 | 30 | 70 - 135 | 20 |
| 1,2,4-Trimethylbenzene | 95-63-6 | 81 | RSL | 2 | 1 | -- | -- | 50 | 65 - 140 | 25 | 70 - 125 | 20 |
| 1,2-Dibromo-3-Chloropropane | 96-12-8 | 0.00014 | RSL | 5 | 2.0 | -- | -- | 50 | 40 - 150 | 30 | 50 - 135 | 30 |
| 1,2-Dibromoethane (EDB) | 106-93-4 | 0.0021 | RSL | 1 | 0.5 | -- | -- | 50 | 65 - 140 | 25 | 70 - 130 | 20 |

**TABLE 4. LEACHING-BASED SOIL ANALYTES AND ANALYTICAL PERFORMANCE CRITERIA**
**QUALITY ASSURANCE PROJECT PLAN**
**Nevada Environmental Response Trust Site; Henderson, Nevada**

| ANALYTES | CAS Number | Screening Level | Screening Level Source[1] | Practical Quantitation Limit (PQL) | Method Detection Limit (MDL) | QUALITY CONTROL LIMITS[2] Surrogate %R | | Duplicate RPD | Matrix Spike %R | Matrix Spike RPD | Blank Spike/LCS %R | Blank Spike/LCS RPD |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| 1,2-Dichlorobenzene | 95-50-1 | 900 | BCL | 1 | 0.5 | -- | -- | 50 | 70 - 130 | 25 | 75 - 120 | 20 |
| 1,2-Dichloroethane | 107-06-2 | 1 | BCL | 1 | 0.5 | -- | -- | 50 | 60 - 150 | 25 | 60 - 140 | 20 |
| 1,2-Dichloropropane | 78-87-5 | 1 | BCL | 1 | 0.5 | -- | -- | 50 | 65 - 130 | 20 | 70 - 130 | 20 |
| 1,3,5-Trimethylbenzene | 108-67-8 | 87 | RSL | 2 | 1.0 | -- | -- | 50 | 65 - 135 | 25 | 70 - 125 | 20 |
| 1,3-Dichlorobenzene | 541-73-1 | 50.8 | CAL | 1 | 0.5 | -- | -- | 50 | 70 - 130 | 25 | 75 - 125 | 20 |
| 1,3-Dichloropropane | 142-28-9 | 1 | BCL | 1 | 0.5 | -- | -- | 50 | 65 - 140 | 25 | 70 - 125 | 20 |
| 1,4-Dichlorobenzene | 106-46-7 | 100 | BCL | 1 | 0.5 | -- | -- | 50 | 70 - 130 | 25 | 75 - 120 | 20 |
| 2,2-Dichloropropane | 594-20-7 | -- | -- | 2 | 1.0 | -- | -- | 50 | 65 - 150 | 25 | 60 - 145 | 20 |
| 2-Butanone | 78-93-3 | 1200 | RSL | 10 | 5 | -- | -- | 50 | 25 - 170 | 40 | 40 - 145 | 35 |
| 2-Chlorotoluene | 95-49-8 | 23 | RSL | 2 | 1.0 | -- | -- | 50 | 60 - 135 | 25 | 70 - 125 | 20 |
| 4-Chlorotoluene | 106-43-4 | 24 | RSL | 2 | 1.0 | -- | -- | 50 | 65 - 135 | 25 | 75 - 125 | 20 |
| Benzene | 71-43-2 | 2 | BCL | 1 | 0.5 | -- | -- | 50 | 65 - 130 | 20 | 65 - 120 | 20 |
| Bromobenzene | 108-86-1 | 4.2 | RSL | 2 | 1.0 | -- | -- | 50 | 65 - 140 | 25 | 75 - 120 | 20 |
| Bromochloromethane | 74-97-5 | 21 | RSL | 2 | 1.0 | -- | -- | 50 | 65 - 145 | 25 | 70 - 135 | 20 |
| Bromodichloromethane | 75-27-4 | 30 | BCL | 1 | 0.5 | -- | -- | 50 | 65 - 145 | 20 | 70 - 135 | 20 |
| Bromoform | 75-25-2 | 40 | BCL | 2 | 1.0 | -- | -- | 50 | 50 - 145 | 30 | 55 - 135 | 25 |
| Bromomethane | 74-83-9 | 10 | BCL | 2 | 1.0 | -- | -- | 50 | 60 - 155 | 25 | 60 - 145 | 20 |
| Carbon tetrachloride | 56-23-5 | 3 | BCL | 2 | 0.5 | -- | -- | 50 | 60 - 145 | 25 | 65 - 140 | 20 |
| Chlorobenzene | 108-90-7 | 70 | BCL | 1 | 0.5 | -- | -- | 50 | 70 - 130 | 25 | 75 - 120 | 20 |
| Chloroethane | 75-00-3 | 5900 | RSL | 2 | 1.0 | -- | -- | 50 | 60 - 150 | 25 | 60 - 140 | 25 |
| Chloroform | 67-66-3 | 30 | BCL | 1 | 0.5 | -- | -- | 50 | 65 - 135 | 20 | 70 - 130 | 20 |
| Chloromethane | 74-87-3 | 49 | RSL | 2 | 1.0 | -- | -- | 50 | 40 - 145 | 25 | 45 - 145 | 25 |
| cis-1,2-Dichloroethene | 156-59-2 | 20 | BCL | 1 | 0.5 | -- | -- | 50 | 65 - 135 | 25 | 70 - 125 | 20 |
| cis-1,3-Dichloropropene | 10061-01-5 | -- | -- | 1 | 0.5 | -- | -- | 50 | 70 - 135 | 25 | 75 - 125 | 20 |
| Dibromochloromethane | 124-48-1 | 20.0 | BCL | 1 | 0.5 | -- | -- | 50 | 60 - 145 | 25 | 65 - 140 | 20 |
| Dibromomethane | 74-95-3 | 2.1 | RSL | 1 | 0.5 | -- | -- | 50 | 65 - 140 | 25 | 70 - 130 | 20 |
| Dichlorodifluoromethane | 75-71-8 | 300 | RSL | 2 | 1.0 | -- | -- | 50 | 30 - 160 | 35 | 35 - 160 | 30 |
| Ethyl tert-butyl ether | 637-92-3 | -- | -- | 2 | 1 | -- | -- | 50 | 60 - 145 | 30 | 60 - 140 | 20 |
| Ethylbenzene | 100-41-4 | 700 | BCL | 1 | 0.5 | -- | -- | 50 | 70 - 135 | 25 | 70 - 125 | 20 |
| Hexachlorobutadiene | 87-68-3 | 100 | BCL | 2 | 1.0 | -- | -- | 50 | 50 - 145 | 35 | 60 - 135 | 20 |

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**QUALITY ASSURANCE PROJECT PLAN**
**Nevada Environmental Response Trust Site; Henderson, Nevada**

| ANALYTES | CAS Number | Screening Level | Screening Level Source[1] | Practical Quantitation Limit (PQL) | Method Detection Limit (MDL) | QUALITY CONTROL LIMITS[2] Surrogate %R | | Duplicate RPD | Matrix Spike %R | | Matrix Spike RPD | Blank Spike/LCS %R | | Blank Spike/LCS RPD |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| Isopropyl benzene | 98-82-8 | 740 | RSL | 1 | 0.5 | -- | -- | 50 | 70 | - 145 | 25 | 75 | - 130 | 20 |
| m,p-Xylene | 179601-23-1 | 10 | BCL | 2 | 1.0 | -- | -- | 50 | 70 | - 130 | 25 | 70 | - 125 | 20 |
| Methylene chloride | 75-09-2 | 1 | BCL | 10 | 5 | -- | -- | 50 | 55 | - 145 | 25 | 55 | - 135 | 20 |
| Naphthalene | 91-20-3 | 4000 | BCL | 2 | 1.0 | -- | -- | 50 | 40 | - 150 | 40 | 55 | - 135 | 25 |
| n-Butylbenzene | 104-51-8 | 3200 | RSL | 2 | 1.0 | -- | -- | 50 | 55 | - 145 | 30 | 70 | - 130 | 20 |
| n-Propylbenzene | 103-65-1 | 1200 | RSL | 1 | 0.5 | -- | -- | 50 | 65 | - 140 | 25 | 70 | - 130 | 20 |
| o-Xylene | 95-47-6 | 9000 | BCL | 1 | 0.5 | -- | -- | 50 | 65 | - 130 | 25 | 70 | - 125 | 20 |
| p-Isopropyltoluene | 99-87-6 | 3910 | CAL | 1 | 0.5 | -- | -- | 50 | 60 | - 140 | 25 | 75 | - 125 | 20 |
| sec-Butylbenzene | 135-98-8 | 5900 | RSL | 2 | 1.0 | -- | -- | 50 | 60 | - 135 | 25 | 70 | - 125 | 20 |
| Styrene | 100-42-5 | 200 | BCL | 1 | 0.5 | -- | -- | 50 | 70 | - 140 | 25 | 75 | - 130 | 20 |
| tert-Butylbenzene | 98-06-6 | 1600 | RSL | 2 | 1.0 | -- | -- | 50 | 60 | - 140 | 25 | 70 | - 125 | 20 |
| Tetrachloroethene | 127-18-4 | 3 | BCL | 1 | 0.5 | -- | -- | 50 | 65 | - 135 | 25 | 70 | - 125 | 20 |
| Toluene | 108-88-3 | 600 | BCL | 1 | 0.5 | -- | -- | 50 | 70 | - 130 | 20 | 70 | - 125 | 20 |
| trans-1,2-Dichloroethene | 156-60-5 | 30 | BCL | 1 | 0.5 | -- | -- | 50 | 70 | - 135 | 25 | 70 | - 125 | 20 |
| trans-1,3-Dichloropropene | 10061-02-6 | -- | -- | 1 | 0.5 | -- | -- | 50 | 60 | - 145 | 25 | 70 | - 135 | 20 |
| Trichloroethene | 79-01-6 | 3.0 | BCL | 1 | 0.5 | -- | -- | 50 | 65 | - 140 | 25 | 70 | - 125 | 20 |
| Trichlorofluoromethane | 75-69-4 | 3300 | RSL | 2 | 1.0 | -- | -- | 50 | 55 | - 155 | 25 | 60 | - 145 | 25 |
| Vinyl chloride | 75-01-4 | 0.7 | BCL | 2 | 1.0 | -- | -- | 50 | 55 | - 140 | 30 | 55 | - 135 | 25 |
| 4-Bromofluorobenzene (Surr) | 460-00-4 | -- | -- | -- | -- | 79 | - 120 | -- | -- | -- | -- | -- | -- | -- |
| Dibromofluoromethane (Surr) | 1868-53-7 | -- | -- | -- | -- | 60 | - 120 | -- | -- | -- | -- | -- | -- | -- |
| Toluene-d8 (Surr) | 2037-26-5 | -- | -- | -- | -- | 79 | - 123 | -- | -- | -- | -- | -- | -- | -- |
| *EPA Method 8260B SIM* | | | | | | | | | | | | | | |
| 1,2,3-Trichloropropane | 96-18-4 | 1.8 | RSL | 0.01 | 0.004 | -- | -- | 50 | 50 | - 150 | 30 | 60 | - 135 | 25 |
| 1,4-Dioxane | 123-91-1 | 0.094 | RSL | 5 | 1.1 | -- | -- | 50 | 70 | - 130 | 30 | 70 | - 130 | 30 |
| Dibromofluoromethane (Surr) | 1868-53-7 | -- | -- | -- | -- | 80 | - 125 | -- | -- | -- | -- | -- | -- | -- |
| **Semi-volatile Organic Compounds (mg/kg)** | | | | | | | | | | | | | | |
| *EPA Method 8270C* | | | | | | | | | | | | | | |
| 1-Methylnaphthalene | 90-12-0 | 0.006 | RSL | 0.35 | 0.15 | -- | -- | 50 | 60 | - 140 | 30 | 60 | - 140 | 30 |

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| ANALYTES | CAS Number | Screening Level | Screening Level Source[1] | Practical Quantitation Limit (PQL) | Method Detection Limit (MDL) | QUALITY CONTROL LIMITS[2] Surrogate %R | | Duplicate RPD | Matrix Spike %R | Matrix Spike RPD | Blank Spike/LCS %R | Blank Spike/LCS RPD |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| 2,4,5-Trichlorophenol | 95-95-4 | 14 | BCL | 0.3 | 0.13 | -- | -- | 50 | 45 - 120 | 20 | 50 - 120 | 20 |
| 2,4,6-Trichlorophenol | 88-06-2 | 0.008 | BCL | 0.3 | 0.075 | -- | -- | 50 | 45 - 120 | 25 | 50 - 120 | 20 |
| 2,4-Dichlorophenol | 120-83-2 | 0.05 | BCL | 0.33 | 0.067 | -- | -- | 50 | 45 - 120 | 25 | 45 - 120 | 20 |
| 2,4-Dimethylphenol | 105-67-9 | 0.4 | BCL | 0.33 | 0.13 | -- | -- | 50 | 30 - 120 | 25 | 40 - 120 | 20 |
| 2,4-Dinitrophenol | 51-28-5 | 0.01 | BCL | 0.66 | 0.33 | -- | -- | 50 | 20 - 120 | 25 | 25 - 120 | 25 |
| 2,4-Dinitrotoluene | 121-14-2 | 0.00004 | BCL | 0.33 | 0.08 | -- | -- | 50 | 50 - 125 | 25 | 55 - 125 | 20 |
| 2,6-Dinitrotoluene | 606-20-2 | 0.00003 | BCL | 0.33 | 0.095 | -- | -- | 50 | 50 - 125 | 20 | 55 - 125 | 20 |
| 2-Chloronaphthalene | 91-58-7 | 3.9 | RSL | 0.33 | 0.067 | -- | -- | 50 | 45 - 120 | 20 | 45 - 120 | 20 |
| 2-Chlorophenol | 95-57-8 | 0.2 | BCL | 0.33 | 0.07 | -- | -- | 50 | 40 - 120 | 20 | 40 - 120 | 20 |
| 2-Methylnaphthalene | 91-57-6 | 0.19 | RSL | 0.33 | 0.07 | -- | -- | 50 | 40 - 120 | 20 | 45 - 120 | 20 |
| 2-Methylphenol | 95-48-7 | 0.8 | BCL | 0.33 | 0.08 | -- | -- | 50 | 40 - 120 | 25 | 40 - 120 | 20 |
| 2-Nitroaniline | 88-74-4 | 0.08 | RSL | 0.33 | 0.067 | -- | -- | 50 | 45 - 120 | 25 | 50 - 125 | 20 |
| 2-Nitrophenol | 88-75-5 | -- | -- | 0.33 | 0.133 | -- | -- | 50 | 40 - 120 | 25 | 45 - 120 | 20 |
| 3,3'-Dichlorobenzidine | 91-94-1 | 0.0003 | BCL | 0.83 | 0.15 | -- | -- | 50 | 20 - 130 | 25 | 20 - 130 | 25 |
| 3-Methylphenol + 4-Methylphenol | 106-44-5 | -- | -- | 0.33 | 0.133 | -- | -- | 50 | 50 - 120 | 25 | 50 - 120 | 20 |
| 3-Nitroaniline | 99-09-2 | -- | -- | 0.33 | 0.133 | -- | -- | 50 | 30 - 120 | 25 | 35 - 120 | 25 |
| 4-Bromophenyl phenyl ether | 101-55-3 | -- | -- | 0.33 | 0.075 | -- | -- | 50 | 45 - 120 | 20 | 45 - 120 | 20 |
| 4-Chloro-3-methylphenol | 59-50-7 | 1.7 | RSL | 0.33 | 0.07 | -- | -- | 50 | 50 - 125 | 25 | 50 - 125 | 20 |
| 4-Chloroaniline | 106-47-8 | 0.03 | BCL | 0.33 | 0.133 | -- | -- | 50 | 20 - 120 | 30 | 20 - 120 | 30 |
| 4-Chlorophenyl phenyl ether | 7005-72-3 | -- | -- | 0.33 | 0.085 | -- | -- | 50 | 50 - 120 | 25 | 55 - 120 | 20 |
| 4-Nitroaniline | 100-01-6 | 0.0016 | RSL | 0.83 | 0.133 | -- | -- | 50 | 40 - 125 | 30 | 45 - 125 | 20 |
| 4-Nitrophenol | 100-02-7 | -- | -- | 0.83 | 0.14 | -- | -- | 50 | 35 - 125 | 30 | 40 - 125 | 20 |
| Acenaphthene | 83-32-9 | 29 | BCL | 0.33 | 0.067 | -- | -- | 50 | 45 - 120 | 25 | 50 - 120 | 20 |
| Acenaphthylene | 208-96-8 | 0.01 | CAL | 0.33 | 0.07 | -- | -- | 50 | 45 - 120 | 20 | 50 - 120 | 20 |
| Aniline | 62-53-3 | 0.001 | RSL | 0.42 | 0.085 | -- | -- | 50 | 25 - 120 | 30 | 25 - 120 | 20 |
| Anthracene | 120-12-7 | 590 | BCL | 0.33 | 0.08 | -- | -- | 50 | 55 - 120 | 25 | 55 - 120 | 20 |
| Benzidine | 92-87-5 | -- | -- | 0.66 | 0.66 | -- | -- | 50 | 20 - 120 | 30 | 20 - 120 | 30 |
| Benzo(a)anthracene | 56-55-3 | 0.08 | BCL | 0.33 | 0.07 | -- | -- | 50 | 50 - 120 | 25 | 55 - 120 | 20 |
| Benzo(a)pyrene | 50-32-8 | 0.4 | BCL | 0.33 | 0.067 | -- | -- | 50 | 45 - 125 | 25 | 50 - 125 | 20 |

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| ANALYTES | CAS Number | Screening Level | Screening Level Source[1] | Practical Quantitation Limit (PQL) | Method Detection Limit (MDL) | QUALITY CONTROL LIMITS[2] Surrogate %R | | Duplicate RPD | Matrix Spike %R | Matrix Spike RPD | Blank Spike/LCS %R | Blank Spike/LCS RPD |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| Benzo(b)fluoranthene | 205-99-2 | 0.2 | BCL | 0.33 | 0.067 | -- | -- | 50 | 45 - 125 | 30 | 45 - 125 | 25 |
| Benzo(g,h,i)perylene | 191-24-2 | -- | -- | 0.33 | 0.11 | -- | -- | 50 | 25 - 130 | 30 | 35 - 130 | 25 |
| Benzo(k)fluoranthene | 207-08-9 | 2 | BCL | 0.33 | 0.07 | -- | -- | 50 | 45 - 125 | 30 | 45 - 125 | 25 |
| Benzoic acid | 65-85-0 | 20 | BCL | 0.83 | 0.15 | -- | -- | 50 | 20 - 120 | 30 | 20 - 120 | 30 |
| Benzyl alcohol | 100-51-6 | 0.48 | RSL | 0.33 | 0.2 | -- | -- | 50 | 20 - 120 | 30 | 35 - 120 | 25 |
| Bis(2-chloroethoxy)methane | 111-91-1 | 0.013 | RSL | 0.33 | 0.133 | -- | -- | 50 | 45 - 120 | 25 | 45 - 120 | 20 |
| Bis(2-chloroethyl)ether | 111-44-4 | 0.00002 | BCL | 0.33 | 0.06 | -- | -- | 50 | 35 - 110 | 25 | 35 - 120 | 25 |
| Bis(2-ethylhexyl) phthalate | 117-81-7 | 180 | BCL | 0.33 | 0.09 | -- | -- | 50 | 45 - 130 | 25 | 50 - 130 | 20 |
| Butyl benzyl phthalate | 85-68-7 | 810 | BCL | 0.33 | 0.08 | -- | -- | 50 | 45 - 125 | 25 | 50 - 125 | 20 |
| Chrysene | 218-01-9 | 8 | BCL | 0.33 | 0.075 | -- | -- | 50 | 55 - 120 | 25 | 55 - 120 | 20 |
| Dibenz(a,h)anthracene | 53-70-3 | 0.08 | BCL | 0.42 | 0.1 | -- | -- | 50 | 25 - 135 | 30 | 40 - 135 | 25 |
| Dibenzofuran | 132-64-9 | 0.15 | RSL | 0.33 | 0.067 | -- | -- | 50 | 50 - 120 | 25 | 55 - 120 | 20 |
| Diethyl phthalate | 84-66-2 | 6.1 | RSL | 0.33 | 0.095 | -- | -- | 50 | 50 - 125 | 25 | 50 - 125 | 20 |
| Dimethylphthalate | 131-11-3 | -- | -- | 0.33 | 0.067 | -- | -- | 50 | 45 - 125 | 25 | 50 - 125 | 20 |
| Di-n-butyl phthalate | 84-74-2 | 270 | BCL | 0.33 | 0.09 | -- | -- | 50 | 50 - 125 | 25 | 50 - 125 | 20 |
| Di-n-octyl phthalate | 117-84-0 | 57 | RSL | 0.33 | 0.09 | -- | -- | 50 | 50 - 135 | 25 | 50 - 135 | 20 |
| Fluoranthene | 206-44-0 | 210 | BCL | 0.33 | 0.07 | -- | -- | 50 | 45 - 120 | 25 | 55 - 120 | 20 |
| Fluorene | 86-73-7 | 28 | BCL | 0.33 | 0.07 | -- | -- | 50 | 50 - 120 | 25 | 55 - 120 | 20 |
| Hexachlorobenzene | 118-74-1 | 0.1 | BCL | 0.33 | 0.07 | -- | -- | 50 | 50 - 120 | 25 | 50 - 120 | 20 |
| Hexachlorocyclopentadiene | 77-47-4 | 20 | BCL | 0.83 | 0.133 | -- | -- | 50 | 20 - 125 | 30 | 30 - 125 | 25 |
| Hexachloroethane | 67-72-1 | 0.02 | BCL | 0.33 | 0.133 | -- | -- | 50 | 35 - 120 | 30 | 40 - 120 | 20 |
| Indeno[1,2,3-cd]pyrene | 193-39-5 | 0.7 | BCL | 0.33 | 0.13 | -- | -- | 50 | 20 - 130 | 30 | 30 - 135 | 25 |
| Isophorone | 78-59-1 | 0.03 | BCL | 0.33 | 0.067 | -- | -- | 50 | 40 - 120 | 25 | 40 - 120 | 20 |
| Naphthalene | 91-20-3 | 4 | BCL | 0.33 | 0.067 | -- | -- | 50 | 40 - 120 | 25 | 45 - 120 | 20 |
| Nitrobenzene | 98-95-3 | 0.007 | BCL | 0.33 | 0.07 | -- | -- | 50 | 40 - 120 | 25 | 45 - 120 | 20 |
| N-Nitrosodi-n-propylamine | 621-64-7 | 0.000002 | BCL | 0.25 | 0.07 | -- | -- | 50 | 35 - 120 | 25 | 40 - 120 | 20 |
| N-Nitrosodiphenylamine | 86-30-6 | 0.06 | BCL | 0.33 | 0.08 | -- | -- | 50 | 45 - 125 | 25 | 50 - 120 | 20 |
| Octachlorostyrene | 29082-74-4 | -- | -- | 3.30 | 2.3 | -- | -- | 50 | 60 - 140 | 30 | 60 - 140 | 30 |
| Pentachlorophenol | 87-86-5 | 0.001 | BCL | 0.8 | 0.15 | -- | -- | 50 | 30 - 120 | 25 | 40 - 120 | 20 |
| Phenanthrene | 85-01-8 | 0.02 | CAL | 0.3 | 0.067 | -- | -- | 50 | 50 - 120 | 25 | 50 - 120 | 20 |

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**QUALITY ASSURANCE PROJECT PLAN**
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| ANALYTES | CAS Number | Screening Level | Screening Level Source[1] | Practical Quantitation Limit (PQL) | Method Detection Limit (MDL) | QUALITY CONTROL LIMITS[2] Surrogate %R | Duplicate RPD | Matrix Spike %R | Matrix Spike RPD | Blank Spike/LCS %R | Blank Spike/LCS RPD |
|---|---|---|---|---|---|---|---|---|---|---|---|
| Phenol | 108-95-2 | 5 | BCL | 0.3 | 0.09 | -- -- | 50 | 40 - 120 | 25 | 40 - 120 | 20 |
| Pyrene | 129-00-0 | 210 | BCL | 0.3 | 0.08 | -- -- | 50 | 40 - 125 | 30 | 45 - 125 | 25 |
| 2-Fluorophenol (Surr) | 367-12-4 | -- | -- | -- | -- | 35 - 120 | -- | -- -- | -- | -- -- | -- |
| 2,4,6-Tribromophenol (Surr) | 118-79-6 | -- | -- | -- | -- | 35 - 120 | -- | -- -- | -- | -- -- | -- |
| Nitrobenzene-d5 (Surr) | 4165-60-0 | -- | -- | -- | -- | 35 - 120 | -- | -- -- | -- | -- -- | -- |
| Terphenyl-d14 (Surr) | 1718-51-0 | -- | -- | -- | -- | 35 - 120 | -- | -- -- | -- | -- -- | -- |
| Phenol-d6 (Surr) | 13127-88-3 | -- | -- | -- | -- | 35 - 120 | -- | -- -- | -- | -- -- | -- |
| *EPA Method 8315A* | | | | | | | | | | | |
| Formaldehyde | 50-00-0 | 0.000087 | RSL | 1 | 0.6 | -- -- | 50 | 50 - 150 | 20 | 50 - 150 | 20 |
| **Polycyclic Aromatic Hydrocarbons (mg/kg)** | | | | | | | | | | | |
| *EPA Method 8270 SIM* | | | | | | | | | | | |
| Acenaphthene | 83-32-9 | 29 | BCL | 0.03 | 0.004 | -- -- | 50 | 45 - 120 | 25 | 50 - 120 | 20 |
| Acenaphthylene | 208-96-8 | 0.0106 | CAL | 0.03 | 0.004 | -- -- | 50 | 45 - 120 | 20 | 50 - 120 | 20 |
| Anthracene | 120-12-7 | 590 | BCL | 0.03 | 0.004 | -- -- | 50 | 55 - 120 | 25 | 55 - 120 | 20 |
| Benzo(a)anthracene | 56-55-3 | 0.08 | BCL | 0.03 | 0.004 | -- -- | 50 | 50 - 120 | 25 | 55 - 120 | 20 |
| Benzo(a)pyrene | 50-32-8 | 0.40 | BCL | 0.03 | 0.004 | -- -- | 50 | 45 - 125 | 25 | 50 - 125 | 20 |
| Benzo(b)fluoranthene | 205-99-2 | 0.20 | BCL | 0.03 | 0.004 | -- -- | 50 | 45 - 125 | 30 | 45 - 125 | 25 |
| Benzo(g,h,i)perylene | 191-24-2 | -- | -- | 0.03 | 0.004 | -- -- | 50 | 25 - 130 | 30 | 35 - 130 | 25 |
| Benzo(k)fluoranthene | 207-08-9 | 2 | BCL | 0.03 | 0.004 | -- -- | 50 | 45 - 125 | 30 | 45 - 125 | 25 |
| Chrysene | 218-01-9 | 8 | BCL | 0.03 | 0.004 | -- -- | 50 | 55 - 120 | 25 | 55 - 120 | 20 |
| Dibenz(a,h)anthracene | 53-70-3 | 0.08 | BCL | 0.03 | 0.004 | -- -- | 50 | 25 - 135 | 30 | 40 - 135 | 25 |
| Fluoranthene | 206-44-0 | 210 | BCL | 0.03 | 0.004 | -- -- | 50 | 45 - 120 | 25 | 55 - 120 | 20 |
| Fluorene | 86-73-7 | 28 | BCL | 0.03 | 0.004 | -- -- | 50 | 50 - 120 | 25 | 55 - 120 | 20 |
| Indeno(1,2,3-cd)pyrene | 193-39-5 | 0.7 | BCL | 0.03 | 0.004 | -- -- | 50 | 20 - 130 | 30 | 30 - 135 | 25 |
| Naphthalene | 91-20-3 | 4 | BCL | 0.03 | 0.004 | -- -- | 50 | 40 - 120 | 25 | 45 - 120 | 20 |
| Phenanthrene | 85-01-8 | 0.024 | CAL | 0.03 | 0.004 | -- -- | 50 | 50 - 120 | 25 | 50 - 120 | 20 |
| Pyrene | 129-00-0 | 210 | BCL | 0.03 | 0.004 | -- -- | 50 | 40 - 125 | 30 | 45 - 125 | 25 |
| 2-Fluorobiphenyl (Surr) | 321-60-8 | -- | -- | -- | -- | 35 - 120 | -- | -- -- | -- | -- -- | -- |
| Nitrobenzene-d5 (Surr) | 4165-60-0 | -- | -- | -- | -- | 35 - 120 | -- | -- -- | -- | -- -- | -- |

**TABLE 4. LEACHING-BASED SOIL ANALYTES AND ANALYTICAL PERFORMANCE CRITERIA**
**QUALITY ASSURANCE PROJECT PLAN**
**Nevada Environmental Response Trust Site; Henderson, Nevada**

| ANALYTES | CAS Number | Screening Level | Screening Level Source[1] | Practical Quantitation Limit (PQL) | Method Detection Limit (MDL) | QUALITY CONTROL LIMITS[2] Surrogate %R | | Duplicate RPD | Matrix Spike %R | | Matrix Spike RPD | Blank Spike/LCS %R | | Blank Spike/LCS RPD |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| Terphenyl-d14 (Surr) | 1718-51-0 | -- | -- | -- | -- | 35 | 120 | -- | -- | -- | -- | -- | -- | -- |
| **Organophosphorus Pesticides (mg/kg)** | | | | | | | | | | | | | | |
| *EPA Method 8141A* | | | | | | | | | | | | | | |
| Atrazine | 1912-24-9 | -- | -- | 0.067 | 0.0121 | -- | -- | 50 | 49 | 115 | 50 | 49 | 115 | 50 |
| Chlorpyrifos | 2921-88-2 | 0.12 | RSL | 0.020 | 0.00646 | -- | -- | 50 | 38 | 130 | 37 | 38 | 130 | 37 |
| Coumaphos | 56-72-4 | -- | -- | 0.013 | 0.0028 | -- | -- | 50 | 50 | 119 | 27 | 50 | 119 | 27 |
| Demeton, Total | 8065-48-3 | -- | -- | 0.039 | 0.00752 | -- | -- | 50 | 36 | 115 | 47 | 36 | 115 | 47 |
| Diazinon | 333-41-5 | 0.065 | RSL | 0.022 | 0.00727 | -- | -- | 50 | 53 | 115 | 40 | 53 | 115 | 40 |
| Dichlorvos | 62-73-7 | 0.000081 | RSL | 0.023 | 0.0074 | -- | -- | 50 | 43 | 139 | 77 | 43 | 139 | 77 |
| Dimethoate | 60-51-5 | 0.00099 | RSL | 0.022 | 0.00708 | -- | -- | 50 | 25 | 138 | 98 | 25 | 138 | 98 |
| Disulfoton | 298-04-4 | 0.00094 | RSL | 0.048 | 0.00773 | -- | -- | 50 | 29 | 115 | 40 | 29 | 115 | 40 |
| EPN | 2104-64-5 | 0.0028 | RSL | 0.013 | 0.00368 | -- | -- | 50 | 58 | 131 | 50 | 58 | 131 | 50 |
| Ethoprop | 13194-48-4 | -- | -- | 0.015 | 0.00493 | -- | -- | 50 | 53 | 115 | 54 | 53 | 115 | 54 |
| Ethyl Parathion | 56-38-2 | 0.43 | RSL | 0.018 | 0.00529 | -- | -- | 50 | 24 | 163 | 47 | 24 | 163 | 47 |
| Famphur | 52-85-7 | -- | -- | 0.013 | 0.00322 | -- | -- | 50 | 49 | 140 | 31 | 49 | 140 | 31 |
| Fensulfothion | 115-90-2 | -- | -- | 0.025 | 0.00815 | -- | -- | 50 | 52 | 121 | 49 | 52 | 121 | 49 |
| Fenthion | 55-38-9 | -- | -- | 0.033 | 0.00874 | -- | -- | 50 | 45 | 115 | 43 | 45 | 115 | 43 |
| Malathion | 121-75-5 | 0.1 | RSL | 0.015 | 0.00464 | -- | -- | 50 | 50 | 122 | 53 | 50 | 122 | 53 |
| Merphos | 150-50-5 | 0.059 | RSL | 0.030 | 0.00514 | -- | -- | 50 | 19 | 115 | 50 | 19 | 115 | 50 |
| Methyl parathion | 298-00-0 | 0.0074 | RSL | 0.020 | 0.00637 | -- | -- | 50 | 46 | 119 | 53 | 46 | 119 | 53 |
| Mevinphos | 7786-34-7 | -- | -- | 0.015 | 0.00462 | -- | -- | 50 | 10 | 226 | 78 | 10 | 226 | 78 |
| Phorate | 298-02-2 | 0.0034 | RSL | 0.020 | 0.0057 | -- | -- | 50 | 40 | 115 | 40 | 40 | 115 | 40 |
| Ronnel | 299-84-3 | 3.7 | RSL | 0.046 | 0.0152 | -- | -- | 50 | 43 | 118 | 41 | 43 | 118 | 41 |
| Simazine | 122-34-9 | -- | -- | 0.067 | 0.0221 | -- | -- | 50 | 11 | 179 | 58 | 11 | 179 | 58 |
| Stirophos | 22248-79-9 | -- | -- | 0.015 | 0.00436 | -- | -- | 50 | 44 | 118 | 24 | 44 | 118 | 24 |
| Sulfotepp | 3689-24-5 | 0.0052 | RSL | 0.020 | 0.00626 | -- | -- | 50 | 55 | 115 | 40 | 55 | 115 | |
| Thionazin | 297-97-2 | -- | -- | 0.018 | 0.00557 | -- | -- | 50 | 46 | 115 | 40 | 46 | 115 | 40 |
| Trichloronate | 327-98-0 | -- | -- | 0.020 | 0.00625 | -- | -- | 50 | 27 | 115 | 43 | 27 | 115 | 43 |
| Chlormefos (Surr) | 24934-91-6 | -- | -- | -- | -- | 42 | 132 | -- | -- | -- | -- | -- | -- | -- |
| Triphenylphosphate (Surr) | 115-86-6 | -- | -- | -- | -- | 47 | 161 | -- | -- | -- | -- | -- | -- | -- |

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**QUALITY ASSURANCE PROJECT PLAN**
**Nevada Environmental Response Trust Site; Henderson, Nevada**

| ANALYTES | CAS Number | Screening Level | Screening Level Source[1] | Practical Quantitation Limit (PQL) | Method Detection Limit (MDL) | QUALITY CONTROL LIMITS[2] Surrogate %R | | Duplicate RPD | Matrix Spike %R | Matrix Spike RPD | Blank Spike/LCS %R | Blank Spike/LCS RPD |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| **Organochlorine Pesticides (mg/kg)** | | | | | | | | | | | | |
| *EPA Method 8081A* | | | | | | | | | | | | |
| 2,4'-DDE | 3424-82-6 | -- | -- | 0.005 | 0.0015 | -- | -- | 50 | 35 - 130 | 30 | 60 - 120 | 30 |
| 4,4'-DDD | 72-54-8 | 0.8 | BCL | 0.005 | 0.0015 | -- | -- | 50 | 40 - 130 | 30 | 60 - 120 | 30 |
| 4,4'-DDE | 72-55-9 | 3 | BCL | 0.005 | 0.0015 | -- | -- | 50 | 35 - 130 | 30 | 60 - 120 | 30 |
| 4,4'-DDT | 50-29-3 | 2 | BCL | 0.005 | 0.0015 | -- | -- | 50 | 35 - 130 | 30 | 65 - 120 | 30 |
| Aldrin | 309-00-2 | 0.02 | BCL | 0.005 | 0.0015 | -- | -- | 50 | 40 - 115 | 30 | 50 - 115 | 30 |
| alpha-BHC | 319-84-6 | 0.0266 | BCL | 0.005 | 0.0015 | -- | -- | 50 | 40 - 115 | 30 | 60 - 115 | 30 |
| alpha-Chlordane | 57-74-9 | 0.50 | BCL | 0.050 | 0.01 | -- | -- | 50 | 60 - 140 | 30 | 60 - 140 | 30 |
| beta-BHC | 319-85-7 | 0.00545 | BCL | 0.005 | 0.0015 | -- | -- | 50 | 40 - 120 | 30 | 60 - 115 | 30 |
| delta-BHC | 319-86-8 | 28.1 | BCL | 0.010 | 0.0015 | -- | -- | 50 | 45 - 120 | 30 | 60 - 115 | 30 |
| Dieldrin | 60-57-1 | 0.00020 | BCL | 0.005 | 0.0015 | -- | -- | 50 | 40 - 125 | 30 | 65 - 115 | 30 |
| Endosulfan I | 959-98-8 | -- | -- | 0.005 | 0.0015 | -- | -- | 50 | 40 - 120 | 30 | 40 - 120 | 30 |
| Endosulfan II | 33213-65-9 | -- | -- | 0.005 | 0.0015 | -- | -- | 50 | 40 - 125 | 30 | 55 - 120 | 30 |
| Endosulfan sulfate | 1031-07-8 | -- | -- | 0.01 | 0.002 | -- | -- | 50 | 45 - 120 | 30 | 65 - 115 | 30 |
| Endrin | 72-20-8 | 0.05 | BCL | 0.005 | 0.0015 | -- | -- | 50 | 45 - 125 | 30 | 55 - 120 | 30 |
| Endrin aldehyde | 7421-93-4 | -- | -- | 0.005 | 0.0015 | -- | -- | 50 | 30 - 120 | 30 | 55 - 115 | 30 |
| Endrin ketone | 53494-70-5 | -- | -- | 0.005 | 0.002 | -- | -- | 50 | 40 - 120 | 30 | 65 - 115 | 30 |
| gamma-BHC (Lindane) | 58-89-9 | 0.0005 | BCL | 0.005 | 0.0015 | -- | -- | 50 | 40 - 120 | 30 | 55 - 115 | 30 |
| gamma-Chlordane | 57-74-9 | 0.50 | BCL | 0.050 | 0.01 | -- | -- | 50 | 60 - 140 | 30 | 60 - 140 | 30 |
| Heptachlor | 76-44-8 | 1.00 | BCL | 0.005 | 0.002 | -- | -- | 50 | 40 - 115 | 30 | 55 - 115 | 30 |
| Heptachlor epoxide | 1024-57-3 | 0.03 | BCL | 0.005 | 0.002 | -- | -- | 50 | 45 - 115 | 30 | 55 - 115 | 30 |
| Methoxychlor | 72-43-5 | 8.00 | BCL | 0.005 | 0.0015 | -- | -- | 50 | 40 - 135 | 30 | 65 - 120 | 30 |
| Toxaphene | 8001-35-2 | 2.00 | BCL | 0.200 | 0.05 | -- | -- | 50 | 60 - 140 | 30 | 60 - 140 | 30 |
| Decachlorobiphenyl (Surr) | 2051-24-3 | -- | -- | -- | -- | 45 - 120 | | -- | -- | -- | -- | -- |

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| ANALYTES | CAS Number | Screening Level | Screening Level Source[1] | Practical Quantitation Limit (PQL) | Method Detection Limit (MDL) | QUALITY CONTROL LIMITS[2] Surrogate %R | | Duplicate RPD | Matrix Spike %R | Matrix Spike RPD | Blank Spike/LCS %R | Blank Spike/LCS RPD |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| **Dioxin/Furans (µg/kg)** [5] | | | | | | | | | | | | |
| *EPA Method 8290 or 8280(7)* | | | | | | | | | | | | |
| 2,3,7,8-Tetrachloro dibenzo-p-dioxin | 1746-01-6 | 0.000059 | RSL | 0.001 | EDL[4] | -- | -- | 50 | 60 - 138 | 20 | 60 - 138 | 20 |
| 1,2,3,7,8-PeCDD | 40321-76-4 | -- | -- | 0.005 | EDL[4] | -- | -- | 50 | 70 - 122 | 20 | 70 - 122 | 20 |
| 1,2,3,4,7,8-HxCDD[6] | 39227-28-6 | 0.017 | RSL | 0.005 | EDL[4] | -- | -- | 50 | 60 - 138 | 20 | 60 - 138 | 20 |
| 1,2,3,4,7,8-HxCDD[6] | 57653-85-7 | 0.017 | RSL | 0.005 | EDL[4] | -- | -- | 50 | 68 - 136 | 20 | 68 - 136 | 20 |
| 1,2,3,4,7,8-HxCDD[6] | 19408-74-3 | 0.017 | RSL | 0.005 | EDL[4] | -- | -- | 50 | 68 - 138 | 20 | 68 - 138 | 20 |
| 1,2,3,4,6,7,8-HpCDD | 35822-46-9 | -- | -- | 0.005 | EDL[4] | -- | -- | 50 | 71 - 128 | 20 | 71 - 128 | 20 |
| OCDD | 3268-87-9 | -- | -- | 0.01 | EDL[4] | -- | -- | 50 | 70 - 128 | 20 | 70 - 128 | 20 |
| 2,3,7,8-TCDF | 51207-31-9 | -- | -- | 0.001 | EDL[4] | -- | -- | 50 | 56 - 158 | 20 | 56 - 158 | 20 |
| 1,2,3,7,8-PeCDF | 57117-41-6 | -- | -- | 0.005 | EDL[4] | -- | -- | 50 | 69 - 134 | 20 | 69 - 134 | 20 |
| 2,3,4,7,8-PeCDF | 57117-31-4 | -- | -- | 0.005 | EDL[4] | -- | -- | 50 | 70 - 131 | 20 | 70 - 131 | 20 |
| 1,2,3,4,7,8-HxCDF | 70648-26-9 | -- | -- | 0.005 | EDL[4] | -- | -- | 50 | 74 - 128 | 20 | 74 - 128 | 20 |
| 1,2,3,6,7,8-HxCDF | 57117-44-9 | -- | -- | 0.005 | EDL[4] | -- | -- | 50 | 67 - 140 | 20 | 67 - 140 | 20 |
| 1,2,3,7,8,9-HxCDF | 72918-21-9 | -- | -- | 0.005 | EDL[4] | -- | -- | 50 | 72 - 134 | 20 | 72 - 134 | 20 |
| 2,3,4,6,7,8-HxCDF | 60851-34-5 | -- | -- | 0.005 | EDL[4] | -- | -- | 50 | 71 - 137 | 20 | 71 - 137 | 20 |
| 1,2,3,4,6,7,8-HpCDF | 67562-39-4 | -- | -- | 0.005 | EDL[4] | -- | -- | 50 | 71 - 134 | 20 | 71 - 134 | 20 |
| 1,2,3,4,7,8,9-HpCDF | 55673-89-7 | -- | -- | 0.005 | EDL[4] | -- | -- | 50 | 68 - 129 | 20 | 68 - 129 | 20 |
| OCDF | 39001-02-0 | -- | -- | 0.01 | EDL[4] | -- | -- | 50 | 63 - 141 | 20 | 63 - 141 | 20 |
| **PCBs as Congeners (µg/kg)** [5] | | | | | | | | | | | | |
| *EPA Method 1668A* | | | | | | | | | | | | |
| Total PCBs | 1336-36-3 | 78 | RSL | 0.2 | EDL[4] | -- | -- | 50 | -- -- | -- | -- -- | -- |
| 3,4,4',5-TeCB (PCB-81) | 70362-50-4 | 0.062 | RSL | 0.002 | EDL[4] | -- | -- | 50 | 50 - 150 | 50 | 50 - 150 | 50 |
| 2,3',4,4',5-PeCB (PCB-118) | 31508-00-6 | 1.0 | RSL | 0.002 | EDL[4] | -- | -- | 50 | 50 - 150 | 50 | 50 - 150 | 50 |
| 3,3',4,4',5-PeCB (PCB-126) | 57465-28-8 | 0.0003 | RSL | 0.002 | EDL[4] | -- | -- | 50 | 50 - 150 | 50 | 50 - 150 | 50 |
| 3,3',4,4',5,5'-HxCB (PCB-169) | 32774-16-6 | 0.0017 | RSL | 0.002 | EDL[4] | -- | -- | 50 | 50 - 150 | 50 | 50 - 150 | 50 |
| DeCB (PCB-209) | 2051-24-3 | 78 | RSL | 0.02 | EDL[4] | -- | -- | 50 | 50 - 150 | 50 | 50 - 150 | 50 |

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|---|---|---|---|---|---|---|---|---|---|---|---|
| **PCBs as Aroclors (µg/kg)** | | | | | | | | | | | |
| *EPA Method 8082* | | | | | | | | | | | |
| Aroclor-1260 | 11096-82-5 | 5.5 | RSL | 50 | 17 | -- -- | 50 | 50 - 125 | 30 | 65 - 115 | 30 |
| DCB Decachlorobiphenyl (Surr) | 2051-24-3 | -- | -- | -- | -- | 45 - 120 | -- | -- -- | -- | -- -- | -- |
| **Organic Acids (mg/kg)** | | | | | | | | | | | |
| *EPA Method 8270C* | | | | | | | | | | | |
| Phthalic acid[7] | 88-99-3 | -- | -- | 2.5 | 0.76 | -- -- | 50 | -- -- | -- | -- -- | -- |
| 2-fluorobiphenyl (Surr) | 321-60-8 | -- | -- | -- | -- | 29 - 120 | -- | -- -- | -- | -- -- | -- |
| **Total Petroleum Hydrocarbons (mg/kg)** | | | | | | | | | | | |
| *EPA Method 8015B* | | | | | | | | | | | |
| Total petroleum hydrocarbon-gasoline | TPH-gasoline | -- | -- | 0.4 | 0.15 | -- -- | 50 | 60 - 140 | 30 | 70 - 135 | 20 |
| 4-Bromofluorobenzene (Surr) | 460-00-4 | -- | -- | -- | -- | 65 - 140 | -- | -- -- | -- | -- -- | -- |
| Diesel Range Organics (C10-C28) | TPH-diesel | -- | -- | 5 | 2.5 | -- -- | 50 | 40 - 120 | 30 | 45 - 115 | 25 |
| Oil Range Organics (C29-C40) | TPH-oil | -- | -- | 5 | 2.5 | -- -- | 50 | 40 - 120 | 30 | 45 - 115 | 25 |
| n-Octacosane (Surr) | 630-02-4 | -- | -- | -- | -- | 40 140 | -- | -- -- | -- | -- -- | -- |
| **Others (mg/kg)** | | | | | | | | | | | |
| *SM 2320B* | | | | | | | | | | | |
| Alkalinity as $CaCO_3$ | -- | -- | -- | 500 | -- | -- -- | 50 | -- -- | -- | 90 110 | 20 |
| Bicarbonate as $HCO_3^-$ | -- | -- | -- | 610 | -- | -- -- | 50 | -- -- | -- | -- -- | -- |
| Carbonate as $CO_3^-$ | -- | -- | -- | 300 | -- | -- -- | 50 | -- -- | -- | -- -- | -- |
| Hydroxide as $OH^-$ | 14280-30-9 | -- | -- | 170 | -- | -- -- | 50 | -- -- | -- | -- -- | -- |
| *SM 4500-NH3 D* | | | | | | | | | | | |
| Ammonia as $NH_3$ | 7664-41-7 | -- | -- | 12 | 2.4 | -- -- | 50 | 75 - 125 | 15 | 85 - 115 | 15 |
| *EPA Method 300.0* | | | | | | | | | | | |
| Bromide | 24959-67-9 | -- | -- | 5 | 3.5 | -- -- | 50 | 80 - 120 | 20 | 90 - 110 | 20 |
| Chloride | 16887-00-6 | -- | -- | 5 | 4 | -- -- | 50 | 80 - 120 | 20 | 90 - 110 | 20 |

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|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| Fluoride | 16984-48-8 | 120 | RSL | 5 | 3.5 | -- | -- | 50 | 80 | - 120 | 20 | 90 | - 110 | 20 |
| Nitrate | 14797-55-8 | 7.0 | BCL | 1.1 | 0.8 | -- | -- | 50 | 80 | - 120 | 20 | 90 | - 110 | 20 |
| Nitrite | 14797-65-0 | -- | -- | 1.5 | 1.1 | -- | -- | 50 | 80 | - 120 | 20 | 90 | - 110 | 20 |
| Ortho-Phosphate as $PO_4$ | 14265-44-2 | -- | -- | 5 | 4 | -- | -- | 50 | 80 | - 120 | 20 | 90 | - 110 | 20 |
| Sulfate | 14808-79-8 | -- | -- | 5 | 4 | -- | -- | 50 | 80 | - 120 | 20 | 90 | - 110 | 20 |
| *EPA Method 300.1* | | | | | | | | | | | | | | |
| Chlorate | 7790-93-4 | -- | -- | 0.2 | 0.05 | -- | -- | 50 | 75 | - 125 | 25 | 75 | - 125 | 25 |
| Dichloroacetic acid (Surr) | 79-43-6 | -- | -- | -- | -- | 90 | - 115 | -- | -- | -- | -- | -- | -- | -- |
| *EPA Method 314.0* | | | | | | | | | | | | | | |
| Perchlorate | 14797-73-0 | 0.0185 | BCL | 0.04 | 0.0095 | -- | -- | 50 | 80 | - 120 | 20 | 85 | - 115 | 15 |
| *EPA Method 9045C (SU)* | | | | | | | | | | | | | | |
| pH | STL00204 | -- | -- | 0.1 | -- | -- | -- | 50 | -- | -- | -- | -- | -- | -- |
| **Radionuclides (pCi/g)[8]** | | | | | | | | | | | | | | |
| *See Table 1 for Individual Methods* | | | | | | | | | | | | | | |
| Radium-226 | 13982-63-3 | 0.016 | RAD | 1 | -- | -- | -- | 50 | 72 | - 140 | 40 | 65 | - 140 | 40 |
| Radium-228 | 15262-20-1 | 0.016 | RAD | 1 | -- | -- | -- | 50 | 30 | - 150 | 40 | 61 | - 139 | 40 |
| Thorium-228 | 14274-82-9 | 0.11 | BCL | 1 | -- | -- | -- | 50 | 70 | - 130 | 40 | 70 | - 130 | 40 |
| Thorium-230 | 14269-63-7 | 0.042 | BCL | 1 | -- | -- | -- | 50 | 76 | - 115 | 40 | 81 | - 118 | 40 |
| Thorium-232 | 7440-29-1 | 0.14 | BCL | 1 | -- | -- | -- | 50 | 70 | - 130 | 40 | 70 | - 130 | 40 |
| Uranium-234 | 13966-29-5 | 0.012 | RAD | 1 | -- | -- | -- | 50 | 70 | - 130 | 40 | 84 | - 120 | 40 |
| Uranium-235 | 15117-96-1 | 0.012 | RAD | 1 | -- | -- | -- | 50 | -- | -- | 40 | -- | -- | 40 |
| Uranium-238 | 7440-61-1 | 0.012 | RAD | 1 | -- | -- | -- | 50 | 70 | - 130 | 40 | 82 | - 122 | 40 |
| **Asbestos (protocol structures)** | | | | | | | | | | | | | | |
| Total Amphibole Protocol Structures | 1332-21-4 | -- | -- | Fiber Count[9] | | -- | -- | 50 | -- | -- | -- | -- | -- | -- |
| Long Amphibole Protocol Structures | 1332-21-4 | -- | -- | Fiber Count[9] | | -- | -- | 50 | -- | -- | -- | -- | -- | -- |
| Total Chrysotile Protocol Structures | 1332-21-4 | -- | -- | Fiber Count[9] | | -- | -- | 50 | -- | -- | -- | -- | -- | -- |
| Long Chrysotile Protocol Structures | 1332-21-4 | -- | -- | Fiber Count[9] | | -- | -- | 50 | -- | -- | -- | -- | -- | -- |

## TABLE 4. LEACHING-BASED SOIL ANALYTES AND ANALYTICAL PERFORMANCE CRITERIA
## QUALITY ASSURANCE PROJECT PLAN
## Nevada Environmental Response Trust Site; Henderson, Nevada

| ANALYTES | CAS Number | Screening Level | Screening Level Source[1] | Practical Quantitation Limit (PQL) | Method Detection Limit (MDL) | QUALITY CONTROL LIMITS[2] Surrogate %R | | Duplicate RPD | Matrix Spike %R | | Matrix Spike RPD | Blank Spike/LCS %R | | Blank Spike/LCS RPD |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| Total Asbestos Protocol Structures | 1332-21-4 | -- | -- | Fiber Count[9] | | -- | -- | 50 | -- | -- | -- | -- | -- | -- |
| Long Asbestos Protocol Structures | 1332-21-4 | -- | -- | Fiber Count[9] | | -- | -- | 50 | -- | -- | -- | -- | -- | -- |

**Notes:**

Shaded PQLs and MDLs exceed the lowest screening criteria.

µg/kg = micrograms per kilogram

mg/kg = milligrams per kilogram

pCi/g = picoCurie per gram

Surr = Surrogate

EPA = United States Environmental Protection Agency

SM = Standard Method

(1) Soil screening levels were selected according to the following hierarchy of criteria:

(a) Basic Comparison Level (BCL): Leaching-based basic comparison levels (LBCL) with dilution attenuation factor (DAF) of 1 in the most recent version of Nevada Division of Environmental Protection (NDEP) documents (July 2017 for non-radionuclides and April 2009 for radionuclides).

(b) Regional Screening Level (RSL): United States Environmental Protection Agency (USEPA) Regional Screening Levels (RSL) for groundwater protection (June 2017), with the maximum contaminant level (MCL) based screening levels selected over the risk-based screening levels, if available (USEPA 2017).

(c) Radiation Criteria (RAD): USEPA Screening criteria from Soil Screening Guidance for Radionuclides: User's Guide, 2000 (USEPA 2013b).

(d) Calculated Criteria (CAL): Generic leaching-based BSLs (LBCLs) calculated using the approach presented in NDEP guidance (NDEP 2013).

All other individual or grouped dioxins or furans don't have screening levels.

All other individual or grouped PCBs use MCL-based screening levels for low risk PCBs in RSL table.

(2) QC Limits = Quality Control Limits for %R (Percent Recovery) of spiked compounds in Laboratory Control Samples (LCS) and surrogate compounds and Relative Percent Difference (RPD) between Matrix Spike (MS) and MS Duplicate (MSD) samples and LCS and LCS duplicate (LCSD) samples. Laboratory historical control limits are subject to change as a result of periodic re-evaluation. Limits in use at the time of sample analysis are available from the laboratory. Duplicate RPDs apply to sample duplicates and field duplicates.

(3) PQLs and MDLs for zirconium are under development by the laboratory and are not yet available.

(4) EDL = Estimated Detection Limit. For each dioxin, furan, or PCB not detected, an EDL is calculated. The sample specific EDL is an estimate made by the laboratory of the concentration of a given chemical that would have to be present to produce a signal with a peak height of at least 2.5 times the background signal level. The estimate is specific to a particular analysis of the sample and will be affected by sample size, dilution, and so forth. Because of the toxicological significance of dioxins, the EDL value is reported for non-detected chemicals rather than reporting the MDL.

(5) Dioxins and PCBs should be reported to the estimated detection limit (EDL). Dioxin toxicity equivalents (TEQ) will be calculated for the 16 dioxin and furan congeners and 12 PCB congeners with toxicity equivalent factors (TEFs) defined by the World Health Organization (Van den Berg et al. 2006) substituting half the EDL for the congeners not detected.

**TABLE 4. LEACHING-BASED SOIL ANALYTES AND ANALYTICAL PERFORMANCE CRITERIA**
**QUALITY ASSURANCE PROJECT PLAN**
**Nevada Environmental Response Trust Site; Henderson, Nevada**

| ANALYTES | CAS Number | Screening Level | Screening Level Source[1] | Practical Quantitation Limit (PQL) | Method Detection Limit (MDL) | QUALITY CONTROL LIMITS[2] | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|
| | | | | | | Surrogate | Duplicate | Matrix Spike | | Blank Spike/LCS | |
| | | | | | | %R | RPD | %R | RPD | %R | RPD |

(6) The total hexachlorodibenzo-p-dioxin (HxCDD) will be compared to an RSL of 0.017 µg/kg.

(7) Phthalic acid will be run with the SVOCs by EPA Method 8270C.

(8) Radionuclide PQLs and MDLs are based on minimum detectable activity (MDA) values. The measured values are reported regardless of sample-specific MDA.

(9) Asbestos data will be reported as raw asbestos fiber counts per sample (NDEP 2008). There are no PQLs for this method, but sensitivity is calculated by the concentration of protocol structures per volume of PM10.

**Sources:**

NDEP. 2009b. Guidance for Evaluating Radionuclide Data, BMI Plant Sites and Common Areas Projects, Henderson, Nevada. February 6.

NDEP. 2017. User's Guide and Background Technical Document for NDEP Basic Comparison Levels (BCLs) for Human Health for the BMI Complex and Common Areas. Revision 14, July.

USEPA. 2013b. Preliminary Remediation Goals for Radionuclides. On-line calculator. http://epa-prgs.ornl.gov/cgi-bin/radionuclides/rprg_search

USEPA. 2017. Regional Screening Levels (RSL) for Chemical Contaminants at Superfund Sites. June.

Van den Berg et al. 2006. The 2005 World Health Organization Reevaluation of Human and Mammalian Toxic Equivalency Factors for Dioxins and Dioxin-Like Compounds. May 20.

**TABLE 5. GROUNDWATER ANALYTES AND ANALYTICAL PERFORMANCE CRITERIA**
**QUALITY ASSURANCE PROJECT PLAN**
**Nevada Environmental Response Trust Site; Henderson, Nevada**

| ANALYTES | CAS Number | Screening Level | Screening Level Source[1] | Practical Quantitation Limit (PQL) | Method Detection Limit (MDL) | QUALITY CONTROL LIMITS[2] Surrogate %R | | Duplicate RPD | Matrix Spike %R | Matrix Spike RPD | Blank Spike/LCS %R | Blank Spike/LCS RPD |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| **Metals (µg/L)** | | | | | | | | | | | | |
| *EPA Method 200.7 / 6010* | | | | | | | | | | | | |
| Aluminum | 7429-90-5 | 50 | MCL | 50 | 25 | -- | -- | 30 | 75 - 125 | 20 | 80 - 120 | 20 |
| Barium | 7440-39-3 | 2,000 | MCL | 10 | 6 | -- | -- | 30 | 75 - 125 | 20 | 80 - 120 | 20 |
| Boron | 7440-42-8 | 6670 | BCL | 50 | 25 | -- | -- | 30 | 75 - 125 | 20 | 80 - 120 | 20 |
| Beryllium | 7440-41-7 | 4 | MCL | 4 | 0.9 | -- | -- | 30 | 75 - 125 | 20 | 80 - 120 | 20 |
| Cadmium | 7440-43-9 | 5 | MCL | 5 | 2 | -- | -- | 30 | 75 - 125 | 20 | 80 - 120 | 20 |
| Calcium | 7440-70-2 | -- | -- | 100 | 50 | -- | -- | 30 | 75 - 125 | 20 | 80 - 120 | 20 |
| Chromium (total) | 7440-47-3 | 100 | MCL | 5 | 2 | -- | -- | 30 | 75 - 125 | 20 | 80 - 120 | 20 |
| Cobalt | 7440-48-4 | 10 | BCL | 10 | 2 | -- | -- | 30 | 75 - 125 | 20 | 80 - 120 | 20 |
| Copper | 7440-50-8 | 1,300 | MCL | 10 | 3 | -- | -- | 30 | 75 - 125 | 20 | 80 - 120 | 20 |
| Iron | 7439-89-6 | 23,400 | BCL | 40 | 20 | -- | -- | 30 | 75 - 125 | 20 | 80 - 120 | 20 |
| Lead | 7439-92-1 | 15 | MCL | 5 | 4 | -- | -- | 30 | 75 - 125 | 20 | 80 - 120 | 20 |
| Magnesium | 7439-95-4 | 207,000 | BCL | 20 | 10 | -- | -- | 30 | 75 - 125 | 20 | 80 - 120 | 20 |
| Manganese | 7439-96-5 | 801 | BCL | 20 | 7 | -- | -- | 30 | 75 - 125 | 20 | 80 - 120 | 20 |
| Molybdenum | 7439-98-7 | 167 | BCL | 20 | 2 | -- | -- | 30 | 75 - 125 | 20 | 80 - 120 | 20 |
| Nickel | 7440-02-0 | 667 | BCL | 10 | 2 | -- | -- | 30 | 75 - 125 | 20 | 80 - 120 | 20 |
| Phosphorus | 7723-14-0 | 0.667 | BCL | 40 | 20 | -- | -- | 30 | 75 - 125 | 20 | 80 - 120 | 20 |
| Potassium | 7440-09-7 | -- | -- | 500 | 250 | -- | -- | 30 | 75 - 125 | 20 | 80 - 120 | 20 |
| Silicon | 7440-21-3 | -- | -- | 50 | 13 | -- | -- | 30 | 75 - 125 | 20 | 80 - 120 | 20 |
| Silver | 7440-22-4 | 100 | BCL | 10 | 6 | -- | -- | 30 | 75 - 125 | 20 | 80 - 120 | 20 |
| Sodium | 7440-23-5 | -- | -- | 500 | 250 | -- | -- | 30 | 75 - 125 | 20 | 80 - 120 | 20 |
| Strontium | 7440-24-6 | 20,000 | BCL | 20 | 5 | -- | -- | 30 | 75 - 125 | 20 | 80 - 120 | 20 |
| Tin | 7440-31-5 | 20,000 | BCL | 100 | 12 | -- | -- | 30 | 75 - 125 | 20 | 80 - 120 | 20 |
| Titanium | 7440-32-6 | 133,000 | BCL | 5 | 2 | -- | -- | 30 | 75 - 125 | 20 | 80 - 120 | 20 |
| Tungsten | 7440-33-7 | 26.7 | BCL | 1000 | 500 | -- | -- | 30 | 75 - 125 | 20 | 80 - 120 | 20 |
| Vanadium | 7440-62-2 | 167 | BCL | 10 | 3 | -- | -- | 30 | 75 - 125 | 20 | 80 - 120 | 20 |
| Zinc | 7440-66-6 | 10,000 | BCL | 20 | 9 | -- | -- | 30 | 75 - 125 | 20 | 80 - 120 | 20 |
| Zirconium | 7440-67-7 | 2.67 | BCL | 0.2 | -- | -- | -- | 30 | 75 - 125 | 20 | 80 - 120 | 20 |

**TABLE 5. GROUNDWATER ANALYTES AND ANALYTICAL PERFORMANCE CRITERIA**
**QUALITY ASSURANCE PROJECT PLAN**
**Nevada Environmental Response Trust Site; Henderson, Nevada**

| | | | | | | QUALITY CONTROL LIMITS[2] | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | | | Screening Level | Practical Quantitation Limit | Method Detection Limit | Surrogate | | Duplicate | Matrix Spike | | Blank Spike/LCS | |
| ANALYTES | CAS Number | Screening Level | Source[1] | (PQL) | (MDL) | %R | | RPD | %R | RPD | %R | RPD |
| *EPA Method 200.8 / 6020* | | | | | | | | | | | | |
| Antimony | 7440-36-0 | 6 | MCL | 10 | 7 | -- | -- | 30 | 75 - 125 | 20 | 80 - 120 | 20 |
| Arsenic | 7440-38-2 | 10 | MCL | 1.0 | 0.50 | -- | -- | 30 | 75 - 125 | 20 | 80 - 120 | 20 |
| Selenium | 7782-49-2 | 50 | MCL | 2 | 0.5 | -- | -- | 30 | 75 - 125 | 20 | 80 - 120 | 20 |
| Thallium | 7440-28-0 | 2 | MCL | 10 | 8 | -- | -- | 30 | 75 - 125 | 20 | 80 - 120 | 20 |
| *EPA Method 6020A* | | | | | | | | | | | | |
| Niobium | 7440-03-1 | 33.4 | BCL | 25 | 2.23 | -- | -- | 30 | 75 - 125 | 20 | 80 - 120 | 20 |
| Palladium | 7440-05-3 | -- | -- | 0.5 | 0.09 | -- | -- | 30 | 75 - 125 | 20 | 80 - 120 | 20 |
| Sulfur | 7704-34-9 | -- | -- | 5000 | 267 | -- | -- | 30 | 75 - 125 | 20 | 80 - 120 | 20 |
| Uranium | 7440-61-1 | 30 | MCL | 1.00 | 0.231 | -- | -- | 30 | 75 - 125 | 20 | 80 - 120 | 20 |
| *EPA Method 218.6* | | | | | | | | | | | | |
| Chromium (hexavalent) | 18540-29-9 | 0.134 | BCL | 1 | 0.25 | -- | -- | 30 | 90 - 110 | 10 | 90 - 110 | 10 |
| *EPA Method 7470A* | | | | | | | | | | | | |
| Mercury | 7439-97-6 | 2 | MCL | 0.2 | 0.1 | -- | -- | 30 | 70 - 130 | 20 | 80 - 120 | 20 |
| *EPA Method 1632* | | | | | | | | | | | | |
| Arsenic III | 7440-38-2 | -- | -- | 0.02 | 0.003 | -- | -- | 30 | 30 - 170 | 35 | 40 - 160 | 25 |
| Total Inorganic Arsenic[3] | 7440-38-2 | 10 | MCL | 0 | 0.003 | -- | -- | 30 | 80 - 120 | 35 | 60 - 140 | 25 |
| **Volatile Organic Compounds (µg/L)** | | | | | | | | | | | | |
| *EPA Method 8260B* | | | | | | | | | | | | |
| 1,1,1,2-Tetrachloroethane | 630-20-6 | 0.587 | BCL | 0.5 | 0.25 | -- | -- | 30 | 60 - 149 | 20 | 60 - 141 | 20 |
| 1,1,1-Trichloroethane | 71-55-6 | 200 | MCL | 0.5 | 0.25 | -- | -- | 30 | 70 - 130 | 20 | 70 - 130 | 20 |
| 1,1,2,2-Tetrachloroethane | 79-34-5 | 0.0752 | BCL | 0.5 | 0.25 | -- | -- | 30 | 63 - 130 | 30 | 63 - 130 | 25 |
| 1,1,2-Trichloroethane | 79-00-5 | 5 | MCL | 0.5 | 0.25 | -- | -- | 30 | 70 - 130 | 25 | 70 - 130 | 20 |
| 1,1-Dichloroethane | 75-34-3 | 2.7 | BCL | 0.5 | 0.25 | -- | -- | 30 | 65 - 130 | 20 | 64 - 130 | 20 |
| 1,1-Dichloroethene | 75-35-4 | 7 | MCL | 0.5 | 0.25 | -- | -- | 30 | 70 - 130 | 20 | 70 - 130 | 20 |
| 1,1-Dichloropropene | 563-58-6 | -- | -- | 0.5 | 0.25 | -- | -- | 30 | 64 - 130 | 20 | 70 - 130 | 20 |
| 1,2,3-Trichlorobenzene | 87-61-6 | 7 | RSL | 1.0 | 0.4 | -- | -- | 30 | 60 - 140 | 20 | 60 - 140 | 20 |
| 1,2,3-Trichloropropane | 96-18-4 | 0.00224 | BCL | 0.5 | 0.25 | -- | -- | 30 | 60 - 130 | 30 | 63 - 130 | 20 |

**TABLE 5. GROUNDWATER ANALYTES AND ANALYTICAL PERFORMANCE CRITERIA**
**QUALITY ASSURANCE PROJECT PLAN**
**Nevada Environmental Response Trust Site; Henderson, Nevada**

| | | | | | | QUALITY CONTROL LIMITS[2] | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | | | Screening Level | Practical Quantitation Limit | Method Detection Limit | Surrogate | Duplicate | Matrix Spike | | Blank Spike/LCS | |
| ANALYTES | CAS Number | Screening Level | Source[1] | (PQL) | (MDL) | %R | RPD | %R | RPD | %R | RPD |
| 1,2,4-Trichlorobenzene | 120-82-1 | 70 | MCL | 1.0 | 0.4 | -- -- | 30 | 60 - 140 | 20 | 60 - 140 | 20 |
| 1,2,4-Trimethylbenzene | 95-63-6 | 14.6 | BCL | 0.5 | 0.25 | -- -- | 30 | 70 - 130 | 25 | 70 - 135 | 20 |
| 1,2-Dibromo-3-Chloropropane | 96-12-8 | 0.2 | MCL | 1.0 | 0.5 | -- -- | 30 | 48 - 140 | 30 | 52 - 140 | 30 |
| 1,2-Dibromoethane (EDB) | 106-93-4 | 0.05 | MCL | 0.5 | 0.25 | -- -- | 30 | 70 - 131 | 25 | 70 - 130 | 20 |
| 1,2-Dichlorobenzene | 95-50-1 | 600 | MCL | 0.5 | 0.5 | -- -- | 30 | 70 - 130 | 20 | 70 - 130 | 20 |
| 1,2-Dichloroethane | 107-06-2 | 5 | MCL | 0.5 | 0.25 | -- -- | 30 | 56 - 146 | 20 | 57 - 138 | 20 |
| 1,2-Dichloropropane | 78-87-5 | 5 | MCL | 0.5 | 0.25 | -- -- | 30 | 69 - 130 | 20 | 67 - 130 | 20 |
| 1,3,5-Trimethylbenzene | 108-67-8 | 334 | BCL | 0.5 | 0.25 | -- -- | 30 | 70 - 130 | 20 | 70 - 136 | 20 |
| 1,3-Dichlorobenzene | 541-73-1 | 80.7 | BCL | 0.5 | 0.25 | -- -- | 30 | 70 - 130 | 20 | 70 - 130 | 20 |
| 1,3-Dichloropropane | 142-28-9 | 667 | BCL | 0.5 | 0.25 | -- -- | 30 | 70 - 130 | 25 | 70 - 130 | 20 |
| 1,4-Dichlorobenzene | 106-46-7 | 75 | MCL | 0.5 | 0.25 | -- -- | 30 | 70 - 130 | 20 | 70 - 130 | 20 |
| 2,2-Dichloropropane | 594-20-7 | -- | -- | 1.0 | 0.25 | -- -- | 30 | 69 - 138 | 25 | 68 - 141 | 25 |
| 2-Butanone | 78-93-3 | 6,860 | BCL | 5.0 | 2.5 | -- -- | 30 | 48 - 140 | 40 | 44 - 150 | 35 |
| 2-Chlorotoluene | 95-49-8 | 667 | BCL | 0.5 | 0.25 | -- -- | 30 | 70 - 130 | 20 | 70 - 130 | 20 |
| 4-Chlorotoluene | 106-43-4 | 667 | RSL | 0.5 | 0.25 | -- -- | 30 | 70 - 130 | 20 | 70 - 130 | 20 |
| Benzene | 71-43-2 | 5 | MCL | 0.5 | 0.25 | -- -- | 30 | 66 - 130 | 20 | 68 - 130 | 20 |
| Bromobenzene | 108-86-1 | 85.2 | BCL | 0.5 | 0.25 | -- -- | 30 | 70 - 130 | 20 | 70 - 130 | 20 |
| Bromochloromethane | 74-97-5 | 83.4 | RSL | 0.5 | 0.25 | -- -- | 30 | 70 - 130 | 25 | 70 - 130 | 20 |
| Bromodichloromethane | 75-27-4 | 0.133 | BCL | 0.5 | 0.25 | -- -- | 30 | 70 - 138 | 20 | 70 - 132 | 20 |
| Bromoform | 75-25-2 | 3.19 | BCL | 1.0 | 0.25 | -- -- | 30 | 59 - 150 | 25 | 60 - 148 | 25 |
| Bromomethane | 74-83-9 | 8.53 | BCL | 0.5 | 0.25 | -- -- | 30 | 62 - 131 | 25 | 64 - 139 | 20 |
| Carbon tetrachloride | 56-23-5 | 5 | MCL | 0.5 | 0.25 | -- -- | 30 | 60 - 150 | 25 | 60 - 150 | 25 |
| Chlorobenzene | 108-90-7 | 100 | MCL | 0.5 | 0.25 | -- -- | 30 | 70 - 130 | 20 | 70 - 130 | 20 |
| Chloroethane | 75-00-3 | 20,900 | BCL | 0.5 | 0.25 | -- -- | 30 | 68 - 130 | 25 | 64 - 135 | 20 |
| Chloroform | 67-66-3 | 0.219 | BCL | 0.5 | 0.25 | -- -- | 30 | 70 - 130 | 20 | 70 - 130 | 20 |
| Chloromethane | 74-87-3 | 188 | BCL | 0.5 | 0.25 | -- -- | 30 | 39 - 144 | 25 | 47 - 140 | 25 |
| cis-1,2-Dichloroethene | 156-59-2 | 66.7 | MCL | 0.5 | 0.25 | -- -- | 30 | 70 - 130 | 20 | 70 - 133 | 20 |
| cis-1,3-Dichloropropene | 10061-01-5 | -- | -- | 0.5 | 0.25 | -- -- | 30 | 70 - 133 | 20 | 70 - 133 | 25 |

**TABLE 5. GROUNDWATER ANALYTES AND ANALYTICAL PERFORMANCE CRITERIA**
**QUALITY ASSURANCE PROJECT PLAN**
**Nevada Environmental Response Trust Site; Henderson, Nevada**

| | | | | | | QUALITY CONTROL LIMITS[2] | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | | | | | | Surrogate | Duplicate | Matrix Spike | | Blank Spike/LCS | | |
| ANALYTES | CAS Number | Screening Level | Screening Level Source[1] | Practical Quantitation Limit (PQL) | Method Detection Limit (MDL) | %R | RPD | %R | RPD | %R | RPD |
| Dibromochloromethane | 124-48-1 | 0.8 | BCL | 0.5 | 0.25 | -- -- | 30 | 70 - 148 | 25 | 69 - 145 | 20 |
| Dibromomethane | 74-95-3 | 8.16 | BCL | 0.5 | 0.25 | -- -- | 30 | 70 - 130 | 25 | 70 - 130 | 20 |
| Dichlorodifluoromethane | 75-71-8 | 202 | BCL | 0.5 | 0.25 | -- -- | 30 | 25 - 142 | 30 | 29 - 150 | 30 |
| Ethyl ter-butyl ether (ETBE) | 637-92-3 | -- | -- | 0.5 | 0.25 | -- -- | 30 | 70 - 130 | 25 | 60 - 136 | 20 |
| Ethylbenzene | 100-41-4 | 700 | MCL | 0.5 | 0.25 | -- -- | 30 | 70 - 130 | 20 | 70 - 130 | 20 |
| Hexachlorobutadiene | 87-68-3 | 0.197 | BCL | 0.5 | 0.25 | -- -- | 30 | 10 - 150 | 20 | 10 - 150 | 20 |
| Isopropyl benzene | 98-82-8 | 3,340 | BCL | 0.5 | 0.25 | -- -- | 30 | 70 - 132 | 20 | 70 - 136 | 20 |
| m,p-Xylene | 179601-23-1 | 10,000 | MCL | 1.0 | 0.5 | -- -- | 30 | 70 - 133 | 25 | 70 - 130 | 20 |
| Methylene chloride | 75-09-2 | 5 | MCL | 2.0 | 0.88 | -- -- | 30 | 52 - 130 | 20 | 52 - 130 | 20 |
| Naphthalene | 91-20-3 | 0.165 | BCL | 1.0 | 0.4 | -- -- | 30 | 60 - 140 | 30 | 60 - 140 | 25 |
| n-Butylbenzene | 104-51-8 | 1,670 | BCL | 1.0 | 0.4 | -- -- | 30 | 61 - 149 | 20 | 65 - 150 | 20 |
| n-Propylbenzene | 103-65-1 | 1,280 | BCL | 0.5 | 0.25 | -- -- | 30 | 66 - 135 | 20 | 67 - 139 | 20 |
| o-Xylene | 95-47-6 | 10,000 | MCL | 0.5 | 0.25 | -- -- | 30 | 70 - 133 | 20 | 70 - 130 | 20 |
| p-Isopropyltoluene | 99-87-6 | 834 | BCL | 0.5 | 0.25 | -- -- | 30 | 70 - 130 | 20 | 70 - 132 | 20 |
| sec-Butylbenzene | 135-98-8 | 3,340 | BCL | 0.5 | 0.25 | -- -- | 30 | 67 - 134 | 20 | 70 - 138 | 20 |
| Styrene | 100-42-5 | 100 | MCL | 0.5 | 0.25 | -- -- | 30 | 29 - 150 | 35 | 70 - 134 | 20 |
| tert-Butylbenzene | 98-06-6 | 3,340 | BCL | 0.5 | 0.25 | -- -- | 30 | 70 - 130 | 20 | 70 - 130 | 20 |
| Tetrachloroethylene (PCE) | 127-18-4 | 5 | MCL | 0.5 | 0.25 | -- -- | 30 | 70 - 137 | 20 | 70 - 130 | 20 |
| Toluene | 108-88-3 | 1,000 | MCL | 0.5 | 0.25 | -- -- | 30 | 70 - 130 | 20 | 70 - 130 | 20 |
| trans-1,2-Dichloroethene | 156-60-5 | 100 | MCL | 0.5 | 0.25 | -- -- | 30 | 70 - 130 | 20 | 70 - 130 | 20 |
| trans-1,3-Dichloropropene | 10061-02-6 | -- | -- | 0.5 | 0.25 | -- -- | 30 | 70 - 138 | 25 | 70 - 132 | 20 |
| Trichloroethylene (TCE) | 79-01-6 | 5 | MCL | 0.5 | 0.25 | -- -- | 30 | 70 - 130 | 20 | 70 - 130 | 20 |
| Trichlorofluoromethane | 75-69-4 | 10,000 | BCL | 0.5 | 0.25 | -- -- | 30 | 60 - 150 | 25 | 60 - 150 | 20 |
| Vinyl chloride | 75-01-4 | 2 | MCL | 0.5 | 0.25 | -- -- | 30 | 50 - 137 | 30 | 59 - 133 | 30 |
| 4-Bromofluorobenzene (Surr) | 460-00-4 | -- | -- | -- | -- | 80 - 120 | -- | -- -- | -- | -- -- | -- |
| Dibromofluoromethane (Surr) | 1868-53-7 | -- | -- | -- | -- | 76 - 132 | -- | -- -- | -- | -- -- | -- |
| Toluene-d8 (Surr) | 2037-26-5 | -- | -- | -- | -- | 80 - 128 | -- | -- -- | -- | -- -- | -- |

**TABLE 5. GROUNDWATER ANALYTES AND ANALYTICAL PERFORMANCE CRITERIA**
**QUALITY ASSURANCE PROJECT PLAN**
**Nevada Environmental Response Trust Site; Henderson, Nevada**

| | | | | | | QUALITY CONTROL LIMITS[2] | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | | | Screening Level | Practical Quantitation Limit | Method Detection Limit | Surrogate | Duplicate | Matrix Spike | | Blank Spike/LCS | |
| ANALYTES | CAS Number | Screening Level | Source[1] | (PQL) | (MDL) | %R | RPD | %R | RPD | %R | RPD |
| *EPA Method 8260B SIM* | | | | | | | | | | | |
| 1,2,3-Trichloropropane | 96-18-4 | 0.00224 | BCL | 0.005 | 0.0035 | -- -- | 30 | 55 - 135 | 30 | 60 - 130 | 20 |
| 1,4-Dioxane | 123-91-1 | 0.672 | BCL | 2 | 0.5 | -- -- | 30 | 70 - 130 | 30 | 70 - 125 | 30 |
| Dibromofluoromethane (Surr) | 1868-53-7 | -- | -- | -- | -- | 80 - 120 | -- | -- -- | -- | -- -- | -- |
| **Semi Volatile Organic Compounds (µg/L)** | | | | | | | | | | | |
| *EPA Method 8270C* | | | | | | | | | | | |
| 1,2,4-Trichlorobenzene | 120-82-1 | 70 | MCL | 1 | 0.5 | -- -- | 30 | 45 - 120 | 20 | 44 - 120 | 20 |
| 1,2-Dichlorobenzene | 95-50-1 | 600 | MCL | 1 | 0.1 | -- -- | 30 | 40 - 120 | 25 | 43 - 120 | 25 |
| 1,2-Diphenylhydrazine(as Azobenzene) | 103-33-3 | 0.14 | BCL | 1 | 0.2 | -- -- | 30 | 60 - 120 | 25 | 59 - 124 | 25 |
| 1,3-Dichlorobenzene | 541-73-1 | 80.7 | BCL | 1 | 0.3 | -- -- | 30 | 35 - 120 | 25 | 41 - 120 | 25 |
| 1,4-Dichlorobenzene | 106-46-7 | 75 | MCL | 1 | 0.3 | -- -- | 30 | 35 - 120 | 25 | 41 - 120 | 25 |
| 1-Methylnaphthalene | 90-12-0 | 1.1 | RSL | 10 | 3.5 | -- -- | 30 | 55 - 120 | 30 | 60 - 140 | 35 |
| 2,4,5-Trichlorophenol | 95-95-4 | 3340 | BCL | 2 | 1.0 | -- -- | 30 | 55 - 120 | 30 | 20 - 138 | 30 |
| 2,4,6-Trichlorophenol | 88-06-2 | 6.11 | BCL | 1 | 0.5 | -- -- | 30 | 55 - 120 | 30 | 20 - 139 | 30 |
| 2,4-Dichlorophenol | 120-83-2 | 100 | BCL | 2 | 1.0 | -- -- | 30 | 55 - 120 | 25 | 21 - 132 | 20 |
| 2,4-Dimethylphenol | 105-67-9 | 667 | BCL | 2 | 1.0 | -- -- | 30 | 40 - 120 | 25 | 51 - 120 | 25 |
| 2,4-Dinitrophenol | 51-28-5 | 66.7 | BCL | 5 | 2.5 | -- -- | 30 | 40 - 120 | 25 | 20 - 134 | 25 |
| 2,4-Dinitrotoluene | 121-14-2 | 0.217 | BCL | 5 | 2.0 | -- -- | 30 | 65 - 120 | 25 | 54 - 121 | 20 |
| 2,6-Dinitrotoluene | 606-20-2 | 0.0448 | BCL | 5 | 1.0 | -- -- | 30 | 65 - 120 | 20 | 54 - 121 | 20 |
| 2-Chloronaphthalene | 91-58-7 | 2670 | BCL | 1 | 0.2 | -- -- | 30 | 60 - 120 | 20 | 54 - 120 | 20 |
| 2-Chlorophenol | 95-57-8 | 167 | BCL | 1 | 0.2 | -- -- | 30 | 45 - 120 | 25 | 20 - 122 | 25 |
| 2-Methylnaphthalene | 91-57-6 | 36 | RSL | 1 | 0.5 | -- -- | 30 | 55 - 120 | 20 | 55 - 120 | 20 |
| 2-Methylphenol | 95-48-7 | 1670 | BCL | 2 | 1.0 | -- -- | 30 | 50 - 120 | 25 | 47 - 120 | 20 |
| 2-Nitroaniline | 88-74-4 | 334 | BCL | 5 | 1.0 | -- -- | 30 | 65 - 120 | 25 | 46 - 126 | 20 |
| 2-Nitrophenol | 88-75-5 | -- | -- | 2 | 1.0 | -- -- | 30 | 50 - 120 | 25 | 21 - 132 | 25 |
| 3,3'-Dichlorobenzidine | 91-94-1 | 0.149 | BCL | 5 | 2.0 | -- -- | 30 | 45 - 135 | 25 | 25 - 135 | 25 |

**TABLE 5. GROUNDWATER ANALYTES AND ANALYTICAL PERFORMANCE CRITERIA**
**QUALITY ASSURANCE PROJECT PLAN**
**Nevada Environmental Response Trust Site; Henderson, Nevada**

| | | | | | | QUALITY CONTROL LIMITS[2] | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | | | Screening Level | Practical Quantitation Limit | Method Detection Limit | Surrogate | Duplicate | Matrix Spike | | Blank Spike/LCS | |
| ANALYTES | CAS Number | Screening Level | Source[1] | (PQL) | (MDL) | %R | RPD | %R | RPD | %R | RPD |
| 3-Methylphenol + 4-Methylphenol | 106-44-5 | -- | -- | 5 | 1.0 | -- -- | 30 | 50 - 120 | 25 | 50 - 120 | 20 |
| 3-Nitroaniline | 99-09-2 | -- | -- | 5 | 2.0 | -- -- | 30 | 60 - 120 | 25 | 42 - 122 | 25 |
| 4,6-Dinitro-2-methylphenol | 534-52-1 | -- | -- | 5 | 0.3 | -- -- | 30 | 45 - 120 | 25 | 22 - 147 | 25 |
| 4-Bromophenyl phenyl ether | 101-55-3 | -- | -- | 1 | 0.5 | -- -- | 30 | 60 - 120 | 25 | 58 - 120 | 25 |
| 4-Chloro-3-methylphenol | 59-50-7 | 1,400 | RSL | 2 | 0.2 | -- -- | 30 | 60 - 120 | 25 | 46 - 123 | 25 |
| 4-Chloroaniline | 106-47-8 | 0.336 | BCL | 2 | 1.0 | -- -- | 30 | 55 - 120 | 25 | 52 - 120 | 25 |
| 4-Chlorophenyl phenyl ether | 7005-72-3 | -- | -- | 1 | 0.2 | -- -- | 30 | 65 - 120 | 25 | 50 - 122 | 20 |
| 4-Nitroaniline | 100-01-6 | 3.8 | RSL | 5 | 2.0 | -- -- | 30 | 55 - 125 | 25 | 46 - 126 | 20 |
| 4-Nitrophenol | 100-02-7 | 267 | BCL | 5 | 2.5 | -- -- | 30 | 45 - 120 | 30 | 20 - 151 | 30 |
| Acenaphthene | 83-32-9 | 2000 | BCL | 1 | 0.2 | -- -- | 30 | 60 - 120 | 25 | 57 - 120 | 20 |
| Acenaphthylene | 208-96-8 | 6.22 | BCL | 1 | 0.2 | -- -- | 30 | 60 - 120 | 25 | 60 - 120 | 20 |
| Aniline | 62-53-3 | 11.8 | BCL | 10 | 0.3 | -- -- | 30 | 35 - 120 | 30 | 53 - 120 | 30 |
| Anthracene | 120-12-7 | 100000 | BCL | 1 | 0.2 | -- -- | 30 | 65 - 120 | 25 | 62 - 120 | 20 |
| Benzidine | 92-87-5 | -- | -- | 5 | 1.0 | -- -- | 30 | 30 - 160 | 35 | 20 - 168 | 35 |
| Benzo[a]anthracene | 56-55-3 | 0.0328 | BCL | 5 | 2.0 | -- -- | 30 | 65 - 120 | 20 | 62 - 120 | 20 |
| Benzo[a]pyrene | 50-32-8 | 0.2 | MCL | 2 | 1.0 | -- -- | 30 | 55 - 130 | 25 | 58 - 103 | 25 |
| Benzo[b]fluoranthene | 205-99-2 | 0.0921 | BCL | 2 | 1.0 | -- -- | 30 | 55 - 125 | 25 | 46 - 125 | 25 |
| Benzo[g,h,i]perylene | 191-24-2 | 1,000 | BCL | 5 | 2.0 | -- -- | 30 | 45 - 135 | 30 | 52 - 136 | 25 |
| Benzo[k]fluoranthene | 207-08-9 | 0.921 | BCL | 1 | 0.3 | -- -- | 30 | 55 - 125 | 30 | 61 - 127 | 20 |
| Benzoic acid | 65-85-0 | 133,000 | BCL | 5 | 3.0 | -- -- | 30 | 25 - 125 | 30 | 20 - 120 | 30 |
| Benzyl alcohol | 100-51-6 | 3,340 | BCL | 5 | 0.1 | -- -- | 30 | 40 - 120 | 30 | 50 - 120 | 20 |
| bis (2-chloroisopropyl) ether | 108-60-1 | 1,330 | BCL | 1 | 0.2 | -- -- | 30 | 45 - 120 | 25 | 45 - 120 | 20 |
| Bis(2-chloroethoxy)methane | 111-91-1 | 59 | RSL | 1 | 0.2 | -- -- | 30 | 50 - 120 | 25 | 57 - 120 | 20 |
| Bis(2-chloroethyl)ether | 111-44-4 | 0.0133 | BCL | 1 | 0.2 | -- -- | 30 | 50 - 120 | 25 | 54 - 120 | 20 |
| Bis(2-ethylhexyl) phthalate | 117-81-7 | 6 | MCL | 5 | 2.0 | -- -- | 30 | 65 - 130 | 25 | 57 - 124 | 20 |
| Butyl benzyl phthalate | 85-68-7 | 35.4 | BCL | 5 | 2.0 | -- -- | 30 | 55 - 130 | 25 | 57 - 129 | 20 |

**TABLE 5. GROUNDWATER ANALYTES AND ANALYTICAL PERFORMANCE CRITERIA**
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| | | | | | | QUALITY CONTROL LIMITS[2] | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | | | | | | Surrogate | Duplicate | Matrix Spike | | | Blank Spike/LCS | |
| ANALYTES | CAS Number | Screening Level | Screening Level Source[1] | Practical Quantitation Limit (PQL) | Method Detection Limit (MDL) | %R | RPD | %R | | RPD | %R | RPD |
| Chrysene | 218-01-9 | 9.21 | BCL | 1 | 0.2 | -- -- | 30 | 65 - 120 | | 25 | 63 - 109 | 20 |
| Dibenz(a,h)anthracene | 53-70-3 | 0.00921 | BCL | 1 | 0.3 | -- -- | 30 | 45 - 135 | | 30 | 56 - 124 | 25 |
| Dibenzofuran | 132-64-9 | 33.4 | BCL | 1 | 0.2 | -- -- | 30 | 65 - 120 | | 25 | 59 - 109 | 20 |
| Diethyl phthalate | 84-66-2 | 26,700 | BCL | 1 | 0.5 | -- -- | 30 | 55 - 120 | | 30 | 44 - 131 | 30 |
| Dimethyl phthalate | 131-11-3 | 334,000 | BCL | 1 | 0.3 | -- -- | 30 | 30 - 120 | | 30 | 33 - 140 | 30 |
| Di-n-butyl phthalate | 84-74-2 | 3,340 | BCL | 2 | 1.0 | -- -- | 30 | 60 - 125 | | 25 | 60 - 126 | 20 |
| Di-n-octyl phthalate | 117-84-0 | 200 | RSL | 5 | 2.0 | -- -- | 30 | 65 - 135 | | 20 | 56 - 117 | 20 |
| Fluoranthene | 206-44-0 | 1,330 | BCL | 1 | 0.2 | -- -- | 30 | 60 - 120 | | 25 | 64 - 120 | 20 |
| Fluorene | 86-73-7 | 1,330 | BCL | 1 | 0.2 | -- -- | 30 | 65 - 120 | | 25 | 52 - 120 | 20 |
| Hexachlorobenzene | 118-74-1 | 1 | MCL | 1 | 0.5 | -- -- | 30 | 60 - 120 | | 25 | 60 - 105 | 20 |
| Hexachlorobutadiene | 87-68-3 | 0.197 | BCL | 2 | 0.5 | -- -- | 30 | 40 - 120 | | 25 | 34 - 120 | 25 |
| Hexachlorocyclopentadiene | 77-47-4 | 50 | MCL | 5 | 2.0 | -- -- | 30 | 25 - 120 | | 30 | 23 - 120 | 30 |
| Hexachloroethane | 67-72-1 | 0.392 | BCL | 3 | 0.5 | -- -- | 30 | 35 - 120 | | 25 | 34 - 120 | 25 |
| Indeno[1,2,3-cd]pyrene | 193-39-5 | 0.0921 | BCL | 2 | 1.0 | -- -- | 30 | 40 - 135 | | 30 | 59 - 128 | 25 |
| Isophorone | 78-59-1 | 70.8 | BCL | 1 | 0.5 | -- -- | 30 | 50 - 120 | | 25 | 50 - 120 | 20 |
| Naphthalene | 91-20-3 | 0.165 | BCL | 1 | 0.5 | -- -- | 30 | 55 - 120 | | 25 | 52 - 120 | 20 |
| Nitrobenzene | 98-95-3 | 0.14 | BCL | 1 | 0.5 | -- -- | 30 | 55 - 120 | | 25 | 52 - 120 | 25 |
| N-Nitrosodi-n-propylamine | 621-64-7 | 0.0096 | BCL | 2 | 1.0 | -- -- | 30 | 45 - 120 | | 25 | 60 - 120 | 20 |
| N-Nitrosodiphenylamine | 86-30-6 | 13.7 | BCL | 1 | 0.5 | -- -- | 30 | 60 - 120 | | 25 | 58 - 120 | 20 |
| Octachlorostyrene | 29082-74-4 | -- | -- | 20 | 6.5 | -- -- | 30 | 60 - 140 | | 30 | 60 - 140 | 30 |
| Pentachlorophenol | 87-86-5 | 1 | MCL | 2 | 1.0 | -- -- | 30 | 24 - 121 | | 25 | 20 - 137 | 25 |
| Phenanthrene | 85-01-8 | 6.22 | BCL | 1 | 0.2 | -- -- | 30 | 65 - 120 | | 25 | 62 - 120 | 20 |
| Phenol | 108-95-2 | 10,000 | BCL | 1 | 0.5 | -- -- | 30 | 40 - 120 | | 25 | 20 - 120 | 25 |
| Pyrene | 129-00-0 | 1,000 | BCL | 1 | 0.2 | -- -- | 30 | 55 - 125 | | 25 | 54 - 120 | 25 |
| 2,4,6-Tribromophenol (Surr) | 118-79-6 | -- | -- | -- | -- | 40 - 120 | -- | -- | -- | -- | -- | -- |
| 2-Fluorophenol (Surr) | 367-12-4 | -- | -- | -- | -- | 30 - 120 | -- | -- | -- | -- | -- | -- |
| Nitrobenzene-d5 (Surr) | 4165-60-0 | -- | -- | -- | -- | 45 - 120 | -- | -- | -- | -- | -- | -- |

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**Nevada Environmental Response Trust Site; Henderson, Nevada**

| | | | | | | QUALITY CONTROL LIMITS[2] | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | | | Screening Level | Practical Quantitation Limit | Method Detection Limit | Surrogate | Duplicate | Matrix Spike | | Blank Spike/LCS | | |
| ANALYTES | CAS Number | Screening Level | Source[1] | (PQL) | (MDL) | %R | RPD | %R | RPD | %R | RPD | |
| Terphenyl-d14 (Surr) | 1718-51-0 | -- | -- | -- | -- | 37 - 144 | -- | -- | -- | -- | -- | -- |
| Phenol-d6 | 13127-88-3 | -- | -- | -- | -- | 35 - 120 | -- | -- | -- | -- | -- | -- |
| *EPA Method 8315A* | | | | | | | | | | | | |
| Formaldehyde | 50-00-0 | 0.432 | BCL | 10 | 5 | -- -- | 30 | 50 - 150 | 20 | 50 - 150 | 20 | |
| **Polycyclic Aromatic Hydrocarbons (µg/L)** | | | | | | | | | | | | |
| *EPA Method 8270 SIM* | | | | | | | | | | | | |
| Acenaphthene | 83-32-9 | 2,000 | BCL | 0.2 | 0.05 | -- -- | 30 | 60 - 120 | 25 | 60 - 120 | 20 | |
| Acenaphthylene | 208-96-8 | 6.22 | BCL | 0.2 | 0.05 | -- -- | 30 | 60 - 120 | 25 | 60 - 120 | 20 | |
| Anthracene | 120-12-7 | 100,000 | BCL | 0.2 | 0.05 | -- -- | 30 | 65 - 120 | 25 | 65 - 120 | 20 | |
| Benzo(a)anthracene | 56-55-3 | 0.0328 | BCL | 0.2 | 0.05 | -- -- | 30 | 65 - 120 | 20 | 65 - 120 | 20 | |
| Benzo(a)pyrene | 50-32-8 | 0.2 | MCL | 0.2 | 0.05 | -- -- | 30 | 55 - 130 | 25 | 55 - 130 | 25 | |
| Benzo(b)fluoranthene | 205-99-2 | 0.0921 | BCL | 0.2 | 0.05 | -- -- | 30 | 55 - 125 | 25 | 55 - 125 | 25 | |
| Benzo(g,h,i)perylene | 191-24-2 | 1,000 | BCL | 0.2 | 0.05 | -- -- | 30 | 45 - 135 | 30 | 45 - 135 | 25 | |
| Benzo(k)fluoranthene | 207-08-9 | 0.921 | BCL | 0.2 | 0.05 | -- -- | 30 | 55 - 125 | 30 | 50 - 125 | 20 | |
| Chrysene | 218-01-9 | 9.21 | BCL | 0.2 | 0.05 | -- -- | 30 | 65 - 120 | 25 | 65 - 120 | 20 | |
| Dibenz(a,h)anthracene | 53-70-3 | 0.00921 | BCL | 0.2 | 0.05 | -- -- | 30 | 45 - 135 | 30 | 50 - 135 | 25 | |
| Fluoranthene | 206-44-0 | 1,330 | BCL | 0.2 | 0.05 | -- -- | 30 | 60 - 120 | 25 | 60 - 120 | 20 | |
| Fluorene | 86-73-7 | 1,330 | BCL | 0.2 | 0.05 | -- -- | 30 | 65 - 120 | 25 | 65 - 120 | 20 | |
| Indeno(1,2,3-cd)pyrene | 193-39-5 | 0.0921 | BCL | 0.2 | 0.05 | -- -- | 30 | 40 - 135 | 30 | 45 - 135 | 25 | |
| Naphthalene | 91-20-3 | 0.165 | BCL | 0.2 | 0.05 | -- -- | 30 | 55 - 120 | 25 | 55 - 120 | 20 | |
| Phenanthrene | 85-01-8 | 6.22 | BCL | 0.2 | 0.05 | -- -- | 30 | 65 - 120 | 25 | 65 - 120 | 20 | |
| Pyrene | 129-00-0 | 1,000 | BCL | 0.2 | 0.05 | -- -- | 30 | 55 - 125 | 25 | 55 - 125 | 25 | |
| 2-Fluorobiphenyl (Surr) | 321-60-8 | -- | -- | -- | -- | 50 - 120 | -- | -- | -- | -- | -- | -- |
| Nitrobenzene-d5 | 4165-60-0 | -- | -- | -- | -- | 45 - 120 | -- | -- | -- | -- | -- | -- |
| Terphenyl-d14 | 1718-51-0 | -- | -- | -- | -- | 17 - 100 | -- | -- | -- | -- | -- | -- |
| **Organophosphorus Peticides (µg/L)** | | | | | | | | | | | | |
| *EPA Method 8141A* | | | | | | | | | | | | |
| Atrazine | 1912-24-9 | -- | -- | 10.0 | 0.293 | -- -- | 30 | 49 - 116 | 50 | 49 - 116 | 50 | |

**TABLE 5. GROUNDWATER ANALYTES AND ANALYTICAL PERFORMANCE CRITERIA**
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**Nevada Environmental Response Trust Site; Henderson, Nevada**

| | | | | | | QUALITY CONTROL LIMITS[2] | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|
| | | | | | | Surrogate | Duplicate | Matrix Spike | | Blank Spike/LCS | |
| ANALYTES | CAS Number | Screening Level | Screening Level Source[1] | Practical Quantitation Limit (PQL) | Method Detection Limit (MDL) | %R | RPD | %R | RPD | %R | RPD |
| Bolstar | 35400-43-2 | -- | -- | 1.0 | 0.314 | -- -- | 30 | | | 61 - 108 | |
| Chlorpyrifos | 2921-88-2 | 33.4 | BCL | 1.5 | 0.360 | -- -- | 30 | 35 - 124 | 34 | 35 - 124 | 34 |
| Coumaphos | 56-72-4 | -- | -- | 1.0 | 0.135 | -- -- | 30 | 39 - 126 | 43 | 39 - 126 | 43 |
| Demeton, Total | 8065-48-3 | -- | -- | 3.0 | 0.209 | -- -- | 30 | 31 - 123 | 50 | 31 - 123 | 50 |
| Diazinon | 333-41-5 | 23.4 | BCL | 0.5 | 0.147 | -- -- | 30 | 46 - 115 | 40 | 46 - 115 | 40 |
| Dichlorvos | 62-73-7 | 0.232 | BCL | 0.5 | 0.162 | -- -- | 30 | 33 - 151 | 49 | 33 - 151 | 49 |
| Dimethoate | 60-51-5 | 44 | RSL | 1.5 | 0.449 | -- -- | 30 | 36 - 127 | 50 | 36 - 127 | 50 |
| Disulfoton | 298-04-4 | 1.33 | BCL | 1.0 | 0.322 | -- -- | 30 | 36 - 115 | 40 | 36 - 115 | 40 |
| EPN | 2104-64-5 | 0.089 | RSL | 1.2 | 0.149 | -- -- | 30 | 54 - 138 | 50 | 54 - 138 | 50 |
| Ethoprop | 13194-48-4 | -- | -- | 1.5 | 0.177 | -- -- | 30 | 51 - 120 | 36 | 51 - 120 | 36 |
| Ethyl Parathion | 56-38-2 | 200 | BCL | 1.0 | 0.144 | -- -- | 30 | 25 - 175 | 40 | 25 - 175 | 40 |
| Famphur | 52-85-7 | -- | -- | 1.0 | 0.179 | -- -- | 30 | 43 - 146 | 88 | 43 - 146 | 88 |
| Fensulfothion | 115-90-2 | -- | -- | 2.5 | 0.544 | -- -- | 30 | 36 - 124 | 62 | 36 - 124 | 62 |
| Fenthion | 55-38-9 | -- | -- | 2.5 | 0.154 | -- -- | 30 | 34 - 120 | 41 | 34 - 120 | 41 |
| Malathion | 121-75-5 | 667 | BCL | 2.0 | 0.133 | -- -- | 30 | 41 - 134 | 28 | 41 - 134 | 28 |
| Merphos | 150-50-5 | 0.6 | RSL | 5.0 | 0.174 | -- -- | 30 | 10 - 123 | 50 | 10 - 123 | 50 |
| Methyl parathion | 298-00-0 | 8.34 | BCL | 4.0 | 0.141 | -- -- | 30 | 42 - 130 | 30 | 42 - 130 | 30 |
| Mevinphos | 7786-34-7 | -- | -- | 6.2 | 0.460 | -- -- | 30 | 10 - 229 | 40 | 10 - 229 | 40 |
| Phorate | 298-02-2 | 3 | RSL | 1.2 | 0.154 | -- -- | 30 | 36 - 115 | 40 | 36 - 115 | 40 |
| Ronnel | 299-84-3 | 1,670 | BCL | 10.0 | 0.116 | -- -- | 30 | 33 - 126 | 39 | 33 - 126 | 39 |
| Simazine | 122-34-9 | -- | -- | 10.0 | 0.223 | -- -- | 30 | 27 - 186 | 31 | 27 - 186 | 31 |
| Stirphos (Tetrachlorovinphos) | 22248-79-9 | -- | -- | 3.5 | 0.124 | -- -- | 30 | 27 - 131 | 40 | 27 - 131 | 40 |
| Sulfotepp | 3689-24-5 | 7.1 | RSL | 1.5 | 0.168 | -- -- | 30 | 48 - 123 | 40 | 48 - 123 | 40 |
| Thionazin | 297-97-2 | -- | -- | 1.0 | 0.312 | -- -- | 30 | 48 - 115 | 40 | 48 - 115 | 40 |
| Trichloronate | 327-98-0 | -- | -- | 1.5 | 0.242 | -- -- | 30 | 14 - 118 | 38 | 14 - 118 | 38 |
| Chlormefos (Surr) | 24934-91-6 | -- | -- | -- | -- | 49 - 171 | -- | -- -- | -- | -- -- | -- |
| Triphenylphosphate (Surr) | 115-86-6 | -- | -- | -- | -- | 60 - 154 | -- | -- -- | -- | -- -- | -- |

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| | | | | | | QUALITY CONTROL LIMITS[2] | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | | | Screening Level | Practical Quantitation Limit | Method Detection Limit | Surrogate | Duplicate | Matrix Spike | | Blank Spike/LCS | | |
| ANALYTES | CAS Number | Screening Level | Source[1] | (PQL) | (MDL) | %R | RPD | %R | RPD | %R | RPD | |
| **Organochlorine Pesticides (µg/L)** | | | | | | | | | | | | |
| *EPA Method 8081A* | | | | | | | | | | | | |
| 2,4'-DDE | 3424-82-6 | -- | -- | 0.1 | 0.02 | -- -- | 30 | 45 - 125 | 30 | 50 - 120 | 30 | |
| 4,4'-DDD | 72-54-8 | 0.0631 | BCL | 0.0 | 0.00 | -- -- | 30 | 50 - 125 | 30 | 55 - 120 | 30 | |
| 4,4'-DDE | 72-55-9 | 0.198 | BCL | 0.005 | 0.003 | -- -- | 30 | 45 - 125 | 30 | 50 - 120 | 30 | |
| 4,4'-DDT | 50-29-3 | 0.198 | BCL | 0.01 | 0.004 | -- -- | 30 | 50 - 125 | 30 | 55 - 120 | 30 | |
| Aldrin | 309-00-2 | 0.000889 | BCL | 0.01 | 0.002 | -- -- | 30 | 35 - 120 | 30 | 40 - 115 | 30 | |
| alpha-BHC | 319-84-6 | 0.0107 | BCL | 0.005 | 0.0025 | -- -- | 30 | 40 - 120 | 30 | 45 - 115 | 30 | |
| beta-BHC | 319-85-7 | 0.0374 | BCL | 0.01 | 0.004 | -- -- | 30 | 50 - 120 | 30 | 55 - 115 | 30 | |
| delta-BHC | 319-86-8 | 10 | BCL | 0.005 | 0.0035 | -- -- | 30 | 50 - 120 | 30 | 55 - 115 | 30 | |
| Dieldrin | 60-57-1 | 0.0042 | BCL | 0.005 | 0.002 | -- -- | 30 | 50 - 120 | 30 | 55 - 115 | 30 | |
| Endosulfan I | 959-98-8 | -- | -- | 0.005 | 0.003 | -- -- | 30 | 50 - 120 | 30 | 55 - 115 | 30 | |
| Endosulfan II | 33213-65-9 | -- | -- | 0.005 | 0.0020 | -- -- | 30 | 50 - 125 | 30 | 55 - 120 | 30 | |
| Endosulfan Sulfate | 1031-07-8 | -- | -- | 0.01 | 0.003 | -- -- | 30 | 55 - 125 | 30 | 60 - 120 | 30 | |
| Endrin | 72-20-8 | 2 | MCL | 0.005 | 0.0020 | -- -- | 30 | 50 - 120 | 30 | 55 - 115 | 30 | |
| Endrin aldehyde | 7421-93-4 | -- | -- | 0.010 | 0.0020 | -- -- | 30 | 45 - 125 | 30 | 50 - 120 | 30 | |
| Endrin Ketone | 53494-70-5 | -- | -- | 0.01 | 0.007 | -- -- | 30 | 50 - 125 | 30 | 55 - 120 | 30 | |
| gamma-BHC | 58-89-9 | 0.2 | MCL | 0.01 | 0.003 | -- -- | 30 | 40 - 120 | 30 | 45 - 115 | 30 | |
| gamma-chlordane | 57-74-9 | 2 | MCL | 0.10 | 0.080 | -- -- | 30 | 60 - 140 | 30 | 60 - 140 | 30 | |
| Heptachlor | 76-44-8 | 0.4 | MCL | 0.01 | 0.003 | -- -- | 30 | 40 - 120 | 30 | 45 - 115 | 30 | |
| Heptachlor epoxide | 1024-57-3 | 0.2 | MCL | 0.005 | 0.0025 | -- -- | 30 | 50 - 120 | 30 | 55 - 115 | 30 | |
| Methoxychlor | 72-43-5 | 40 | MCL | 0.005 | 0.0035 | -- -- | 30 | 55 - 125 | 30 | 60 - 120 | 30 | |
| Toxaphene | 8001-35-2 | 3 | MCL | 0.500 | 0.2500 | -- -- | 30 | 60 - 140 | 30 | 60 - 140 | 30 | |
| DCB Decachlorobiphenyl (Surr) | 2051-24-3 | -- | -- | -- | -- | 45 - 120 | -- | -- -- | -- | -- -- | -- | |
| **PCBs as Congeners (µg/L)[5]** | | | | | | | | | | | | |
| *EPA Method 1668A* | | | | | | | | | | | | |
| Total PCBs | 1336-36-3 | 0.5 | MCL | 0.000002 | EDL[4] | -- -- | 30 | -- -- | -- | -- -- | -- | |
| 3,4,4',5-TeCB (PCB-81) | 70362-50-4 | 0.0004 | RSL | 0.00000002 | EDL[4] | -- -- | 30 | 50 - 150 | 50 | 50 - 150 | 50 | |

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| | | | | | | QUALITY CONTROL LIMITS[2] | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | | | | Practical Quantitation Limit | Method Detection Limit | Surrogate | Duplicate | Matrix Spike | | Blank Spike/LCS | |
| ANALYTES | CAS Number | Screening Level | Screening Level Source[1] | (PQL) | (MDL) | %R | RPD | %R | RPD | %R | RPD |
| 2,3',4,4',5-PeCB (PCB-118) | 31508-00-6 | 0.004 | RSL | 0.00000002 | EDL[4] | -- -- | 30 | 50 - 150 | 50 | 50 - 150 | 50 |
| 3,3',4,4',5-PeCB (PCB-126) | 57465-28-8 | 0.0000012 | RSL | 0.00000002 | EDL[4] | -- -- | 30 | 50 - 150 | 50 | 50 - 150 | 50 |
| 3,3',4,4',5,5'-HxCB (PCB-169) | 32774-16-6 | 0.000004 | RSL | 0.00000002 | EDL[4] | -- -- | 30 | 50 - 150 | 50 | 50 - 150 | 50 |
| DeCB (PCB-209) | 2051-24-3 | 0.5 | RSL | 0.0000002 | EDL[4] | -- -- | 30 | 50 - 150 | 50 | 50 - 150 | 50 |
| **PCBs as Aroclors (µg/L)** | | | | | | | | | | | |
| *EPA Method 8082* | | | | | | | | | | | |
| Aroclor 1260 | 11096-82-5 | 0.00493 | BCL | 0.500 | 0.2500 | -- -- | 30 | 55 - 125 | 25 | 60 - 120 | 25 |
| **Dioxins/Furans (pg/L)[5]** | | | | | | | | | | | |
| *EPA Method 8290 or 8280(7)* | | | | | | | | | | | |
| 2,3,7,8- TCDD | 1746-01-6 | 30 | MCL | 10 | EDL[4] | -- -- | 30 | 72 - 144 | 20 | 72 - 144 | 20 |
| OCDF | 39001-02-0 | -- | -- | 100 | EDL[4] | -- -- | 30 | 65 - 145 | 20 | 65 - 145 | 20 |
| OCDD | 3268-87-9 | -- | -- | 100 | EDL[4] | -- -- | 30 | 80 - 129 | 20 | 80 - 129 | 20 |
| 1,2,3,4,6,7,8-HpCDF | 67562-39-4 | -- | -- | 50 | EDL[4] | -- -- | 30 | 81 - 135 | 20 | 81 - 135 | 20 |
| 1,2,3,4,6,7,8-HpCDD | 35822-46-9 | -- | -- | 50 | EDL[4] | -- -- | 30 | 81 - 132 | 20 | 81 - 132 | 20 |
| 1,2,3,4,7,8,9-HpCDF | 55673-89-7 | -- | -- | 50 | EDL[4] | -- -- | 30 | 72 - 140 | 20 | 72 - 140 | 20 |
| 1,2,3,4,7,8-HxCDF | 70648-26-9 | -- | -- | 50 | EDL[4] | -- -- | 30 | 86 - 126 | 20 | 86 - 126 | 20 |
| 1,2,3,4,7,8-HxCDD | 39227-28-6 | -- | -- | 50 | EDL[4] | -- -- | 30 | 65 - 144 | 20 | 65 - 144 | 20 |
| 1,2,3,6,7,8-HxCDF | 57117-44-9 | -- | -- | 50 | EDL[4] | -- -- | 30 | 79 - 137 | 20 | 79 - 137 | 20 |
| 1,2,3,6,7,8-HxCDD | 57653-85-7 | -- | -- | 50 | EDL[4] | -- -- | 30 | 78 - 137 | 20 | 78 - 137 | 20 |
| 1,2,3,7,8,9-HxCDF | 72918-21-9 | -- | -- | 50 | EDL[4] | -- -- | 30 | 72 - 145 | 20 | 72 - 145 | 20 |
| 1,2,3,7,8,9-HxCDD | 19408-74-3 | -- | -- | 50 | EDL[4] | -- -- | 30 | 74 - 142 | 20 | 74 - 142 | 20 |
| 1,2,3,7,8-PeCDF | 57117-41-6 | -- | -- | 50 | EDL[4] | -- -- | 30 | 79 - 137 | 20 | 79 - 137 | 20 |
| 1,2,3,7,8-PeCDD | 40321-76-4 | -- | -- | 50 | EDL[4] | -- -- | 30 | 79 - 125 | 20 | 79 - 125 | 20 |
| 2,3,4,6,7,8-HxCDF | 60851-34-5 | -- | -- | 50 | EDL[4] | -- -- | 30 | 80 - 138 | 20 | 80 - 138 | 20 |
| 1,2,3,6,7,8-HxCDF | 57117-44-9 | -- | -- | 50 | EDL[4] | -- -- | 30 | 79 - 137 | 20 | 79 - 137 | 20 |

**TABLE 5. GROUNDWATER ANALYTES AND ANALYTICAL PERFORMANCE CRITERIA**
**QUALITY ASSURANCE PROJECT PLAN**
**Nevada Environmental Response Trust Site; Henderson, Nevada**

| | | | | | | QUALITY CONTROL LIMITS[2] | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|
| | | | | | | Surrogate | Duplicate | Matrix Spike | | Blank Spike/LCS | |
| ANALYTES | CAS Number | Screening Level | Screening Level Source[1] | Practical Quantitation Limit (PQL) | Method Detection Limit (MDL) | %R | RPD | %R | RPD | %R | RPD |
| 2,3,7,8-TCDF | 51207-31-9 | -- | -- | 10 | EDL[4] | -- -- | 30 | 73 - 150 | 20 | 73 - 150 | 20 |
| **Organic Acids (µg/L)** | | | | | | | | | | | |
| *EPA Method 8321A* | | | | | | | | | | | |
| 4-Chlorobenzenesulfonic acic | 98-66-8 | 33,400 | BCL | 1 | 0.097 | -- -- | 30 | 60 - 127 | 20 | 60 - 127 | 20 |
| 4-Bromobenzenesulfonic Acid (Surr) | 79326-93-5 | -- | -- | -- | -- | 63 - 123 | -- | -- -- | -- | -- -- | -- |
| *EPA Method 8270C* | | | | | | | | | | | |
| Phthalic Acid[6] | 88-99-3 | 66,700 | BCL | 400 | 5.84 | -- -- | 30 | -- -- | -- | -- -- | -- |
| 2-fluorobiphenyl (Surr) | 321-60-8 | -- | -- | -- | -- | 29 - 120 | -- | -- -- | -- | -- -- | -- |
| **Total Petroleum Hydrocarbons and Fuel Alcohols (mg/L)** | | | | | | | | | | | |
| *EPA Method 8015B* | | | | | | | | | | | |
| Gasoline Range Organics (C6-C10) | TPH-gasoline | -- | -- | 0.05 | 0.025 | -- -- | 30 | 65 - 140 | 20 | 80 - 120 | 20 |
| 4-Bromofluorobenzene (Surr) | 460-00-4 | -- | -- | -- | -- | 80 - 120 | -- | -- -- | -- | -- -- | -- |
| Diesel Range Organics (C10-C28) | TPH-disel | -- | -- | 0.05 | 0.025 | -- -- | 30 | 40 - 120 | 30 | 40 - 115 | 25 |
| Oil Range Organics (C29-C40) | TPH-diesel | -- | -- | 0.05 | 0.025 | -- -- | 30 | 40 - 120 | 30 | 40 - 115 | 25 |
| n-Octacosane (Surr) | 630-02-4 | -- | -- | -- | -- | 45 - 120 | -- | -- -- | -- | -- -- | -- |
| **Methane (mg/L)** | | | | | | | | | | | |
| *Method RSK 175* | | | | | | | | | | | |
| Methane (FID) | 74-82-8 | -- | -- | 0.000990 | 0.000250 | -- -- | 30 | -- -- | -- | 80 - 120 | 20 |
| Methane (TCD) | 74-82-8 | -- | -- | 1.00 | 0.500 | -- -- | 30 | -- -- | -- | 80 - 120 | 20 |
| **Others (µg/L)** | | | | | | | | | | | |
| *SM 2320B* | | | | | | | | | | | |
| Alkalinity as $CaCO_3$ | -- | -- | -- | 4000 | -- | -- -- | 30 | -- -- | -- | 80 - 120 | 20 |
| Bicarbonate as $HCO_3^-$ | -- | -- | -- | 4800 | -- | -- -- | 30 | -- -- | -- | -- -- | -- |

**TABLE 5. GROUNDWATER ANALYTES AND ANALYTICAL PERFORMANCE CRITERIA**
**QUALITY ASSURANCE PROJECT PLAN**
**Nevada Environmental Response Trust Site; Henderson, Nevada**

| | | | | | | QUALITY CONTROL LIMITS[2] | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | | | | Practical Quantitation Limit | Method Detection Limit | Surrogate | Duplicate | Matrix Spike | | | Blank Spike/LCS | | |
| ANALYTES | CAS Number | Screening Level | Screening Level Source[1] | (PQL) | (MDL) | %R | RPD | %R | | RPD | %R | | RPD |
| Carbonate as $CO_3^-$ | -- | -- | -- | 2400 | -- | -- -- | 30 | -- | -- | -- | -- | -- | -- |
| Hydroxide as $OH^-$ | -- | -- | -- | 1400 | -- | -- -- | 30 | -- | -- | -- | -- | -- | -- |
| *SM 4500-NH3 D* | | | | | | | | | | | | | |
| Ammonia | 7664-41-7 | 209 | BCL | 1200 | 600 | -- -- | 30 | 75 | - 125 | 15 | 85 | - 115 | 15 |
| *EPA Method 300.0* | | | | | | | | | | | | | |
| Bromide | 24959-67-9 | -- | -- | 500 | 250 | -- -- | 30 | 80 | - 120 | 20 | 90 | - 110 | 20 |
| Chloride | 16887-00-6 | 250,000 | 2nd MCL | 500 | 250 | -- -- | 30 | 80 | - 120 | 20 | 90 | - 110 | 20 |
| Fluoride | 16984-48-8 | 4,000 | MCL | 500 | 250 | -- -- | 30 | 80 | - 120 | 20 | 90 | - 110 | 20 |
| Total Nitrogen | 7727-37-9 | -- | -- | 260 | 70 | -- -- | 30 | 80 | - 120 | 20 | 90 | - 110 | 20 |
| Nitrate | 14797-55-8 | 10,000 | MCL | 110 | 55 | -- -- | 30 | 80 | - 120 | 20 | 90 | - 110 | 20 |
| Nitrite | 14797-65-0 | 1,000 | MCL | 150 | 70 | -- -- | 30 | 80 | - 120 | 20 | 90 | - 110 | 20 |
| Sulfate | 14808-79-8 | 250,000 | 2nd MCL | 500 | 250 | -- -- | 30 | 80 | - 120 | 20 | 90 | - 110 | 20 |
| Orthophosphate as $PO_4$ | 14265-44-2 | -- | -- | 500 | 250 | -- -- | 30 | 80 | - 120 | 20 | 90 | - 110 | 20 |
| *EPA Method 300.1* | | | | | | | | | | | | | |
| Chlorate | 7790-93-4 | -- | -- | 20 | 10 | -- -- | 30 | 75 | - 125 | 25 | 75 | - 125 | 25 |
| Chlorite | 14998-27-7 | 1000 | MCL | 20 | 10 | -- -- | 30 | 75 | - 125 | 25 | 85 | - 115 | 25 |
| *EPA Method 314.0* | | | | | | | | | | | | | |
| Perchlorate | 14797-73-0 | 23.4 | BCL | 4 | 0.95 | -- -- | 30 | 80 | - 120 | 20 | 85 | - 115 | 15 |
| *SM 2340C* | | | | | | | | | | | | | |
| Hardness as $CaCO_3$ | STL00009 | -- | -- | 0.004 | -- | -- -- | 30 | -- | -- | -- | -- | -- | -- |
| *EPA Method 351.2* | | | | | | | | | | | | | |
| Total Kjeldahl Nitrogen | 7727-37-9 | -- | -- | 200 | 100 | -- -- | 30 | 90 | - 110 | 20 | 90 | - 110 | 20 |
| *EPA Method 4500S-2 D* | | | | | | | | | | | | | |
| Sulfide | 18496-25-8 | -- | -- | 50 | 27 | -- -- | 30 | 70 | - 130 | 30 | 80 | 120 | 20 |
| Sulfide, Dissolved | 18496-25-8 | -- | -- | 50 | 27 | -- -- | 30 | 70 | - 130 | 30 | 80 | 120 | 20 |
| *EPA Method 9014B* | | | | | | | | | | | | | |
| Cyanide (total) | 57-12-5 | 200 | MCL | 25 | 12.5 | -- -- | 30 | 70 | - 115 | 15 | 90 | - 110 | 10 |
| *EPA Method 365.3* | | | | | | | | | | | | | |
| Phosphorus (total) | 7723-14-0 | 0.667 | BCL | 50 | 25 | -- -- | 30 | 75 | - 125 | 20 | 80 | - 120 | 20 |

**TABLE 5. GROUNDWATER ANALYTES AND ANALYTICAL PERFORMANCE CRITERIA**
**QUALITY ASSURANCE PROJECT PLAN**
**Nevada Environmental Response Trust Site; Henderson, Nevada**

| | | | | | | QUALITY CONTROL LIMITS[2] | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | | | Screening Level | Practical Quantitation Limit | Method Detection Limit | Surrogate | | Duplicate | Matrix Spike | | | Blank Spike/LCS | |
| ANALYTES | CAS Number | Screening Level | Source[1] | (PQL) | (MDL) | %R | | RPD | %R | | RPD | %R | RPD |
| *SM 5310B* | | | | | | | | | | | | | |
| Total Organic Carbon | 7440-44-0 | -- | -- | 1000 | 650 | -- | -- | 30 | 80 | - 120 | 20 | 90 - 110 | 20 |
| Dissolved Organic Carbon | 7440-44-0 | -- | -- | 1000 | 650 | -- | -- | 30 | 80 | - 120 | 20 | 90 - 110 | 20 |
| *SM 2540C* | | | | | | | | | | | | | |
| Total Dissolved Solids | 10-33-3 | 500,000 | 2nd MCL | 10000 | 5000 | -- | -- | 30 | -- | -- | -- | 90 - 110 | 10 |
| **Volatile Fatty Acids (mg/L)** | | | | | | | | | | | | | |
| *Lab SOP by Ion Chromatography SOP No. BF-MB-009, Rev 3* | | | | | | | | | | | | | |
| Acetic acid | 64-19-7 | -- | -- | 1.00 | 0.29 | -- | -- | 30 | 80 | - 120 | 20 | 80 - 120 | 20 |
| Formic-acid | 64-18-6 | -- | -- | 1.00 | 0.26 | -- | -- | 30 | 80 | - 120 | 20 | 80 - 120 | 20 |
| Lactic acid | 50-21-5 | -- | -- | 1.00 | 0.31 | -- | -- | 30 | 80 | - 120 | 20 | 80 - 120 | 20 |
| n-Butyric Acid | 107-92-6 | -- | -- | 1.00 | 0.26 | -- | -- | 30 | 80 | - 120 | 20 | 80 - 120 | 20 |
| Propionic acid | 79-09-4 | -- | -- | 1.00 | 0.35 | -- | -- | 30 | 80 | - 120 | 20 | 80 - 120 | 20 |
| Pyruvic Acid | 127-17-3 | -- | -- | 1.5 | 0.37 | -- | -- | 30 | 80 | - 120 | 20 | 80 - 120 | 20 |
| **Radionuclides (pCi/L)** | | | | | | | | | | | | | |
| *See Table 1 for Individual Methods* | | | | | | | | | | | | | |
| Radium-226 | 13982-63-3 | 5[7] | MCL | 1.00 | -- | -- | -- | 30 | 75 | - 138 | 40 | 68 - 137 | 40 |
| Radium-228 | 15262-20-1 | 5[7] | MCL | 1.00 | -- | -- | -- | 30 | 45 | - 150 | 40 | 56 - 140 | 40 |
| Thorium-228 | 14274-82-9 | 0.11 | Other | 1.00 | -- | -- | -- | 30 | 70 | - 130 | 40 | 70 - 130 | 40 |
| Thorium-230 | 14269-63-7 | 0.042 | Other | 1.00 | -- | -- | -- | 30 | 82 | - 139 | 40 | 81 - 125 | 40 |
| Thorium-232 | 7440-29-1 | 0.14 | Other | 1.00 | -- | -- | -- | 30 | 70 | - 130 | 40 | 70 - 130 | 40 |
| Uranium-234 | 13966-29-5 | 187,000 | Other | 1.00 | -- | -- | -- | 30 | 65 | - 146 | 40 | 84 - 120 | 40 |
| Uranium-235 | 15117-96-1 | 64.8 | Other | 1.00 | -- | -- | -- | 30 | -- | -- | 40 | -- -- | 40 |
| Uranium-238 | 7440-61-1 | 10.1 | Other | 1.00 | -- | -- | -- | 30 | 68 | - 143 | 40 | 83 - 121 | 40 |

**Notes:**

Shaded PQLs and MDLs exceed the lowest screening criteria.

-- = no value

µg/L = micrograms per liter

## TABLE 5. GROUNDWATER ANALYTES AND ANALYTICAL PERFORMANCE CRITERIA
## QUALITY ASSURANCE PROJECT PLAN
## Nevada Environmental Response Trust Site; Henderson, Nevada

| ANALYTES | CAS Number | Screening Level | Screening Level Source[1] | Practical Quantitation Limit (PQL) | Method Detection Limit (MDL) | QUALITY CONTROL LIMITS[2] | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|
| | | | | | | Surrogate | Duplicate | Matrix Spike | | Blank Spike/LCS | |
| | | | | | | %R | RPD | %R | RPD | %R | RPD |

pCi/L = picoCurie per liter

FID = flame ionization detector

TCD = thermal conductivity detector

(1) Groundwater screening levels were selected according to the following hierarchy of criteria:
- (a) Maximum Contaminant Level (MCL): Primary United States Environmental Protections Agency (USEPA) maximum contaminant level (USEPA 40 CFR Part 141).
- (b) Basic Contaminant Level (BCL): Residential water basic comparison levels in NDEP August 2013 BCL Spreadsheet (NDEP 2013).
- (c) Regional Screening Level (RSL): Tap water regional screening levels in USEPA Pacific Southwest, Region 9, Regional Screening Levels Chemical Specific Parameters table, Nov 2013. The screening levels were selected as the minimal values of carcinogenic screening level and noncarcinogenic screening level (USEPA 2013a).
- (d) 2nd Maximum Contaminant Level (2nd MCL): National Secondary Drinking Water Regulations (USEPA, 40 CFR Part 143).
- (e) Other criteria for radionuclides, including target activities for radium and thorium isotopes (NDEP, 2009) and for uranium isotopes (USEPA 2013b).

(2) QC Limits = Quality Control Limits for %R (Percent Recovery) of spiked compounds in Laboratory Control Samples (LCS) and surrogate compounds and Relative Percent Difference (RPD) between Matrix Spike (MS) and MS Duplicate (MSD) samples and LCS and LCS duplicate (LCSD) samples. Laboratory historical control limits are subject to change as a result of periodic re-evaluation. Limits in use at the time of sample analysis are available from the laboratory. Duplicate RPDs apply to sample duplicates and field duplicates.

(3) According to the laboratory's standard operating procedure (SOP) Arsenate (Arsenic V) is determined by calculating the difference between Total Inorganic Arsenic and Arsenic III.

(4) EDL = Estimated Detection Limit. For each dioxin, furan, or PCB not detected, an EDL is calculated. The sample specific EDL is an estimate made by the laboratory of the concentration of a given chemical that would have to be present to produce a signal with a peak height of at least 2.5 times the background signal level. The estimate is specific to a particular analysis of the sample and will be affected by sample size, dilution, and so forth. Because of the toxicological significance of dioxins, the EDL value is reported for non-detected chemicals rather than reporting the MDL.

(5) Dioxins and PCB congeners shall be reported to the estimated detection limit (EDL). Dioxin toxicity equivalents (TEQ) will be calculated for the 16 dioxin and furan congeners and 12 PCB congeners with toxicity equivalent factors (TEFs) defined by the World Health Organization (Van den Berg et al. 2006) substituting half of the EDL for the congeners not detected.

(6) Phthalic acid will be run with the other SVOCs by EPA Method 8270C.

(7) The screening level listed for Radium-226 and Radium-228 is the BCL for a combination of Radium-226 and Radium-228.

Sources:

NDEP. 2009b. Guidance for Evaluating Radionuclide Data, BMI Plant Sites and Common Areas Projects, Henderson, Nevada. February 6.

NDEP. 2013. User's Guide and Background Technical Document for NDEP Basic Comparison Levels (BCLs) for Human Health for the BMI Complex and Common Areas. Revision 12, August.

**TABLE 5. GROUNDWATER ANALYTES AND ANALYTICAL PERFORMANCE CRITERIA**
**QUALITY ASSURANCE PROJECT PLAN**
**Nevada Environmental Response Trust Site; Henderson, Nevada**

| ANALYTES | CAS Number | Screening Level | Screening Level Source[1] | Practical Quantitation Limit (PQL) | Method Detection Limit (MDL) | QUALITY CONTROL LIMITS[2] | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|
| | | | | | | Surrogate | Duplicate | Matrix Spike | | Blank Spike/LCS | |
| | | | | | | %R | RPD | %R | RPD | %R | RPD |


USEPA. 2013a. Regional Screening Levels (RSL) for Chemical Contaminants at Superfund Sites. November.

USEPA. 2013b. Preliminary Remediation Goals for Radionuclides. On-line calculator. http://epa-prgs.ornl.gov/cgi-bin/radionuclides/rprg_search

USEPA. National Primary Drinking Water Regulations. Code of Federal Regulations, 40 CFR Part 141.

USEPA. National Secondary Drinking Water Regulations. Code of Federal Regulations, 40 CFR Part 143.

Van den Berg et al., 2006. The 2005 World Health Organization Reevaluation of Human and Mammalian Toxic Equivalency Factors for Dioxins and Dioxin-Like Compounds. May 20.

## TABLE 6. FREQUENCY OF QA/QC SAMPLES
## QUALITY ASSURANCE PROJECT PLAN
## Nevada Environmental Response Trust; Henderson, Nevada

| SAMPLE TYPE | FREQUENCY OF ANALYSIS |
|---|---|
| **Contamination Control Samples** | |
| Laboratory Method Blank | One per each analytical method. One in every batch of samples (not to exceed 20 samples). |
| Trip Blank | One per cooler/shipment if VOCs are tested; analyze for VOCs only. |
| Equipment Blank | One per each analytical method. One in every batch of samples (not to exceed 20 samples). EBs will not be collected when dedicated single-use equipment is used for sample collection (e.g., new bailers and filters used to collect grab groundwater samples at boring locations). |
| Field Blank | One per each analytical method. One in every batch of samples (not to exceed 20 samples). FBs will not be collected from soil boring locations. |
| **Accuracy Control Samples** | |
| Laboratory Control Samples | One per each analytical method. One in every preparation batch (not to exceed 20 samples). |
| Surrogate Spiked Samples | For methods that use surrogate(s), the surrogate(s) will be spiked and analyzed in all samples and in all blanks. (1) |
| Matrix Spike Samples(2) | Analyzed in each batch, where applicable to the method (not to exceed 20 samples). |
| **Precision Control Samples** | |
| Field Duplicate Sample | One per each analytical method. One in every batch of samples collected (not to exceed 10 samples). |
| Laboratory Control Sample Duplicates | One per each analytical method. One in every preparation batch (not to exceed 20 samples). |
| Matrix Spike Duplicate Samples(2) | Analyzed in each batch, where applicable to the method (not to exceed 20 samples). |

**NOTE:**

(1) Not all methods use surrogates. See Tables 2, 3, 4, and 5 for specific surrogates to be used.

(2) Not all analytical methods or sample matrices have Matrix Spikes.

**TABLE 7. SAMPLE PRESERVATION, CONTAINERS, AND HOLDING TIMES**
**QUALITY ASSURANCE PROJECT PLAN**
**Nevada Environmental Response Trust; Henderson, Nevada**

| MATRIX | ANALYTES | ANALYTICAL METHOD | PRESERVATION | CONTAINER[1][2] | TAT | HOLD TIME[3] Prior to Extraction | HOLD TIME[3] After Extraction |
|---|---|---|---|---|---|---|---|
| Water | Sulfide | EPA Method 4500S-2 D | 4 drops of $2NZn(C_2H_3O_2)_2$ and NaOH to pH >9; cool to ≤6˚C | 500 mL HDPE | 10d | 7d | |
| Water | Total Dissolved Solids (TDS) | SM 2540C | Cool to ≤6 °C | 500 mL HDPE | 10d | 7d | |
| Water | Total and/or Dissolved Organic Carbon | SM 5310B | HCl to pH <2; cool to ≤6 ˚C | 1 x 1 L amber glass with Teflon-lined lids | 10d | 28d | |
| Water | Radium 226 | EPA Method 903.0 | None | 2 x 1 L HDPE | 22d | 180d | |
| Water | Radium 228 | EPA Method 904.0 | None | 2 x 1 L HDPE | 22d | 180d | |
| Water | Thorium 228, 230, 232 and Uranium 234, 235, and 238 | DOE EML HASL 300 A-01-R (alpha spectroscopy) | None | 500 mL HDPE | 22d | 180d | |
| Soil | Metals | EPA Method 6010 | Cool to ≤6 °C | 1 X 4 oz glass jar with Teflon-lined cap | 10d | 180d | |
| Soil | Metals | EPA Method 6020 | Cool to ≤6 °C | 1 X 4 oz glass jar with Teflon-lined cap | 10d | 180d | |
| Soil | Rare Earth Metals[4] | EPA Method 6020A | Cool to ≤6 °C | 1 X 2 oz glass jar with Teflon-lined cap | 11d | 180d | |
| Soil | Hexavalent chromium | EPA Method 7199 | Cool to ≤6 °C | 1 X 4 oz glass jar with Teflon-lined cap | 10d | 30d to digestion; 7d from digestion to analysis | |
| Soil | Mercury | EPA Method 7471A | Cool to ≤6 °C | 1 X 4 oz glass jar with Teflon-lined cap | 10d | 28d | |
| Soil | Volatile Organic Compounds (VOCs) | EPA Method 8260B and 8260 SIM | Cool to ≤6 °C | Preserved in Accordance with EPA Method 5035 (3x 40 mL glass vials w/ $H_2O$, 1x 40mL glass vial w/ MeOH) | 10d | Frozen or preserved within 48h of collection, 14d from preservation to analysis | |

**TABLE 7. SAMPLE PRESERVATION, CONTAINERS, AND HOLDING TIMES**
**QUALITY ASSURANCE PROJECT PLAN**
**Nevada Environmental Response Trust; Henderson, Nevada**

| | | | | | | HOLD TIME[3] | |
|---|---|---|---|---|---|---|---|
| MATRIX | ANALYTES | ANALYTICAL METHOD | PRESERVATION | CONTAINER[1][2] | TAT | Prior to Extraction | After Extraction |
| Soil | Semivolatile Organic Compounds (SVOCs) and Phthalic Acid | EPA Method 8270C | Cool to ≤6 °C | 1 X 8 oz glass jar with Teflon-lined cap | 10d | 14d | 40d |
| Soil | Formaldehyde | EPA Method 8315A | Cool to ≤6 °C | 1 X 8 oz glass jar with Teflon-lined cap | 10d | 7d | 3d |
| Soil | Polyaromatic Hydrocarbons (PAHs) | EPA Method 8270 SIM | Cool to ≤6 °C | 1 X 8 oz glass jar with Teflon-lined cap | 10d | 14d | 40d |
| Soil | Organophosphorus Pesticides | EPA Method 8141A | Cool to ≤6 °C | 1 X 8 oz glass jar with Teflon-lined cap | 10d | 14d | 40d |
| Soil | Organochlorine Pesticides | EPA Method 8081A | Cool to ≤6 °C | 1 X 8 oz glass jar with Teflon-lined cap | 10d | 14d | 40d |
| Soil | PCBs as Congeners | EPA Method 1668A | ≤6 °C, from field, lab storage < -10 °C | 1 X 8 oz glass jar with Teflon-lined cap | 20d | 1y | 45d[8] |
| Soil | PCBs as Aroclors | EPA Method 8082 | Cool to ≤6 °C | 1 X 8 oz glass jar with Teflon-lined cap | 10d | 14d | 40d |
| Soil | Dioxins/Furans | EPA Method 8290 or 8280(7) | Cool to ≤6 °C | 1 X 8 oz glass jar with Teflon-lined cap | 15d | 30d[8] | 45d[8] |
| Soil | Gasoline Range Organics (GROs) | EPA Method 8015B | Cool to ≤6 °C | Preserved in Accordance with EPA Method 5035 | 10d | Frozen or preserved within 48h of collection, 14d from preservation to analysis | |
| Soil | Diesel/Oil Range Organics (DROs/OROs) | EPA Method 8015B | Cool to ≤6 °C | 1 X 8 oz glass jar with Teflon-lined cap | 10d | 14d | 40d |
| Soil | Alkalinity and Carbonate | SM 2320B | Cool to ≤6 °C | 1 X 4 oz glass jar with Teflon-lined cap | 10d | None for soil. Use water holding time for leachates. | |
| Soil | Ammonia | SM 4500-NH3 D | Cool to ≤6 °C | 1 X 4 oz glass jar with Teflon-lined cap | 10d | None for soil. Use water holding time for leachates. | |

**TABLE 7. SAMPLE PRESERVATION, CONTAINERS, AND HOLDING TIMES**
**QUALITY ASSURANCE PROJECT PLAN**
**Nevada Environmental Response Trust; Henderson, Nevada**

| MATRIX | ANALYTES | ANALYTICAL METHOD | PRESERVATION | CONTAINER[1][2] | TAT | HOLD TIME[3] Prior to Extraction | HOLD TIME[3] After Extraction |
|---|---|---|---|---|---|---|---|
| Soil | Inorganic Anions[5] | EPA Method 300.0 | 4 °C | 1 X 4 oz glass jar with Teflon-lined cap | 10d | None for soil. Use water holding time for leachates. | |
| Soil | Chlorate | EPA Method 300.1 | Cool to ≤6 °C | 1 X 4 oz glass jar with Teflon-lined cap | 10d | None for soil. Use water holding time for leachates. | |
| Soil | Perchlorate | EPA Method 314.0 | Cool to ≤6 °C | 1 X 4 oz glass jar with Teflon-lined cap | 10d | 28d | |
| Soil | Cyanide | EPA Method 9014B | Cool to ≤6 °C | 1 X 4 oz glass jar with Teflon-lined cap | 10d | 14d | |
| Soil | pH | EPA Method 9045C | Cool to ≤6 °C | 1 X 4 oz glass jar with Teflon-lined cap | 10d | Immediate | |
| Soil | Specific Conductance | EPA Method 120.1 / SM 2510B | Cool to ≤6 °C | 1 X 4 oz glass jar with Teflon-lined cap | 10d | None for soil. Use water holding time for leachates. | |
| Soil | Surfactants | SM 5540C | Cool to ≤6 °C | 1 X 4 oz glass jar with Teflon-lined cap | 10d | None for soil. Use water holding time for leachates. | |
| Soil | Total and Dissolved Organic Carbon | SM 5310B | Cool to ≤6 °C | 1 X 4 oz glass jar with Teflon-lined cap | 10d | 28d | |
| Soil | Radium 226 | EPA Method 903.0 | None | 1 X 500 mL HDPE | 22d | 180d | |
| Soil | Radium 228 | EPA Method 904.0 | None | 1 X 500 mL HDPE | 22d | 180d | |
| Soil | Thorium 228, 230, 232 and Uranium 234, 235, and 238 | DOE EML HASL 300 A-01-R (alpha spectroscopy) | None | 1 X 50 mL HDPE | 22d | 180d | |
| Soil | Asbestos | EPA Method 540-R-97-028 modified per Berman & Kolk (2000) | None | 1 X 250 mL glass with Teflon-lined cap | 30d | None established for soil. | |

**TABLE 7. SAMPLE PRESERVATION, CONTAINERS, AND HOLDING TIMES**
**QUALITY ASSURANCE PROJECT PLAN**
**Nevada Environmental Response Trust; Henderson, Nevada**

| | | | | | | HOLD TIME[3] | |
|---|---|---|---|---|---|---|---|
| **MATRIX** | **ANALYTES** | **ANALYTICAL METHOD** | **PRESERVATION** | **CONTAINER[1][2]** | **TAT** | **Prior to Extraction** | **After Extraction** |
| Soil Gas | Volatile Organic Compounds (VOCs) | EPA Method TO-15 | None | SUMMA canister | 5d | 30d | |
| Soil Gas | Helium | ASTM D1946 | None | SUMMA canister | 5d | 30d | |

**Notes:**

ASTM = American Society for Testing and Materials
DOE = Department of Energy
HDPE = high-density polyethylene
HASL = Health and Safety Laboratory
EML = Environmental Measurements Laboratory
EPA = United States Environmental Protection Agency
KPA = Kinetic Phosphorescense Analyzer
SIM = Single Ion Monitoring
SM = Standard Method
TAT = Turnaround Time

EDA = Ethylene Diamine
HCL = Hydrochloric Acid
$H_2SO_4$ = Sulfuric Acid
$HNO_3$ = Nitric Acid
NaOH = Sodium Hydroxide

d = day(s)
h = hours
mL = milliliters
L = liter
oz = ounces
y = year

(1) Additional volume will be collected for MS/MSD samples.
(2) Laboratory may provide alternate containers as long as the containers meet the requirements of the method and allow the collection of sufficient volume to perform the analysis.
(3) Holding time begins from date of sample collection. Leachate holding times must conform to water holding time or the requirements of EPA Method 1312.
(4) Niobium, palladium, sulfur and/or total uranium
(5) Fluoride, chloride, bromide, sulfate, ortho-phosphate as $PO_4$, nitrite, and nitrate.
(6) 28 days for fluoride, chloride, bromide, and sulfate; 48 hours for nitrate, nitrate, and orthophosphate
(7) With proper storage, hold times for unextracted and extracted PCBs and dioxins/furans can be extended to one year. The hold times listed here correspond to those listed in the laboratory's standard operating procedure (SOP).

Immediate means within 15 minutes from sampling or field test

**TABLE 8. CALIBRATION AND MAINTENANCE OF FIELD EQUIPMENT**
**QUALITY ASSURANCE PROJECT PLAN**
**Nevada Environmental Response Trust; Henderson, Nevada**

| INSTRUMENT | TASK | FREQUENCY |
|---|---|---|
| Organic Vapor Meter OVM[1] | (a) Inspect and calibrate<br>(b) Charge batteries | (a) Daily<br>(b) Each night prior to operation |
| Particulate monitor[2] | (a) Inspect and calibrate<br>(b) Charge batteries | (a) Daily<br>(b) Each night prior to operation |
| Asbestos monitor[3] | (a) Inspect and calibrate<br>(b) Charge batteries | (a) Daily<br>(b) Each night prior to operation |
| Conductivity, Dissolved Oxygen (DO), Oxygen Reduction Potential (ORP), pH, and Temperature Meter[4] | (a) Inspect and calibrate<br>(b) Test batteries | (a) Daily<br>(b) Each night prior to operation |
| Turbidity Meter[5] | (a) Inspect and calibrate<br>(b) Test batteries | (a) Daily<br>(b) Each night prior to operation |
| Alkalinity Test Kit[6] | (a) Inspect kit integrity | (a) Daily prior to testing |
| Ferrous Iron Test Kit[7] | (a) Inspect kit integrity | (a) Daily prior to testing |
| Sulfide Test Kit[8] | (a) Inspect kit integrity | (a) Daily prior to testing |
| Water Level Indicator[9] | (a) Inspect<br>(b) Test batteries<br>(c) Calibrate | (a) Daily<br>(b) Each night prior to operation<br>(c) Annually with steel tape |
| Low flow adjustable-rate sampling pump[10] | (a) Change bladder<br>(b) Change tubing[11] | (a) Each sample location<br>(b) Each sample location |
| Low flow adjustable-rate sampling pump | (a) Inspect<br>(b) Calibrate | (a) Individually prior to operation<br>(b) Factory calibrated prior to shipment to site |
| Pressure Transducers[12] | (a) Inspect data log<br>(b) Check batteries and o-rings<br>(c) Perform depth and drift tests<br>(d) Calibrate | (a) Daily<br>(b) Prior to installation<br>(c) Prior to installation<br>(d) Factory calibrated prior to shipment to site |

**Notes:**

(1) MiniRAE 2000 Photoionization Detector (PID) with 10.6 eV lamp or similar

(2) DataRAM pDR-1000AN or similar

(3) Gilian BDX II Personal Abatement Air Sampler or similar

(4) YSI 556 MPS or similar

(5) HACH 2100P Turbidity Meter or similar

(6) HACH Digital Titrator or similar

(7) HACH, CHEMetrics, or similar. Method based on ASTM D 1068-77.

(8) HACH, CHEMetrics, or similar. Method based on USEPA Method 376.2 and Apha Method 4500-$S^2$-D.

(9) Solinst Water Level Indicator or similar having gradations marked at 0.01-foot intervals.

(10) QED Sample Pro or similar

(11) Teflon® or Teflon®-lined

(12) In Situ Level Troll 500 vented water level/temperature monitor or similar.

**TABLE 9. ANALYTICAL LABORATORY CALIBRATION FREQUENCIES**
**QUALITY ASSURANCE PROJECT PLAN**
**Nevada Environmental Response Trust; Henderson, Nevada**

| | | QUALITY CONTROL CHECK[1] | |
|---|---|---|---|
| **LABORATORY ANALYSIS** | **ANALYTICAL METHOD** | **Initial Calibration Type/Frequency** | **Continuing Calibration Type/Frequency** |
| Volatile Organic Compounds (VOCs) by EPA 8260B | Gas Chromatography/ Mass Spectroscopy | Minimum five points on an as needed basis with daily verification before sample analysis. | Standard analyzed at the beginning of every sequence. |
| Semivolatile Organic Compounds (SVOCs) by EPA Method 8270C | Gas Chromatography/ Mass Spectroscopy | Minimum five points on an as needed basis with daily verification before sample analysis. | Standard analyzed at beginning of the sequence. |
| Organochlorine Pesticides by EPA Method 8081A | Gas Chromatography | Minimum five point calibration daily prior to analysis. | Standard analyzed prior to each 12-hour shift, at least once every 20 samples, and at the end of the sequence. |
| PCBs as Aroclors by EPA Method 8082 | Gas Chromatography | Seven point calibration on an as needed basis with daily verification before sample analysis. | Standard analyzed prior to each 12-hour shift, at least once every 20 samples, and at the end of the sequence. |
| Gasoline Range Organics by EPA Method 8015B | Gas Chromatography | Minimum five point calibration daily prior to analysis. | Standard analyzed after every 10 sample injections or 12 hours, which ever is sooner and at the end of the sequence. |
| Diesel Range Organics by EPA Method 8015B | Gas Chromatography | Minimum five point calibration daily prior to analysis. | Standard analyzed prior to each 12-hour shift, at least once every 20 samples, and at the end of the sequence. |
| Metals by EPA Method 6010B | Inductively Coupled Plasma Atomic Emission Spectroscopy | Minimum two point and a blank calibration daily prior to analysis. | Standard analyzed at a minimum after every 10 samples and end of the sequence. |
| Metals by EPA Method 6020 | Inductively Coupled Plasma/ Mass Spectroscopy | Four point (three standard + blank) calibration daily prior to analysis. | Standard analyzed after every 10 samples. |
| Rare Earth Metals by EPA Method 6020A | Inductively Coupled Plasma/ Mass Spectroscopy | Four point (three standard + blank) calibration daily prior to analysis. | Standard analyzed after every 10 samples. |
| PCBs as Congeners by EPA Method 1668A | High-Resolution Gas Chromatography/High-Resolution Mass Spectrometry | Minimium five point calibration daily prior to analysis. | Standard analyzed at the beginning of and after each 12-hour shift. |
| Organophosphorus Pesticides by EPA Method 8141A | High-Resolution Gas Chromatography/High-Resolution Mass Spectrometry | Minimium five point calibration daily prior to analysis. | Standard analyzed at the beginning of and after each 12-hour shift. |
| Dioxins/Furans by EPA Method 8290 | High-Resolution Gas Chromatography/High-Resolution Mass Spectrometry | Five point calibration daily prior to analysis. | Standard analyzed at the beginning of and after each 12-hour shift. |
| Mercury by EPA Method 7471A and 7470A | Cold-Vapor Atomic Absorption Spectroscopy | Minimum three points plus a blank daily prior to analysis | Standard analyzed after every 10 samples and end of the sequence. |
| Inorganic Anions by EPA Method 300.0 and 300.1 | Ion Chromatography | Minimum three points plus a blank on an as needed basis with daily verification before sample analysis. | Standard analyzed after every 10 samples and end of sequence. |

**TABLE 9. ANALYTICAL LABORATORY CALIBRATION FREQUENCIES**
**QUALITY ASSURANCE PROJECT PLAN**
**Nevada Environmental Response Trust; Henderson, Nevada**

| | | QUALITY CONTROL CHECK[1] | |
|---|---|---|---|
| **LABORATORY ANALYSIS** | **ANALYTICAL METHOD** | **Initial Calibration Type/Frequency** | **Continuing Calibration Type/Frequency** |
| Hexavalent Chromium by EPA Method 7199 | Ion Chromatography | Minimum three points plus a blank on an as needed basis with daily verification before sample analysis. | Standard analyzed at least once every 10 samples and end of the sequence. |
| Perchlorate by EPA Method 314.0 | Ion Chromatography | Minimum five points plus a blank on an as needed basis with daily verification before sample analysis. | Standard analyzed after every 10 samples and end of the sequence. |
| Volatile Fatty Acids by Lab SOP by Ion Chromatography | Ion Chromatography | Minimum five points plus a blank at a minimum of once every six months. | Standard analyzed at least once every 10 samples and end of the sequence. |
| Total Kjedahl Nitrogen by EPA Method 351.2 | Spectroscopy | Minimum three points plus a blank on an as needed basis with daily verification before sample analysis. | Standard analyzed at least once every 10 samples and end of the sequence. |
| Surfactants by SM 5540C | Spectroscopy | Minimum five points plus a blank on an as needed basis with daily verification before sample analysis. | Standard analyzed at least once every 10 samples and end of the sequence. |
| Phosphorus by EPA Method 365.3 | Spectroscopy | Minimum three points plus a blank on an as needed basis with daily verification before sample analysis. | Standard analyzed at least once every 10 samples and end of the sequence. |
| Cyanide by EPA Method 9014B | Spectroscopy | Minimum three points plus a blank on an as needed basis with daily verification before sample analysis. | Standard analyzed at least once every 10 samples and end of the sequence. |
| Sulfide by EPA Method 4500S-2 D | Spectroscopy | Minimum six points plus a blank on an as needed basis with daily verification before sample analysis. | Standard analyzed at least once every 10 samples and end of the sequence. |
| Alkalinity by SM 2320B | Titration | Minimum three points on an as needed basis with daily verification before sample analysis. | Standard analyzed at least once every 10 samples and end of the sequence. |
| 4-chlorobenzenesulfonic acid by EPA 8321A | Gas Chromatography/ Mass Spectroscopy | Minimum five point calibration daily prior to analysis. | Standard analyzed at the beginning of and after each 12-hour shift. |
| Formaldehyde by EPA Method 8315A | High-Performance Liquid Chromatography-Ultraviolet Detection | Minimum five point calibration daily prior to analysis. | Standard analyzed at least once every 10 samples, not to exceed 12 hours, and end of the sequence. |
| Specific Conductance by EPA Method 120.1 | Conductivity Bridge with platinum electrode | Two point calibration daily prior to analysis | Standard analyzed after every 10 samples and end of the sequence. |
| Ammonia by SM 4500-$NH_3$ | Determined Potentiometrically with an Ion Selective Ammonia Electrode | Minimum five points plus a blank on an as needed basis with daily verification before sample analysis. | Standard analyzed at least once every 10 samples and end of the sequence. |
| Total Organic Carbon and Dissolved Organic Carbon | Non-Dispersive Infrared Analyzer | Minimum three points plus a blank on an as needed basis with daily verification before sample analysis. | Standard analyzed daily. |

**TABLE 9. ANALYTICAL LABORATORY CALIBRATION FREQUENCIES**
**QUALITY ASSURANCE PROJECT PLAN**
**Nevada Environmental Response Trust; Henderson, Nevada**

| | | QUALITY CONTROL CHECK[1] | |
|---|---|---|---|
| **LABORATORY ANALYSIS** | **ANALYTICAL METHOD** | **Initial Calibration Type/Frequency** | **Continuing Calibration Type/Frequency** |
| pH by EPA Method 9045C | Electrometric | Standard analyzed on an as needed basis with daily verification before sample analysis. | Standard analyzed after every 10 samples and end of the sequence. |
| Radium 226 by EPA Method 903.0 | Gamma Spectroscopy | Annual calibration against standards with daily verification before sample analysis. | Source standard analyzed daily. |
| Radium 228 by EPA Method 904.0 | Gamma Spectroscopy | Annual calibration against standards with daily verification before sample analysis. | Source standard analyzed daily. |
| Uranium 234, 235, 238, and Thorium 228, 230, 232 by Method HASL 300 modified | Alpha Spectroscopy | Annual calibration against standards with daily verification before sample analysis. | Source standard analyzed daily. |

**Notes:**

ASTM = American Society for Testing and Materials

EPA = United States Environmental Protection Agency

HASL = Health and Safety Laboratory

KPA = Kinetic Phosphorescense Analyzer

SM = Standard Method

(1) These Quality Control checks are to be considered the minimum frequency and scope of checks and calibrations to be performed. Laboratories may have more stringent requirements as part of their Standard Operating Procedures.

**TABLE 10. DATA VALIDATION QUALIFIERS AND REASON CODES**
**QUALITY ASSURANCE PROJECT PLAN**
**Nevada Environmental Response Trust; Henderson, Nevada**

**Data Validation Codes for Organics**

| Qualifier | Definition |
|---|---|
| U | The analyte was analyzed for, but was not detected above the level of the reported sample quantitation limit. |
| J | The result is an estimated quantity. The associated numerical value is the approximate concentration of the analyte in the sample. |
| J+ | The result is an estimated quantity, but the result may be biased high. |
| J- | The result is an estimated quantity, but the result may be biased low. |
| NJ | The analyte has been “tentatively identified” or “presumptively” as present and the associated numerical value is the estimated concentration in the sample. |
| UJ | The analyte was analyzed for, but was not detected. The reported quantitation limit is approximate and may be inaccurate or imprecise. |
| R | The data are unusable. The sample results are rejected due to serious deficiencies in meeting QC criteria. The analyte may or may not be present in the sample. |
| C | The target Pesticide or Aroclor analyte identification has been confirmed by Gas Chromatography/Mass Spectrometry (GC/MS). |
| X | The target Pesticide or Aroclor analyte identification was not confirmed when GC/MS analysis was performed. |

**Data Validation Codes for Inorganics**

| Qualifier | Definition |
|---|---|
| U | The analyte was analyzed for, but was not detected above the level of the reported sample quantitation limit. |
| J | The result is an estimated quantity. The associated numerical value is the approximate concentration of the analyte in the sample. |
| J+ | The result is an estimated quantity, but the result may be biased high. |
| J- | The result is an estimated quantity, but the result may be biased low. |
| UJ | The analyte was analyzed for, but was not detected. The reported quantitation limit is approximate and may be inaccurate or imprecise. |
| R | The data are unusable. The sample results are rejected due to serious deficiencies in meeting QC criteria. The analyte may or may not be present in the sample. |

**Data Validation Reason Codes**

| Reason | Explanation |
|---|---|
| a | qualified due to low abundance (radiochemical activity) |
| be | qualified due to equipment blank contamination |
| bf | qualified due to field blank contamination |
| bl | qualified due to lab blank contamination |
| bt | qualified due to trip blank contamination |
| bp | qualified due to pump blank contamination (wells w/o dedicated pumps, when contamination is detected in the Pump Blk) |
| br | qualified due to filter blank contamination (aqueous Hexavalent Chromium and Dissolved sample fractions) |
| c | qualified due to calibration problems |
| cp | qualified due to insufficient ingrowth (radiochemical only) |
| dc | duel column confirmation %D exceeded |
| e | concentration exceeded the calibration range |

**Data Validation Reason Codes**

| Reason | Explanation |
|---|---|
| fd | qualified due to field duplicate imprecision |
| h | qualified due to holding time exceedance |
| i | qualified due to internal standard areas |
| k | qualified as Estimated Maximum Possible Concentrations (dioxins and PCB congeners) |
| l | qualified due to LCS recoveries |
| ld | qualified due to lab duplicate imprecision (matrix duplicate, MSD, LCSD) |
| m | qualified due to matrix spike recoveries |
| nb | qualified due to negative lab blank contamination (nondetect results only) |
| nd | qualified due to non-detected target analyte |
| o | other |
| p | qualified due to quantitation during shipping |
| pH | sample preservation not within acceptance range |
| q | qualified due to quantitation problem |
| s | qualified due to surrogate recoveries |
| sd | serial dilution did not meet control criteria |
| sp | detected value report >SQL <PQL |
| st | sample receipt temperature exceeded |
| t | qualified due to elevated helium tracer concentrations |
| vh | volatile headspace detected in aqueous sample containers submitted for VOC analysis |
| x | qualified due to low % solids |
| z | qualified due to ICS results |

**Sources:**

USEPA. 2017. National Functional Guidelines for Organic Superfund Data Review. OLEM 9355.0-136. EPA-540-R-2017-002. January.
USEPA. 2017. National Functional Guidelines for Inorganic Superfund Data Review. OLEM 9355.0-136. EPA-540-R-2017-001. January.

# APPENDIX A
# PROJECT ORGANIZATION/ROLES AND RESPONSIBILITIES

## Appendix A. QAPP Project Organization / Roles and Responsibilities

| Organization | Name | Project Role / Title |
|---|---|---|
| **Nevada Division of Environmental Protection** | Weiquan Dong, PhD | NDEP Remedial Project Manager |
| **Nevada Environmental Response Trust** | Steve Clough, PG, CEM | NERT Remediation Director |
| **Ramboll Environ** | John M. Pekala, PG, CEM | Project Manager |
| **Ramboll Environ** | Ross Russell, PG | Project Quality Assurance/Quality Control Officer |
| **Ramboll Environ** | Greg Kinsall, PG | Field Task Leader |
| **Ramboll Environ** | Elizabeth Miesner, MPH | Health Risk Assessment Task Leader |
| **Ramboll Environ** | Craig Knox | Analytical Task Leader |
| **Tetra Tech** | Dan Pastor, PE | Project Manager |
| **Tetra Tech** | Gina Heaton | Project Quality Assurance/Quality Control Officer |
| **Tetra Tech** | Kyle Hansen, CEM | Field Task Leader |
| **Tetra Tech** | Valerie Bogle | Database Administrator |
| **Tetra Tech** | Michael Wilson | Data Validation Coordinator |
| **AECOM** | Sally Bilodeau, PG, CEM | Project Manager |
| **AECOM** | Leta Maclean, CHMM | Project Quality Assurance/Quality Control Officer |
| **AECOM** | Carmen Caceres-Schnell | Field Task Leader – Subsurface Investigation |
| **AECOM** | Kristen Durocher | Field Task Leader – Surface Water |
| **AECOM** | Chad Roper, PhD | Analytical Task Leader/ Database Administrator |
| **TestAmerica Laboratories** | Patty Mata | Laboratory Project Manager |
| **EMSL Analytical Inc.** | Daniel Kocher | Laboratory Project Manager |
| **Laboratory Data Consultants** | Stella Cuenco | Data Validation Project Manager |
| **Neptune and Company** | Patti Meeks | Data Validation Project Manager |

# APPENDIX B
# LABORATORY QUALITY ASSURANCE MANUALS (QAMs)

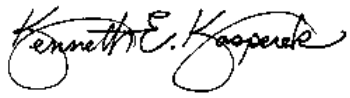
# Cover Page:

# Quality Assurance Manual

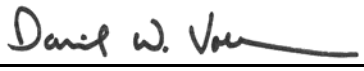
**TestAmerica Buffalo**
**10 Hazelwood Drive**
**Amherst, New York 14228**
**716.504.9800**
**716.691.7991**

**www.testamericainc.com**

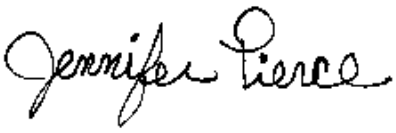
# Title Page:
# Quality Assurance Manual
# Approval Signatures



Laboratory Director – Kene' Kasperek — 10/05/2016 Date



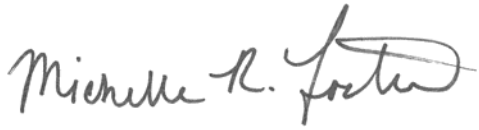
Quality Assurance Manager - Daniel Vollmer — 10/12/2016 Date



Inorganics Operations Manager – Jennifer Pierce — 10/04/2016 Date

Organic Operations Manager – Gary Rudz — 10/04/2016 Date

Organic Preparation Manager – Vikrambhai Patel — 10/07/2016 Date



Wet Chemistry Manager – Michelle Foster — 10/04/2016 Date

Metals Manager – Todd Brandt

10/04/2016
Date

GC/MS Semivolatiles – Michelle Page

10/07/2016
Date

GC/MS Volatiles – Leah Hill

10/11/2016
Date

Facilities Manager – Ken Kinecki

10/05/2016
Date

## SECTION 2

## TABLE OF CONTENTS

## LIST OF TABLES

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# LIST OF APPENDICES



# REFERENCED CORPORATE SOPs AND POLICIES

| SOP / Policy Reference | Title |
|---|---|
| CA-I-P-002 | Electronic Reporting and Signature Policy |
| CA-L-P-002 | Contract Compliance Policy |
| CW-L-S-004 | Subcontracting Procedures |
| CA-Q-M-002 | Corporate Quality Management Plan |
| CA-Q-S-001 | Solvent and Acid Lot Testing and Approval |
| CA-Q-S-002 | Acceptable Manual Integration Practices |
| CA-Q-S-006 | Detection Limits |
| CA-Q-S-009 | Root Cause Analysis |
| CA-T-P-001 | Qualified Products List |
| CW-E-M-001 | Corporate Environmental Health & Safety Manual |
| CW-F-P-002 | Company-Wide Authorization Matrix |
| CW-F-P-004 | Procurement and Contracts Policy |
| CW-F-S-007 | Capital Expenditure, Controlled Purchase Requests and Fixed Asset Capitalization |
| CW-L-P-004 | Ethics Policy |
| CW-L-S-002 | Internal Investigation |
| CW-Q-S-001 | Corporate Document Control and Archiving |
| CW-Q-S-002 | Writing a Standard Operating Procedure (SOPs) |
| CW-Q-S-003 | Internal Auditing |
| CW-Q-S-004 | Management Systems Review |
| CW-Q-S-005 | Data Recall Process |
| CA-C-S-001 | Work Sharing Process |

## REFERENCED LABORATORY SOPs

| SOP Reference | Title |
|---|---|
| BF-GP-001 | Calibration of Autopipettes and Repipetters |
| BF-GP-002 | Support Equipment: Maintenance, Record Keeping and Corrective Actions |
| BF-GP-005 | Sample Homogenization and Subsampling |
| BF-GP-012 | Technical Data Review |
| BF-GP-013 | Manual Integration |
| BF-GP-015 | Record Storage and Retention |
| BF-GP-018 | Strict Internal Chain or Custody |
| `BF-GP-019 | Standard Traceability and Preparation |

| | |
|---|---|
| BF-GP-020 | Thermometer Calibration |
| BF-PM-001 | Project Information Requirements |
| BF-PM-003 | Bottle Order Set-up |
| BF-PM-005 | Correctness of Analysis |
| BF-PM-008 | Massachusetts DEP Notification Procedures |
| BF-QA-001 | Determination of Method Detection Limits |
| BF-QA-002 | Quality Control Limits |
| BF-QA-003 | Procedure for Writing, Reviewing and Revising Controlled Documents |
| BF-QA-004 | Laboratory Personnel Training |
| BF-QA-005 | Preventative and Corrective Action |
| BF-QA-006 | Data Quality Review |
| BF-SR-001 | Cooler Shipping - Bottle Kits and Samples |
| BF-SR-002 | Receipt of Analytical Samples |

- The full list of Laboratory SOPs is maintained in the Quality Assurance Department
- The full list of analytical methods performed in the Laboratory is can be exported from the Laboratory Information Management System's Total Access Database

# SECTION 3

# INTRODUCTION, SCOPE AND APPLICABILITY

## 3.1 INTRODUCTION AND COMPLIANCE REFERENCES

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

The QAM has been prepared to assure compliance with 2003 National Environmental Laboratory Accreditation Conference (NELAC) standards, The NELAC Institute (TNI) Standard, dated 2009, Volume 1 Modules 2 and 4, and ISO/IEC Guide 17025(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.

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

- ANSI/ASQC, E4-1994, "Specifications and Guidelines for Quality Management Systems for Environmental Data Collection and Environmental Technology Programs" (American National Standard, January 5, 1995, or most recent version)
- "EPA Requirements for Quality Management Programs" (QA/R-2) (EPA/240/B-01/002, May 31, 2006).
- EPA 600/4-88/039, *Methods for the Determination of Organic Compounds in Drinking Water,* EPA, Revised July 1991.
- EPA 600/R-95/131, *Methods for the Determination of Organic Compounds in Drinking Water,* Supplement III, EPA, August 1995.
- EPA 600/4-79-019, *Handbook for Analytical Quality Control in Water and Wastewater Laboratories,* EPA, March 1979.
- Test Methods for Evaluating Solid Waste Physical/Chemical Methods (SW846), Third Edition September 1986, Final Update I, July 1992, Final Update II A, August 1993, Final Update II, September 1994; Final Update IIB, January 1995; Final Update III, December 1996; Final Update IV, January 2008.
- Federal Register, 40 CFR Parts 136, 141, 172, 173, 178, 179 and 261. New York State Analytical Services Protocol, July 2005
- Manual for the Certification of Laboratories Analyzing Drinking Water (EPA 815-R-05-004, January 2005).
- 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,* 18th Edition, 19th, 20th, and on-line Editions. 21st.

- *U.S. Department of Energy* Order 414.1B, *Quality Assurance*, Approved April 29, 2004.
- *U.S. Department of Energy* Order 414.1C, *Quality Assurance*, June 17, 2005.
- *U.S. Department of Energy Order 414.1D, Quality Assurance, Aril, 25, 2011.*
- Toxic Substances Control Act (TSCA).

## 3.2 TERMS AND DEFINITIONS

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

Refer to Appendix 2 for the Glossary/Acronyms.

## 3.3 SCOPE / FIELDS OF TESTING

The laboratory analyzes a broad range of environmental and industrial samples every month. Sample matrices vary among air, drinking water, effluent water, groundwater, hazardous waste, sludge and soils. The Quality Assurance Program contains specific procedures and methods to test samples of differing matrices for chemical, physical and biological parameters. The Program also contains guidelines on maintaining documentation of analytical 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 Section 19.0. 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/Manager and the Quality Assurance (QA) Manager. In some cases, QAPPs and DQOs may specify less stringent requirements. The Laboratory Director/Manager and the QA Manager must determine if it is in the lab's best interest to follow the less stringent requirements.

## 3.4 MANAGEMENT OF THE MANUAL

### 3.4.1 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. The manual itself is reviewed every two years by senior laboratory management to assure that it reflects current practices and meets the requirements of the laboratory's clients and regulators as well as the CQMP. Occasionally, the manual may need changes in order to meet new or changing regulations and operations. The QA Manager will review the changes in the normal course of business and incorporate changes into revised sections of the document. All updates will be reviewed by the senior laboratory management staff. The laboratory updates and approves such changes according to our Document Control & updating procedures (refer to BF-QA-003)

# SECTION 4

# MANAGEMENT REQUIREMENTS

## 4.1 OVERVIEW

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

## 4.2 ROLES AND RESPONSIBILITIES

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

### 4.2.1 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 CQMP. This manual is specific to the operations of TestAmerica's Buffalo laboratory.

### 4.2.2 Laboratory Director

***TestAmerica Buffalo***'s Laboratory Director is responsible for the overall quality, safety, financial, technical, human resource and service performance of the whole laboratory and reports to their respective GM. The Laboratory Director provides the resources necessary to implement and maintain an effective and comprehensive Quality Assurance and Data Integrity Program.

The Laboratory Director has the authority to affect those policies and procedures to ensure that only data of the highest level of excellence are produced. As such, the Laboratory Director is responsible for maintaining a working environment which encourages open, constructive problem solving and continuous improvement.

Specific responsibilities include, but are not limited to:

- Provides one or more department managers for the appropriate fields of testing. If the Department Manager is absent for a period of time exceeding 15 consecutive calendar

days, the Laboratory Director must designate another full time staff member meeting the qualifications of the Department Manager to temporarily perform this function. If the absence exceeds 65 consecutive calendar days, the primary NELAP accrediting authority must be notified in writing.

- Ensures that all analysts and supervisors have the appropriate education and training to properly carry out the duties assigned to them and ensures that this training has been documented.
- Ensures that personnel are free from any commercial, financial and other undue pressures which might adversely affect the quality of their work.
- Ensures TestAmerica's human resource policies are adhered to and maintained.
- Ensures that sufficient numbers of qualified personnel are employed to supervise and perform the work of the laboratory.
- Ensures that appropriate corrective actions are taken to address analyses identified as requiring such actions by internal and external performance or procedural audits. Procedures that do not meet the standards set forth in the QAM or laboratory SOPs may be temporarily suspended by the Laboratory Director.
- Reviews and approves all SOPs prior to their implementation and ensures all approved SOPs are implemented and adhered to.
- Pursues and maintains appropriate laboratory certification and contract approvals. Supports ISO 17025 requirements.
- Ensures client specific reporting and quality control requirements are met.
- Leads the management team, consisting of the QA Manager, the Technical Manager, and the Operations Manager as direct reports.

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

The QA manager has responsibility and authority to ensure the continuous implementation of the quality system.

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

- Serves as the focal point for QA/QC in the laboratory.
- Having functions independent from laboratory operations for which he/she has quality assurance oversight.
- Maintaining and updating the QAM.

- Monitoring and evaluating laboratory certifications; scheduling proficiency testing samples.
- Monitoring and communicating regulatory changes that may affect the laboratory to management.
- Training and advising the laboratory staff on quality assurance/quality control procedures that are pertinent to their daily activities.
- Have documented training and/or experience in QA/QC procedures and the laboratory's Quality System.
- Having a general knowledge of the analytical test methods for which data audit/review is performed (and/or having the means of getting this information when needed).
- Arranging for or conducting internal audits on quality systems, data authenticity and the technical operation.
- The laboratory QA Manager will maintain records of all ethics-related training, including the type and proof of attendance.
- Maintain, improve, and evaluate the corrective action and preventive action systems.
- Notifying laboratory management of deficiencies in the quality system and ensuring corrective action is taken. 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 monitor standards of performance in quality control and quality assurance without outside (e.g., managerial) influence.
- Coordinating of document control of SOPs, MDLs, control limits, and miscellaneous forms and information.
- Review a subset of all final data reports for internal consistency. Review of Chain of Custody (COC), correspondence with the analytical request, batch QC status, completeness of any corrective action statements, evaluate manual calculations, format, holding time, sensibility and completeness of the project file contents.
- Review of external audit reports and data validation requests.
- Follow-up with audits to ensure client QAPP requirements are met.
- Establishment of reporting schedule and preparation of various quality reports for the Laboratory Director, clients and/or Corporate QA.
- Development of suggestions and recommendations to improve quality systems.
- Research of current state and federal requirements and guidelines.
- Leads the QA team to enable communication and to distribute duties and responsibilities.
- Ensuring Communication & monitoring standards of performance to ensure that systems are in place to produce the level of quality as defined in this document.

- Notifying laboratory management of deficiencies in the quality system and ensuring corrective action is taken. Procedures that do not meet the standards set forth in the QAM or laboratory SOPs are temporarily suspended following the procedures outlined in Section 12.

- Evaluation of the thoroughness and effectiveness of training.

- Compliance with ISO 17025.

### 4.2.4 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 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, and 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.
- Reviewing and approving, with input from the QA Manager, proposals from marketing, in accordance with an established procedure for the review of requests and contracts. This procedure addresses the adequate definition of methods to be used for analysis and any limitations, the laboratory's capability and resources, the client's expectations. Differences are resolved before the contract is signed and work begins. A system documenting any significant changes is maintained, as well as pertinent discussions with the client regarding their requirements or the results of the analyses during the performance of the contract. All work subcontracted by the laboratory must be approved by the client. Any deviations from the contract must be disclosed to the client. Once the work has begun, any amendments to the contract must be discussed with the client and so documented.
- Monitoring the validity of the analyses performed and data generated in the laboratory. This activity begins with reviewing and supporting all new business contracts, insuring data quality, analyzing internal and external non-conformances to identify root cause issues and implementing the resulting corrective and preventive actions, facilitating the data review process (training, development, and accountability at the bench), and providing technical and troubleshooting expertise on routine and unusual or complex problems.
- Providing 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.

- Enhancing efficiency and improving quality through technical advances and improved LIMS utilization. Capital forecasting and instrument life cycle planning for second generation methods and instruments as well as asset inventory management.
- Coordinating sample management from "cradle to grave," insuring that no time is lost in locating samples.
- Scheduling 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.

### 4.2.5 Operations Manager

The Operations Manager manages and directs the analytical production sections of the laboratory. He/She reports directly to the Laboratory Director. He/She assists the Technical Manager in determining the most efficient instrument utilization. More specifically, he/she:

- Evaluates the level of internal/external non-conformances for all departments.
- Continuously evaluates production capacity and improves capacity utilization.
- Continuously evaluates turnaround time and addresses any problems that may hinder meeting the required and committed turnaround time from the various departments.
- Develops and improves the training of all analysts in cooperation with the Technical Manager and QA Manager and in compliance with regulatory requirements.
- Is responsible for efficient utilization of supplies.
- Constantly monitors and modifies the processing of samples through the departments.
- Fully supports the quality system and, if called upon in the absence of the QA Manager, serves as his substitute in the interim.

### 4.2.6 Department Managers

Department Managers report to the Operations Manager. The Department Managers serve as the technical experts on assigned projects, provide technical liaison, assist in resolving any technical issues within the area of their expertise; and implement established policies and procedures to assist the Operations Manager in achieving section goals. Each one is responsible to:

- Ensure that analysts in their department adhere to applicable SOPs and the QA Manual. They perform frequent SOP and QA Manual review to determine if analysts are in compliance and if new, modified, and optimized measures are feasible and should be added to these documents.
- With regard to analysts, participates in the selection, training, and development of performance objectives and standards of performance, appraisal (measurement of objectives), scheduling, counseling, discipline, and motivation of analysts and documents these activities in accordance with systems developed by the QA and Human Resources

Departments. They evaluate staffing sufficiency and overtime needs. Training consists of familiarization with SOP, QC, Safety, and computer systems.

- Encourage the development of analysts to become cross-trained in various methods and/or operate multiple instruments efficiently while performing maintenance and documentation, self-supervise, and function as a department team.
- Provide guidance to analysts in resolving problems encountered daily during sample prep/analysis in conjunction with the Technical Manager, Operations Manager, and/or QA Manager. Each is responsible for 100% of the data review and documentation, non-conformance and CPAR issues, the timely and accurate completion of performance evaluation samples and MDLs, for his department.
- Ensure all logbooks are maintained, current, and properly labeled or archived.
- Report all non-conformance conditions to the QA Manager, Technical Manager, Operations Manager, and/or Laboratory Director.
- Ensure that preventive maintenance is performed on instrumentation as detailed in the QA Manual or SOPs. He is responsible for developing and implementing a system for preventive maintenance, troubleshooting, and repairing or arranging for repair of instruments.
- Maintain adequate and valid inventory of reagents, standards, spare parts, and other relevant resources required to perform daily analysis.
- Achieve optimum turnaround time on analyses and compliance with holding times.
- Conduct efficiency and cost control evaluations on an ongoing basis to determine optimization of labor, supplies, overtime, first-run yield, capacity (designed vs. demonstrated), second- and third-generation production techniques/instruments, and long-term needs for budgetary planning.
- Develop, implement, and enhance calibration programs.
- Provide written responses to external and internal audit issues.

### 4.2.7 Hazardous Waste Coordinator

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

- Staying current with the hazardous waste regulations.
- Continuing training on hazardous waste issues.
- Reviewing and updating annually the Hazardous Waste Contingency Plan in the Environmental Health & Safety Manual.
- Auditing the staff with regard to compliance with the Hazardous Waste Contingency Plan.
- Contacting the hazardous waste subcontractors for review of procedures and opportunities for minimization of waste.

### 4.2.8 Environmental Health & Safety Coordinator

The Environmental Health and Safety Coordinator reports to the Laboratory Director and ensures that systems are maintained for the safe operation of the laboratory. The Safety Officer is responsible to:

- Conduct ongoing, necessary safety training and conduct new employee safety orientation.
- Assist in developing and maintaining the Chemical Hygiene/Safety Manual.
- Administer dispersal of all Safety Data Sheet (SDS) information.
- Perform regular chemical hygiene and housekeeping instruction.
- Give instruction on proper labeling and practice.
- Serve as chairman of the laboratory safety committee.
- Provide and train personnel on protective equipment.
- Oversee the inspection and maintenance of general safety equipment – fire extinguishers, safety showers, eyewash fountains, etc. and ensure prompt repairs as needed.
- Supervise and schedule fire drills and emergency evacuation drills.
- Determine what initial and subsequent exposure monitoring, if necessary to determine potential employee exposure to chemicals used in the laboratory.
- When determined necessary, conduct exposure monitoring assessments.
- Determine when a complaint of possible over-exposure is “reasonable” and should be referred for medical consultation.
- Assist in the internal and external coordination of the medical consultation/monitoring program conducted by TestAmerica’s medical consultants.

### 4.2.9 Laboratory Analysts

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

- Perform analyses by adhering to analytical and quality control protocols prescribed by current SOPs, this QA Manual, and project-specific plans honestly, accurately, timely, safely, and in the most cost-effective manner.
- Document standard and sample preparation, instrument calibration and maintenance, data calculations, sample matrix effects, and any observed non-conformance on worklists, benchsheets, lab notebooks and/or the Non-Conformance Database.
- Report all non-conformance situations, instrument problems, matrix problems and QC failures, which might affect the reliability of the data, to their supervisor, the Technical Manager, and/or the QA Manager or member of QA staff.
- Perform 100% review of the data generated prior to entering and submitting for secondary level review.

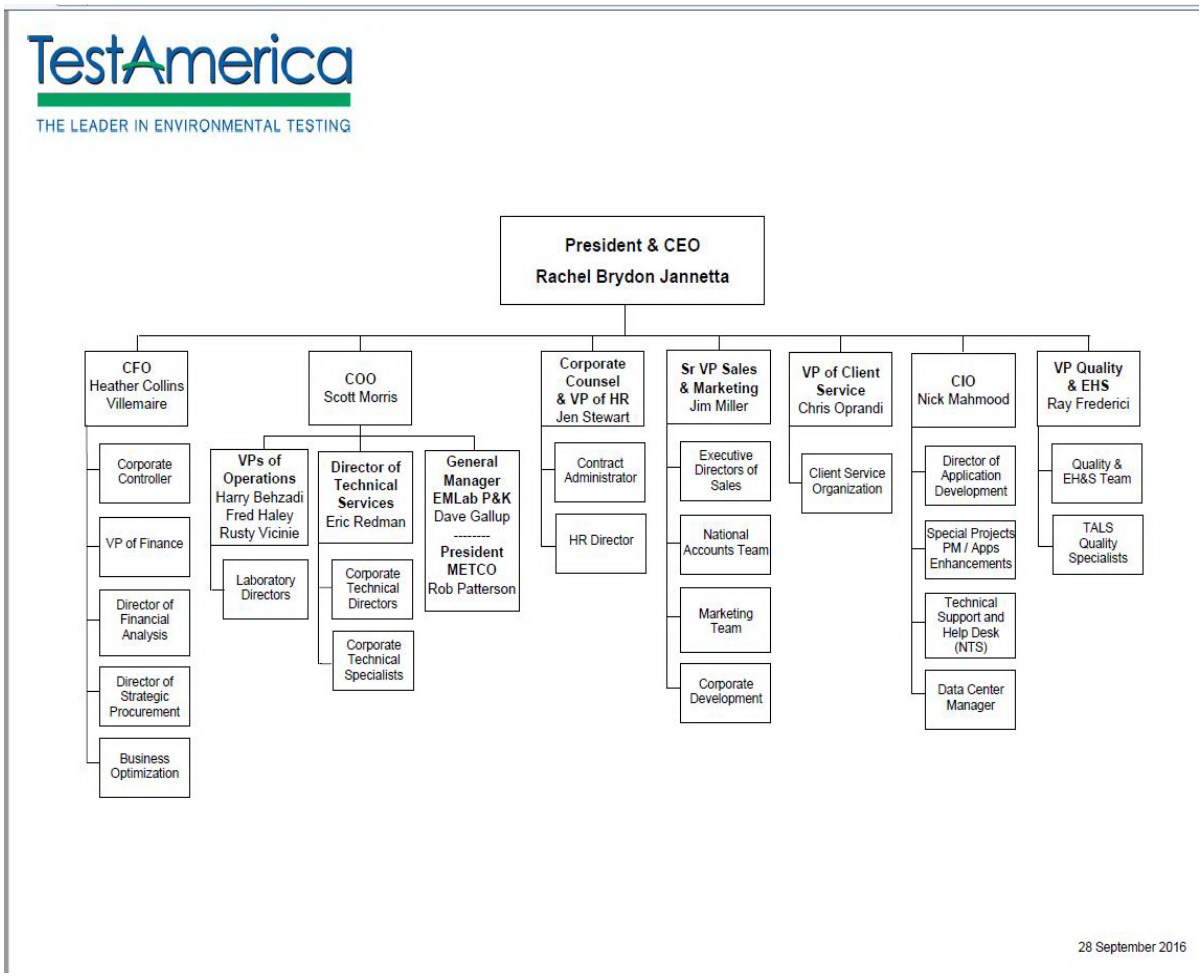
- Suggest method improvements to their supervisor, the Technical Manager, and the QA Manager. These improvements, if approved, will be incorporated. Ideas for the optimum performance of their assigned area, for example, through the proper cleaning and maintenance of the assigned instruments and equipment, are encouraged.
- Work cohesively as a team in their department to achieve the goals of accurate results, optimum turnaround time, cost effectiveness, cleanliness, complete documentation, and personal knowledge of environmental analysis.

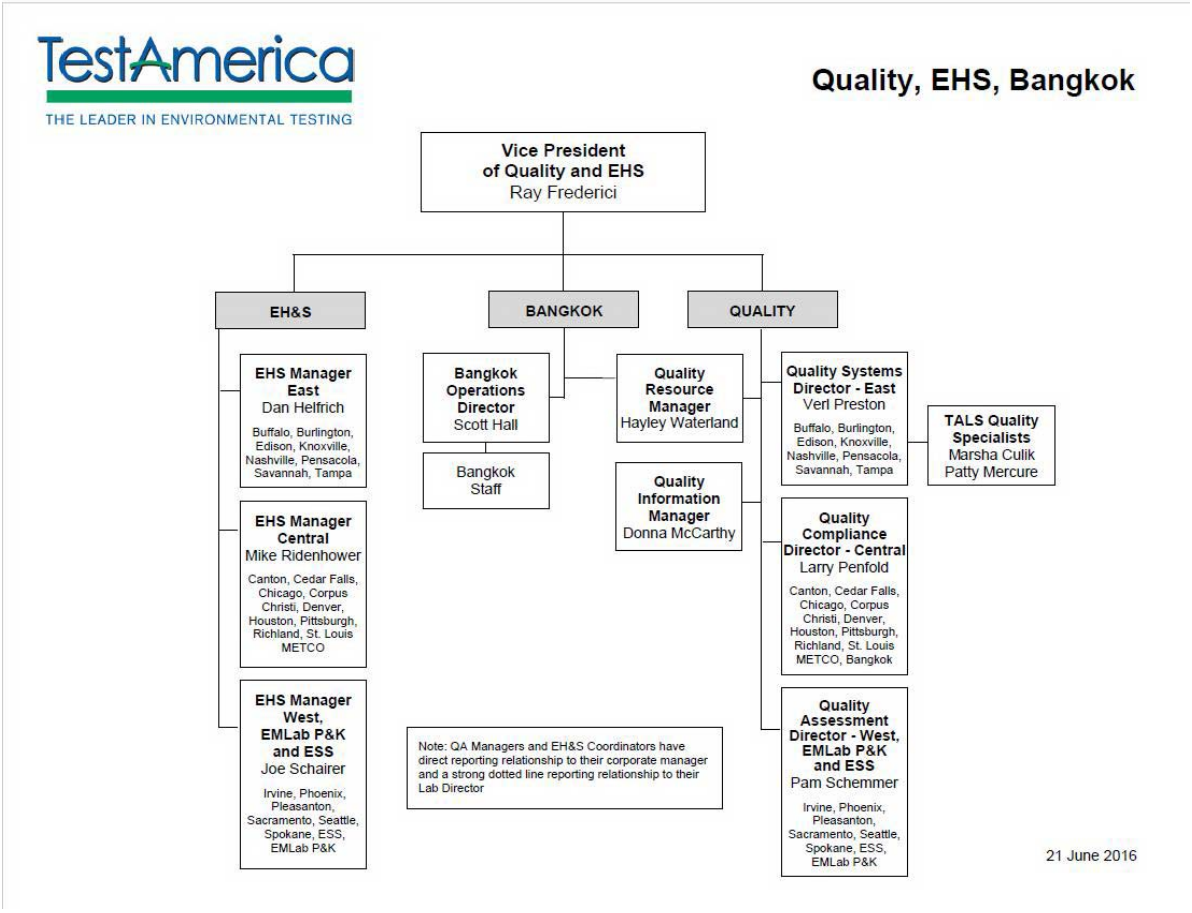
### 4.3 DEPUTIES

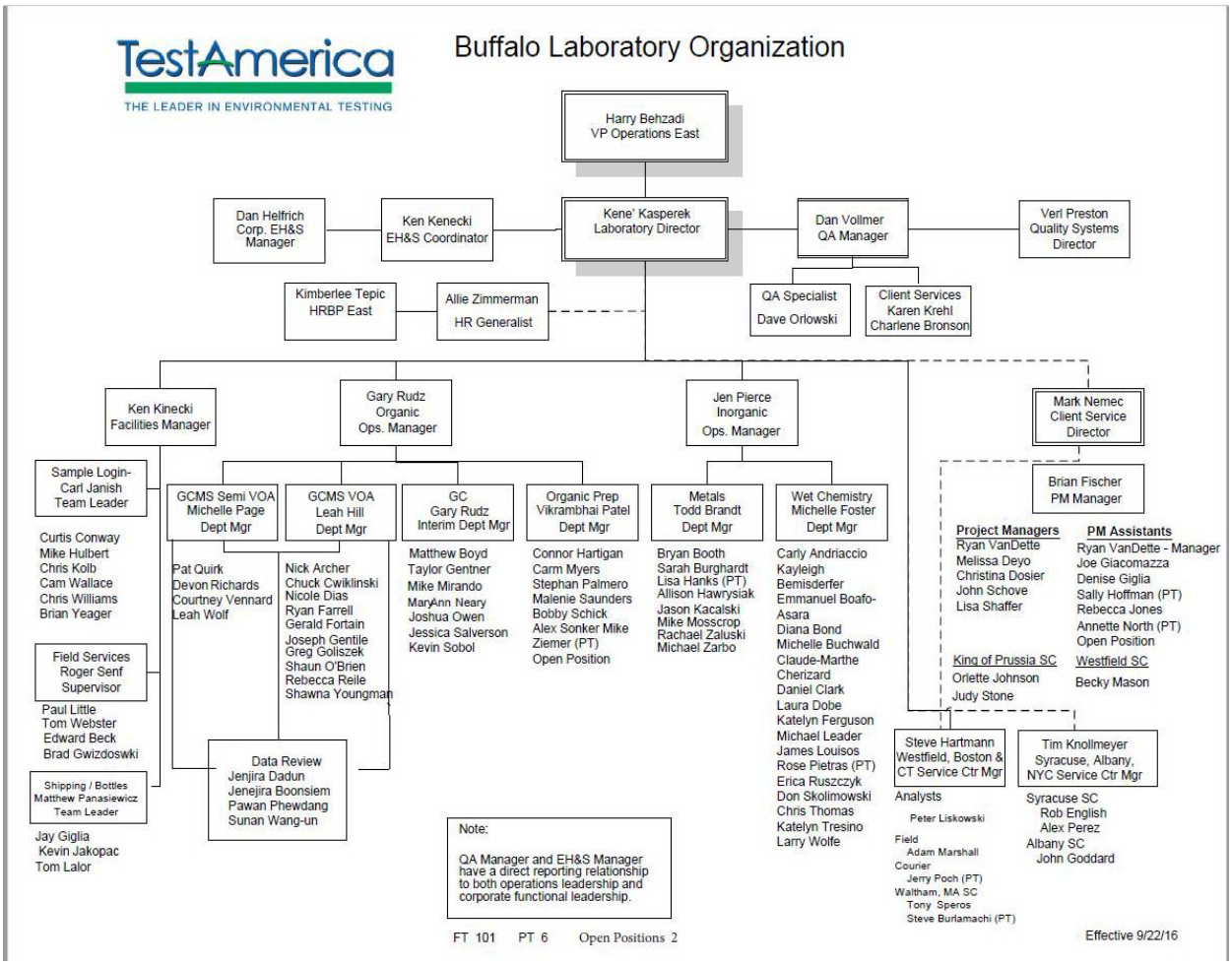
The following table defines who assumes the responsibilities of key personnel in their absence:

| Key Personnel | Deputy | Comment |
|---|---|---|
| Laboratory Director | Operations Manager (1)<br>Technical Manager (2) | |
| QA Manager | QA Specialist (1)<br>Operations Manager (2) | |
| Technical Manager | Laboratory Director (1)<br>Operations Manager (2) | |
| Operations Manager | Department Manager (1)<br>Department Manager (2) | Selected based on availability |
| Manager of Project Management | Project Manager (1)<br>Client Services Director (2) | Selected based on availability |
| Project Manager | Project Manager (1)<br>Project Management Asst. (2) | (1) 2° team PM<br>(2) Team PMA |
| Organic Department Manager | Analyst (1)<br>Analyst (2) | Selected based on department, experience and availability |
| Inorganic Department Manager | Analyst (1)<br>Analyst (2) | Selected based on department, experience and availability |
| Data Validation / Data Packaging Manager | Data Validation Specialist<br>Data Packaging Specialist | Selected based on department and availability |
| EHS Coordinator | Laboratory Director (1)<br>EHS Manager (2) | |
| Sample Management Manager | Sample Custodian (1)<br>EHS Coordinator (2) | |
| Bottle Preparation / Shipping Manager | Bottle Prep Technician (1)<br>Sample Mng't Manager (2) | |

**Figure 4-1.**
**Corporate and Laboratory Organization Charts**








Note: Organizational Charts are current at the date of publication of this manual. Updated charts may be obtained by contacting the TestAmerica Buffalo Quality 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.
- To comply with the NELAC Standards (2003), 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 7 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-Q-S-005).

- 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 laboratories 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).

### 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 maintains Quality Control Limit Data in their LIMS system. A summary report is generated from LIMS to check the precision and accuracy acceptability limits for performed analyses on request. The summary report is generated and is managed by the laboratory's QA department. Some acceptability limits are derived from US EPA methods when they are required. Where US EPA method limits are not required, the laboratory has developed limits from evaluation of data from similar matrices. Criteria for development of control limits are contained in Section 24.

## 5.6 STATISTICAL QUALITY CONTROL

Statistically-derived precision and accuracy limits are required by selected methods (such as SW-846) and programs [such as the Ohio Voluntary Action Plan (VAP)]. The laboratory routinely utilizes statistically-derived limits to evaluate method performance and determine when corrective action is appropriate. The procedure for determining the statistical limits may be found in SOP BF-QA-002, Quality Control Limits. The analysts are instructed to use the current limits in the laboratory (dated and approved the QA Manager) and entered into the Laboratory Information Management System (LIMS). The Quality Assurance department maintains an archive of all limits used within the laboratory through date sensitive tables within the LIMs System. If a method defines the QC limits, the method limits are used.

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

Surrogate recoveries are determined for a specific time period as defined above. The resulting ranges are entered in LIMS.

Current QC limits are entered and maintained in the LIMS analyte database. As sample results and the related QC are entered into LIMS, the sample QC values are compared with the limits in LIMS to determine if they are within the acceptable range. The analyst then evaluates if the sample needs to be rerun or re-extracted/rerun or if a comment should be added to the report explaining the reason for the QC outlier.

### 5.6.1 QC Charts

The QA Manager periodically evaluates these to determine if adjustments need to be made or for corrective actions to methods (SOP No. BF-QA-002). All findings are documented and kept on file.

## 5.7 QUALITY SYSTEM METRICS

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

# 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 No. BF-QA-003.

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

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

## 6.2 DOCUMENT APPROVAL AND ISSUE

The pertinent elements of a document control system for each document include a unique document title and number, 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 Quality personnel are responsible for the maintenance of the system.

Controlled documents are authorized by the QA Department. In order to develop a new document, a Department Manager submits an electronic draft to the QA Department for

suggestions and approval before use. Upon approval, QA personnel add the identifying version information to the document and retain that document as the official document on file. That document is then provided to all applicable operational units. Controlled documents are identified as such and records of their distribution are kept by the QA Department. Document control may be achieved by either electronic or hardcopy distribution.

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

Quality System Policies and Procedures will be reviewed at a minimum of every two years for the majority of procedures. Exceptions include review every 1 year for Drinking Water programs and the Kentucky CWA program. Changes to documents occur when a procedural change warrants.

## 6.3 PROCEDURES FOR DOCUMENT CONTROL POLICY

For changes to the QA Manual, refer to SOP No. BF-QA-003, “Writing, Reviewing and Revising Controlled Documents”. Uncontrolled copies must not be used within the laboratory. Previous revisions and back-up data are stored by the QA department. A controlled electronic copy of the current version is maintained on the laboratory Intranet site and is available to all personnel.

For changes to SOPs, refer to SOP No. BF-QA-003, “Writing, Reviewing and Revising Controlled Documents”.

Forms, worksheets, work instructions and information are organized by department and are maintained electronically by QA. There is a table of contents. As revisions are required, a new version number and revision date is assigned. Controlled electronic copies are made available on a public server for laboratory staff to access.

## 6.4 OBSOLETE DOCUMENTS

When revisions are implemented for an SOP, form or work instruction, the previous document becomes obsolete and is archived. All invalid or obsolete documents are removed, or otherwise prevented from unintended use. The laboratory has specific procedures as described above to accomplish this. In general, obsolete documents are collected from employees according to distribution lists and are destroyed. At least one copy of the obsolete document is archived according to SOPs No. BF-GP-015 and BF-QA-003. All archived SOPs, manuals, forms or work instructions are considered obsolete.

# 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 Client Relations Manager or Proposal Team, who will decide which lab will receive the work based on the scope of work and other requirements, including certification, testing methodology, and available capacity to perform the work. The contract review process is outlined in TestAmerica's Corporate SOP No. CA-L-P-002**,** Contract Compliance Policy.

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

- Contact Administrator
- VP of Operations
- Laboratory Project Manager
- Laboratory and/or Corporate Technical Managers
- Corporate Information Technology Managers/Directors
- Regional and/or National Account representatives
- Laboratory and/or Corporate Quality
- Laboratory and/or Corporate Environmental Health and Safety Managers/Directors
- The Laboratory Director reviews the formal laboratory quote and makes final acceptance for their facility.

The Sales Director, Contract Administrator, Account Executive or Proposal Coordinator 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 Contracts Department maintains copies of all signed contracts. The Project Managers at the TestAmerica Buffalo facility also maintains copies of these documents.

## 7.3 DOCUMENTATION

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

The contract will be distributed to and maintained by the appropriate sales/marketing personnel and the Account Executive. A copy of the contract and formal quote will be filed with the laboratory PM and the Laboratory Director.

Records are maintained of pertinent discussions with a client relating to the client's requirements or the results of the work during the period of execution of the contract. The PM keeps a phone log of conversations with the client.

### 7.3.1 Project-Specific Quality Planning

Communication of contract specific technical and QC criteria is an essential activity in ensuring the success of site specific testing programs. To achieve this goal a PM is assigned to each client. The PM is the first point of contact for the client. It is the PM's responsibility to ensure that project specific technical and QC requirements are effectively evaluated and communicated to the laboratory personnel before and during the project. QA department involvement may be needed to assist in the evaluation of custom QC requirements. Specific information related to project planning may be found in SOP BF-PM-001, Project Information Requirements.

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

Prior to work on a new project, the dissemination of project information and/or project opening meetings may occur to discuss schedules and unique aspects of the project. Items to be discussed may include the project technical profile, turnaround times, holding times, methods, analyte lists, reporting limits, deliverables, sample hazards, or other special requirements. The PM introduces new projects to the laboratory staff through project kick-off meetings or to the management staff during production meetings. These meetings provide direction to the laboratory staff in order to maximize production and client satisfaction, while maintaining quality. In addition, project notes may be associated with each sample batch as a reminder upon sample receipt and analytical processing.

During the project, any change that may occur within an active project is agreed upon between the client/regulatory agency and the PM/laboratory. These changes (e.g., use of a non-standard method or modification of a method) and approvals must be documented prior to implementation. Documentation pertains to any document, e.g., letter, e-mail, variance, contract addendum, which has been signed by both parties.

Such changes are also communicated to the laboratory during production meetings. Such changes are updated to the project notes and are introduced to the managers at these meetings. The laboratory staff is then introduced to the modified requirements via the PM or the individual laboratory Department Manager.

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

## 7.4 SPECIAL SERVICES

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

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 Managers/Designees 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 (CW-L-S-004) 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 TNI/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 accredited work where required.

Project Managers (PMs), Client Service Managers (CSM), or Account Executives (AE) for the Export Lab (TestAmerica laboratory that transfers samples to another laboratory) are responsible for obtaining client approval prior to subcontracting 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. Standard TestAmerica Terms & Conditions include the flexibility to subcontract samples within the TestAmerica laboratories. Therefore, additional advance notification to clients for intra-laboratory subcontracting is not necessary unless specifically required by a client contract.

**Note:** In addition to the client, some regulating agencies, such as the Department of Energy and the USDA, may require notification prior to placing such work**.**

Approval may be documented through reference in a quote / contract or e-mail correspondence.

## 8.2 QUALIFYING AND MONITORING SUBCONTRACTORS

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

- The first priority is to attempt to place the work in a qualified TestAmerica laboratory;
- Firms specified by the client for the task (Documentation that a subcontractor was designated by the client must be maintained with the project file. This documentation can be
- as simple as placing a copy of an e-mail from the client in the project folder);
- Firms listed as pre-qualified and currently under a subcontract with TestAmerica. A listing of all approved subcontracting laboratories 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 A2LA accredited laboratories.
- In addition, the firm must hold the appropriate certification to perform the work required.

All TestAmerica laboratories are pre-qualified for work-sharing provided they hold the appropriate accreditations, can adhere to the project/program requirements, and the client approved sending samples to that laboratory. The client must provide acknowledgement that the samples can be sent to that facility (an e-mail is sufficient documentation or if acknowledgement is verbal, the date, time, and name of person providing acknowledgement must be documented). The originating laboratory is responsible for communicating all technical, quality, and deliverable requirements as well as other contract needs. (Corporate SOP No. CA-C-S-001, Work Sharing Process).

When the potential sub-contract laboratory has not been previously approved, Account Executives or PMs may nominate a laboratory as a subcontractor based on need. The decision to nominate a laboratory must be approved by the Laboratory Director. The Laboratory Director requests that the QA Manager/Designee begin the process of approving the subcontract laboratory as outlined in Corporate SOP No. CA-L-S-002, Subcontracting Procedures.

**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 the Corporate Quality Information Manager (QIM) for review. Once all documents are reviewed for completeness, the Corporate QIM will forward the documents to the Purchasing Manager for formal signature and contracting with the laboratory. The approved vendor will be added to the approved subcontractor list on the intranet site and the finance group is concurrently notified 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 Corporate Quality Departments. Any problems identified will be brought to the attention of TestAmerica's Corporate Finance or Corporate Quality personnel.

- Complaints shall be investigated. Documentation of the complaint, investigation and
- Corrective action will be maintained in the subcontractor's file on the intranet site. Complaints are posted using the Vendor Performance Report (Form No. CW-F-WI-009)**.**
- Information shall be updated on the intranet when new information is received from the subcontracted laboratories.
- Subcontractors in good standing will be retained on the intranet listing. The CSO personnel will notify all TestAmerica laboratories and Corporate Quality and Corporate Contracts if any laboratory requires removal from the intranet site. This notification will be posted on the intranet site and e-mailed to all CSO Personnel, Laboratory Directors/Managers, QA Managers and Sales Personnel.

## 8.3 OVERSIGHT AND REPORTING

The PM must request that the selected subcontractor be presented with a subcontract, if one is not already executed between the laboratory and the subcontractor. The subcontract must include terms which flow down the requirements of our clients, either in the subcontract itself or through the mechanism of work orders relating to individual projects. A standard subcontract and the Lab Subcontractor Vendor Package (posted on the intranet) can be used to accomplish this, and the Corporate Counsel can tailor the document or assist with negotiations, if needed. The PM (or AE or CSM, etc.) responsible for the project must advise and obtain client consent to the subcontract as appropriate, and provide the scope of work to ensure that the proper requirements are made a part of the subcontract and are made known to the subcontractor.

Prior to sending samples to the subcontracted laboratory, the PM confirms their certification status to determine if it's current and scope-inclusive. The information is documented within the project records. For TestAmerica laboratories, certifications can be viewed on the company TotalAccess Database.

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

All subcontracted samples must be accompanied by a TestAmerica Chain of Custody (COC). A copy of the original COC sent by the client must be available in TALS for 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, facilities successful execution of the work, and ensures the timeliness and completeness of the analytical report.

Non-TNI accredited work must be identified in the subcontractor's report as appropriate. If TNI accreditation is not required, the report does not need to include this information.

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

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

## 8.4 CONTINGENCY PLANNING

The Laboratory Director may waive the full qualification of a subcontractor process temporarily to meet emergency needs; 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 time. . The comprehensive approval process must then be initiated within 30 calendar days of subcontracting.

# 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 Capital Expenditure, Controlled Purchase Requests and Fixed Asset Capitalization, SOP No. CW-F-S-007.

Contracts will be signed in accordance with TestAmerica's Company-Wide 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, consumables 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 and TestAmerica Buffalo SOP on Solvent Purity, SOP BF-OP-013. Approval information for the solvents and acids tested under SOP CA-Q-S-001 is stored on the TestAmerica Sharepoint, under Solvent Approvals. A master list of all tested materials, as well as the certificates of analysis for the materials, is stored in the same location. [

### 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. Purchase requisitions are placed into the J.D. Edwards system by designated departmental personnel. The listing of items available in the J.D. Edwards system has been approved for use by the corporate purchasing staff. Each purchase requisition receives final approval by the laboratory Operations Manager or purchasing coordinator before the order is submitted.

The analyst may also check the item out of the on-site consignment system that contains items approved for laboratory use.

### 9.3.2 Receiving

It is the responsibility of the purchasing manager/designee to receive the shipment. It is the responsibility of the department that ordered the materials to document the date the materials were received. Once the ordered reagents or materials are received, the department that submitted the order compares the information on the label or packaging to the original order to ensure that the purchase meets quality level specified. This is documented through the addition of the received date and initials to the information present on the daily order log.

The purchasing manager/designee verifies the lot numbers of received solvents and acids against the pre-approval lists. If a received material is listed as unapproved, or is not listed, it is sequestered and returned to the vendor. Alternatively, the laboratory may test the material for the intended use, and if it is acceptable, document the approval on the approval list. Records of any testing performed locally are maintained on the shared “public” folder on the computer network.

Materials may not be released for use in the laboratory until they have been inspected, verified as suitable for use, and the inspection/verification has been documented.

Safety Data Sheets (SDSs) are available online through the Company’s intranet website. Anyone may review these for relevant information on the safe handling and emergency precautions of on-site chemicals

### 9.3.3 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. 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 manufacturer's or SOP expiration date unless 'verified' (refer to item 3 listed below).

- An expiration date cannot not 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 meets CCV limits. The comparison studies are maintained along with the calibration raw data for which the reagent was used.

Wherever possible, standards must be traceable to national or international standards of measurement or to national or international reference materials. Records to that effect are available to the user.

Compressed gases in use are checked for pressure and secure positioning daily. 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.

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

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

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

Purchased 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 maintained in the LIMS system, files or binders in each laboratory section. These records include date of receipt, lot number (when applicable), and expiration date (when applicable). Incorporation of the item into the record indicates that the analyst has compared the new certificate with the previous one for the same purpose and that no difference is noted, unless approved and so documented by the Technical Manager or QA Manager.

### 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 Manager and/or the Laboratory Director. If they agree with the request the procedures outlined in TestAmerica's Corporate Policy No. CA-T-P-001, Qualified Products List, is followed. A decision is made as to which piece of equipment can best satisfy the requirements. The appropriate written requests are completed and purchasing places the order.

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

## 9.5 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 Department Managers. The service providers that perform the services are approved by the Department Managers, Operations Manager and/or Technical Manager.

Analytical balances are serviced and calibrated annually in accordance with SOP BF-GP-002,. The calibration and maintenance services are performed on-site, and the balances are returned to use immediately following successful calibration. When the calibration certificates are received (usually within two weeks of the service), they are reviewed, and documentation of the

review is filed with the certificates. If the calibration was unsuccessful, the balance is immediately removed from service and segregated pending either further maintenance or disposal.

Calibration services for support equipment such as NIST thermometers, weight sets, etc, are obtained from vendors with current and valid ISO 17025 accreditation for calibration of the specific piece of equipment. Prior to utilizing the vendor's services, the vendor's accreditation status is verified. Once the equipment has been calibrated, the calibration certificates are reviewed by the QA department, and documentation of the review is filed with the calibration certificates. The equipment is then returned to service within the laboratory

## 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 Procurements & Contracts Policy (Policy No. CW-F-P-004). The level of control used in the selection process is dependent on the anticipated spending amount and the potential impact on TestAmerica business. Vendors that provide test and measuring equipment, solvents, standards, certified containers, instrument related service contracts or subcontract laboratory services shall be subject to more rigorous controls than vendors that provide off-the-shelf items of defined quality that meet the end use requirements. The JD Edwards purchasing system includes all suppliers /vendors that have been approved for use.

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

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

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

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

The laboratory has access to a listing of all approved suppliers of critical consumables, supplies and services. This information is provided through the JD Edwards purchasing system.

### 9.6.1 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 (available on the intranet site).

New vendors are evaluated based upon criteria appropriate to the products or services provided as well as their ability to provide those products and services at a competitive cost. Vendors are also evaluated to determine if there are ethical reasons or potential conflicts of interest with TestAmerica employees that would make it prohibitive to do business with them as well as their financial stability. The QA Department and/or the Technical Manager 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 with both external and internal complaints with the goal of providing satisfactory resolution to complaints in a timely and professional manner.

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

The process of complaint resolution and documentation utilizes the procedures outlined in Section 12 (Corrective Actions) and is documented in the laboratory SOP related Corrective Action (BF-QA-005).

## 10.2 EXTERNAL COMPLAINTS

An employee that receives a complaint initiates the complaint resolution process by first documenting the complaint according to BF-QA-005.

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

The general steps in the complaint handling process are:

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

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

## 10.3 INTERNAL COMPLAINTS

Internal complaints include, but are not limited to: errors and non-conformances, training issues, internal audit findings, and deviations from methods. Corrective actions may be initiated by any staff member who observes a nonconformance and shall follow the procedures outlined in Section 13. In addition, Corporate Management, Sales and Marketing and Information Technology (IT) may initiate a complaint by contacting the laboratory or through the corrective action system described in Section 12.

## 10.4 MANAGEMENT REVIEW

The number and nature of client complaints is reported by the QA Manager to the laboratory and Quality 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 standard procedures, policies and/or client requests have occurred, corrective action is taken immediately. First, the laboratory evaluates the significance of the nonconforming work. Then, a corrective action plan is initiated based on the outcome of the evaluation. If it is determined that the nonconforming work is an isolated incident, the plan could be as simple as adding a qualifier to the final results and/or making a notation in the case narrative. If it is determined that the nonconforming work is a systematic or improper practices issue, the corrective action plan could include a more in depth investigation and a possible suspension of an analytical method. In all cases, the actions taken are documented using the laboratory's corrective action system (refer to Section 12).

Due to the frequently unique nature of environmental samples, sometimes departures from documented policies and procedures are needed. When an analyst encounters such a situation, the problem is presented to the department manager for resolution. The department manager may elect to discuss it with the Technical Manager, QA Manager or have a representative contact the client to decide on a logical course of action. Once an approach is agreed upon, the analyst documents it using the laboratory's non-conformance and corrective action system described in Section 12. This information can then be supplied to the client in the form of a footnote or a case narrative with the report.

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

## 11.2 RESPONSIBILITIES AND AUTHORITIES

Under certain circumstances the Laboratory Director, the Technical Manager, the Operations Manager or a member of the QA team may exceptionally authorize departures from documented procedures or policies. The departures may be a result of procedural changes due to the nature of the sample; a one-time procedure for a client; QC failures with insufficient sample to reanalyze, etc. In most cases, the client will be informed of the departure prior to the reporting of the data. Any departures must be well documented using the laboratory's non-conformance and corrective action procedures described in Section 12. This information may also need to be documented in logbooks and/or data review checklists as appropriate. Any

impacted data must be referenced in a case narrative and/or flagged with an appropriate data qualifier.

Any misrepresentation or possible misrepresentation of analytical data discovered by any laboratory staff member must be reported to facility senior laboratory management within 24-hours. The Senior Management staff is comprised of the Laboratory Director, Technical Manager, and QA Manager. Suspected misrepresentation issues may also be reported to any member of the corporate staff as identified in Ethics Policy, CW-L-P-004. The data integrity hotline (1-800-736-9407) may also be used. The reporting of issues involving alleged violations of the company's Data Integrity or Manual Integration procedures must be conveyed to an Ethics and Compliance Officer (ECO), (e.g., the VP-QA/EHS)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, Executive VP of Operations and the Quality Directors have the authority and responsibility to halt work, withhold final reports, or suspend an analysis for due cause as well as authorize the resumption of work.

### 11.3 EVALUATION OF SIGNIFICANCE AND ACTIONS TAKEN

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

Corporate SOP entitled Data Recalls (CW-Q-S-005) is the procedure to be followed when it is discovered that erroneous or biased data may have been reported to clients or regulatory agencies.

Corporate SOP entitled Internal Investigations (CW-L-S-002) is the procedure to be followed for investigation and correction of situations involved alleged incidents of misconduct or violation of the company's ethics policy.

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-Q-S-005.

### 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 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 VP of Operations and member of Corporate QA. This fax/e-mail acts as notification of the incident.

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

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

# 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. Corrective actions are documented using Non-Conformance Memo (NCM) and 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 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 Non-Conformance Memo (NCM)** - is used to document the following types of corrective actions:

- Deviations from an established procedure or SOP
- QC outside of limits (non matrix related)
- Isolated reporting / calculation errors
- Client complaints
- Project Management concerns regarding specific analytical results
- Discrepancies in materials / goods received vs. manufacturer packing slips.

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

- Questionable trends that are found in the monthly review of NCMs.
- Issues found while reviewing NCMs that warrant further investigation.
- Internal and External Audit Findings

- Failed or Unacceptable PT results.
- Corrective actions that cross multiple departments in the laboratory.
- Systematic Reporting / Calculation Errors
- Client complaints
- Data recall investigations
- Identified poor process or method performance trends
- Excessive revised reports

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

### 12.3.2 Selection and Implementation of Corrective Actions

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

### 12.3.3 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. Corporate SOP Root Cause Analysis (No. CA-Q-S-009) describes the procedure.

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 Department Manager, Operations Manager and QA Manager are responsible to ensure that the corrective action taken was effective.
- Ineffective actions are documented and re-evaluated until acceptable resolution is achieved. Department Managers and the Operations Manager are accountable to the Laboratory Director to ensure final acceptable resolution is achieved and documented appropriately.
- Each NCM is entered into the Laboratory Information Management System (LIMS) and each CAR is entered into the Incident and Corrective Action Tracker (iCAT) database for tracking and trending purposes for review to aid in ensuring that the corrective actions have taken effect.
- TestAmerica laboratories began using the Incident/Corrective Action Tracker (iCAT) database developed by the company in 2015. (Previously, a local spreadsheet database served this purpose.) An incident is an event triggering the need for one or more corrective actions as distinct from a corrective action, a potential deficiency stemming from an incident that requires investigation and possibly fixing. The database is independent of TALS, available to all local and corporate managers, and capable of notifying and tracking multiple corrective actions per event, dates, and personnel. iCAT allows associated document upload, categorization (such as, external/internal audit, client service concerns, data quality issues, proficiency testing, etc.), and trend analysis. Refer to Figure 12-1.

- The QA Manager reviews monthly NCMs and CARs for trends. Highlights are included in the QA monthly report (refer to Section 16). If a significant trend develops that adversely affects quality, an audit of the area is performed and corrective action implemented.
- Any out-of-control situations that are not addressed acceptably at the laboratory level may be reported to the Corporate Quality Director by the QA Manager, indicating the nature of the 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 NCM or CAR.

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

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

To the extent possible, samples shall be reported only if all quality control measures are acceptable. If the deficiency does not impair the usability of the results, data will be reported with an appropriate data qualifier and/or the deficiency will be noted in the case narrative. Where sample results may be impaired, the Project Manager is notified by an NCM and appropriate corrective action (e.g., reanalysis) is taken and documented.

## 12.5 BASIC CORRECTIONS

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

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

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 – iCAT Corrective Action Notice**

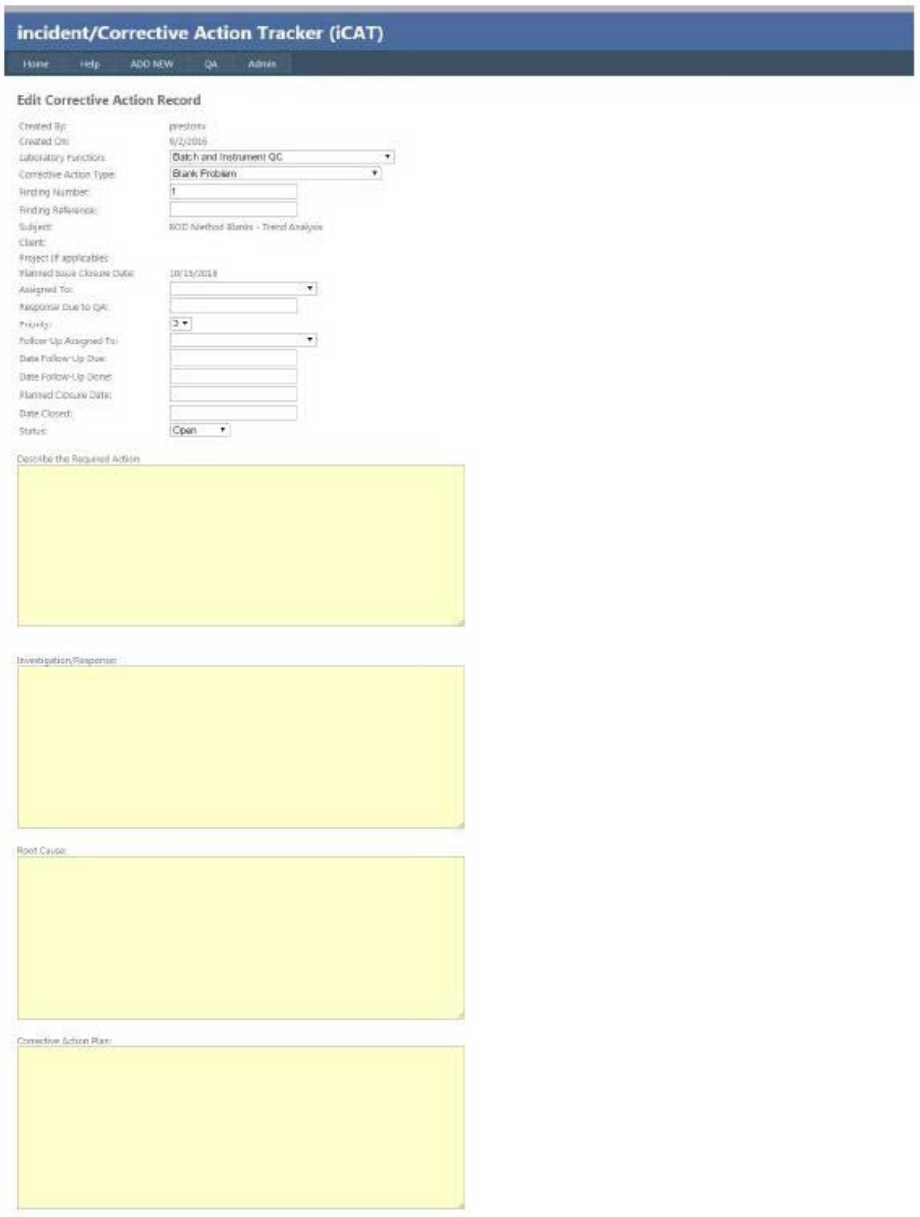


**Table 12-1.**

**Example – General Corrective Action Procedures**

| QC Activity *(Individual Responsible for Initiation/Assessment)* | Acceptance Criteria | Recommended Corrective Action |
|---|---|---|
| Initial Instrument Blank<br><br>*(Analyst)* | - Instrument response < MDL. | - Prepare another blank.<br>- If same response, determine cause of contamination: reagents, environment, instrument equipment failure, etc. |
| Initial Calibration Standards<br><br>*(Analyst, Department Manager)* | - Correlation coefficient > 0.99 or standard concentration value.<br>- % Recovery within acceptance range.<br>- See details in Method SOP. | - Reanalyze standards.<br>- If still unacceptable, remake standards and recalibrate instrument. |
| Independent Calibration Verification (Second Source)<br><br>*(Analyst, Department Manager)* | - % Recovery within control limits. | - Remake and reanalyze standard.<br>- If still unacceptable, then remake calibration standards or use new primary standards and recalibrate instrument. |
| Continuing Calibration Standards<br><br>*(Analyst, Data Reviewer)* | % Recovery within control limits. | - Reanalyze standard.<br>- If still unacceptable, then recalibrate and rerun affected samples. |
| Matrix Spike / Matrix Spike Duplicate (MS/MSD)<br><br>*(Analyst, Data Reviewer)* | - % Recovery within limits documented in LIMs. | - 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.<br>- If the LCS is within acceptable limits the batch is acceptable.<br>- The results of the duplicates, matrix spikes and the LCS are reported with the data set.<br>-For matrix spike or duplicate results outside criteria the data for 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 (LCS)<br><br>*(Analyst, Data Reviewer)* | - % Recovery within limits specified in LIMs. | - Batch must be re-prepared and re-analyzed. This includes any allowable marginal exceedance.<br>When not using marginal exceedances, the following exceptions apply:<br>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;<br>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.<br><br>**Note:** If there is insufficient sample or the holding time cannot be met, contact client and report with flags. |
| Surrogates<br><br>*(Analyst, Data Reviewer)* | - % Recovery within limits of method or within three standard deviations of the historical mean. | - Individual sample must be repeated. Place comment in LIMS.<br>- Surrogate results outside criteria shall be reported with qualifiers. |
| Method Blank (MB)<br><br>*(Analyst, Data Reviewer)* | < Reporting Limit [1] | - Reanalyze blank.<br>- If still positive, determine source of contamination. If necessary, reprocess (i.e. digest or extract) entire sample batch. Report blank results.<br>- Qualify the result(s) if the concentration of a targeted analyte in the MB is at or above the reporting limit AND is > 1/10 of the amount measured in the sample. |
| Proficiency Testing (PT) Samples<br><br>*(QA Manager, Department Manager)* | - Criteria supplied by PT Supplier. | - Any failures or warnings must be investigated for cause. Failures may result in the need to repeat a PT sample to show the problem is corrected. |

| QC Activity *(Individual Responsible for Initiation/Assessment)* | Acceptance Criteria | Recommended Corrective Action |
|---|---|---|
| Internal / External Audits<br><br>*(QA Manager, Department Manager, Operations Manager, Technical Manager, Laboratory Director)* | - Defined in Quality System documentation such as SOPs, QAM, etc. | - Non-conformances must be investigated through CAR system and necessary corrections must be made. |
| Reporting / Calculation Errors<br><br>*(Depends on issue – possible individuals include: Analysts, Data Reviewers, Project Managers, Department Manager, QA Manager, Corporate QA, Corporate Management)* | - SOP CW-Q-S-005, Data Recall. | - Corrective action is determined by type of error. Follow the procedures in SOP CW-Q-S-005 or lab SOP BF-QA-005 |
| Client Complaints<br><br>*(Project Managers, Lab Director, Sales and Marketing, QA Manager)* | - | - Corrective action is determined by the type of complaint. For example, a complaint regarding an incorrect address on a report will result in the report being corrected and then follow-up must be performed on the reasons the address was incorrect (e.g., database needs to be updated). |
| QA Monthly Report (Refer to Section 17 for an example)<br><br>*(QA Manager, Lab Director, Operations Manager Department Managers)* | - QAM, SOPs. | - Corrective action is determined by the type of issue. For example, CARs for the month are reviewed and possible trends are investigated. |
| Health and Safety Violation<br><br>*(EH&S Coordinator, Lab Director, Operations Manager, Department Manager)* | - Environmental Health and Safety (EHS) Manual. | - Non-conformance is investigated and corrected through EH&S office. |

**Note:** 1. Except as noted below for certain compounds, the method blank should be below the reporting limit. Concentrations up to five times the reporting limit will be allowed for the

ubiquitous laboratory and reagent contaminants: methylene chloride, acetone, 2-butanone and phthalates provided they appear in similar levels in the reagent blank and samples. This allowance presumes that the reporting limit is significantly below any regulatory limit to which the data are to be compared and that blank subtraction will not occur. For benzene and ethylene dibromide (EDB) and the other analytes for which regulatory limits are extremely close to the detection limit, the method blank must be below the method detection limit.

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## SECTION 13.0

## PREVENTIVE ACTION / 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, the laboratory continually strives to improve customer service and client satisfaction through continuous improvements to laboratory systems.

Opportunities for improvement may be discovered through any of the following:

- review of the monthly QA Metrics Report,
- trending NCMs,
- review of control charts and QC results,
- trending proficiency testing (PT) results,
- performance of management system reviews,
- trending client complaints,
- review of processing operations, or
- staff observations.

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. The metrics report is reviewed monthly be the laboratory management, Corporate QA and TestAmerica's Executive Committee. 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.

Items identified as continuous improvement opportunities to the management system may be issued as goals from the annual management systems review, recommendations from internal audits, white papers, Lesson Learned, Technical Services audit report, Technical Best Practices, or as Corporate or management initiatives.

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 and non-conformances provides a valuable mechanism for identifying preventive action opportunities.

**13.1.1** The following elements are part of a preventive action system/process improvement system:

- Identification of an opportunity for preventive action or process improvement.
- Process for the preventive action or improvement.
- Define the measurements of the effectiveness of the process once undertaken.
- Execution of the preventive action or improvment.
- Evaluation of the plan using the defined measurements.
- Verification of the effectiveness of the preventive action or improvement.
- Close-Out by documenting any permanent changes to the Quality System as a result of the Preventive Action or Process Improvement. Documentation of Preventive Action/Process Improvement is incorporated into the monthly QA reports, corrective action process and management review

**13.1.2** Any Preventive Actions/Process Improvements undertaken or attempted shall be taken into account during the Annual Management Systems Review (Section 17). A highly detailed report is not required; however a summary of success and failure within the preventive action program is sufficient to provide management with a measurement for evaluation.

## 13.2 MANAGEMENT OF CHANGE

The Management of Change process is designed to manage significant events and changes that occur within the laboratory. Through these procedures, the potential risks inherent with a new event or change are identified and evaluated. The risks are minimized or eliminated through pre-planning and the development of preventive measures. The types of changes covered under this system include: Facility Changes, Major Accreditation Changes, Addition or Deletion to Division's Capabilities or Instrumentation, Key Personnel Changes, Laboratory Information Management System (LIMS) changes.

## SECTION 14.0

## 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. Exceptions for programs with longer retention requirements are discussed in Section 14.1.2. TestAmerica Buffalo SOP BF-GP-015, Record Storage and Retention, specifies additional storage, archiving and retention procedures.

### 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 in a database which is backed up as past of the regular laboratory backup. Records are of two types; either electronic or hard copy paper formats depending on whether the record is computer or hand generated (some records may be in both formats). Hardcopy technical records are maintained by the Laboratory Director and the QA Department while electronic technical records are maintained by the IT Administrator.

**Table 14-1. Record Index[1]**

| | Record Types [1]: | Retention Time: |
|---|---|---|
| **Technical Records** | - Raw Data<br>- Logbooks[2]<br>- Standards<br>- Certificates<br>- Analytical Records<br>- MDLs/IDLs/DOCs<br>- Lab Reports | 5 Years from analytical report issue* |
| **Official Documents** | - Quality Assurance Manual (QAM)<br>- Work Instructions<br>- Policies<br>- Policy Memorandums<br>- SOPs<br>- Manuals | 5 Years from document retirement date* |
| **QA Records** | - Internal & External Audits/Responses<br>- Certifications<br>- Corrective/Preventive Actions<br>- Management Reviews<br>- Method & Software Validation / Verification Data<br>- Data Investigation | 5 Years from archival*<br><br>**Data Investigation:** 5 years or the life of the affected raw data storage whichever is greater (beyond 5 years if ongoing project or pending investigation) |

| | Record Types [1]: | Retention Time: |
|---|---|---|
| **Project Records** | - Sample Receipt & COC Documents<br>- Contracts and Amendments<br>- Correspondence<br>- QAPP<br>-SAP<br>- Telephone Logbooks<br>- Lab Reports | 5 Years from analytical report issue* |
| **Administrative Records** | 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) | All HR docs have different retention times: Refer to HR Manual |
| | Administrative Policies<br>Technical Training Records | 7 years |

[1] Record Types encompass hardcopy and electronic records.
[2] 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 according to BF-GP-015 and in such a way that they are secure and readily retrievable at the laboratory facility that provides a suitable environment to prevent damage or deterioration and to prevent loss. Records are maintained for a minimum of five years unless other wise specified by a client or regulatory requirement. All records shall be protected against fire, theft, loss, environmental deterioration and vermin. In the case of electronic records, electronic or magnetic sources, storage media are protected from deterioration caused by magnetic fields and/or electronic deterioration. Access to the data is limited to laboratory and company employees and shall be documented with an access log.

If records are archived off-site they are to be stored in a secure location where a record is maintained of any entry into the storage facility.

For raw data and project records, record retention shall be calculated from the date the project report is issued. For other records, such as Controlled Documents, QA, or Administrative Records, the retention time is calculated from the date the record is formally retired. Records related to the programs listed in Table 14-2 have lengthier retention requirements and are subject to the requirements in Section 14.1.3.

**14.1.2 Programs with Longer Retention Requirements**

Some regulatory programs have longer record retention requirements than the standard record retention time. These are detailed in Table 14-2 with their retention requirements. In these cases, the longer retention requirement is enacted. If special instructions exist such that client data cannot be destroyed prior to notification of the client, the container or box containing that data is marked as to who to contact for authorization prior to destroying the data. Specific Information related to archival of data for greater than 5 years may be found in TestAmerica Buffalo SOP BF-GP-015.

**Table 14-2. Special Record Retention Requirements**

| Program | [1]Retention Requirement |
|---|---|
| Drinking Water – All States | 5 years (project records)<br>10 years-Radiochemistry (project records) |
| Drinking Water Lead and Copper Rule | 12 years (project records) |
| Commonwealth of MA – All environmental data 310 CMR 42.14 | 10 years |
| FIFRA – 40 CFR Part 160 | Retain for life of research or marketing permit for pesticides regulated by EPA |
| Housing and Urban Development (HUD) Environmental Lead Testing | 10 years |
| Alaska | 10 years |
| Louisiana – All | 10 years |
| Michigan Department of Environmental Quality – all environmental data | 10 years |
| Navy Facilities Engineering Service Center (NFESC) | 5 years |
| NY Potable Water NYCRR Part 55-2 | 10 years |
| TSCA - 40 CFR Part 792 | 10 years after publication of final test rule or negotiated test agreement |

[1]Note: Extended retention requirements are noted with the archive documents or addressed in TestAmerica Buffalo facility-specific records retention procedure BF-GP-015.

**14.1.3** The laboratory has procedures to protect and back-up records stored electronically and to prevent unauthorized access to or amendment of these records. All analytical data is maintained as hard copy or in a secure readable electronic format. TestAmerica Buffalo SOP BF-GP-015 also contains specific information for archival of scanned data.

**14.1.4** 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 (any records stored off site should be accessible within 2 business days of a request for such records). The history of the sample from when the laboratory took possession of the samples must be readily understood through the documentation. This shall include inter-laboratory transfers of samples and/or extracts.

- The records include the identity of personnel involved in sampling, sample receipt, preparation, or testing. All analytical work contains the initials (at least) of the personnel involved. The laboratory's copy of the chain of custody is stored with the project file and the Job Number in TALS. The chain of custody would indicate the name of the sampler. If any sampling notes are provided with a work order, they are kept with this package.

- All information relating to the laboratory facilities equipment, analytical test methods, and related laboratory activities, such as sample receipt, sample preparation, or data verification are documented.

- The record keeping system facilitates the retrieval of all working files and archived records for inspection and verification purposes (e.g., set format for naming electronic files, set format for what is included with a given analytical data set). Instrument data is stored sequentially by instrument. Calibration data for a given sequence are maintained in the order of the analysis. Sample data are stored on a job number basis in the project file or as part of the daily batch or sequence. Run logs are maintained for each instrument or method; a copy of each day's run log or instrument sequence is stored with the data to aid in re-constructing an analytical sequence. Where an analysis is performed without an instrument, bound logbooks, bench sheets or excel spreadsheets are used to record and file data. Standard and reagent information is recorded in logbooks or on the raw data for each method as required.

- Changes to hardcopy records shall follow the procedures outlined in Section 13 and 20. Changes to electronic records in LIMS or instrument data are recorded in audit trails.

- The reason for a signature or initials on a document is clearly indicated in the records such as "sampled by," "prepared by," "reviewed by", or "analyzed by".

- All generated data except those that are generated by automated data collection systems, are recorded directly, promptly and legibly in permanent dark ink.

- Hard copy data may be scanned into PDF format for record storage as long as the scanning process can be verified in order to ensure that no data is lost and the data files and storage media must be tested to verify the laboratory's ability to retrieve the information prior to the destruction of the hard copy that was scanned. The procedure for this verification can be found in TestAmerica SOP BF-GP-015.

- Also refer to Section 19.14.1 'Computer and Electronic Data Related Requirements'**.**

## 14.2 TECHNICAL AND ANALYTICAL RECORDS

**14.2.1** The laboratory retains records of original observations, derived data and sufficient information to establish an audit trail, calibration records, staff records and a copy of each analytical report issued, for a minimum of five years unless otherwise specified by a client or regulatory requirement. The records for each analysis shall contain sufficient information to

enable the analysis to be repeated under conditions as close as possible to the original. The

records shall include the identity of laboratory personnel responsible for the sampling, performance of each analysis and reviewing of results.

**14.2.2** Observations, data and calculations are recorded real-time.

**14.2.3** Changes to hardcopy records shall follow the procedures outlined in Section 13 and 20. Changes to electronic records in LIMS or instrument data are recorded in audit trails.
The essential information to be associated with analysis, such as strip charts, tabular printouts, computer data files, analytical notebooks, and run logs, include:

- laboratory sample ID code**;**
- Date of analysis; time of analysis is also required if the holding time is seventy-two (72) hours or less, or when time critical steps are included in the analysis (e.g., drying times, incubations, etc.); instrumental analyses have the date and time of analysis recorded as part of their general operations. Where a time critical step exists in an analysis, location for such a time is included as part of the documentation in a specific logbook or on a bench sheet.
- Instrumentation identification and instrument operating conditions/parameters. Operating conditions/parameters are typically recorded in the method specific SOPs, in the instrument method detail records or the instrument maintenance logs where available.
- analysis type**;**
- all manual calculations and manual integrations**;**
- analyst's or operator's initials/signature**;**
- sample preparation including cleanup, separation protocols, incubation periods, ID codes, volumes, weights, instrument printouts, meter readings, temperatures, calculations, reagents**;**
- test results**;**
- standard and reagent origin, receipt, preparation, and use**;**
- calibration criteria, frequency and acceptance criteria**;**
- data and statistical calculations, review, confirmation, interpretation, assessment and reporting conventions**;**
- quality control protocols and assessment**;**
- electronic data security, software documentation and verification, software and hardware audits, backups, and records of any changes to automated data entries.
- Method performance criteria including expected quality control requirements. These are indicated both in the LIMS and on specific analytical report formats**.**

## 14.3 LABORATORY SUPPORT ACTIVITIES

In addition to documenting all the above-mentioned activities, the following are retained QA records and project records (previous discussions in this section relate where and how these data are stored):

- all original raw data, whether hard copy or electronic, for calibrations, samples and quality control measures, including analysts' work sheets and data output records (chromatograms, strip charts, and other instrument response readout records);
- a written description or reference to the specific test method used which includes a description of the specific computational steps used to translate parametric observations into a reportable analytical value;
- copies of final reports;
- archived SOPs;
- correspondence relating to laboratory activities for a specific project;
- all corrective action reports, audits and audit responses;
- proficiency test results and raw data; and
- results of data review, verification, and crosschecking procedures

### 14.3.1 Sample Handling Records

Records of all procedures to which a sample is subjected while in the possession of the laboratory are maintained. These include but are not limited to records pertaining to:

- sample preservation including appropriateness of sample container and compliance with holding time requirement;
- sample identification, receipt, acceptance or rejection and login;
- sample storage and tracking including shipping receipts, sample transmittal / COC forms; and
- Procedures for the receipt and retention of samples, including all provisions necessary to protect the integrity of samples.

## 14.4 ADMINISTRATIVE RECORDS

The laboratory also maintains the administrative records in either electronic or hard copy form. Refer to Table 14-1.

## 14.5 RECORDS MANAGEMENT, STORAGE AND DISPOSAL

**14.5.1** All records (including those pertaining to test equipment), certificates and reports are safely stored, held secure and in confidence to the client. Certification related records are available upon request.

**14.5.2** All information necessary for the historical reconstruction of data is maintained by the laboratory. Records that are stored only on electronic media must be supported by the hardware and software necessary for their retrieval.

**14.5.3** Records that are stored or generated by computers or personal computers have hard copy, write-protected backup copies, or an electronic audit trail controlling access**.**

**14.5.4** The laboratory has a record management system (also known as document control) for control of laboratory notebooks, instrument logbooks, standards logbooks, and records for data reduction, validation, storage and reporting. Laboratory notebooks are issued on a per instrument or analysis basis, and are numbered sequentially as they are issued. No instrument or analysis has more than one active notebook at a time, so all data are recorded sequentially within a series of sequential notebooks. Bench sheets and raw data sequence files are filed sequentially by date. Standard and reagent information is maintained in LIMS and logbooks which are maintained on a departmental basis and are numbered sequentially as they are issued or as they are archived by QA.

**14.5.5** Records are considered archived when noted as such in the records management system (also known as document control). Access to archived hard-copy information is documented with an access log and in/out records is used to note data that is removed and returned.

### 14.5.6 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.

### 14.5.7 Records Disposal

**14.5.7.1** Records are removed from the archive and destroyed after 5 years unless otherwise specified by a client or regulatory requirement. On a project specific or program basis, clients may need to be notified prior to record destruction. Records are destroyed in a manner that ensures their confidentiality such as shredding, mutilation or incineration. (Refer to Tables 14-1 and 14-2).

**14.5.7.2** Electronic copies of records must be destroyed by erasure or physically damaging off-line storage media so no records can be read.

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. CW-Q-S-003. 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<br>QA Technical Data Audits<br>SOP Compliance Audits | Joint responsibility:<br>a) QA Manager or designee<br>b) Technical Manager or Designee<br>(Refer to CW-Q-S-003) | QA Methods Audits Frequency: All methods are reviewed annually<br>50% of methods receive a QA Technical Audit<br>50% of methods receive a SOP Method Compliance Audit |
| Special | QA Department or Designee | Surveillance or spot checks performed as needed to monitor specific issues |
| Performance Testing | Coordinated by Corporate QA | Two successful per year for each TNI -NELAP 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 assess data authenticity and analyst integrity. These audits are based on client projects, associated sample delivery groups, and the methods performed. Reported results are compared to raw data to verify the authenticity of results. The validity of calibrations and QC results are compared to data qualifiers, footnotes, and case narratives. Documentation is assessed by examining run logs and records of manual integrations. Manual calculations are checked. Where possible, Chrom AuditMiner is used to identify unusual manipulations of the data deserving closer scrutiny. QA technical audits will include all methods within a two-year period. All analysts should be reviewed over the course of a two year period through at least one QA Technical Audit

### 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 Manager or qualified designee at least every two years. It is also recommended that the work of each newly hired analyst 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 semi-annually in performance audits conducted through the analysis of PT samples provided by a third party. The laboratory generally participates in the following types of PT studies: Drinking Water, Non-potable Water, Soil, and Air.

It is TestAmerica's policy that PT samples be treated as typical samples in the production process. Furthermore, where PT samples present special or unique problems, in the regular production process they may need to be treated differently, as would any special or unique request submitted by any client. The QA Manager must be consulted and in agreement with any decisions made to treat a PT sample differently due to some special circumstance.

Written responses to unacceptable PT results are required. In some cases it may be necessary for blind QC samples to be submitted to the laboratory to show a return to control.

## 15.2 EXTERNAL AUDITS

External audits are performed when certifying agencies or clients conduct on-site inspections or submit performance testing samples for analysis. It is TestAmerica's policy to cooperate fully with regulatory authorities and clients. The laboratory makes every effort to provide the auditors with access to personnel, documentation, and assistance. Laboratory supervisors are responsible for providing corrective actions to the QA Manager who coordinates the response for any deficiencies discovered during an external audit. Audit responses are due in the time allotted by the client or agency performing the audit. When requested, a copy of the audit report and the labs corrective action plan will be forwarded to Corporate Quality.

The laboratory cooperates with clients and their representatives to monitor the laboratory's performance in relation to work performed for the client. The client may only view data and systems related directly to the client's work. All efforts are made to keep other client information confidential.

### 15.2.1 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 database. The laboratory's corrective action responses for both types of audits may include action plans that could not be completed within a predefined timeframe. In these instances, a completion date must set and agreed to by operations management and the QA Manager.

Developing and implementing corrective actions to findings is the responsibility of the Department Manager where the finding originated. Findings that are not corrected by specified due dates are reported monthly to management in the QA monthly report. . When requested, a copy of the audit report and the labs corrective action plan will be forwarded to Corporate Quality.

If any audit finding casts doubt on the effectiveness of the operations or on the correctness or validity of the laboratory's test results, the laboratory shall take timely corrective action, and shall notify clients in writing if the investigations show that the laboratory results have been affected. Once corrective action is implemented, a follow-up audit is scheduled to ensure that the problem has been corrected.

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, their Quality Director as well as the VP of Operations. All aspects of the QA system are reviewed to evaluate the suitability of policies and procedures. During the course of the year, the Laboratory Director, General Manager or Corporate QA may request that additional information be added to the report.

On a monthly basis, Corporate QA compiles information from all the monthly laboratory reports. The Corporate Quality Director prepares a report that includes a compilation of all metrics and notable information and concerns regarding the QA programs within the laboratories. The report also includes a listing of new regulations that may potentially impact the laboratories. This report is presented to the Senior Management Team and VPs of Operations.

### 16.2 ANNUAL MANAGEMENT REVIEW

The senior lab management team (Laboratory Director, Technical Manager, Operations Manager, and QA Manager) conducts a review annually of its quality systems and LIMS to ensure its continuing suitability and effectiveness in meeting client and regulatory requirements and to introduce any necessary changes or improvements. It will also provide a platform for defining goals, objectives and action items that feed into the laboratory planning system. Corporate Operations and Corporate QA personnel may be included in this meeting at the discretion of the Laboratory Director. The LIMS review consists of examining any audits, complaints or concerns that have been raised through the year that are related to the LIMS. The laboratory will summarize any critical findings that can not be solved by the lab and report them to Corporate IT.

This management systems review (Corporate SOP No. CW-Q-S-004 & Work Instruction No. CW-Q-WI-003) 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 VP of Operations and the Quality Director. The report includes, but is not limited to:

- The date of the review and the names and titles of participants.
- A reference to the existing data quality related documents and topics that were reviewed.
- Quality system or operational changes or improvements that will be made as a result of the review [e.g., an implementation schedule including assigned responsibilities for the changes.

Changes to the quality systems requiring update to the laboratory QA Manual shall be included in the next revision of the QA Manual.

### 16.3 POTENTIAL INTEGRITY RELATED MANAGERIAL REVIEWS

Potential integrity issues (data or business related) must be handled and reviewed in a confidential manner until such time as a follow-up evaluation, full investigation, or other appropriate actions have been completed and issues clarified. The TestAmerica Corporate Internal InvestigationsSOP 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 President and CEO, COO, Technical & Operations Support, VP of Client and Technical Services, VPs of Operations and Quality Directors receive a monthly report from the VP QA/EHS summarizing any current data integrity or data recall investigations. The VPs of Operations 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 staff that possesses a college degree (AA, BA, BS) in an applied science with some chemistry in the curriculum. Exceptions can be made based upon the individual's experience and ability to learn. Selection of qualified candidates for laboratory employment begins with documentation of minimum education, training, and experience prerequisites needed to perform the prescribed task. Minimum education and training requirements for TestAmerica employees are outlined in job descriptions and are generally summarized for analytical staff in the table below.

The laboratory maintains job descriptions for all personnel who manage, perform or verify work affecting the quality of the environmental testing the laboratory performs. Job Descriptions are

located in the TestAmerica intranet site's Human Resources web-page (Also see Section 4 for position descriptions/responsibilities).

Experience and specialized training are occasionally accepted in lieu of a college degree (basic lab skills such as using a balance, pipette, quantitation techniques, etc. are also considered).

As a general rule for analytical staff:

| Specialty | Education | Experience |
|---|---|---|
| Extractions, Digestions, some electrode methods (pH, DO, Redox, etc.), or Titrimetric and Gravimetric Analyses | H.S. Diploma | On the job training (OJT) |
| CVAA, Single component or short list Chromatography (e.g., Fuels, BTEX-GC, IC) | A college degree in an applied science or 2 years of college and at least 1 year of college chemistry | Or 2 years prior analytical experience is required |
| ICP, ICPMS, Long List or complex chromatography (e.g., Pesticides, PCB, Herbicides, HPLC, etc.), GCMS | A college degree in an applied science or 2 years of college chemistry | or 5 years of prior analytical experience |
| Spectra Interpretation | A college degree in an applied science or 2 years of college chemistry | And 2 years relevant experience<br>Or<br>5 years of prior analytical experience |
| Technical Managers/Department Managers – **General** | Bachelors Degree in an applied science or engineering with 24 semester hours in chemistry<br><br>An advanced (MS, PhD.) degree may substitute for one year of experience | And 2 years experience in environmental analysis of representative analytes for which they will oversee |

When an analyst does not meet these requirements, they can perform a task under the direct supervision of a qualified analyst, peer reviewer or Department Manager, and are considered an analyst in training. The person supervising an analyst in training is accountable for the quality of the analytical data and must review and approve data and associated corrective actions.

### 17.3 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. Below are examples of various areas of required employee training:

| Required Training | Time Frame | Employee Type |
|---|---|---|
| Environmental Health & Safety | Prior to lab work | All |
| Ethics – New Hires | 1 week of hire | All |
| Ethics - Comprehensive | 90 days of hire | All |
| Data Integrity | 30 days of hire | Technical and PMs |
| Quality Assurance | 90 days of hire | All |
| Ethics – Refresher | Annually | All |
| Initial Demonstration of Capability (DOC) | Prior to unsupervised method performance | Technical |

The laboratory maintains records of relevant authorization/competence, education, professional qualifications, training, skills and experience of technical personnel (including contracted personnel) as well as the date that approval/authorization was given. These records are kept on file at the laboratory. Also refer to "Demonstration of Capability" in Section 19.

The training of technical staff is kept up to date by:

- Each employee must have documentation in their training file that they have read, understood and agreed to follow the most recent version of the laboratory QA Manual and SOPs in their area of responsibility. This documentation is updated as SOPs are updated.
- Documentation from any training courses or workshops on specific equipment, analytical techniques or other relevant topics are maintained in their training file.
- Documentation of proficiency (refer to Section 20).
- An Ethics Agreement signed by each staff member (renewed each year) and evidence of annual ethics training.
- A Confidentiality Agreement signed by each staff member signed at the time of employment.
- The Human Resource office maintains documentation and attestation forms on employment status & records; benefit programs; timekeeping/payroll; and employee conduct (e.g., ethics violations). This information is maintained in the employee's secured personnel file.

Further details of the laboratory's training program are described in TestAmerica Buffalo SOP BF-QA-004, Laboratory Personnel Training.

### 17.4 DATA INTEGRITY AND ETHICS TRAINING PROGRAM

Establishing and maintaining a high ethical standard is an important element of a Quality System. Ethics and data integrity training is integral to the success of TestAmerica and is provided for each employee at TestAmerica. It is a formal part of the initial employee orientation within 1 week of hire followed by technical data integrity training within 30 days, comprehensive

training within 90 days, and an annual refresher for all employees. Senior management at each facility performs the ethics training for their staff.

In order to ensure that all personnel understand the importance TestAmerica places on maintaining high ethical standards at all times; TestAmerica has established a Corporate Ethics Policy No. CW-L-P-004 and an Ethics Statement. All initial and annual training is documented by signature on the signed Ethics demonstrating that the employee has participated in the training and understands their obligations related to ethical behavior and data integrity.

Violations of this Ethics Policy will not be tolerated. Employees who violate this policy will be subject to disciplinary actions up to and including termination. Criminal violations may also be referred to the Government for prosecution. In addition, such actions could jeopardize TestAmerica's ability to do work on Government contracts, and for that reason, TestAmerica has a Zero Tolerance approach to such violations.

Employees are trained as to the legal and environmental repercussions that result from data misrepresentation. Key topics covered in the presentation include:

- Organizational mission and its relationship to the critical need for honesty and full disclosure in all analytical reporting.
- Ethics Policy
- How and when to report ethical/data integrity issues. Confidential reporting.
- Record keeping.
- Discussion regarding data integrity procedures.
- Specific examples of breaches of ethical behavior (e.g. peak shaving, altering data or computer clocks, improper macros, etc., accepting/offering kickbacks, illegal accounting practices, unfair competition/collusion)
- Internal monitoring. Investigations and data recalls.
- Consequences for infractions including potential for immediate termination, debarment, or criminal prosecution.
- Importance of proper written narration / data qualification by the analyst and project manager with respect to those cases where the data may still be usable but are in one sense or another partially deficient.

Additionally, a data integrity hotline (1-800-736-9407) is maintained by TestAmerica and administered by the Corporate Quality Department.

# SECTION 18

# ACCOMMODATIONS AND ENVIRONMENTAL CONDITIONS

## 18.1 OVERVIEW

TestAmerica Buffalo is a 32,000 $ft^2$ secure laboratory facility with controlled access and designed to accommodate an efficient workflow and to provide a safe and comfortable work environment for employees. All visitors sign in and are escorted by laboratory personnel. Access is controlled by various measures.

The laboratory is equipped with structural safety features. Each employee is familiar with the location, use, and capabilities of general and specialized safety features associated with their workplace. The laboratory provides and requires the use of protective equipment including safety glasses, protective clothing, gloves, etc. OSHA and other regulatory agency guidelines regarding required amounts of bench and fume hood space, lighting, ventilation (temperature and humidity controlled), access, and safety equipment are met or exceeded.

Traffic flow through sample preparation and analysis areas is minimized to reduce the likelihood of contamination. Adequate floor space and bench top area is provided to allow unencumbered sample preparation and analysis space. Sufficient space is also provided for storage of reagents and media, glassware, and portable equipment. Ample space is also provided for refrigerated sample storage before analysis and archival storage of samples after analysis. Laboratory HVAC and deionized water systems are designed to minimize potential trace contaminants.

The laboratory is separated into specific areas for field operations, bottle kit preparation, sample receiving, sample preparation, volatile organic sample analysis, non-volatile organic sample analysis, inorganic sample analysis and administrative functions.

## 18.2 ENVIRONMENT

Laboratory accommodation, test areas, energy sources, lighting are adequate to facilitate proper performance of tests. The facility is equipped with heating, ventilation, and air conditioning (HVAC) systems appropriate to the needs of environmental testing performed at this laboratory.

The environment in which these activities are undertaken does not invalidate the results or adversely affect the required accuracy of any measurements.

The laboratory provides for the effective monitoring, control and recording of environmental conditions that may affect the results of environmental tests as required by the relevant specifications, methods, and procedures. Such environmental conditions include humidity, voltage, temperature, and vibration levels in the laboratory. Key equipment has been provided with back-up power supply in the event of a power outage.

When any of the method or regulatory required environmental conditions change to a point where they may adversely affect test results, analytical testing will be discontinued until the environmental conditions are returned to the required levels.

Environmental conditions of the facility housing the computer network and LIMS are regulated to protect against raw data loss.

## 18.3 WORK AREAS

There is effective separation between neighboring areas when the activities therein are incompatible with each other. Examples include:

- Volatile organic chemical handling areas, including sample preparation and waste disposal, and volatile organic chemical analysis areas.

Access to and use of all areas affecting the quality of analytical testing is defined and controlled by secure access to the laboratory building as described below in the Building Security section.

Adequate measures are taken to ensure good housekeeping in the laboratory and to ensure that any contamination does not adversely affect data quality. These measures include regular cleaning to control dirt and dust within the laboratory.

Work areas are available to ensure an unencumbered work area. Work areas include:

- Access and entryways to the laboratory.
- Sample receipt areas.
- Sample storage areas.
- Chemical and waste storage areas.
- Data handling and storage areas.
- Sample processing areas.
- Sample analysis areas.

## 18.4 FLOOR PLAN

A floor plan can be found in Appendix 1.

## 18.5 BUILDING SECURITY

Building pass cards and alarm codes are distributed to all facility employees.

Visitors to the laboratory sign in and out in a visitor's logbook. A visitor is defined as any person who visits the laboratory who is not an employee of the laboratory. [The reason for this is that it is important to know who is in the building in case of a safety emergency. The visitors logbook is used to ensure that everyone got out of the building safely.] In addition to signing into the laboratory, the Environmental, Health and Safety Manual contains requirements for visitors and

vendors. There are specific safety forms that must be reviewed and signed. Visitors (with the exception of company employees) are escorted by laboratory personnel at all times, or the location of the visitor is noted in the visitor's logbook.

# SECTION 19.0

# TEST METHODS AND METHOD VALIDATION

## 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 in the laboratory:

- All SOPs contain a revision number, effective date, and appropriate approval signatures. Controlled copies are available to all staff.
- Procedures for writing an SOP are incorporated by reference to TestAmerica's Corporate SOP CW-Q-S-002, Writing a Standard Operating Procedure (SOP) and Laboratory SOP BF-QA-003, Procedure for Writing, Reviewing and Revising Controlled Quality Documents (QAM, SOP, etc)
- SOPs are reviewed at a minimum of every 2 years (annually for Drinking Water SOPs), and where necessary, revised to ensure continuing suitability and compliance with applicable requirements.

## 19.3 LABORATORY METHODS MANUAL

For each test method, the laboratory shall have available the published referenced method as well as the laboratory developed SOP.

**Note:** If more stringent standards or requirements are included in a mandated test method or regulation than those specified in this manual, the laboratory shall demonstrate that such requirements are met. If it is not clear which requirements are more stringent, the standard from the method or regulation is to be followed. Any exceptions or deviations from the referenced methods or regulations are noted in the specific analytical SOP.

The laboratory maintains an SOP Index for both technical and non-technical SOPs. Technical SOPs are maintained to describe a specific test method. Non-technical SOPs are maintained to describe functions and processes not related to a specific test method.

## 19.4 SELECTION OF METHODS

Since numerous methods and analytical techniques are available, continued communication between the client and laboratory is imperative to assure the correct methods are utilized. Once client methodology requirements are established, this and other pertinent information is summarized by the Project Manager. These mechanisms ensure that the proper analytical methods are applied when the samples arrive for log-in. For non-routine analytical services (e.g., special matrices, non-routine compound lists, etc.), the method of choice is selected based on client needs and available technology. The methods selected should be capable of measuring the specific parameter of interest, in the concentration range of interest, and with the required precision and accuracy.

### 19.4.1 Sources of Methods

Routine analytical services are performed using standard EPA-approved methodology. In some cases, modification of standard approved methods may be necessary to provide accurate analyses of particularly complex matrices. When the use of specific methods for sample analysis is mandated through project or regulatory requirements, only those methods shall be used.

When clients do not specify the method to be used or methods are not required, the methods used will be clearly validated and documented in an SOP and available to clients and/or the end user of the data.

**19.4.1.1** The analytical methods used by the laboratory are those currently accepted and approved by the U. S. EPA and the state or territory from which the samples were collected. Reference methods include:

- *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*
- *Compendium of Methods for the Determination of Toxic Organic Compounds in Ambient Air, US EPA, January 1996.*
- *Guidelines Establishing Test Procedures for the Analysis of Pollutants Under the Clean Water Act; Analysis and Sampling Procedures; 40CFR Part 136 as amended by Method Update Rule; May 18, 2012*
- *Methods for Chemical Analysis of Water and Wastes, EPA 600 (4-79-020), 1983.*
- *Methods for the Determination of Inorganic Substances in Environmental Samples, EPA-600/R-93/100, August 1993.*
- *Methods for the Determination of Metals in Environmental Samples, EPA/600/4-91/010, June 1991. Supplement I: EPA-600/R-94/111, May 1994.*

- *Methods for the Determination of Organic Compounds in Drinking Water, EPA-600/4-88-039, December 1988, Revised, July 1991, Supplement I, EPA-600-4-90-020, July 1990, Supplement II, EPA-600/R-92-129, August 1992. Supplement III EPA/600/R-95/131 - August 1995 (EPA 500 Series) (EPA 500 Series methods)*
- *Technical Notes on Drinking Water Methods, EPA-600/R94-173, October 1994*
- *NIOSH Manual of Analytical Methods, 4th ed., August 1994.*
- *Statement of Work for Inorganics & Organics Analysis, SOM and ISM, current versions, USEPA Contract Laboratory Program Multi-media, Multi-concentration.*
- *Standard Methods for the Examination of Water and Wastewater, 18th/19th /20th / 21st/ 22nd/on-line edition; Eaton, A.D. Clesceri, L.S. Greenberg, A.E. Eds; American Water Works Association, Water Pollution Control Federation, American Public Health Association: Washington, D.C.*
- *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; Final Update V, August 2015.*
- *Annual Book of ASTM Standards, American Society for Testing & Materials (ASTM), Philadelphia, PA.*
- *National Status and Trends Program, National Oceanographic and Atmospheric Administration, Volume I-IV, 1985-1994.*
- *Manual for the Certification of Laboratories Analyzing Drinking Water (EPA 815-R-05-004, January 2005)* ***(DW labs only)***
- *Code of Federal Regulations (CFR) 40, Parts 136, 141, 172, 173, 178, 179 and 261*
- *New York State DEC Analytical Services Protocol, 2005*
- *New York State DOH Methods Manual*
- *Massachusetts Contingency Plan 310 CMR 40, April 25, 2014*
- *Connecticut Reasonable Confidence Protocol, July 2006*

The laboratory reviews updated versions to all the aforementioned references for adaptation based upon capabilities, instrumentation, etc., and implements them as appropriate. As such, the laboratory strives to perform only the latest versions of each approved method as regulations allow or require.

Other reference procedures for non-routine analyses may include methods established by specific states (e.g., Underground Storage Tank methods), ASTM or equipment manufacturers. Sample type, source, and the governing regulatory agency requiring the analysis will determine the method utilized.

The laboratory shall inform the client when a method proposed by the client may be inappropriate or out of date. After the client has been informed, and they wish to proceed contrary to the laboratory's recommendation, it will be documented.

### 19.4.2 Demonstration of Capability

Before the laboratory may institute a new method and begin reporting results, the laboratory shall confirm that it can properly operate the method. In general, this demonstration does not test the performance of the method in real world samples, but in an applicable and available clean matrix sample. If the method is for the testing of analytes that are not conducive to spiking, demonstration of capability may be performed on quality control samples.

**19.4.2.1** A demonstration of capability (BF-QA-004) is performed whenever there is a significant change in instrument type (e.g., new instrumentation), method or personnel.

**Note**: The laboratory shall have a DOC for all analytes included in the methods that the laboratory performs, and proficiency DOCs for each analyst shall include all analytes that the laboratory routinely performs. Addition of non-routine analytes does not require new DOCs for all analysts if those analysts are already qualified for routine analytes tested using identical chemistry and instrument conditions.

**19.4.2.2** The initial demonstration of capability must be thoroughly documented and approved by the Operations Manager/Designee and QA Manager prior to independently analyzing client samples. All associated documentation must be retained in accordance with the laboratories archiving procedures.

**19.4.2.3** The laboratory must have an approved SOP, demonstrate satisfactory performance, and conduct a method detection limit study (when applicable). There may be other requirements as stated within the published method or regulations (i.e., retention time window study).

**Note:** In some instances, a situation may arise where a client requests that an unusual analyte be reported using a method where this analyte is not normally reported. If the analyte is being reported for regulatory purposes, the method must meet all procedures outlined within this QA Manual (SOP, MDL, and Demonstration of Capability). If the client states that the information is not for regulatory purposes, the result may be reported as long as the following criteria are met:

- The instrument is calibrated for the analyte to be reported using the criteria for the method and ICV/CCV criteria are met (unless an ICV/CCV is not required by the method or criteria are per project DQOs).
- The laboratory's nominal or default reporting limit (RL) is equal to the quantitation limit (QL), must be at or above the lowest non-zero standard in the calibration curve and must be reliably determined. Project RLs are client specified reporting levels which may be higher than the QL. Results reported below the QL must be qualified as estimated values. Also see Section 19.6.1.3, Relationship of Limit of Detection (LOD) to Quantitation Limit (QL).

- The client request is documented and the lab informs the client of its procedure for working with unusual compounds. The final report must be footnoted: *Reporting Limit based on the low standard of the calibration curve.*

### 19.4.3 Initial Demonstration of Capability (IDOC) Procedures

Procedures for generation of IDOCs are detailed below and in laboratory SOP BF-QA-004, Laboratory Personnel Training.

**19.4.3.1** The spiking standard used must be prepared independently from those used in instrument calibration.

**19.4.3.2** The analyte(s) shall be diluted in a volume of clean matrix sufficient to prepare four aliquots at the concentration specified by a method or the laboratory SOP.

**19.4.3.3** At least four aliquots shall be prepared (including any applicable clean-up procedures) and analyzed according to the test method (either concurrently or over a period of days).

**19.4.3.4** Using all of the results, calculate the mean recovery in the appropriate reporting units and the standard deviations for each parameter of interest.

**19.4.3.5** When it is not possible to determine the mean and standard deviations, such as for presence, absence and logarithmic values, the laboratory will assess performance against criteria described in the Method SOP.

**19.4.3.6** Compare the information obtained above to the corresponding acceptance criteria for precision and accuracy in the test method (if applicable) or in laboratory generated acceptance criteria (LCS or interim criteria) if there is no mandatory criteria established. If any one of the parameters do not meet the acceptance criteria, the performance is unacceptable for that parameter.

**19.4.3.7** When one or more of the tested parameters fail at least one of the acceptance criteria, the analyst must proceed according to either option listed below:

- Locate and correct the source of the problem and repeat the test for all parameters of interest beginning with 19.4.3.3 above.
- Beginning with 19.4.3.3 above, repeat the test for all parameters that failed to meet criteria. Repeated failure, however, will confirm a general problem with the measurement system. If this occurs, locate and correct the source of the problem and repeat the test for all compounds of interest beginning with 19.4.3.1 above.

Note: Results of successive LCS analyses can be used to fulfill the DOC requirement.

A certification statement (see Figure 19-1) shall be used to document the completion of each initial demonstration of capability. A copy of the certification is archived in the analyst's training folder.

## 19.5 LABORATORY DEVELOPED METHODS AND NON-STANDARD METHODS

Any new method developed by the laboratory must be fully defined in an SOP and validated by qualified personnel with adequate resources to perform the method. Method specifications and the relation to client requirements must be clearly conveyed to the client if the method is a non-standard method (not a published or routinely accepted method). The client must also be in agreement to the use of the non-standard method.

## 19.6 VALIDATION OF METHODS

Validation is the confirmation by examination and the provision of objective evidence that the particular requirements for a specific intended use are fulfilled.

All non-standard methods, laboratory designed/developed methods, standard methods used outside of their scope, and major modifications to published methods must be validated to confirm they are fit for their intended use. The validation will be as extensive as necessary to meet the needs of the given application. The results are documented with the validation procedure used and contain a statement as to the fitness for use.

### 19.6.1 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 can be differentiated from blanks. The MDL is determined for each analyte initially during the method validation process and updated as required in the analytical methods, whenever there is a significant change in the procedure or equipment, or based on project specific requirements (refer to 19.7.10). Generally the analyst prepares at least seven replicates of solution spiked at one to five times the estimated method detection limit (most often at the lowest standard in the calibration curve) into the applicable matrix with all the

analytes of interest. Each of these aliquots is extracted (including any applicable clean-up procedures) and analyzed in the same manner as the samples. Where possible, the seven replicates should be analyzed over 2-4 days to provide a more realistic MDL. To allow for some flexibility, this low level standard may be analyzed every batch or every week or some other frequency rather than doing the study all at once. In addition, a larger number of data points may be used if the appropriate t-value multiplier is used.

Refer to the Corporate SOP No. CA-Q-S-006 or the laboratory's SOP No. BF-QA-001 for details on the laboratory's MDL process.

## 19.8 INSTRUMENT DETECTION LIMITS (IDL)

**19.8.1** The IDL is sometimes used to assess the reasonableness of the MDLs or in some cases required by the analytical method or program requirements. IDLs are most used in metals analyses but may be useful in demonstration of instrument performance in other areas.

**19.8.2** IDLs are calculated to determine an instrument's sensitivity independent of any preparation method. IDLs are calculated either using 7 replicate spike analyses, like MDL but without sample preparation, or by the analysis of 10 instrument blanks and calculating 3 x the absolute value of the standard deviation. (For CLP procedures, the IDL is determined using the standard deviation of 7 replicate spike analyses on each of 3 non-consecutive days.)

**19.8.3** If IDL is > than the MDL, it may be used as the reported MDL.

## 19.9 VERIFICATION OF DETECTION AND REPORTING LIMITS

**19.9.1** Once an MDL is established, it must be verified, on each instrument, by analyzing a quality control sample (prepared as a sample) at no more than 3 times the calculated MDL for single analyte analyses (e.g. most wet chemistry methods, CVAA, 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 or see section 20.7.9 for other options. This verification does not apply to methods that are not readily spiked (e.g. pH, turbidity, etc.) or where the lab does not report to the MDL. If the MDL does not verify, then the lab will not report to the MDL, or redevelop their MDL or use the level where qualitative identification is established. MDLs must be verified at least annually.

**19.9.2** When the laboratory establishes a quantitation limit, it must be initially verified by the analysis of a low level standard or QC sample at 1-2 the reporting limit and annually thereafter. The annual requirement is waved for methods that have an annually verified MDL. The laboratory will comply with any regulatory requirement.

## 19.10 RETENTION TIME WINDOWS

Most organic analyses and some inorganic analyses use chromatography techniques for qualitative and quantitative determinations. For every chromatography analysis each analyte will have a specific time of elution from the column to the detector. This is known as the analyte's retention time. The variance in the expected time of elution is defined as the retention time

window. As the key to analyte identification in chromatography, retention time windows must be established on every column for every analyte used for that method. These records are kept with the files associated with an instrument for later quantitation of the analytes. Complete details are available in the laboratory's SOPs.

## 19.11 EVALUATION OF SELECTIVITY

The laboratory evaluates selectivity by following the checks within the applicable analytical methods, which include mass spectral tuning, second column confirmation, ICP interelement interference checks, chromatography retention time windows, sample blanks, and specific electrode response factors.

## 19.12 ESTIMATION OF UNCERTAINTY OF MEASUREMENT

**19.12.1** Uncertainty is "a parameter associated with the result of a measurement, that characterizes the dispersion of the values that could reasonably be attributed to the measurand" (as defined by the International Vocabulary of Basic and General Terms in Metrology, ISO Geneva, 1993, ISBN 92-67-10175-1). Knowledge of the uncertainty of a measurement provides additional confidence in a result's validity. Its value accounts for all the factors which could possibly affect the result, such as adequacy of analyte definition, sampling, matrix effects and interferences, climatic conditions, variances in weights, volumes, and standards, analytical procedure, and random variation. Some national accreditation organizations require the use of an "expanded uncertainty": the range within which the value of the measurand is believed to lie within at least a 95% confidence level with the coverage factor k=2.

**19.12.2** Uncertainty is not error. Error is a single value, the difference between the true result and the measured result. On environmental samples, the true result is never known. The measurement is the sum of the unknown true value and the unknown error. Unknown error is a combination of systematic error, or bias, and random error. Bias varies predictably, constantly, and independently from the number of measurements. Random error is unpredictable, assumed to be Gaussian in distribution, and reducible by increasing the number of measurements.

**19.12.3** The minimum uncertainty associated with results generated by the laboratory can be determined by using the Laboratory Control Sample (LCS) accuracy range for a given analyte. The LCS limits are used to assess the performance of the measurement system since they take into consideration all of the laboratory variables associated with a given test over time (except for variability associated with the sampling and the variability due to matrix effects). The percent recovery of the LCS is compared either to the method-required LCS accuracy limits or to the statistical, historical, in-house LCS accuracy limits.

**19.12.4** To calculate the uncertainty for the specific result reported, multiply the result by the decimal of the lower end of the LCS range percent value for the lower end of the uncertainty range, and multiply the result by the decimal of the upper end of the LCS range percent value for the upper end of the uncertainty range. These calculated values represent uncertainties at approximately the 99% confidence level with a coverage factor of k = 3. As an example, for a reported result of 1.0 mg/L with an LCS recovery range of 50 to 150%, the estimated uncertainty in the result would be 1.0 ±0.5 mg/L.

**19.12.5** In the case where a well recognized test method specifies limits to the values of major sources of uncertainty of measurement (e.g. 524.2, 525, etc) and specifies the form of presentation of calculated results, no further discussion of uncertainty is required.

## 19.13 SAMPLE REANALYSIS GUIDELINES

Because there is a certain level of uncertainty with any analytical measurement, a sample 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 Supervisor or Laboratory Director/Manager if unsure.

## 19.14 CONTROL OF DATA

The laboratory has policies and procedures in place to ensure the authenticity, integrity, and accuracy of the analytical data generated by the laboratory.

### 19.14.1 Computer and Electronic Data Related Requirements

The three basic objectives of our computer security procedures and policies are shown below. The laboratory is currently running the 'TALS Data System' which is a LIMs system that has been highly customized to meet the needs of the laboratory. It is referred to as LIMS for the remainder of this section. The LIMS utilizes a SQL server which is an industry standard relational database platform. It is referred to as Database for the remainder of this section.

#### 19.14.1.1 Maintain the Database Integrity

Assurance that data is reliable and accurate through data verification (review) procedures, password-protecting access, anti-virus protection, and 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, 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 data review sheets, or any other type of applicable documents, are signed by both the analyst and alternate reviewer to confirm the accuracy of the manual entry(s).

Manual integration of peaks will be documented and reviewed and the raw data will be flagged in accordance with the TestAmerica Corporate SOP CA-Q-S-002, *Acceptable Manual Integration Practices*.

Analytical results are reduced to appropriate concentration units specified by the analytical method, taking into account factors such as dilution, sample weight or volume, etc. Blank correction will be applied only when required by the method or per manufacturer's indication; otherwise, it should not be performed. Calculations are independently verified by appropriate laboratory staff. Calculations and data reduction steps for various methods are summarized in the respective analytical SOPs or program requirements.

**19.14.2.1** All raw data must be retained in the project job folder, computer file, and/or run log. All criteria pertinent to the method must be recorded. The documentation is recorded at the time observations or calculations are made and must be signed or initialed/dated (month/day/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 (µg/l) for liquids and milligrams per kilogram (mg/kg) or micrograms per kilogram (µg/kg) for solids. For values greater than 10,000 mg/l, results can be reported in percent, i.e., 10,000 mg/l = 1%. Units are defined in each lab SOP.

**19.14.2.3** In reporting, the analyst or the instrument output records the raw data result using values of known certainty plus one uncertain digit. If final calculations are performed external to LIMS, the results should be entered in LIMS with at least three significant figures. In general, final inorganic results are reported to 2 significant figures for values less than 10 and 3 significant figures for values greater than 10 on the final report. Organic results are generally reported to 1 significant figure for values less than 10 and 2 significant figures for values greater than 10 on the final report. The number of significant figures may be adjusted based on client or project requirements.

**19.14.2.4** For those methods that do not have an instrument printout, an instrumental output or a calculation spreadsheet upload compatible with the LIMS System, the final results and dilution factors are entered directly into LIMS by the analyst, and the software formats the final result for the analytical report. LIMS has a defined significant figure criterion for each analyte.

**19.14.2.5** The laboratory strives to import data directly from instruments or calculation spreadsheets to ensure that the reported data are free from transcription and calculation errors. For those analyses with an instrumental output compatible with the LIMS, the raw results and dilution factors are transferred into LIMS electronically after reviewing the quantitation report, and removing unrequested or poor spectrally-matched compounds. The analyst prints a copy of what has been entered to check for errors. This printout and the instrument's printout of calibrations, concentrations, retention times, chromatograms, and mass spectra, if applicable, are retained with the data file. The data file is automatically transferred to the network server and, eventually, to a back-up tape file.

### 19.14.3 Logbook / Worksheet Use Guidelines

Logbooks and worksheets are filled out 'real time' and have enough information on them to trace the events of the applicable analysis/task. (e.g. calibrations, standards, analyst, sample ID, date, time on short holding time tests, temperatures when applicable, calculations are traceable, etc.)

- Corrections are made following the procedures outlined in Section 12.
- Logbooks are controlled by the QA department. A record is maintained of all logbooks in the lab.
- Unused portions of pages must be "Z"'d out, signed and dated.
- Worksheets are created with the approval of the Technical 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 out lined in several laboratory SOPs (e.g. BF-SR-002, "Receipt of Analytical Samples", BF-GP-012, "Technical Data Review", and BF-PM-001, "Project Information Requirements") to ensure that reported data are free from calculation and transcription errors, that QC parameters have been reviewed and evaluated before data is reported. The laboratory also has an SOP discussing Manual Integrations to ensure the authenticity of the data (BF-GP-013, Manual Integration). The general review concepts are discussed below, more specific information can be found in the SOPs.

**19.14.4.1** Log-In Review - The data review process starts at the sample receipt stage. Sample control personnel review chain-of-custody forms and project instructions from the project management group. This is the basis of the sample information and analytical instructions entered into the LIMS. The log-in instructions are reviewed by the personnel entering the information, and a second level review is conducted by the project management staff.

**19.14.4.2** First Level Data Review –The next level of data review occurs with the analysts. As data are generated, analysts review their work to ensure that the results meet project and SOP requirements. First level reviews include inspection of all raw data (e.g., instrument output for continuous analyzers, chromatograms, spectra, and manual integrations), evaluation of calibration/calibration verification data in the day's analytical run, evaluation of QC data, and reliability of sample results. The analyst transfers data into LIMS, data qualifiers are added as needed. All first level reviews are documented.

**19.14.4.3** Second Level Data Review – All analytical data are subject to review by a second qualified analyst or supervisor. Second level reviews include inspection of all raw data (e.g., instrument output, chromatograms, and spectra) including 100% of data associated with any changes made by the primary analyst, such as manual integrations or reassignment of peaks to different analytes, or elimination of false negative analytes. The second review also includes evaluation of initial calibration/calibration verification data in the day's analytical run, evaluation of QC data, reliability of sample results, qualifiers and NCM narratives. Manual calculations are checked in second level review. All second level reviews are documented.

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

**19.14.4.4** Unacceptable analytical results may require reanalysis of the samples. Any problems are brought to the attention of the Laboratory Director, Project Manager, Quality Director/Manager, Technical Manager, or Supervisor for further investigation. Corrective action is initiated whenever necessary.

**19.14.4.5** The results are then entered or directly transferred into the computer database and a hard copy (or .pdf) is printed for the client.

**19.14.4.6** 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 COC is followed, cover letters/ narratives are present, flags are appropriate, and project specific requirements are met. The Project Manager may also evaluate the validity of results for different test methods given expected chemical relationships.

**19.14.4.7** Any project that requires a data package is subject to a tertiary data review for transcription errors and acceptable quality control requirements. The Project Manager then signs the final report and creates the invoice. When complete, the report is issued to the client.

### 19.14.5 Manual Integrations

Computerized data systems provide the analyst with the ability to re-integrate raw instrument data in order to optimize the interpretation of the data. Though manual integration of data is an invaluable tool for resolving variations in instrument performance and some sample matrix problems, when used improperly, this technique would make unacceptable data appear to meet quality control acceptance limits. Improper re-integrations lead to legally indefensible data, a poor reputation, or possible laboratory decertification. Because guidelines for re-integration of data are not provided in the methods and most methods were written prior to widespread implementation of computerized data systems, the laboratory trains all analytical staff on proper manual integration techniques using SOP CA-Q-S-002 as the guidelines.

**19.14.5.1** The analyst must adjust baseline or the area of a peak in some situations, for example when two compounds are not adequately resolved or when a peak shoulder needs to be separated from the peak of interest. The analyst must use professional judgment and common sense to determine when manual integrating is required. Analysts are encouraged to ask for assistance from a senior analyst or manager when in doubt.

**19.14.5.2** Analysts shall not increase or decrease peak areas for the sole purpose of achieving acceptable QC recoveries that would have otherwise been unacceptable. The intentional recording or reporting of incorrect information (or the intentional omission of correct information) is against company principles and policy and is grounds for immediate termination.

**19.14.5.3** Client samples, performance evaluation samples, and quality control samples are all treated equally when determining whether or not a peak area or baseline should be manually adjusted.

**19.14.5.4** All manual integrations receive a second level review. Manual integrations must be indicated on an expanded scale “after” chromatograms such that the integration performed can be easily evaluated during data review. Expanded scale “before” chromatograms are also required for all manual integrations on QC parameters (calibrations, calibration verifications, laboratory control samples, internal standards, surrogates, etc.) unless the laboratory has another documented corporate approved procedure in place that can demonstrate an active process for detection and deterrence of improper integration practices.

**Figure 19-1.**
**Example - Demonstration of Capability Documentation**

TestAmerica
THE LEADER IN ENVIRONMENTAL TESTING

BF-QA-DOC-004
DOC Cert. Statement
Rev. 3 9/28/2016

***TESTAMERICA LABORATORIES, INC.***

***DEMONSTRATION OF CAPABILITY CERTIFICATION STATEMENT***

Employee Name (print): _____

Method Number: _____ Matrix (circle): water / soil / air

Parameters or Analytes: _____

Date Submitted: _____

**Initial Demonstration of Capability:**

SOP Number: _____Revision # _____ Date Read_____

Trained By (print name): _____

Date training began: _____

Date training completed: _____

**Continued Demonstration of Capability:**

SOP Number: _____Revision # _____ Date Read_____

**Demonstration of Capability Reviewed and Analyst Authorized to Perform Method:**

_____ _____ _____
Department Manager/Designee Signature Date

_____ _____ _____
QA Manager/Designee Signature Date

# 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 laboratory SOPs. A list of laboratory equipment and instrumentation is presented in Table 20-1.

Equipment is only operated by authorized and trained personnel. Manufacturer's instructions for equipment use are readily accessible to all appropriate laboratory personnel.

## 20.2 PREVENTIVE MAINTENANCE

**20.2.1** The laboratory follows a well-defined maintenance program to ensure proper equipment operation and to prevent the failure of laboratory equipment or instrumentation during use. This program of preventive maintenance helps to avoid delays due to instrument failure.

**20.2.2** Routine preventive maintenance procedures and frequency, such as lubrication, cleaning, and replacements, should be performed according to the procedures outlined in the manufacturer's manual. Qualified personnel must also perform maintenance when there is evidence of degradation of peak resolution, a shift in the calibration curve, loss of sensitivity, or failure to continually meet one of the quality control criteria.

**20.2.3** Table 20-2 lists examples of scheduled routine maintenance. It is the responsibility of each Department Manager to ensure that instrument maintenance logs are kept for all equipment in his/her department. Preventative maintenance procedures may also be outlined in analytical SOPs or instrument manuals. (Note: for some equipment, the log used to monitor performance is also the maintenance log. Multiple pieces of equipment may share the same log as long as it is clear as to which instrument is associated with an entry.)

**20.2.4** Instrument maintenance logs are controlled and are used to document instrument problems, instrument repair and maintenance activities. Maintenance logs shall be kept for all major pieces of equipment. Instrument maintenance logs may also be used to specify instrument parameters.

**20.2.4.1** Documentation must include all major maintenance activities such as contracted preventive maintenance and service and in-house activities such as the replacement of electrical components, lamps, tubing, valves, columns, detectors, cleaning and adjustments.

**20.2.4.2** Each entry in the instrument log includes the Analyst's initials, the date, a detailed description of the problem (or maintenance needed/scheduled), a detailed explanation of the solution or maintenance performed, and a verification that the equipment is functioning properly (state what was used to determine a return to control. e.g. CCV run on *'date'* was acceptable, or instrument recalibrated on 'date' with acceptable verification, etc.) must also be documented in the instrumentation records.

**20.2.4.3** When maintenance or repair is performed by an outside agency, service receipts detailing the service performed can be affixed into the logbooks adjacent to pages describing the maintenance performed. This stapled in page must be signed across the page entered and the logbook so that it is clear that a page is missing if only half a signature is found in the logbook.

**20.2.5** If an instrument requires repair (subjected to overloading or mishandling, gives suspect results, or otherwise has shown to be defective or outside of specified limits) it shall be taken out of operation and tagged as out of service or otherwise isolated until such a time as the repairs have been made and the instrument can be demonstrated as operational by calibration and/or verification or other test to demonstrate acceptable performance. The laboratory shall examine the effect of this defect on previous analyses

**20.2.6** In the event of equipment malfunction that cannot be resolved, service shall be obtained from the instrument vendor manufacturer, or qualified service technician, if such a service can be tendered. If on-site service is unavailable, arrangements shall be made to have the instrument shipped back to the manufacturer for repair. Back up instruments, which have been approved, for the analysis shall perform the analysis normally carried out by the malfunctioning instrument. If the back up is not available and the analysis cannot be carried out within the needed timeframe, the samples shall be subcontracted.

At a minimum, if an instrument is sent out for service or transferred to another facility, it must be recalibrated and the laboratory MDL verified (using an MDLV) 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 and volumetric dispensing devices if quantitative results are dependent on their accuracy, as in standard preparation and dispensing or dilution into a specified volume. All raw data records associated with the support equipment are retained to document instrument performance. Laboratory SOPs BF-GP-001,"Calibration of Autopipettes and Repipetters" and BF-GP-002, "Support Equipment: Maintenance, Record Keeping and Corrective Actions of Analytical Balances, Temperature Control Devises and Reagent Water" provide additional detail on the monitoring and record keeping for support equipment.

### 20.3.1 Weights and Balances

The accuracy of the balances used in the laboratory is checked every working day, before use. All balances are placed on stable counter tops.

Each balance is checked prior to initial serviceable use with at least two certified ASTM type 1 weights spanning its range of use (weights that have been calibrated to ASTM type 1 weights may also be used for daily verification). ASTM type 1 weights used only for calibration of other weights (and no other purpose) are inspected for corrosion, damage or nicks at least annually and if no damage is observed, they are calibrated at least every 5 years by an outside calibration laboratory. Any weights (including ASTM Type 1) used for daily balance checks or other purposes are recalibrated/recertified annually to NIST standards (this may be done internally if laboratory maintains "calibration only" ASTM type 1 weights).

All balances are serviced annually by a qualified service representative, who supplies the laboratory with a certificate that identifies traceability of the calibration to the NIST standards.

All of this information is recorded in logs, and the recalibration/recertification certificates are kept on file.

### 20.3.2 pH, Conductivity, and Turbidity Meters

The pH meters used in the laboratory are accurate to ± 0.1 pH units, and have a scale readability of at least 0.05 pH units. The meters automatically compensate for the temperature, and are calibrated with at least two working range buffer solutions before each use.

Conductivity meters are also calibrated before each use with a known standard to demonstrate the meters do not exceed an error of 1% or one umhos/cm.

Turbidity meters are also calibrated before each use. All of this information is documented in logs.

Consult pH and Conductivity, and Turbidity SOPs for further information.

### 20.3.3 Thermometers

All reusable thermometers are calibrated on an annual basis with a NIST-traceable thermometer.

- If the temperature measuring device is used over a range of 10°C or less, then a single point verification within the range of use is acceptable;
- If the temperature measuring device is used over a range of greater than 10°C, then the verification must bracket the range of use.

Disposable thermometers are discarded upon expiration and replaced with newly purchased thermometers. IR thermometers should be calibrated over the full range of use, including ambient, iced (4 degrees) and frozen (0 to -5 degrees), per the Drinking Water Manual. The IR thermometers are verified daily and calibrated quarterly. Digital probes and thermocouples are calibrated quarterly.

The NIST Mercury thermometer is recalibrated every five years (unless thermometer has been exposed to temperature extremes or apparent separation of internal liquid) by an approved outside service and the provided certificate of traceability is kept on file. The NIST digital

thermometer is recalibrated every one year (unless thermometer has been exposed to temperature extremes or apparent separation of internal liquid) by an approved outside service and the provided certificate of traceability is kept on file The NIST thermometer(s) have increments of 1 degree (0.5 degree or less increments are required for drinking water microbiological laboratories) and have ranges applicable to method and certification requirements. The NIST traceable thermometer is used for no other purpose than to calibrate other thermometers.

All of this information is documented in logbooks. Monitoring method-specific temperatures, including incubators, heating blocks, water baths, and ovens, is documented in method-specific logbooks. More information on this subject can be found in the laboratory SOP BF-GP-020, "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.

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 > 0ºC and ≤ 6 ºC.

Specific temperature settings/ranges for other refrigerators, ovens waterbaths, and incubators can be found in method specific SOPs.

All of this information is documented in Daily Temperature Logbooks and method-specific logbooks.

### 20.3.5 Autopipettors, Dilutors, and Syringes

Mechanical volumetric dispensing devices including burettes (except Class A Glassware 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 applied to the device stating that it is not calibrated. Any device not regularly verified can not be used for any quantitative measurements.

Micro-syringes are purchased from Hamilton Company. Each syringe is traceable to NIST. The laboratory keeps on file an "Accuracy and Precision Statement of Conformance" from Hamilton attesting established accuracy.

### 20.3.6 Field Sampling Devices (Isco Auto Samplers)

Each Auto Sampler (ISCO) is assigned a unique identification number in order to keep track of the calibration. This number is also recorded on the sampling documentation.

The Auto Sampler is calibrated monthly (or if not utilized monthly, immediately prior to its usage) by setting the sample volume to 100ml and recording the volume received. The results are filed in a logbook/binder. The Auto Sampler is programmed to run three (3) cycles and each of the three cycles is measured into a graduated cylinder to verify 100ml are received.

If the RSD (Relative Standard Deviation) between the 3 cycles is greater than 10%, the procedure is repeated and if the result is still greater than 10%, then the Auto Sampler is taken out of service until it is repaired and calibration verification criteria can be met. The results of this check are kept in a logbook/binder.

Additional calibration and use information is detailed in laboratory SOP BF-FS-006, “Calibration of Field Meter”.

## 20.4 INSTRUMENT CALIBRATIONS

Calibration of analytical instrumentation is essential to the production of quality data. Strict calibration procedures are followed for each method. These procedures are designed to determine and document the method detection limits, the working range of the analytical instrumentation and any fluctuations that may occur from day to day.

Sufficient raw data records are retained to allow an outside party to reconstruct all facets of the initial calibration. Records contain, but are not limited to, the following: calibration date, method, instrument, analyst(s) initials or signatures, analysis date, analytes, concentration, response, type of calibration (Avg RF, curve, or other calculations that may be used to reduce instrument responses to concentration.)

Sample results must be quantitated from the initial calibration and may not be quantitated from any continuing instrument calibration verification unless otherwise required by regulation, method or program.

If the initial calibration results are outside of the acceptance criteria, corrective action is performed and any affected samples are reanalyzed if possible. If the reanalysis is not possible, any data associated with an unacceptable initial calibration will be reported with appropriate data qualifiers (refer to Section 12).

**Note:** Instruments are calibrated initially and as needed after that and at least annually.

### 20.4.1 Calibration Standards

Calibration standards are prepared using the procedures indicated in the Reagents and Standards section of the determinative method SOP. If a reference method does not specify the number of calibration standards, a minimum of 3 calibration points will be used.

**20.4.1.1** Standards for instrument calibration are obtained from a variety of sources. All standards are traceable to national or international standards of measurement, or to national or international standard reference materials.

**20.4.1.2** The lowest concentration calibration standard that is analyzed during an initial calibration must be at or below the stated reporting limit for the method based on the final volume of extract (or sample).

**20.4.1.3** The other concentrations define the working range of the instrument/method or correspond to the expected range of concentrations found in actual samples that are also within the working range of the instrument/method. Results of samples not bracketed by initial instrument calibration standards (within calibration range to 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 exceptions to these rules is ICP and ICPMS methods which define the working range with periodic linear dynamic range studies, rather than through the range of concentrations of daily calibration standards.

**20.4.1.4** All initial calibrations are verified with a standard obtained from a second source and traceable to a national standard, when available (or vendor certified different lot if a second source is not available). For unique situations, such as air analysis where no other source or lot is available, a standard made by a different analyst would be considered a second source. This verification occurs immediately after the calibration curve has been analyzed, and before the analysis of any samples.

### 20.4.2 Calibration Verification

The calibration relationship established during the initial calibration must be verified at least daily as specified in the laboratory method SOPs in accordance with the referenced analytical methods and 2009 TNI Std. EL-V1M4, section 1.7.1. The process of calibration verification applies to both external standard and internal standard calibration techniques, as well as to linear and non-linear calibration models. Initial calibration verification is with a standard source secondary (second source standard) to the calibration standards, but continuing calibration verifications may use the same source standards as the calibration curve.

**Note:** The process of calibration verification referred to is fundamentally different from the approach called "calibration" in some methods. As described in those methods, the calibration factors or response factors calculated during calibration are used to update the calibration factors or response factors used for sample quantitation. This approach, while employed in other EPA programs, amounts to a daily single-point calibration.

All target analytes and surrogates, including those reported as non-detects, must be included in periodic calibration verifications for purposes of retention time confirmation and to demonstrate that calibration verification criteria are being met i.e., RPD, per NELAC (2003) Standard, Section 5.5.5.10 and 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.

**Note:** If an internal standard calibration is being used then bracketing calibration verification standards are not required, only daily verifications are needed. The results from these verification standards must meet the calibration verification criteria and the retention time criteria (if applicable).

Generally, the initial calibrations must be verified at the beginning of each 12-hour analytical shift during which samples are analyzed. (Some methods may specify more or less frequent verifications). The 12-hour analytical shift begins with the injection of the calibration verification standard (or the MS tuning standard in MS methods). The shift ends after the completion of the analysis of the last sample, QC, or standard that can be injected within 12 hours of the beginning of the shift.

A continuing instrument calibration verification (CCV) must be repeated at the beginning and, for methods that have quantitation by external calibration models, at the end of each analytical batch. Some methods have more frequent CCV requirements see specific SOPs. Most Inorganic methods require the CCV to be analyzed after ever 10 samples or injections, including matrix or batch QC samples.

Note: If an internal standard calibration is being used (basically GCMS) then bracketing standards are not required, only daily verifications are needed. The results from these verification standards must meet the calibration verification criteria and the retention time criteria (if applicable).

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.

#### 20.4.2.1 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.

- When the acceptance criteria for the calibration verification are exceeded high, i.e., high bias, and there are associated samples that are non-detects, then those non-detects may be reported. Otherwise, the samples affected by the unacceptable calibration verification shall be reanalyzed after a new calibration curve has been established, evaluated and accepted.

- When the acceptance criteria for the calibration verification are exceeded low, i.e., low bias, those sample results may be reported if they exceed a maximum regulatory limit/decision level. Otherwise, the samples affected by the unacceptable verification shall be reanalyzed after a new calibration curve has been established, evaluated and accepted. Alternatively, a reporting limit standard may be analyzed to demonstrate that the laboratory can still support non-detects at their reporting limit.

### 20.5 TENTATIVELY IDENTIFIED COMPOUNDS (TICS) – GC/MS ANALYSIS

For samples containing components not associated with the calibration standards, a library search may be made for the purpose of tentative identification. The necessity to perform this type of identification will be determined by the purpose of the analyses being conducted. Data system library search routines should not use normalization routines that would misrepresent the library or unknown spectra when compared to each other.

**Note:** If the TIC compound is not part of the client target analyte list but is calibrated by the laboratory and is both qualitatively and/or quantitatively identifiable, it should not be reported as a TIC. If the compound is reported on the same form as true TICs, it should be qualified and/or narrated that the reported compound is qualitatively and quantitatively (if verification in control) reported compared to a known standard that is in control (where applicable).

For example, the RCRA permit or waste delisting requirements may require the reporting of non-target analytes. Only after visual comparison of sample spectra with the nearest library searches may the analyst assign a tentative identification. See laboratory SOP's BF-MB-005 and BF-MV-007 for guidelines for making tentative identifications

**Note:**
For general reporting if TICs are requested, the ten (10), largest non-target analyte peaks whose area count exceeds 10% of the nearest internal standard will be termed "Tentatively Identified Compounds" (TICs). More or fewer TICs may be identified based on client requirements.

## 20.6 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. Laboratory Equipment and Instrumentation – TestAmerica Buffalo

TestAmerica Buffalo
Equipment/Instrument List

Page 1

| Equipment/Instrument | Manufacturer | Model Number | S/N from instrument | Year Put into Service | Condition When Received |
|---|---|---|---|---|---|
| GC/MS Instrumentation | Agilent | 5975 | US83110163 | 2013 | good |
| GC/MS Instrumentation | Agilent | 5973 | US05605976 | 2001 | good |
| GC/MS Instrumentation | Agilent | 5973 | US44621446 | 2005 | good |
| GC/MS Instrumentation | Agilent | 5973 | US52420646 | 2005 | good |
| GC/MS Instrumentation | Agilent | 5973 | US05060084 | 2001 | good |
| GC/MS Instrumentation | Agilent | 5973 | US03950346 | 2001 | good |
| GC/MS Instrumentation | Agilent | 5973 | US82321636 | 2001 | good |
| GC/MS Instrumentation | Agilent | 5973 | US21854062 | 2003 | good |
| GC/MS Instrumentation | Agilent | 5973 | US41720721 | 2009 | good |
| GC/MS Instrumentation | Agilent | 5973 | US30965692 | 2003 | good |
| GC/MS Instrumentation | Agilent | 5973 | US30965634 | 2003 | good |
| GC/MS Instrumentation | Agilent | 5973 | US35120354 | 2004 | good |
| GC/MS Instrumentation | Agilent | 5973 | US41720707 | 2004 | good |
| GC/MS Instrumentation | Agilent | 5975 | US80838844 | 2001 | good |
| GC/MS Instrumentation | Agilent | 5975 | US83130241 | 2013 | good |
| GC/MS Instrumentation | Agilent | 5973 | US02450141 | 2012 | good |
| GC Instrumentation | Agilent | 6890 dual uECD | CN10520009 | 2005 | good |
| GC Instrumentation | Agilent | 6890 dual uECD | CN10520010 | 2005 | good |
| GC Instrumentation | Agilent | 6890 dual uECD | CN10448015 | 2005 | good |
| GC Instrumentation | Hewlett Packard | 5890II dual ECD | 3310A47661 | 1993 | good |
| GC Instrumentation | Hewlett Packard | 5890II dual ECD | 3336A53325 | 1993 | good |
| GC Instrumentation | Hewlett Packard | 5890II dual ECD | 3336A53464 | 1994 | good |
| GC Instrumentation | Hewlett Packard | 5890II dual ECD | 3336A53463 | 1994 | good |
| GC Instrumentation | Hewlett Packard | 5890II dual FID | 3336A53727 | 1994 | good |
| GC Instrumentation | Hewlett Packard | 5890II dual FID | 3019A28433 | 1991 | good |
| GC Instrumentation | Hewlett Packard | 5890II FID/FID | 3336A53729 | 1994 | good |
| GC Instrumentation | Hewlett Packard | 5890II Hall/PID | 3121A35782 | 1990 | good |
| GC Instrumentation | Hewlett Packard | 5890II PID/FID | 3336A60622 | 1994 | good |
| GC Instrumentation | Hewlett Packard | 5890II PID/FID | 3133A37157 | 1993 | good |
| GC Instrumentation | Hewlett Packard | 5890II PID/FID | 3336a53465 | 1994 | good |
| GC Instrumentation | Agilent | 6890N dual uECD | CN10839003 | 2005 | good |
| GC Instrumentation | Agilent | 7890N dual FID | CN10833020 | 2005 | good |
| GC Instrumentation | Perkin Elmer | Clarus 600 dual FID | 665S10020401 | 2012 | good |
| GC Instrumentation | Perkin Elmer | Clarus 600 dual FID | 680s10101807 | 2013 | good |
| GC Instrumentation | Perkin Elmer | Clarus 608 dual uECD | 680S10042901 | 2012 | good |
| Ion Chromatography Instrumentation | Dionex | Ion Chromatograph #DX-120 | 99110569 | 1999 | good |
| Ion Chromatography Instrumentation | Dionex | Ion Chromatograph #DX-120 | 02060196 | 2002 | good |
| Ion Chromatography Instrumentation | Dionex | Ion Chromatograph #DX-120 | 20126 | 2004 | good |
| Ion Chromatography Instrumentation | Dionex | Ion Chromatograph #DX-120 | 98050413 | 1999 | good |
| Metals Instrumentation | Environmental Express | AutoBlock Plus | AB4001-1213-042 | 2013 | Good |

Revision 09-2016

**TestAmerica Buffalo**
**Equipment/Instrument List**

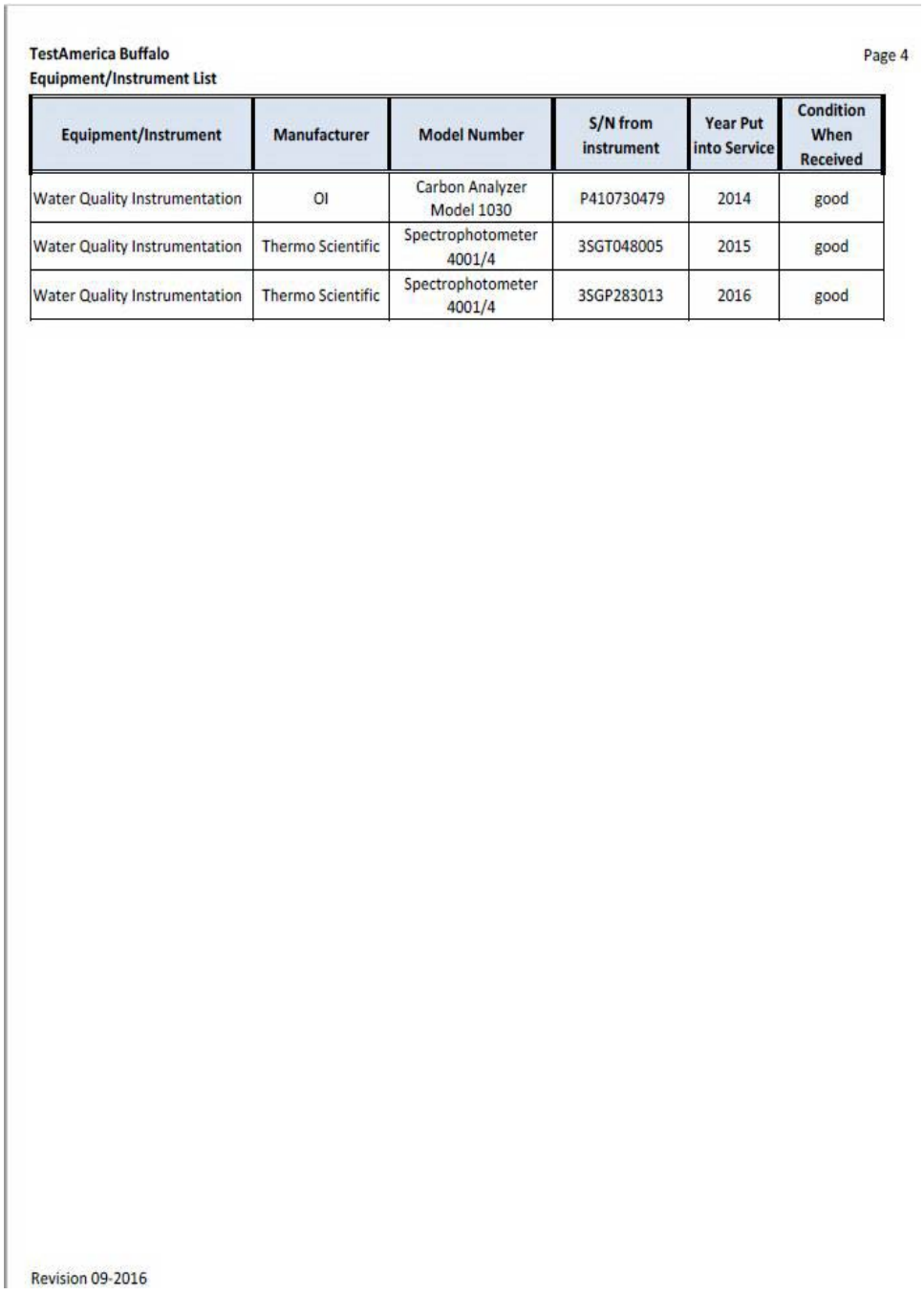


| Equipment/Instrument | Manufacturer | Model Number | S/N from instrument | Year Put into Service | Condition When Received |
|---|---|---|---|---|---|
| Metals Instrumentation | Leeman | PS200 II | HG0033 | 2000 | good |
| Metals Instrumentation | Leeman | PS200 II | 4026 | 2000 | good |
| Metals Instrumentation | Perkin Elmer | Elan 9000 ICP-MS | P0230202 | 2002 | good |
| Metals Instrumentation | Thermo | ICAP 6000 Duo | ICP-20094603 | 2010 | good |
| Metals Instrumentation | Thermo | ICAP 6000 Duo | ICP-20094602 | 2010 | good |
| Sample Preparation Equipment | CEM | Microwave MARS | MD3978 | 2013 | good |
| Sample Preparation Equipment | Gilson | Fractionator Model GX-274 | 40579 | 2013 | good |
| Sample Preparation Equipment | Heat Systems | Sonicator #XL-2020 | G1647/C5659 | 1994 | good |
| Sample Preparation Equipment | Heat Systems | Sonicator #XL-2020 | G2665/C5674 | 1994 | good |
| Sample Preparation Equipment | Heat Systems | Sonicator #XL-2020 | G2620/C5660 | 1994 | good |
| Sample Preparation Equipment | Heat Systems | Sonicator #XL-2020 | G2245/C6328 | 1995 | good |
| Sample Preparation Equipment | Heat Systems | Sonicator #XL-2020 | G2621/C6733 | 1995 | good |
| Sample Preparation Equipment | Heat Systems | Sonicator #XL-2020 | G2713/C6732 | 1995 | good |
| Sample Preparation Equipment | Heat Systems | Sonicator #XL-2020 | G1643/C6837 | 1995 | good |
| Sample Preparation Equipment | Heat Systems | Sonicator #XL-2020 | G2742/C6842 | 1995 | good |
| Sample Preparation Equipment | Organomation | Rot-X-Tractor | 16902 | 1999 | good |
| Sample Preparation Equipment | Organomation | Rot-X-Tractor | 16907 | 1999 | good |
| Sample Preparation Equipment | Organomation | Rot-X-Tractor | 16913 | 1999 | good |
| Sample Preparation Equipment | TurboVap | II | TV0529N12427 | 2006 | good |
| Sample Preparation Equipment | TurboVap | II | TV0529N12428 | 2006 | good |
| Sample Preparation Equipment | TurboVap | II | TV9445N5816 | 1996 | good |
| Sample Preparation Equipment | TurboVap | II | TV9427N4133 | 1996 | good |
| Sample Preparation Equipment | TurboVap | II | TV944N5819 | 1996 | good |
| Sample Preparation Equipment | TurboVap | II | TV944N5820 | 1996 | good |
| Sample Preparation Equipment | TurboVap | II | TV0024N9623 | 2000 | good |

**TestAmerica Buffalo**
**Equipment/Instrument List**



| Equipment/Instrument | Manufacturer | Model Number | S/N from instrument | Year Put into Service | Condition When Received |
|---|---|---|---|---|---|
| Sample Preparation Equipment | TurboVap | II | TV0022N9604 | 2000 | good |
| Sample Preparation Equipment | TurboVap | II | TV0312N11592 | 2003 | good |
| Sample Preparation Equipment | TurboVap | II | TV0312N11591 | 2003 | good |
| Water Quality Instrumentation | Flash Point Analyzer | HFP 339 | 73390092 | 2007 | good |
| Water Quality Instrumentation | Flash Point Analyzer | Optiflash 104002 | Herzog PAC 000334 | 2015 | good |
| Water Quality Instrumentation | Glastron | CN Midi-distillation | 2502 | 2003 | good |
| Water Quality Instrumentation | Glastron | Phenol Midi-distillation | 2069 | 2003 | good |
| Water Quality Instrumentation | Glastron | Phenol Midi-distillation | 2053 | 2003 | good |
| Water Quality Instrumentation | Horizon | Speed Vap | 03-0415 | 2005 | good |
| Water Quality Instrumentation | Konelab | 20 | S5019455 | 2004 | good |
| Water Quality Instrumentation | Konelab | 20XT | E3719731 | 2005 | good |
| Water Quality Instrumentation | Konelab | Aqua20 | SEA032 | 2009 | good |
| Water Quality Instrumentation | Lachat | Quickchem 8000 Autoanalyzer | A83000-1527 | 2000 | good |
| Water Quality Instrumentation | Lachat | Quickchem 8500 Autoanalyzer | 40300001665 | 2014 | good |
| Water Quality Instrumentation | Lachat | Quickchem 8500 Autoanalyzer | 1106 0000 1336 | 2013 | good |
| Water Quality Instrumentation | Mantech | BOD Analyzer | MT-0B4-215 | 2015 | good |
| Water Quality Instrumentation | Mantech | BOD Autoanalyzer | MS-1LO-157 | 2014 | good |
| Water Quality Instrumentation | ManTech | PC Titrator | MS-OK2-607 | 2003 | good |
| water Quality Instrumentation | Mantech | PC Titrator | MT-1H5-971 | 2016 | good |
| Water Quality Instrumentation | OI | Carbon Analyzer Model 1030 | A547730578 | 2005 | good |
| Water Quality Instrumentation | OI | Carbon Analyzer Model 1030 | E616730030 | 2006 | good |



TestAmerica Buffalo
Equipment/Instrument List

Page 4

| Equipment/Instrument | Manufacturer | Model Number | S/N from instrument | Year Put into Service | Condition When Received |
|---|---|---|---|---|---|
| Water Quality Instrumentation | OI | Carbon Analyzer Model 1030 | P410730479 | 2014 | good |
| Water Quality Instrumentation | Thermo Scientific | Spectrophotometer 4001/4 | 3SGT048005 | 2015 | good |
| Water Quality Instrumentation | Thermo Scientific | Spectrophotometer 4001/4 | 3SGP283013 | 2016 | good |

Revision 09-2016

Note: The Equipment List is current at the date of publication of this manual. An updated list may be obtained by contacting the TestAmerica Buffalo Quality Department.

**Table 20-2.**

**Schedule of Routine Maintenance**

| Instrument | Procedure | Frequency |
|---|---|---|
| Leeman Mercury Analyzer | Check tubing for wear<br>Fill rinse tank with 10% HCl<br>Change dryer tube<br>Fill reductant bottle with 10% Stannous Chloride | Daily<br>Daily<br>As Needed<br><br>Daily |
| ICP & ICP/MS | Check pump tubing<br>Check liquid argon supply<br>Check fluid level in waste container<br>Check re-circulator levels<br>Clean or replace filters<br>Check torch<br>Check sample spray chamber for debris<br>Clean and align nebulizer<br>Change pump oil<br>Change Cones<br>Change printer cartridge<br>Replace pump tubing | Daily<br>Daily<br>Daily<br>Monthly<br>As required<br>Daily<br>Monthly<br>Monthly<br>Monthly<br>As required<br>As required<br>As required |
| UV-Vis Spectrophotometer | Clean ambient flow cell<br>Precision check/alignment of flow cell<br>Wavelength verification check | As required<br>As required<br>Annually |
| Auto Analyzers | Clean sampler<br>Check all tubing<br>Clean inside of colorimeter<br>Clean pump well and pump rollers<br>Clean wash fluid receptacle<br>Oil rollers/chains/side rails<br>Clean optics and cells | Daily<br>Daily<br>Daily<br>Quarterly<br>Weekly<br>Weekly<br>Quarterly |

| Instrument | Procedure | Frequency |
|---|---|---|
| Agilent GC/MS | Pump oil-level check<br>Pump oil changing<br>Analyzer bake-out<br>Analyzer cleaning<br>Resolution adjustment<br><br>COMPUTER SYSTEM AND PRINTER:<br>Air filter cleaning<br>Change data system air filter<br>Printer head carriage lubrication<br>Paper sprocket cleaning<br>Drive belt lubrication | Monthly<br>Annually<br>As required<br>As required<br>As required<br><br><br>As required<br>As required<br>As required<br>As required<br>As required |
| Gas Chromatograph | Compare standard response to previous day or since last initial calibration<br>Check carrier gas flow rate in column<br><br>Check temp. of detector, inlet, column oven<br>Septum replacement<br>Glass wool replacement<br>Check system for gas leaks with SNOOP<br><br>Check for loose/frayed power wires and insulation<br>Bake injector/column<br>Change/remove sections of guard column<br>Replace connectors/liners<br>Change/replace column(s) | Daily<br><br>Daily via use of known compound retention<br>Daily<br>As required<br>As required<br>W/cylinder change as required<br>As Required<br>As Required<br>As Required<br>As Required<br>As Required |
| Electron Capture Detector (ECD) | Detector wipe test (Ni-63)<br>Detector cleaning | Semi-annually<br>As required |
| Flame Ionization Detector (FID) | Detector cleaning | As required |
| Photoionization Detector (PID) | Change O-rings<br>Clean lamp window | As required<br>As required |
| HPLC | Change guard columns<br>Change lamps<br>Change pump seals<br><br>Replace tubing<br>Change fuses in power supply<br>Filter all samples and solvents<br>Change autosampler rotor/stator | As required<br>As required<br>Semi-annually or as required<br>As required<br>As required<br>Daily<br>As required |

| Instrument | Procedure | Frequency |
|---|---|---|
| Vacuum Pumps/<br>Air Compressor | Drained<br>Belts checked<br>Lubricated | Weekly<br>Monthly<br>Semi-annually |
| Centrifuge | Check brushes and bearings | Every 6 months or as needed |

**Table 20-3.**

**Periodic Calibration**

| Instrument | Type of Calibration/ Number of Standards | Frequency | Acceptance Limits | Corrective Action |
|---|---|---|---|---|
| Analytical Balance | Accuracy determined using "S" NIST traceable weights. Minimum of 2 standards bracketing the weight of interest.<br><br>Inspected and calibrated by A2LA accredited person annually. | Daily, when used<br><br>Annual | ± 0.2% | Clean, check level, insure lack of drafts, and that unit is warmed up, recheck. If fails, call service. |
| Top Loading Balance | Accuracy determined using "S" NIST traceable. Minimum of 2 standards bracketing the weight of interest.<br><br>Inspected and calibrated by A2LA accredited person annually. | Daily, when used<br><br>Annual | ± 0.5% | Clean. Replace. |
| NIST Certified Weights | Accuracy determined by accredited weights and measurement laboratory. | 1 year | As per certificate. | Replace. |
| NIST-Traceable Thermometer-Mercury | Accuracy determined by accredited measurement laboratory. | 3 years | As per certificate. | Replace. |
| NIST-Traceable Thermometer-Digital | Accuracy determined by accredited measurement laboratory. | 1 year | As per certificate | Replace. |
| Thermometer | Against NIST-traceable thermometer | Yearly at appropriate temperature range for intended use | ± 2.0°C | Replace |
| Minimum-Maximum Thermometers | Against NIST-traceable thermometer | Yearly | ± 2.0°C | Replace |

| Instrument | Type of Calibration/ Number of Standards | Frequency | Acceptance Limits | Corrective Action |
|---|---|---|---|---|
| InfraRed Temperature Guns | Against NIST-traceable thermometer<br><br>Accuracy determined by accredited measurement laboratory. | Daily at appropriate temperature range for intended use.<br><br>Annual | ± 2.0°C | Repair/replace |
| Dial-type Thermometers | Against NIST-traceable thermometer | Quarterly at appropriate temperature range for intended use. | ± 2.0°C | Replace |
| Refrigerator | Temperature checked using NIST-traceable thermometer. | Daily. If out of range, check again in two hours. | 0-6°C | Adjust. Repair. While waiting for repair, seal door, attach "Out of Service" sign, move items to functional unit. Notify supervisor. |
| Freezer | Temperature checked using NIST-traceable thermometer | Daily. If out of range, check again in two hours. | (-10)-(-20)°C | Adjust. Repair. While waiting for repair, seal door, attach "Out of Service" sign, move items to functional unit. Notify supervisor. |
| Oven | Temperature checked using NIST-traceable thermometer. | When in use. | 104 ± 1°C (drying)<br>180 ± 2°C (TDS) | Adjust. Replace. |
| Water Bath | Temperature checked using NIST-traceable thermometer. | When in use. | ± 2°C | Adjust. Replace. |
| Volumetric Dispensing Devices (Eppendorf ® pipette, automatic dilutor or dispensing devices) | One delivery by weight. Using DI water or solvent of use, dispense into tared vessel. Record weight with device ID number.<br><br>Calibrate using 4 replicate gravimetric measurements | Each day of use<br><br>Quarterly | ± 2%<br>Calculate accuracy by dividing weight by stated volume times 100 for percent. | Adjust. Replace. |

| Instrument | Type of Calibration/ Number of Standards | Frequency | Acceptance Limits | Corrective Action |
|---|---|---|---|---|
| Glass Microliter Syringes | None | Accuracy must be initially demonstrated if syringe was not received with a certificate attesting to established accuracy. | ± 1% | Not applicable. |
| Deionized Water | Check in-line conductivity meter on system with conductivity meter in Inorganics Department. | Daily | <1.0 µmho at 25°C | Record on log. Report discrepancies to QA Manager, Operations Manager or Technical Manager. |

# 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. For certain programs Microsyringes are verified semi-annually or disposed of after 6 months of use. Wherever possible, subsidiary or peripheral equipment is checked against standard equipment or standards that are traceable to national or international standards. Class A Glassware 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), or another accreditation organization that is a signatory to a MRA (Mutual Recognition Arrangement) of one or more of the following cooperations – ILAC (International Laboratory accreditation Cooperation) or APLAC (Asia – Pacific Laboratory Accreditation Cooperation)..A certificate and scope of accreditation is kept on file at the laboratory.

The calibration report or certificate submitted to ***TestAmerica Buffalo*** contains, in a well designed format, a traceability statement, the conditions under which the calibrations were made in the context of any potential influence, a compliance statement with an identified metrological specification and the pertinent clauses, a clearly identified record of the quantities and functional test results before and after re-calibration, and no recommendation on the calibration interval. Opinions and interpretations of results are presented along with the basis upon which they were made and identified as such. The report may be submitted by facsimile or other electronic means as long as the requirements of the International Standard are achieved. If significant amendments are made to a calibration certificate, a supplemental certificate for the serial-number-specified piece of equipment is so identified. When a new certificate is offered, it uniquely identifies and references the one it replaces. All calibration reports are filed in the QA Office.

An external certified service engineer services laboratory balances on an annual basis. This service is documented on each balance with a signed and dated certification sticker. Balance calibrations are checked each day of use. All mercury thermometers are calibrated annually against a traceable reference thermometer. Temperature readings of ovens, refrigerators, and incubators are checked on each day of use.

## 21.3 REFERENCE STANDARDS / MATERIALS

Reference standards/materials, where commercially available, are traceable to certified reference materials. Commercially prepared standard materials are purchased from vendors accredited by ISO Guide 34 and ISO/IEC Guide 17025. All reference standards from commercial vendors shall be accompanied with a certificate that includes at least the following information:

- Manufacturer
- Analytes or parameters calibrated
- Identification or lot number
- Calibration method
- Concentration with associated uncertainties
- Purity

If a standard cannot be purchased from a vendor that supplies a Certificate of Analysis, the purity of the standard is documented by analysis. The receipt of all reference standards must be documented. Reference standards are labeled with a unique Standard Identification Number and expiration date. All documentation received with the reference standard is retained as a QC record and references the Standard Identification Number.

All reference, primary and working standards/materials, whether commercially purchased or laboratory prepared, must be checked regularly to ensure that the variability of the standard or material from the 'true' value does not exceed method requirements. The accuracy of calibration standards is checked by comparison with a standard from a second source. In cases where a second standard manufacturer is not available, a vendor certified different lot is acceptable for use as a second source. For unique situations, such as air analysis where no other source or lot is available, a standard made by a different analyst would be considered a second source. The appropriate Quality Control (QC) criteria for specific standards are defined in laboratory SOPs. In most cases, the analysis of an Initial Calibration Verification (ICV) or LCS (where there is no sample preparation) is used as the second source confirmation. These checks are generally performed as an integral part of the analysis method (e.g. calibration checks, laboratory control samples).

All standards and materials must be stored and handled according to method or manufacturer's requirements in order to prevent contamination or deterioration. Refer to the Corporate Environmental Health & Safety Manual or laboratory SOPs. Method specific information may also be found in the laboratory method SOPs in the "Standards and Reagents" sections. For safety requirements, please refer to method SOPs and the laboratory Environmental Health and Safety Manual.

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 SOP No. CA-Q-S-001, Solvent and Acid Lot Testing and Approval.

All manufacturer or vendor supplied Certificate of Analysis or Purity must be retained, stored appropriately, and readily available for use and inspection. These records are maintained by each department in bound or electronic folders. Records must be kept of the date of receipt and date of expiration of standards, reagents and reference materials. In addition, records of preparation of laboratory standards, reagents, and reference materials must be retained, stored appropriately, and be readily available for use and inspection. For detailed information on documentation and labeling, please refer laboratory SOP BF-GP-019, “Standard Traceability and Preparation” and also to the method specific SOPs.

Commercial materials purchased for preparation of calibration solutions, spike solutions, etc.., are usually accompanied with an assay certificate or the purity is noted on the label. If the assay purity is 96% or better, the weight provided by the vendor may be used without correction. If the assay purity is less than 96% a correction will be made to concentrations applied to solutions prepared from the stock commercial material. Blended gas standard cylinders use a nominal concentration if the certified value is within +/-15%, otherwise the certified values is used for the canister concentration.

**21.4.1** All standards, reagents, and reference materials must be labeled in an unambiguous manner. Standards are logged into the laboratory department’s chemical history log and are assigned a unique identification number. Preparation of working standards or reagents prepared from the stock is documented in the laboratory Department’s Standard Preparation Log. The following information is typically recorded:

- Standard ID
- Description of Standard
- Department
- Preparer’s name
- Final volume and number of vials prepared
- Solvent type and lot number
- Preparation Date

- Expiration Date
- Standard source type (stock or daughter)
- Standard type (spike, surrogate, other)
- Parent standard ID (if applicable)
- Parent Standard Analyte Concentration (if applicable)
- Parent Standard Amount used (if applicable)
- Component Analytes
- Final concentration of each analyte
- Comment section

Records are maintained for standard and reference material preparation. These records show the traceability to purchased stocks or neat compounds. These records also include method of preparation, date of preparation, expiration date and preparer's name or initials. Preparation procedures are provided in the Method SOPs.

**21.4.2** All standards, reagents, and reference materials must be clearly labeled with a minimum of the following information:

- Expiration Date
- Standard ID 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 in the LIMS system.

**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 preparation/analytical batch records.

All reagents and standards must be stored in accordance to the following priority: 1) with the manufacturer's recommendations; 2) with requirements in the specific analytical methods as specified in the laboratory SOPs.

# SECTION 22.0

# SAMPLING

## 22.1 OVERVIEW

The laboratory provides sampling services. Sampling procedures are described in the following SOPs:

| | |
|---|---|
| **BF-FS-001** | Chain of Custody Documentation |
| **BF-FS-003** | Groundwater Sampling Field Data Collection |
| **BF-FS-004** | Equipment Decontamination |
| **BF-FS-005** | Groundwater/Surface Water Sampling |
| **BF-FS-006** | Calibration of Field Meter |
| **BF-FS-007** | Low Flow Sampling Procedures |
| **BF-FS-008** | Surface and Subsurface Soil/Sediment Sampling |

## 22.2 SAMPLING CONTAINERS

The laboratory offers clean sampling containers for use by clients. These containers are obtained from reputable container manufacturers and meet EPA specifications as required. Certificates of cleanliness for bottles and preservatives are provided by the supplier and are maintained at the laboratory. Alternatively, the certificates may be maintained by the supplier and available to the laboratory online.

### 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:

- Hydrochloric Acid – Reagent ACS (Certified VOA Free) or equivalent
- Methanol – Purge and Trap grade
- Nitric Acid – Instra-Analyzed or equivalent
- Sodium Bisulfate – ACS Grade or equivalent
- Sodium Hydroxide – Instra-Analyzed or equivalent
- Sulfuric Acid – Instra-Analyzed or equivalent
- Sodium Thiosulfate – ACS Grade or equivalent

## 22.3 DEFINITION OF HOLDING TIME

The date and time of sampling documented on the chain-of-custody (COC) form establishes the day and time zero. As a general rule, when the maximum allowable holding time is expressed in

"days" (e.g. 14 days, 28 days), the holding time is based on calendar day measured. Holding times expressed in "hours" (e.g. 6 hours, 24 hours, etc.) are measured from date and time zero. Holding times for analysis include any necessary reanalysis. However there are some programs that determine holding time compliance based on the date and specific time of analysis compared to the time of sampling regardless of how long the holding time is. These programs will be addressed on a case-by-case basis.

## 22.4 SAMPLING CONTAINERS, PRESERVATION REQUIREMENTS, HOLDING TIMES

The preservation and holding time criteria specified in the laboratory SOPs are derived from the source documents for the methods. If method required holding times, this info is in the SOP or preservation requirements are not met, the reports will be qualified using a flag, footnote or case narrative. As soon as possible or "ASAP" is an EPA designation for tests for which rapid analysis is advised, but for which neither EPA nor the laboratory have a basis for a holding time.

## 22.5 SAMPLE ALIQUOTS / SUBSAMPLING

Taking a representative sub-sample from a container is necessary to ensure that the analytical results are representative of the sample collected in the field. The size of the sample container, the quantity of sample fitted within the container, and the homogeneity of the sample need consideration when sub-sampling for sample preparation. It is the laboratory's responsibility to take a representative subsample or aliquot of the sample provided for analysis.

Analysts should handle each sample as if it is potentially dangerous. At a minimum, safety glasses, gloves, and lab coats must be worn when preparing aliquots for analysis.

The following information provides general guidance for homogenization and subsampling. For laboratory specific procedures refer to SOP BF-GP-005, "Sample Homogenization and Subsampling".

# 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.

## 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. 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 shipping documents are retained with the project files.

### 23.1.2 Legal / Evidentiary Chain-of-Custody

If samples are identified for legal/evidentiary purposes on the COC or in the project notes, sample management will initiate Strict Chain of Custody procedures as defined in SOP BF-GP-018, "Strict Internal Chain-of-Custody".

## 23.2 SAMPLE RECEIPT

Samples are received at the laboratory by designated sample receiving personnel and a unique laboratory project identification number is assigned. Each sample container shall be assigned a unique sample identification number that is cross-referenced to the client identification number such that traceability of test samples is unambiguous and documented. Each sample container is affixed with a durable sample identification label. Sample acceptance, receipt, tracking and storage procedures are summarized in the following sections.

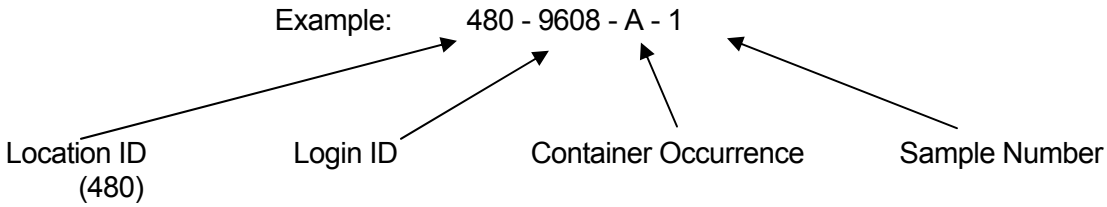
### 23.2.1 Laboratory Receipt

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 the Sample Login Form – and brought to the immediate attention of the client. The COC, shipping documents, documentation of any non-conformance, irregularity, or compromised sample receipt, record of client contact, and resulting instructions become part of the project record.

#### 23.2.1.1 Unique Sample Identification

All samples that are processed through the laboratory receive a unique sample identification to ensure that there can be no confusion regarding the identity of such samples at anytime. This system includes identification for all samples, subsamples and subsequent extracts and/or digestates.

The laboratory assigns a unique identification (e.g., Sample ID) code to each sample container received at the laboratory. This Primary ID is made up of the following information (consisting of 4 components):




The above example states that TestAmerica Buffalo Laboratory (Location 480). Login ID is 9608 (unique to a particular client/job occurrence). The container code indicates it is the first container ("A") of Sample #1.

If the primary container goes through a prep step that creates a "new" container, then the new container is considered secondary and gets another ID. An example of this being a client sample in a 1-Liter amber bottle is sent through a Liquid/Liquid Extraction and an extraction vial is created from this step. The vial would be a SECONDARY container. The secondary ID has 5 components.

Example: XXX - 9608 - A - 1 - **A** ← **Secondary Container Occurrence**

Example: 220-9608-A-1-A, would indicate the PRIMARY container listed above that went through a step that created the 1st occurrence of a Secondary container.

With this system, a client sample can literally be tracked throughout the laboratory in every step from receipt to disposal.

### 23.3 SAMPLE ACCEPTANCE POLICY

The laboratory has a written sample acceptance policy (Figure 23-2) that clearly outlines the circumstances under which samples shall be accepted or rejected. These include:

- a COC filled out completely;
- samples must be properly labeled;
- proper sample containers with adequate volume for the analysis (Sampling Guide) and necessary QC;
- samples must be preserved according to the requirements of the requested analytical method (Sampling Guide);

- sample holding times must be adhered to (Sampling Guide);
- every sample cooler is given a radiation screen with a standardized Radiation Monitor (Monitor 4 model). This screen has no analytical repercussions; it is just a gross screen for employee safety purposes. Contact TestAmerica Buffalo's Technical Manager, Environmental Health and Safety Coordinator or Sample Control Manager immediately if screening indicates radioactivity in excess of 0.02 mR/hr.;
- 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** Any deviations from these checks described in Section 23.1.1.1 that question the suitability of the sample for analysis, or incomplete documentation as to the tests required will be resolved by consultation with the client. If the sample acceptance policy criteria are not met, the laboratory shall either:

- Retain all correspondence and/or records of communications with the client regarding the disposition of rejected samples, or

- Fully document any decision to proceed with sample analysis that does not meet sample acceptance criteria.

Once sample acceptance is verified, the samples are logged into the LIMS according SOP No. BF-SR-002.

## 23.4 SAMPLE STORAGE

In order to avoid deterioration, contamination or damage to a sample during storage and handling, from the time of receipt until all analyses are complete, samples are stored in refrigerators, freezers or protected locations suitable for the sample matrix. Aqueous samples designated for metals analysis are stored at ambient temperature. In addition, samples to be analyzed for volatile organic parameters are stored in separate refrigerators designated for volatile organic parameters only. Samples are never to be stored with reagents, standards or materials that may create contamination.

To ensure the integrity of the samples during storage, refrigerator blanks are maintained in the volatile sample refrigerators and analyzed at a minimum of every two weeks.

Analysts and technicians provide a request form to the cooler custodian who then retrieves the requested samples. In the absence of the cooler custodian, the analysts may personally retrieve the sample containers allocated to their analysis from the designated refrigerator. The samples are placed on carts, transported the analytical area and analyzed. Following analysis

the remaining sample is returned to the refrigerator from which it originally came. All unused portions of samples are returned to the secure sample control area. All samples are kept in the refrigerators for two to four weeks after analysis, which meets or exceeds most sample holding times. After two to four weeks the samples are moved to dry room temperature, sample archive area where they are retained a minimum of 2 weeks after the final report has been issued to the client at which time disposal occurs. Special arrangements may be made to store samples for longer periods of time. Extended archival periods allow additional metal analyses to be performed on the archived sample and assists clients in dealing with legal matters or regulatory issues.

Access to the laboratory is controlled such that sample storage need not be locked at all times unless a project specifically demands it. Samples are accessible to laboratory personnel only. Visitors to the laboratory are prohibited from entering the refrigerator and laboratory areas unless accompanied by an employee of TestAmerica.

## 23.5 HAZARDOUS SAMPLES AND FOREIGN SOILS

To minimize exposure to personnel and to avoid potential accidents, samples which are known or suspected to be hazardous are segregated and a notification is issued to all laboratory personnel.
All hazardous samples are either returned to the client or disposed of appropriately through a hazardous waste disposal firm. All soil samples, including foreign soil samples are heat treated or incinerated in accordance with USDA permit requirements and are transported / disposed by USEPA approved facilities.

Unused portions of samples found or suspected to be hazardous according to state or federal guidelines may be returned to the client upon completion of the analytical work.

## 23.6 SAMPLE SHIPPING

In the event that the laboratory needs to ship samples, the samples are placed in a cooler with enough ice to ensure the samples remain just above freezing and at or below 6.0°C during transit. The samples are carefully surrounded by packing material to avoid breakage (yet maintain appropriate temperature). For sample shipments which include water/solid volatile organic analyses (see Note), a trip blank is enclosed when required by method specifications or state or regulatory programs. The chain-of-custody form is signed by the sample control technician and attached to the shipping paperwork. Samples are generally shipped overnight express or hand-delivered by a TestAmerica courier to maintain sample integrity. All personnel involved with shipping and receiving samples must be trained to maintain the proper chain-of-custody documentation and to keep the samples intact and on ice. The Environmental, Health and Safety Manual contains additional shipping requirements.

Note: If a client does not request trip blank analysis on the COC or other paperwork, the laboratory will analyze the trip blanks that were supplied.

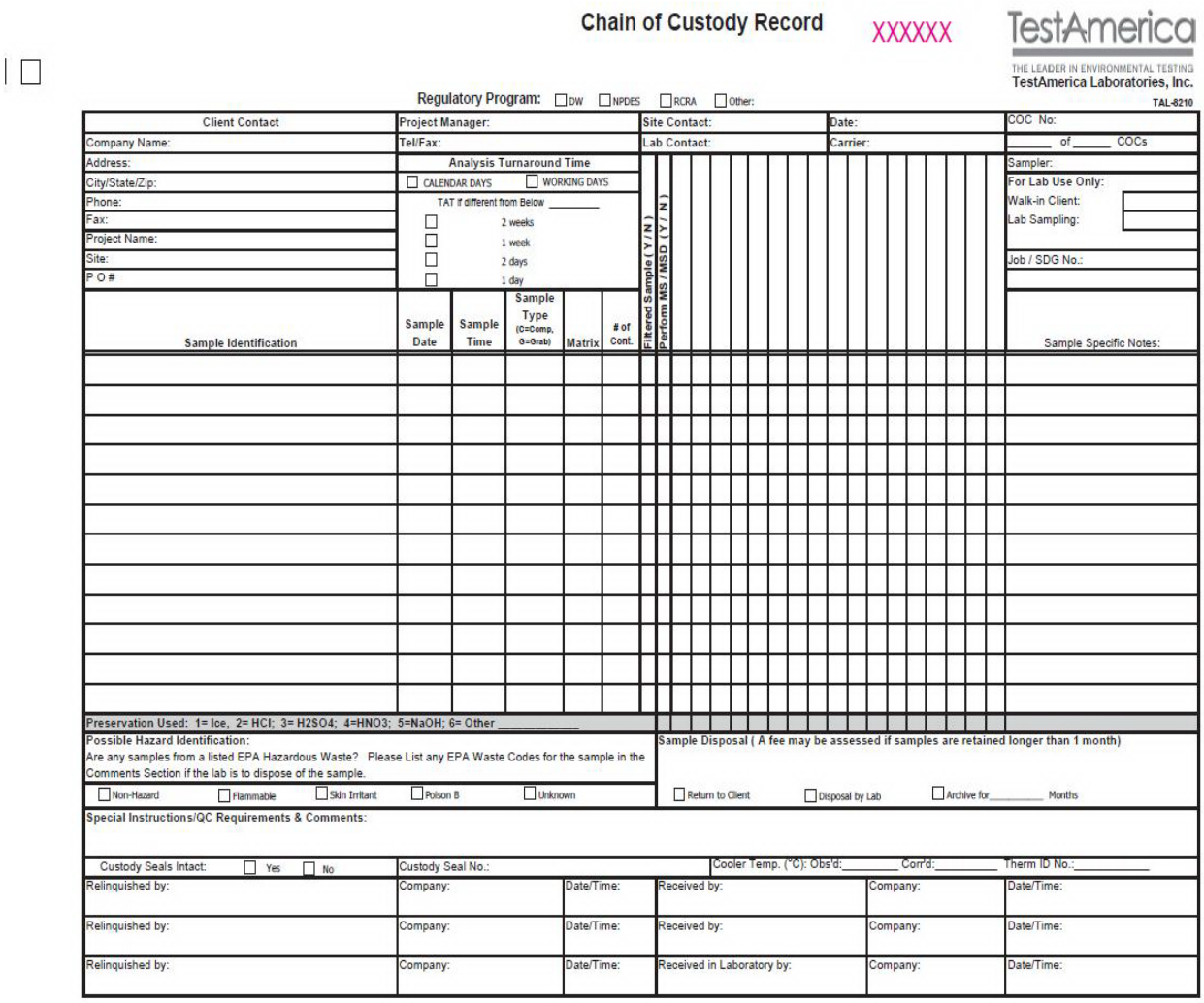
## 23.7 SAMPLE DISPOSAL

Samples should be retained for a minimum of 30 days after the project report is sent, however, provisions may be made for earlier disposal of samples once the holding time is exceeded. Some samples are required to be held for longer periods based on regulatory or client requirements (e.g., 60 days after project report is sent). The laboratory must follow the longer sample retention requirements where required by regulation or client agreement. Several possibilities for sample disposal exist: the sample may be consumed completely during analysis, the sample may be returned to the customer or location of sampling for disposal, or the sample may be disposed of in accordance with the laboratory's waste disposal procedures (SOP: BF-WM-001, "Waste Management".) All procedures in the laboratory Environmental, Health and Safety Manual are followed during disposal. Samples are normally maintained in the laboratory no longer than six weeks from receipt unless otherwise requested. Unused portions of samples found or suspected to be hazardous according to state or federal guidelines may be returned to the client upon completion of the analytical work.

If a sample is part of a known litigation, the affected legal authority, sample data user, and/or submitter of the sample may request to participate in the decision about the sample's disposal. All documentation and correspondence concerning the disposal decision process must be kept on file. Pertinent information includes the date of disposal and nature of disposal (such as sample depletion, hazardous waste facility disposal, and return to client). All disposal of sample containers is accomplished through incineration. A Waste Disposal Record should be completed.

**Figure 23-1.**

**Example: Chain of Custody (COC)**



Chain of Custody Record XXXXXX

TestAmerica
THE LEADER IN ENVIRONMENTAL TESTING
TestAmerica Laboratories, Inc.
TAL-8210

Regulatory Program: ☐ DW ☐ NPDES ☐ RCRA ☐ Other:

| Client Contact | Project Manager: | Site Contact: | Date: | COC No: |
|---|---|---|---|---|
| Company Name: | Tel/Fax: | Lab Contact: | Carrier: | ____ of ____ COCs |
| Address: | Analysis Turnaround Time | | | Sampler: |
| City/State/Zip: | ☐ CALENDAR DAYS ☐ WORKING DAYS | | | For Lab Use Only: |
| Phone: | TAT if different from Below ____ | | | Walk-in Client: |
| Fax: | ☐ 2 weeks | | | Lab Sampling: |
| Project Name: | ☐ 1 week | | | |
| Site: | ☐ 2 days | | | Job / SDG No.: |
| P O # | ☐ 1 day | | | |

| Sample Identification | Sample Date | Sample Time | Sample Type (C=Comp, G=Grab) | Matrix | # of Cont. | Filtered Sample ( Y / N ) | Perform MS / MSD ( Y / N ) | Sample Specific Notes: |
|---|---|---|---|---|---|---|---|---|
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Preservation Used: 1= Ice, 2= HCl; 3= H2SO4; 4=HNO3; 5=NaOH; 6= Other _____

Possible Hazard Identification:
Are any samples from a listed EPA Hazardous Waste? Please List any EPA Waste Codes for the sample in the Comments Section if the lab is to dispose of the sample.
☐ Non-Hazard ☐ Flammable ☐ Skin Irritant ☐ Poison B ☐ Unknown

Sample Disposal ( A fee may be assessed if samples are retained longer than 1 month)
☐ Return to Client ☐ Disposal by Lab ☐ Archive for_____ Months

Special Instructions/QC Requirements & Comments:

Custody Seals Intact: ☐ Yes ☐ No | Custody Seal No.: | Cooler Temp. (°C): Obs'd:_____ Corr'd:_____ Therm ID No.:_____

| Relinquished by: | Company: | Date/Time: | Received by: | Company: | Date/Time: |
|---|---|---|---|---|---|
| Relinquished by: | Company: | Date/Time: | Received by: | Company: | Date/Time: |
| Relinquished by: | Company: | Date/Time: | Received in Laboratory by: | Company: | Date/Time: |

**Figure 23-2.**

**Example: Sample Acceptance Policy**

All incoming work will be evaluated against the criteria listed below. Where applicable, data from any samples that do not meet the criteria listed below will be noted on the laboratory report defining the nature and substance of the variation. In addition the client will be notified either by telephone, fax or e-mail ASAP after the receipt of the samples.

1) Samples must arrive with labels intact with a Chain of Custody filled out completely. The following information must be recorded.
   - *Client name, address, phone number and fax number (if available)*
   - *Project name and/or number*
   - *The sample identification*
   - *Date, time and location of sampling*
   - *The collectors name*
   - *The matrix description*
   - *The container description*
   - *The total number of each type of container*
   - *Preservatives used*
   - *Analysis requested*
   - *Requested turnaround time (TAT)*
   - *Any special instructions*
   - *Purchase Order number or billing information (e.g. quote number) if available*
   - *The date and time that each person received or relinquished the sample(s), including their signed name.*
   - ***The date and time of receipt must be recorded between the last person to relinquish the samples and the person who receives the samples in the lab, and they must be exactly the same.***
   - **Information must be legible**

2) Every sample cooler is given a radiation screen with a standardized Radiation Monitor (Monitor 4 model). This screen has no analytical repercussions; it is just a gross screen for employee safety purposes. Contact TestAmerica Buffalo's Technical Manager, Environmental Health and Safety Coordinator or Sample Control Manager immediately if screening indicates radioactivity in excess of 0.02 mR/hr.

3) Per State and/or Federal Regulation, the client is responsible to ensure that samples are shipped in accordance with DOT/IATA requirements, and that radioactive materials may only be delivered to licensed facilities. Any samples containing (or suspected to contain) Source, Byproduct, or Special Nuclear Material as defined by 10 CFR should be delivered directly to facilities licensed to handle such radioactive material. Natural material or ores containing naturally occurring radionuclides may be delivered to any TestAmerica facility or

courier as long as the activity concentration of the material does not exceed 270 pCi/g alpha or 2700 pCi/g beta (49 CFR Part 173).

4) Samples must be properly labeled.
   - Use durable labels (labels provided by TestAmerica are preferred)
   - Include a unique identification number
   - Include sampling date and time & sampler ID
   - Include preservative used.
   - Use indelible ink
   - **Information must be legible**

5) Proper sample containers with adequate volume for the analysis and necessary QC are required for each analysis requested.

6) Samples must be preserved according to the requirements of the requested analytical method. See lab Sampling Guide.
   **Note:** Samples that are hand delivered to the laboratory immediately after collection may not have had time to cool sufficiently. In this case the samples will be considered acceptable as long as there is evidence that the chilling process has begun (arrival on ice).

   - Chemical preservation (pH) will be verified prior to analysis and documented, either in sample control or at the analyst's level. 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 either ascorbic acid or sodium thiosulfate prior to adding HCl.
     - 2. Use VOA vials pre-preserved with sodium thiosulfate or ascorbic acid and add HCl after filling the VOA vial with the sample.
   - **FOR WATER SAMPLES TESTED FOR CYANIDE – for NPDES samples by Standard Methods or EPA 335**
     - In the Field: Samples are to be tested for Sulfide using lead acetate paper prior to the addition of Sodium Hydroxide (NaOH). If sulfide is present, the sample must be treated with Cadmium Chloride and filtered prior to the addition of NaOH.

       - If the sulfide test and treatment is not performed in the field, the lab will test the samples for sulfide using lead acetate paper at the time of receipt and if sulfide is present in the sample, the client will be notified and given the option of retaking the sample and treating in the field per the method requirements

or the laboratory can analyze the samples as delivered and qualify the results in the final report.

- It is the responsibility of the client to notify the laboratory if thiosulfate, sulfite, or thiocyanate are known or suspected to be present in the sample. This notification may be on the chain of custody. The samples may need to be subcontracted to a laboratory that performs a UV digestion. If the lab does not perform the UV digestion on samples that contain these compounds, the results must be qualified in the final report.

- The laboratory must test the sample for oxidizing agents (e.g. Chlorine) prior to analysis and treat according to the methods prior to distillation. (ascorbic acid or sodium arsenite are the preferred choice).

7) Sample Holding Times

- TestAmerica will make every effort to analyze samples within the regulatory holding time. Samples must be received in the laboratory with enough time to perform the sample analysis. Except for short holding time samples (< 48hr HT) sample must be received with at least 48 hrs (2 working days) remaining on the holding time to ensure analysis.

- Analyses that are designated as “field” analyses (Odor, pH, Dissolved Oxygen, Disinfectant Residual; a.k.a. Residual Chlorine, and Redox Potential) should be analyzed ASAP by the field sampler prior to delivering to the lab (within 15 minutes). However, if the analyses are to be performed in the laboratory, TestAmerica will make every effort to analyze the samples within 24 hours from receipt of the samples in the testing laboratory. Samples for “field” analyses received after 4:00 pm on Friday or on the weekend will be analyzed no later than the next business day after receipt (Monday unless a holiday). Samples will remain refrigerated and sealed until the time of analysis.

8) All samples submitted for Volatile Organic analyses must have a Trip Blank submitted at the same time. TestAmerica will supply this blank with the bottle order.

9) The project manager will be notified if any sample is received in damaged condition. TestAmerica will request that a sample be resubmitted for analysis.

10) Recommendations for packing samples for shipment.

- Pack samples in Ice rather than “Blue” ice packs.

- Soil samples should be placed in plastic zip-lock bags. The containers often have dirt around the top and do not seal very well and are prone to intrusion from the water from melted ice.

- Water samples would be best if wrapped with bubble-wrap or paper (newspaper, or paper towels work) and then placed in plastic zip-lock bags.

- Fill extra cooler space with bubble wrap.

**Figure 23-3.**
**Example: Cooler Receipt Form (Optional)**

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Rev. 1
6/10/2013

**SAMPLE LOGIN**

Project_____Event _____

Analysis Groups_____

TAT ____ # SAMPLES: _____ TRIP BLANK? Y/N ___ #/date _____

Custody Seal Intact Y/N NONE Rad Check <0.02 mR/hr Y/N

Residual Chlorine Check Y/N/ NA Pres Checked Y/N/NA

Workshare/Subcontract Y/N Lab_____ _____ SO/ICOC #_____

Received out of hold: Samples _____Analysis_____

Checklist/NCM's_____

Temperature(s) #of coolers_____ IR Gun 1 2 3

**RUSH** **TIME CRITICAL**

## Section 24.0

## 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 measurements (e.g. Blanks, Laboratory Control Samples (LCS), Matrix Spikes (MS), duplicates (DUP), surrogates, Internal Standards (IS)). These quality control checks are performed as required by the method or regulations to assess precision and accuracy. Quality control samples are to be treated in the exact same manner as the associated field samples being tested. In addition to the routine process quality control samples, Proficiency Testing (PT) Samples (concentrations unknown to laboratory) are analyzed to help ensure laboratory performance.

### 24.2 CONTROLS

Sample preparation or pre-treatment is commonly required before analysis. Typical preparation steps include homogenization, grinding, solvent extraction, sonication, acid digestion, distillation, reflux, evaporation, drying and ashing. During these pre-treatment steps, samples are arranged into discreet manageable groups referred to as preparation (prep) batches. Prep batches provide a means to control variability in sample treatment. Control samples are added to each prep batch to monitor method performance and are processed through the entire analytical procedure with investigative/field samples.

### 24.3 NEGATIVE CONTROLS

**Table 24-1.**

| Control Type | Details |
|---|---|
| Method Blank (MB) | Are used to assess preparation and analysis for possible contamination during the preparation and processing steps. |
| | The specific frequency of use for method blanks during the analytical sequence is defined in the specific standard operating procedure for each analysis. Generally it is 1 for each batch of samples; not to exceed 20 environmental samples. |
| | The method blank is prepared from a clean matrix similar to that of the associated samples that is free from target analytes (e.g., Reagent water, Ottawa sand, glass beads, etc.) and is processed along with and under the same conditions as the associated samples.<br><br>The method blank goes through all of the steps of the process (including as necessary: filtration, clean-ups, etc.). |
| | 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. |

**Table 24-1.**

| Control Type | Details |
|---|---|
| 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 [1] | 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. |
| Field Blanks [1] | Are sometimes used for specific projects by the field samplers. A field blank prepared in the field by filling a clean container with pure reagent water and appropriate preservative, if any, for the specific sampling activity being undertaken. (EPA OSWER) |
| Equipment Blanks [1] | 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) |
| 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 |

[1] When known, these field QC samples should not be selected for matrix QC as it does not provide information on the behavior of the target compounds in the field samples. Usually, the client sample ID will provide information to identify the field blanks with labels such as "FB", "EB", or "TB."

Evaluation criteria and corrective action for these controls are defined in the specific standard operating procedure for each analysis.

## 24.4 POSITIVE CONTROLS

Control samples (e.g., QC indicators) are analyzed with each batch of samples to evaluate data based upon (1) Method Performance (Laboratory Control Sample (LCS) or Blank Spike (BS)), which entails both the preparation and measurement steps; and (2) Matrix Effects (Matrix Spike (MS) (Matrix spikes are not applicable to air) or Sample Duplicate (MD, DUP), which evaluates field sampling accuracy, precision, representativeness, interferences, and the effect of the matrix on the method performed. Each regulatory program and each method within those programs specify the control samples that are prepared and/or analyzed with a specific batch

Note that frequency of control samples vary with specific regulatory, methodology and project specific criteria. Complete details on method control samples are as listed in each analytical SOP.

**24.4.1 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 may be reported as the LCS. In some instances where there is no practical clean solid matrix available, aqueous LCS's may be processed for solid matrices; final results may be calculated as mg/kg or ug/kg, assuming 100% solids and a weight equivalent to the aliquot used for the corresponding field samples, to facilitate comparison with the field samples.

**24.4.1.3** Certified pre-made reference material purchased from a NIST/A2LA accredited vendor may also be used for the LCS when the material represents the sample matrix or the analyte is not easily spiked (e.g. solid matrix LCS for metals, TDS, etc.).

**24.4.1.4** The specific frequency of use for LCS during the analytical sequence is defined in the specific standard operating procedure for each analysis. It is generally 1 for each batch of samples; not to exceed 20 environmental samples.

**24.4.1.5** If the mandated or requested test method, or project requirements, do not specify the spiking components, the laboratory shall spike all reportable components to be reported in the Laboratory Control Sample (and Matrix Spike) where applicable (e.g. no spike of pH). In order to meet this requirement, TestAmerica Buffalo spikes with the Corporate Standard Standards primary mix for each analysis. 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.

**24.4.1.5.1** For methods that have 1-10 target analytes, spike all components.

**24.4.1.5.2** For methods that include 11-20 target analytes, spike at least 10 or 80%, whichever is greater.

**24.4.1.5.3** For methods with more than 20 target analytes, spike at least 16 components.

**24.4.1.5.4** Exception: Due to analyte incompatibility in pesticides, Toxaphene and Chlordane are only spiked at client request based on specific project needs.

**24.4.1.5.5** Exception: Due to analyte incompatibility between the various PCB aroclors, aroclors 1016 and 1260 are used for spiking as they cover the range of all of the aroclors. Specific aroclors may be used by request on a project specific basis.

## 24.5 SAMPLE MATRIX CONTROLS

**Table 24-5. Sample Matrix Control**

| Control Type | Details | |
|---|---|---|
| Matrix Spikes (MS) | Use | Used to assess the effect sample matrix of the spiked sample has on the precision and accuracy of the results generated by the method used; |
| | Typical Frequency [1] | 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 [1] | 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[2] | 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 [1] | 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 [1] | 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. |

[1] See the specific analytical SOP for type and frequency of sample matrix control samples.

[2] LCSD's are normally not performed except when regulatory agencies or client specifications require them. The recoveries for the spiked duplicate samples must meet the same laboratory established recovery limits as the accuracy QC samples. If an LCSD is analyzed both the LCS and LCSD must meet the same recovery criteria and be included in the final report. The precision measurement is reported as "Relative Percent Difference" (RPD). Poor precision between duplicates (except LCS/LCSD) may indicate non-homogeneous matrix or sampling.

## 24.6 ACCEPTANCE CRITERIA (CONTROL LIMITS)

**24.6.1** As mandated by the test method and regulation, each individual analyte in the LCS, MS, or Surrogate Spike is evaluated against the control limits published in the test method. Where there are no established acceptance criteria, the laboratory calculates in-house control limits with the use of control charts or, in some cases, utilizes client project specific control limits. When this occurs, the regulatory or project limits will supersede the laboratory's in-house limits.

**Note:** For methods, analytes and matrices with very limited data (e.g., unusual matrices not analyzed often), interim limits are established using available data or by analogy to similar methods or matrices.

**24.6.2** Once control limits have been established, they are verified, reviewed, and updated if necessary on an annual basis unless the method requires more frequent updating. Control limits are established per method (as opposed to per instrument) regardless of the number of instruments utilized.

**24.6.3** Laboratory generated % Recovery acceptance (control) limits are generally established by taking $\pm$ 3 Standard Deviations (99% confidence level) from the average recovery of a minimum of 20-30 data points (more points are preferred).

**24.6.3.1** Regardless of the calculated limit, the limit should be no tighter than the Calibration Verification (ICV/CCV). (Unless the analytical method specifies a tighter limit).

**24.6.3.2** In-house limits cannot be any wider than those mandated in a regulated analytical method. Client or contract required control limits are evaluated against the laboratory's statistically derived control limits to determine if the data quality objectives (DQOs) can be achieved. If laboratory control limits are not consistent with DQOs, then alternatives must be considered, such as method improvements or use of an alternate analytical method.

**24.6.3.3** The lowest acceptable recovery limit will be 10% (the analyte must be detectable). Exception: The lowest acceptable recovery limit for Benzidine will be 5% and the analyte must be detectable.

**24.6.3.4** The maximum acceptable recovery limit will be 150%.

**24.6.3.5** The maximum acceptable RPD limit will be 35% for waters and 40% for soils. The minimum RPD limit is 10%.

**24.6.3.6** If either the high or low end of the control limit changes by $\leq$ 5% from previous, the data points are inspected and, using professional judgment, the limits may be left unchanged if there is no affect on laboratory ability to meet the existing limits.

**24.6.4** The lab must be able to generate a current listing of their control limits and track when the updates are performed. In addition, the laboratory must be able to recreate historical control limits. This process is outline in BF-QA-002.

**24.6.4.1** The control limits are maintained in the laboratory LIMs system. The limits for each analyte/method/matrix combination are assigned effective and expiration dates. The QA department is able to query the LIMs system and print an active list of control limits based on this database. The most current laboratory limits (based on the effective/expiration dates) are reflected on the laboratory worksheets and final reports unless superseded by project specific limits.

**24.6.5** A LCS that is within the acceptance criteria establishes that the analytical system is in control and is used to validate the process. Samples that are analyzed with an LCS with recoveries outside of the acceptance limits may be determined as out of control and should be reanalyzed if possible. If reanalysis is not possible, then the results for all affected analytes for samples within the same batch must be qualified when reported. The internal corrective action process (see Section 13) is also initiated if an LCS exceeds the acceptance limits. Sample results may be qualified and reported without reanalysis if:

**24.6.5.1** The analyte results are below the reporting limit and the LCS is above the upper control limit.

**24.6.5.2** If the analytical results are above the relevant regulatory limit and the LCS is below the lower control limit.

**24.6.6** If the MS/MSDs do not meet acceptance limits, the MS/MSD and the associated spiked sample is reported with a qualifier for those analytes that do not meet limits. If obvious preparation errors are suspected, or if requested by the client, unacceptable MS/MSDs are reprocessed and reanalyzed to prove matrix interference. A more detailed discussion of acceptance criteria and corrective action can be found in the lab's method SOPs and in Section 12.

**24.6.7** If a surrogate standard falls outside the acceptance limits, if there is not obvious chromatographic matrix interference, reanalyze the sample to confirm a possible matrix effect. If the recoveries confirm or there was obvious chromatographic interference, results are reported from the original analysis and a qualifier is added. If the reanalysis meets surrogate recovery criteria, the second run is reported (or both are reported if requested by the client). Under certain circumstances, where all of the samples are from the same location and share similar chromatography, the reanalysis may be performed on a single sample rather than all of the samples and if the surrogate meets the recovery criteria in the reanalysis, all of the affected samples would require reanalysis.

## 24.7 ADDITIONAL PROCEDURES TO ASSURE QUALITY CONTROL

**24.7.1** The laboratory has written and approved method SOPs to assure the accuracy of the test method including calibration (see Section 20), use of certified reference materials (see Section 21) and use of PT samples.

**24.7.2** A discussion regarding MDLs, Limit of Detection (LOD) and Limit of Quantitation (LOQ) can be found in Section 19.

**24.7.3** Use of formulae to reduce data is discussed in the method SOPs and in Section 20.

**24.7.4** Selection of appropriate reagents and standards is included in Section 9 and 22.

**24.7.5** A discussion on selectivity of the test is included in Section 5.

**24.7.6** Constant and consistent test conditions are discussed in Section 19.

**24.7.7** The laboratories sample acceptance policy is included in Section 23.

# SECTION 25.0

# 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. A variety of report formats are available to meet specific needs. Analytical results are issued in a format that is intended to satisfy customer and laboratory accreditation requirements as well as provide the end user with the information needed to properly evaluate the results. Where there is conflict between client requests and laboratory ethics or regulatory requirements, the laboratory's ethical and legal requirements are paramount, and the laboratory will work with the client during project set up to develop an acceptable solution. Refer to Section 7.

In cases where a client asks for simplified reports, there must be a written request from the client. There still must be enough information that would show any analyses that were out of conformance (QC out of limits) and there should be a reference to a full report that is made available to the client.

The laboratory complies with any state reporting requirements. An example is located in BF-PM-008 – Massachusetts DEP Notification Procedures.

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. Analytical Report) with a "sample results" column header.

**25.2.2** Each report cover page is printed on company letterhead which includes the laboratory name, address and telephone number.

**25.2.3** A unique identification of the report (e.g. job number) and on each page an identification in order to ensure the page is recognized as part of the report and a clear identification of the end.

**Note:** Page numbers of report are represented as # / ##. Where the first number is the page number and the second is the total number of pages.

**25.2.4** A copy of the chain of custody (COC).

- Any COCs involved with Subcontracting are included.

**25.2.5** The name and address of client and a project name/number, if applicable.

**25.2.6** Client project manager or other contact

**25.2.7** Description and unambiguous identification of the tested sample(s) including the client identification code.

**25.2.8** Date of receipt of sample, date and time of collection, and date(s) of test preparation and performance, and time of preparation or analysis if the required holding time for either activity is less than or equal to 72 hours.

**25.2.9** Date reported or date of revision, if applicable.

**25.2.10** Method of analysis including method code (EPA, Standard Methods, etc).

**25.2.11** Practical quantitation limits or client reporting limit.

**25.2.12** Method detection limits (if requested)

**25.2.13** Definition of Data qualifiers and reporting acronyms (e.g. ND).

**25.2.14** Sample results.

**25.2.15** QC data consisting of method blank, surrogate, LCS, and MS/MSD recoveries and control limits (if requested).

**25.2.16** Condition of samples at receipt including temperature. This may be accomplished in a narrative or by attaching sample login sheets (Refer to Sec. 25.2.4 – Item 3 regarding additional addenda). Sample temperatures are recorded in the report case narrative and on the COC. Deviations from normal conditions (e.g., preservation, breakage) are recorded in the report case narrative.

**25.2.17** A statement expressing the validity of the results, that the source methodology was followed and all results were reviewed for error.

**25.2.18** A statement to the effect that the results relate only to the items tested and the sample as received by the laboratory.

**25.2.19** A statement that the report shall not be reproduced except in full, without prior express written approval by the laboratory coordinator.

**25.2.20** A signature and title of the person(s) accepting responsibility for the content of the report and date of issue. Authorized signatories are qualified Project Managers appointed by the Manager of Project Managers.

**25.2.21** When NELAP accreditation is required, the lab shall certify that the test results meet all requirements of NELAP or provide reasons and/or justification if they do not.

**25.2.22** The laboratory includes a cover letter.

**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). 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 an addendum to the report on the official letterhead of the subcontractor. All TestAmerica subcontracting is clearly identified on the report as to which laboratory performed a specific analysis.

**25.2.28** Certification Summary report, where required, will document that unless otherwise noted, all analytes tested and reported by the laboratory were covered by the noted certifications.

## 25.3 REPORTING LEVEL OR REPORT TYPE

***TestAmerica Buffalo*** offers four levels of quality control reporting. Each level, in addition to its own specific requirements, contains all the information provided in the preceding level. The packages provide the following information in addition to the information described above:

- Level 1 is a report with all of the elements outlined in Section 25.2 above, excluding 25.2.15 (QC data)
- Level II is a Level I report plus summary information, including results for the method blank, percent recovery for laboratory control samples and matrix spike samples, and the RPD values for all MSD and sample duplicate analyses.
- Level III contains all the information supplied in Level II, but presented on CLP-like summary forms, and relevant calibration information. A Level II report is not included, unless specifically requested. No raw data is provided.
- Level IV is the same as Level III with the addition of all raw supporting data.

In addition to the various levels of QC packaging, the laboratory also provides reports in diskette deliverable form. Initial reports may be provided to clients by facsimile. Procedures used to ensure client confidentiality are outlined in Section 26.7.

### 25.3.1 Electronic Data Deliverables (EDDs)

EDDs are routinely offered as part of TestAmerica's services in addition to the test report as described in section 25.2. When NELAP accreditation is required and both a test report and EDD are provided to the client, the official version of the test report will be the combined information of the report and the EDD . TestAmerica Buffalo offers a variety of EDD formats including Environmental Restoration Information Management System (ERPIMS), Excel, Dbase, GISKEY, and Text Files.

EDD specifications are submitted to the IT department by the PM for review and undergo the contract review process. Once the facility has committed to providing data in a specific electronic format, the coding of the format may need to be performed. This coding is documented and validated. The validation of the code is retained by the IT staff coding the EDD.

EDDs shall be subject to a review to ensure their accuracy and completeness. If EDD generation is automated, review may be reduced to periodic screening if the laboratory can demonstrate that it can routinely generate that EDD without errors. Any revisions to the EDD format must be reviewed until it is demonstrated that it can routinely be generated without errors. If the EDD can be reproduced accurately and if all subsequent EDDs can be produced error-free, each EDD does not necessarily require a review.

## 25.4 SUPPLEMENTAL INFORMATION FOR TEST

The lab identifies any unacceptable QC analyses or any other unusual circumstances or observations such as environmental conditions and any non-standard conditions that may have affected the quality of a result. This is typically in the form of a footnote or a qualifier and/or a narrative explaining the discrepancy in the front of the report

**25.4.1** Numeric results with values outside of the calibration range, either high or low are qualified as 'estimated'.

**25.4.2** Where quality system requirements are not met, a statement of compliance/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.

**25.4.3** Where applicable, a statement on the estimated uncertainty of measurements; information on uncertainty is needed when a client's instructions so require.

**25.4.4** Opinions and Interpretations - The test report contains objective information, and generally does not contain subjective information such as opinions and interpretations. If such information is required by the client, the Laboratory Director will determine if a response can be prepared. If so, the Laboratory Director will designate the appropriate member of the management team to prepare a response. The response will be fully documented, and reviewed

by the Laboratory Director, before release to the client. There may be additional fees charged to the client at this time, as this is a non-routine function of the laboratory.

**Note:** Review of data deliverable packages for submittal to regulatory authorities requires responses to non-conforming data concerning potential impact on data quality. This necessitates a limited scope of interpretation, and this work is performed by the QA Department. This is the only form of "interpretation" of data that is routinely performed by the laboratory.

When opinions or interpretations are included in the report, the laboratory provides an explanation as to the basis upon which the opinions and interpretations have been made. Opinions and interpretations are clearly noted as such and where applicable, a comment should be added suggesting that the client verify the opinion or interpretation with their regulator.

## 25.5 ENVIRONMENTAL TESTING OBTAINED FROM SUBCONTRACTORS

If the laboratory is not able to provide the client the requested analysis, the samples would be subcontracted following the procedures outlined in Section 8.

Data reported from analyses performed by a subcontractor laboratory are clearly identified as such on the analytical report provided to the client. Results from a subcontract laboratory outside of TestAmerica are reported to the client on the subcontract laboratory's original report stationary and the report includes any accompanying documentation.

## 25.6 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*

## 25.7 FORMAT OF REPORTS

The format of reports is designed to accommodate each type of environmental test carried out and to minimize the possibility of misunderstanding or misuse.

## 25.8 AMENDMENTS TO TEST REPORTS

Corrections, additions, or deletions to reports are only made when justification arises through supplemental documentation. Justification is documented using the laboratory's corrective action system (refer to Section 12).

The revised report is retained on the Archive data server, as is the original report. The revised report is stored in the Archive data server under the sample number followed by "R". The revised report will have the word "revised" appended to the cover letter.

When the report is re-issued, a notation of "revised" is placed on the cover/signature page of the report. A brief explanation of reason for the re-issue is included in the report case narrative.

## 25.9 POLICIES ON CLIENT REQUESTS FOR AMENDMENTS

### 25.9.1 Policy on Data Omissions or Reporting Limit Increases

Fundamentally, our policy is simply to not omit previously reported results (including data qualifiers) or to not raise reporting limits and report sample results as ND. This policy has few exceptions. Exceptions are:

- Laboratory error.
- Sample identification is indeterminate (confusion between COC and sample labels).
- An incorrect analysis (not analyte) was requested (e.g., COC lists 8315 but client wanted 8310). A written request for the change is required.
- Incorrect limits reported based on regulatory requirements.
- The requested change has absolutely *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.

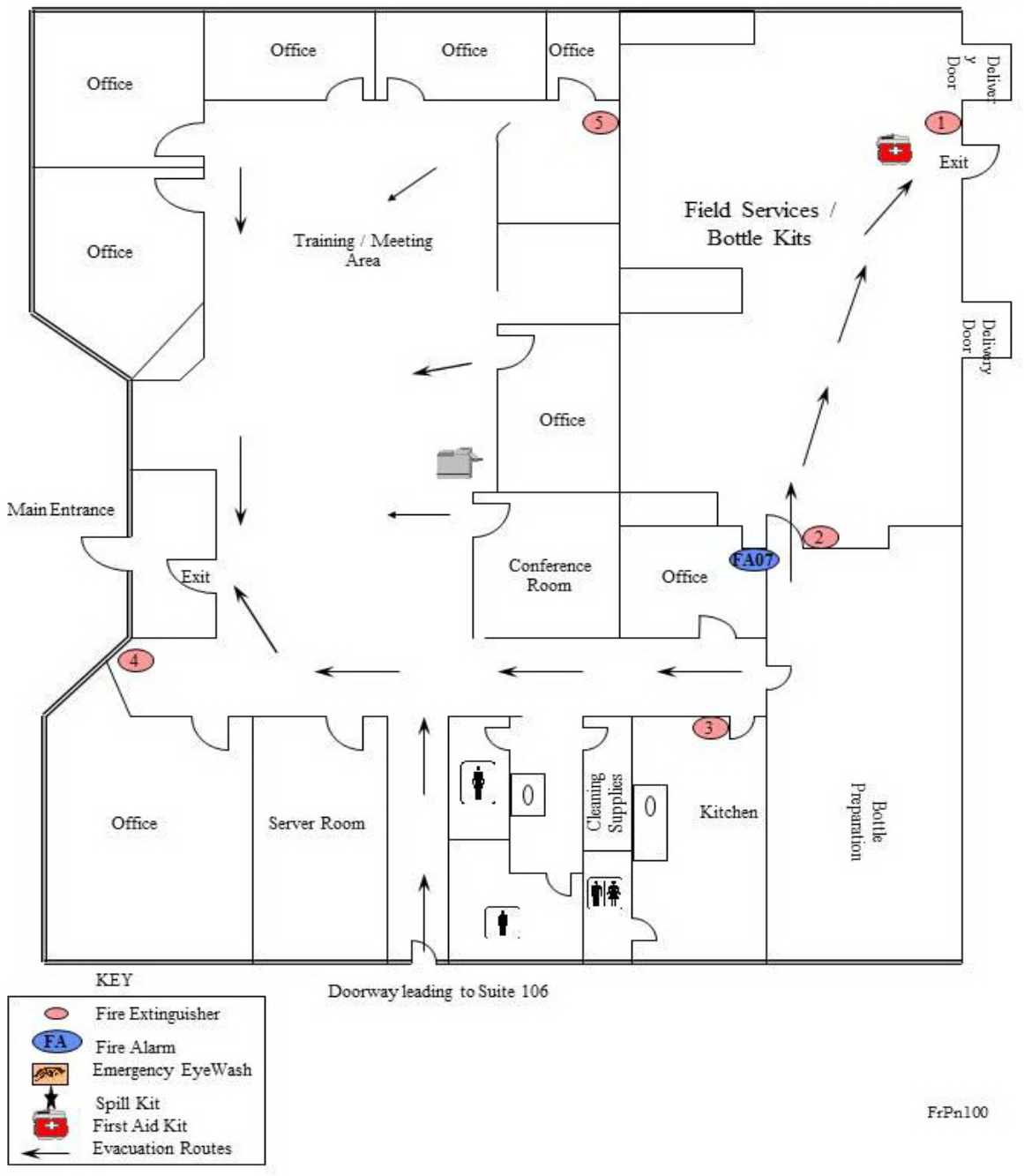
### 25.9.2 Multiple Reports

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.**

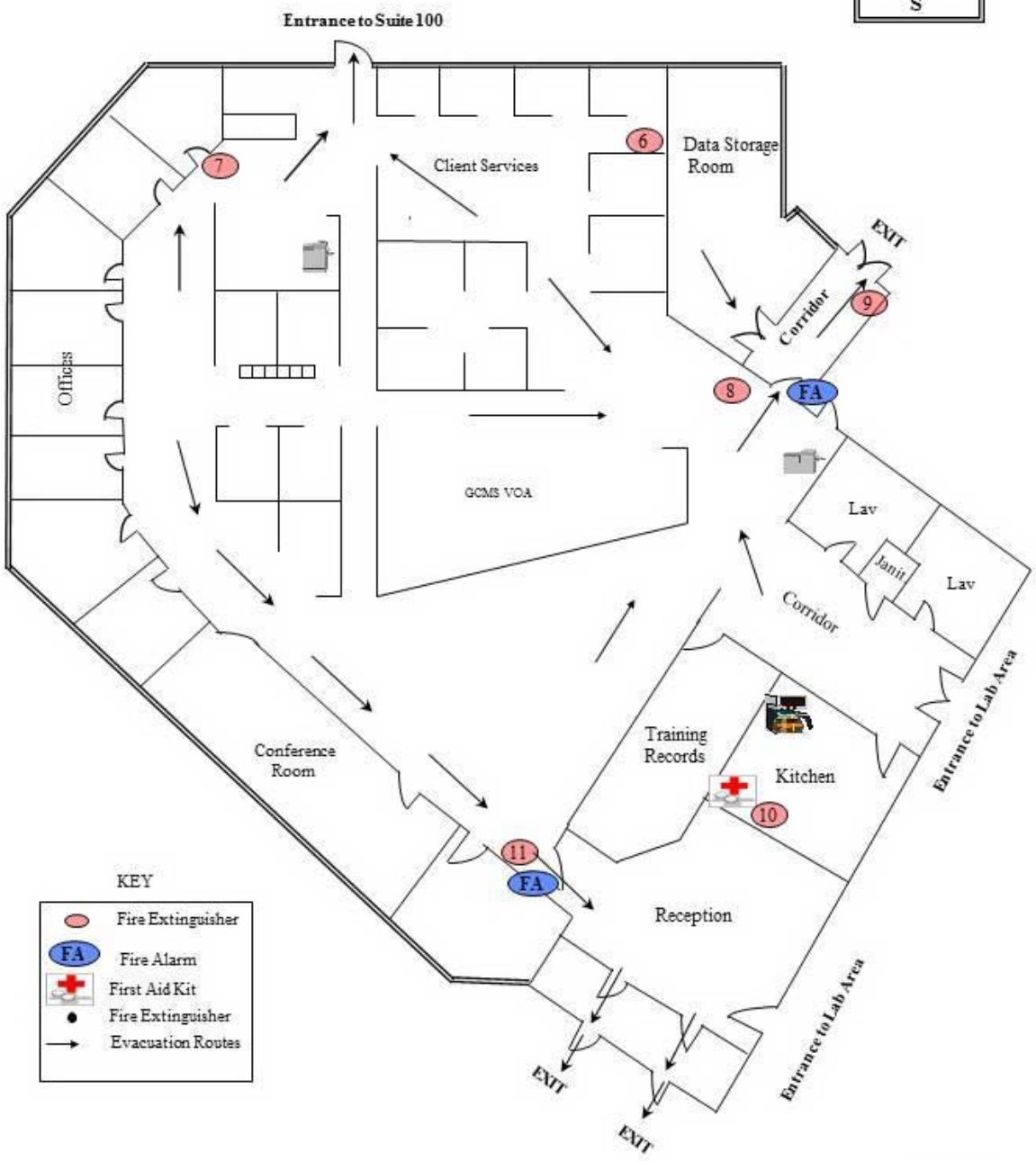
TestAmerica
THE LEADER IN ENVIRONMENTAL TESTING

**TAL BUFFALO**
**HAZELWOOD DR. OFFICES, SUITE 100**
**FLOOR PLAN**



TestAmerica
THE LEADER IN ENVIRONMENTAL TESTING

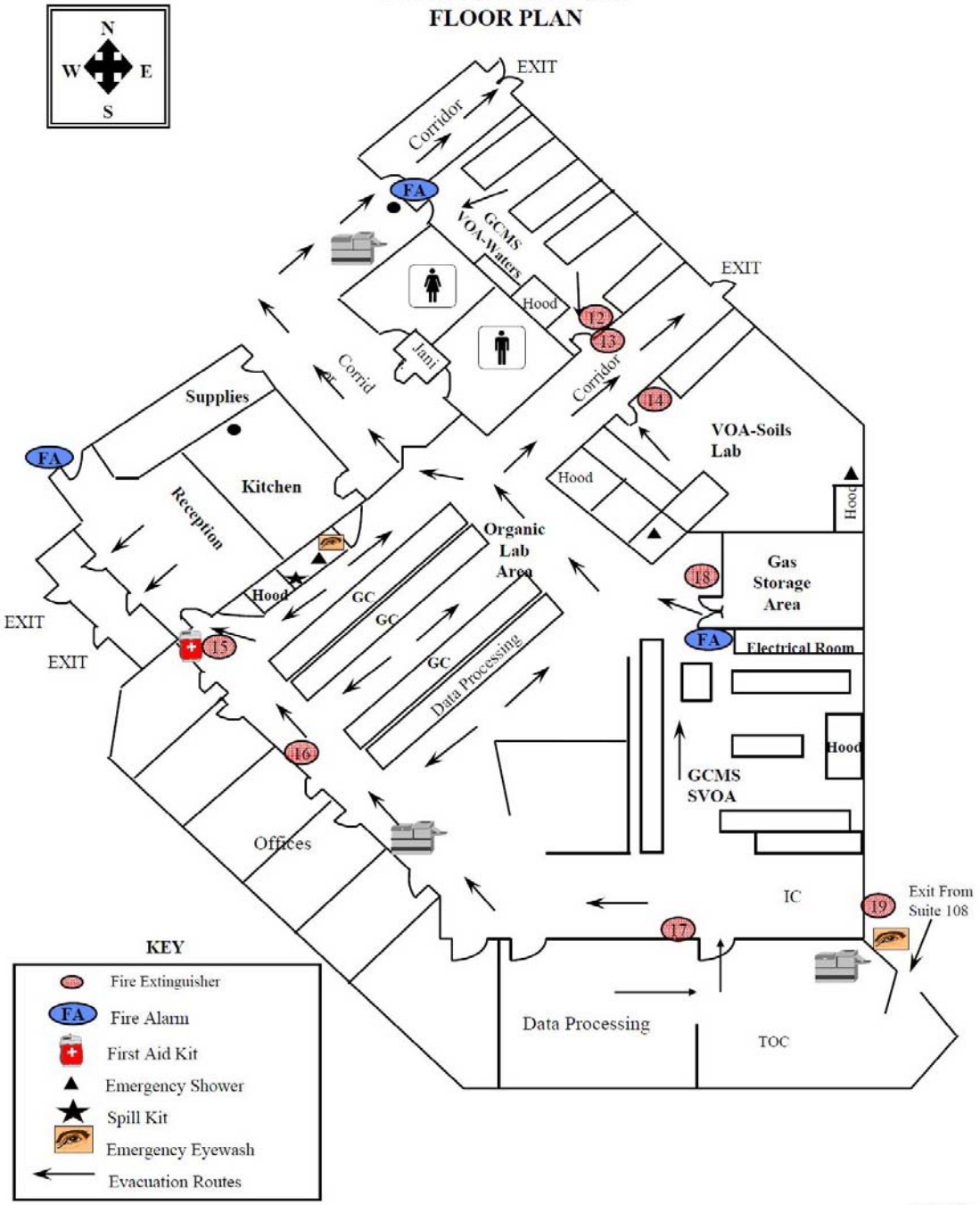
# TAL BUFFALO
# HAZELWOOD DR. OFFICES, SUITE 106
# CLIENT SERVICES/REPORT PREP
# FLOOR PLAN



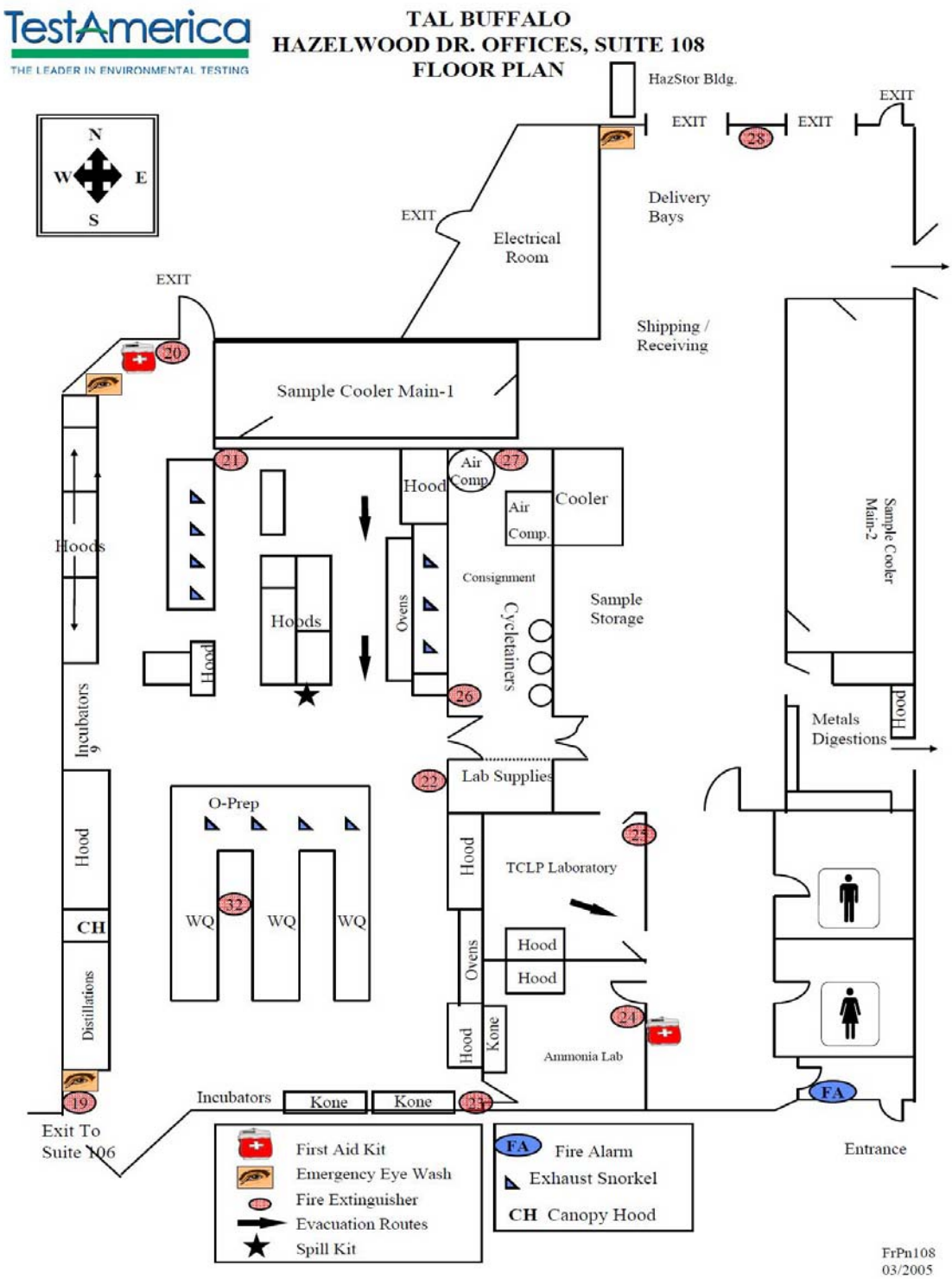

FrP1106L
3/2005

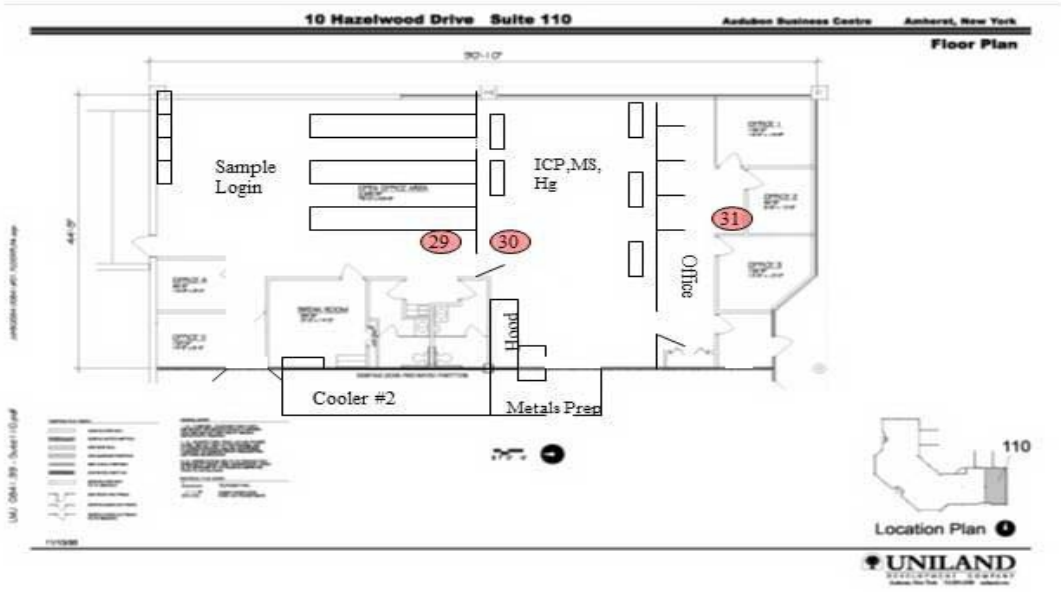
TestAmerica
THE LEADER IN ENVIRONMENTAL TESTING

**TAL BUFFALO**
**HAZELWOOD DR. NY OFFICES, SUITE 106**
**LABORATORY AREA**
**FLOOR PLAN**




FrPn106r
03/2005


TestAmerica
THE LEADER IN ENVIRONMENTAL TESTING
TAL BUFFALO
HAZELWOOD DR. OFFICES, SUITE 108
FLOOR PLAN
N
W
E
S
HazStor Bldg.
EXIT
EXIT
EXIT
EXIT
28
Delivery Bays
Electrical Room
EXIT
Shipping / Receiving
20
Sample Cooler Main-1
21
Hood
Air Comp.
27
Air Comp.
Cooler
Hoods
Consignment
Sample Storage
Ovens
Cycletainers
Hoods
Hood
26
Sample Cooler Main-2
Incubators
Hood
Metals Digestions
22
Lab Supplies
O-Prep
25
Hood
TCLP Laboratory
WQ
32
WQ
WQ
CH
Ovens
Hood
Hood
Distillations
Hood
Kone
24
Ammonia Lab
19
Incubators
Kone
Kone
23
FA
Exit To Suite 106
Entrance
First Aid Kit
Emergency Eye Wash
Fire Extinguisher
Evacuation Routes
Spill Kit
FA Fire Alarm
Exhaust Snorkel
CH Canopy Hood
FrPn108
03/2005


10 Hazelwood Drive Suite 110
Floor Plan
Sample Login
ICP,MS, Hg
29
30
31
Office
Hood
Cooler #2
Metals Prep
110
Location Plan
UNILAND

## Appendix 2. Glossary/Acronyms

### Glossary:

**Acceptance Criteria:** Specified limits placed on characteristics of an item, process, or service defined in requirement documents. (ASQC)

**Accreditation:** The process by which an agency or organization evaluates and recognizes a laboratory as meeting certain predetermined qualifications or standards, thereby accrediting the laboratory. In the context of the National Environmental Laboratory Accreditation Program (NELAP), this process is a voluntary one. (TNI)

**Accrediting Authority:** The Territorial, State, or Federal Agency having responsibility and accountability for environmental laboratory accreditation and which grants accreditation (TNI)

**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. (TNI)

**Analytical Uncertainty:** A subset of Measurement Uncertainty that includes all laboratory activities performed as part of the analysis. (TNI)

**Anomaly:** A condition or event, other than a deficiency, that may affect the quality of the data, whether in the laboratory's control or not.

**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 personnel, using the same lot(s) of reagents. A preparation batch is composed of one to 20 environmental samples of the same matrix, meeting the above mentioned criteria and with a maximum time between the start of processing of the first and last sample in the batch to be 24 hours. An analytical batch is composed of prepared environmental samples (extracts, digestates or concentrates) and /or those samples not requiring preparation, which are analyzed together as a group using the same calibration curve or factor. An analytical batch can include samples originating from various environmental matrices and can exceed 20 samples. (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 Curve:** The mathematical relationship between the known values, such as concentrations, of a series of calibration standards and their instrument response. (TNI)

**Calibration Standard:** A substance or reference material used to calibrate an instrument (QAMS)

**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 (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. (TNI)

**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 safeguarding identified CBI and to maintain all information identified as such in full confidentiality.

**Confirmation:** Verification of the identity of a component through the use of an approach with a different scientific principle from the original method. These may include, but are not limited to:

Second column confirmation
Alternate wavelength
Derivitization
Mass spectral interpretation
Alternative detectors or
Additional Cleanup procedures
(TNI)

**Conformance:** An affirmative indication or judgment that a product or service has met the requirements of the relevant specifications, contract, or regulation; also the state of meeting the requirements. (ANSI/ASQC E4-1994)

**Corrective Action:** The action taken to eliminate the causes of an existing nonconformity, defect or other undesirable situation in order to prevent recurrence. (ISO 8402)

**Data Audit:** A qualitative and quantitative evaluation of the documentation and procedures associated with environmental measurements to verify that the resulting data re of acceptable quality (i.e., that they meet specified acceptance criteria). (TNI)

**Data Reduction:** The process of transforming the number of data items by arithmetic or statistical calculations, standard curves, concentration factors, etc., and collation into a more useable form. (TNI)

**Deficiency:** An unauthorized deviation from acceptable procedures or practices, or a defect in an item (ASQC), whether in the laboratory's control or not.

**Demonstration of Capability:** A procedure to establish the ability of the analyst to generate analytical results of acceptable accuracy and precision. (TNI)

**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. (TNI)

**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 filing a clean container with pure de-ionized water and appropriate preservative, if any, for the specific sampling activity being undertaken (EPA OSWER)

**Field of Accreditation:** Those matrix, technology/method, and analyte combinations for which the accreditation body offers accreditation.

**Holding Times:** The maximum time that samples may be held prior to analyses and still be considered valid or not compromised. (40 CFR Part 136)

**Internal Standard:** A known amount of standard added to a test portion of a sample 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 ± 100%. The IDL represents a range where qualitative detection occurs on a specific instrument. Quantitative results are not produced in this range.

**Laboratory Control Sample** (however named, such as laboratory fortified blank, spiked blank, or QC check sample): A sample matrix, free from the analytes of interest, spiked with verified known amounts of analytes or a material containing known and verified amounts of analytes, taken through all preparation and analysis steps 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.

**Least Squares Regression (1$^{st}$ 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)

**LOD Verification [a.k.a., MDL Verification]:** A processed QC sample in the matrix of interest, spiked with the analyte at no more than 3X the LOD for single analyte tests and 4X the LOD for multiple analyte tests and processed through the entire analytical procedure.

**Limit(s) of Quantitation (LOQ) [a.k.a., Reporting Limit]:** The minimum levels, concentrations, or quantities of a target variable (e.g., target analyte) that can be reported with a specified degree of confidence. (TNI)

**(QS) 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 & Emissions*: Whole gas or vapor samples including those contained in flexible or rigid wall containers and the extracted concentrated analytes of interest from a gas or vapor that are collected with a sorbant tube, impinger solution, filter, or other device. (TNI)

**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 replicate matrix spike prepared and analyzed to obtain a measure of the precision of the recovery for each analyte.

**Method Blank:** A sample of a matrix similar to the batch of associated samples (when available) that is free from the analytes of interest and is processed simultaneously with and under the same conditions as samples through all steps of the analytical procedures, and in which no target analytes or interferences are present at concentrations that impact the analytical results for sample analyses. (TNI)

**Method Detection Limit:** The minimum concentration of a substance (an analyte) that can be measured and reported with 99% confidence that the analyte concentration is greater than zero and is determined from analysis of a sample in a given matrix containing the analyte. (40 CFR Part 136, Appendix B)

**Negative Control:** Measures taken to ensure that a test, its components, or the environment do not cause undesired effects, or produce incorrect test results. (TNI)

**Non-conformance:** An indication, judgment, or state of not having met the requirements of the relevant specifications, contract, or regulation.

**Observation:** A record of phenomena that (1) may assist in evaluation of the sample data; (2) may be of importance to the project manager and/or the client, and yet not at the time of the observation have any known effect on quality.

**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. (TNI)

**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. (TNI)

**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) [2.1]

**Proficiency Testing Program:** The aggregate of providing rigorously controlled and standardized environmental samples to a laboratory for analysis, reporting of results, statistical evaluation of the results and the collective demographics and results summary of all participating laboratories. (TNI)

**Proficiency Test Sample (PT):** A sample, the composition of which is unknown to the laboratory 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. (TNI)

**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)

**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. (TNI)

**Reference Standard:** Standard used for the calibration of working measurement standards in a given organization or a given location. (TNI)

**Sampling:** Activity related to obtaining a representative sample of the object of conformity assessment, according to a procedure.

**Second Order Polynomial Curve (Quadratic):** The 2nd 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 2nd 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. (TNI)

**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 and which is accepted as the method 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

**Technology:** A specific arrangement of analytical instruments, detection systems, and/or preparation techniques.

**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)

**Uncertainty:** A parameter associated with the result of a measurement that characterizes the dispersion of the value that could reasonably be attributed to the measured value.

**Acronyms:**

CAR – Corrective Action Report
CCV – Continuing Calibration Verification
CF – Calibration Factor
CFR – Code of Federal Regulations
COC – Chain of Custody
DOC – Demonstration of Capability
DQO – Data Quality Objectives
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
LOD – Limit of Detection

LOQ – Limit of Quantitation
MDL – Method Detection Limit
MDLCK – MDL Check Standard
MDLV – MDL Verification Check Standard
MRL – Method Reporting Limit Check Standard
MS – Matrix Spike
MSD – Matrix Spike Duplicate
NELAP - National Environmental Laboratory Accreditation Program
PT – Performance Testing
QAM – Quality Assurance Manual
QA/QC – Quality Assurance / Quality Control
QAPP – Quality Assurance Project Plan
RF – Response Factor
RPD – Relative Percent Difference
RSD – Relative Standard Deviation
SD – Standard Deviation
SDS - Safety Data Sheet
SOP: Standard Operating Procedure
TAT – Turn-Around-Time
TNI – The NELAC Institute
VOA – Volatiles
VOC – Volatile Organic Compound

**Appendix 3.** Laboratory Certifications, Accreditations, Validations

***TestAmerica Buffalo*** maintains certifications, accreditations, certifications, and validations with numerous state and national entities. Programs vary but may include on-site audits, reciprocal agreements with another entity, performance testing evaluations, review of the QA Manual, Standard Operating Procedures, Method Detection Limits, training records, etc. At the time of this QA Manual revision, the laboratory has accreditation/certification/licensing with the following organizations:

TestAmerica
THE LEADER IN ENVIRONMENTAL TESTING

## TestAmerica Certifications

| Laboratory | Program | Authority | Identification | Expiration Date |
|---|---|---|---|---|
| TestAmerica Buffalo | Federal | USDA | P330-11-00386 | 11/26/2017 |
| TestAmerica Buffalo | NELAP | Florida | E87672 | 06/30/2017 |
| TestAmerica Buffalo | NELAP | Illinois | 200003 | 09/30/2016 |
| TestAmerica Buffalo | NELAP | Kansas | E-10187 | 10/31/2016 |
| TestAmerica Buffalo | NELAP | Louisiana | 02031 | 06/30/2017 |
| TestAmerica Buffalo | NELAP | Minnesota | 036-999-337 | 12/31/2016 |
| TestAmerica Buffalo | NELAP | New Jersey | NY455 | 06/30/2017 |
| TestAmerica Buffalo | NELAP | New York | 10026 | 03/31/2017 |
| TestAmerica Buffalo | NELAP | Oregon | NY200003 | 06/09/2017 |
| TestAmerica Buffalo | NELAP | Pennsylvania | 68-00281 | 07/31/2017 |
| TestAmerica Buffalo | NELAP | Texas | T104704412-15-6 | 07/31/2017 |
| TestAmerica Buffalo | NELAP | Virginia | 460185 | 09/14/2017 |
| TestAmerica Buffalo | NELAP Primary AB | New Hampshire | 2973 | 09/11/2017 |
| TestAmerica Buffalo | NELAP Secondary AB | New Hampshire | 2337 | 11/17/2016 |
| TestAmerica Buffalo | State Program | Arkansas DEQ | 88-0686 | 07/06/2017 |
| TestAmerica Buffalo | State Program | California | 1169CA | 09/30/2017 |
| TestAmerica Buffalo | State Program | Connecticut | PH-0568 | 09/30/2016 |
| TestAmerica Buffalo | State Program | Georgia | 956 | 03/31/2017 |
| TestAmerica Buffalo | State Program | Georgia | N/A | 03/31/2017 |
| TestAmerica Buffalo | State Program | Iowa | 374 | 03/01/2017 |
| TestAmerica Buffalo | State Program | Kentucky (DW) | 90029 | 12/31/2016 |
| TestAmerica Buffalo | State Program | Kentucky (UST) | 30 | 03/31/2017 |
| TestAmerica Buffalo | State Program | Kentucky (WW) | 90029 | 12/31/2016 |
| TestAmerica Buffalo | State Program | Maine | NY00044 | 12/04/2016 |
| TestAmerica Buffalo | State Program | Maryland | 294 | 03/31/2017 |
| TestAmerica Buffalo | State Program | Massachusetts | M-NY044 | 06/30/2017 |
| TestAmerica Buffalo | State Program | Michigan | 9937 | 03/31/2016 * |
| TestAmerica Buffalo | State Program | North Dakota | R-176 | 03/31/2017 |
| TestAmerica Buffalo | State Program | Oklahoma | 9421 | 08/31/2017 |
| TestAmerica Buffalo | State Program | Rhode Island | LAO00328 | 12/30/2016 |
| TestAmerica Buffalo | State Program | Tennessee | TN02970 | 03/31/2017 |
| TestAmerica Buffalo | State Program | Washington | C784 | 02/10/2017 |
| TestAmerica Buffalo | State Program | West Virginia DEP | 252 | 09/30/2016 |
| TestAmerica Buffalo | State Program | Wisconsin | 998310390 | 08/31/2017 |

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* Certification Valid - Laboratory is Pending Renewal with the Program Authority

For more information, or to contact a local TestAmerica representative nearest you, please visit our website at www.testamericainc.com



The certificates and accredited parameter lists are available for each State/Program at www.testamericainc.com under Analytical Services Search – Certifications.




EMSL Analytical, Inc. Management




# MODULE A

## Asbestos Analysis

**PHASE CONTRAST MICROSCOPY**

**TRANSMISSION ELECTRON MICROSCOPY**

**POLARIZED LIGHT MICROSCOPY**

# Module A – Asbestos – PLM, PCM and TEM

Table of Contents

# Module A – Asbestos – PCM, PLM and TEM

## A.1.0 Scope

This module defines the policies and procedures specific for laboratories performing analysis for asbestos samples under AIHA-LAP, A2LA, NVLAP, CALA, TNI or State Accreditation requirements or for laboratories conducting this analysis without accreditation. The requirements documented in this module are supplemental to the policies and procedures documented in the main section of the Quality Management System (QMS) Manual. This Module maintains the same format as the main QMS Manual for easy reference of additional requirements.

## A.2.0 Normative References

- AIHA-LAP, LLC – Accreditation Policy Modules – October 2016
- ISO/IEC 17025:2005
- TNI Standards – July 1, 2009
- NIST 150 Handbook & Checklist – July 1, 2016
- NIST 150-3 Handbook (2006) & Checklist (2011)
- NIST 15-13 Handbook (2006) & Checklist (2007)
- A2LA General Requirements: Accreditation of 17025 Laboratories (R101), Feb. 2016

## A.3.0 Terms and Definitions

See QMS Manual, Appendix A – Glossary.

## A.4.0 Management Requirements

### A.4.1 Organization

#### *A.4.1.1 NVLAP Sub-Facilities*

**A.4.1.1.1 Relationship with Main Facility**

Following the NVLAP requirements, sub-facilities are to be supported by the main facility. The EMSL main facility includes the corporate Quality Assurance Department, vice president/asbestos division, as well as the corporate laboratory facility. The Quality Assurance Department, vice president, and regional managers provide support to the sub-facilities including:

- Developing, implementing and maintaining quality assurance procedures and policies
- Standardizing reporting policies and standard operating procedures
- Implementing and enforcing the quality control program
- Providing monthly review of quality control data (including calibrations, re-analysis data, standards, contamination data, etc.)
- Completing annual reports to upper management on laboratory quality activities
- Providing technical direction and support

- Staying current with the technical, analytical, and policy developments of the industry aided by involvement in technical committees such as ASTM, AIHA, EIA, etc.
- Distribution of proficiency testing samples
- Selection of supplies and equipment

#### A.4.1.1.2 Sub-Facility Internal Audits

The internal audit procedures for the sub-facility follow those policies written in the EMSL Internal Audit SOP.

#### A.4.1.1.3 Sub-Facility Proficiency Testing

Regional managers ensure that the NVLAP sub-facilities receive and analyze NVLAP proficiency testing rounds. All active analysts participate in the testing, when possible, following the requirements of the NVLAP program.

The QA Department later forwards a copy of the scored results to the sub-facilities. If necessary, the laboratory manager initiates corrective action procedures. A copy of these results is maintained at the sub-facility and is made available for analysts to review.

Results of the proficiency analysis of the sub-facility laboratories are not submitted to NVLAP.

### A.4.2 Management System

No additional requirements. See § 4.2 of QMS Manual.

### A.4.3 Document Control

No additional requirements. See § 4.3 of QMS Manual.

### A.4.4 Review of Requests, Tenders and Contracts

No additional requirements. See § 4.4 of QMS Manual.

### A.4.5 Subcontracting of Tests and Calibrations

Laboratories that choose to subcontract any sample analysis to an outside laboratory shall ensure that the subcontract laboratory maintains the same relevant accreditations as that of the contracting laboratory. Qualifications of EMSL laboratories are included in the "Laboratory Qualifications" pages on the EMSL website. Use of non-EMSL subcontract laboratories must be approved by the Vice President of Asbestos Division.

**Note:** EMSL laboratories are considered independent facilities following NVLAP and AIHA-LAP policy, therefore, subcontracting policies apply. See *EMSL Subcontracting SOP* and § 4.5 of QMS Manual for additional detail.

### A.4.6 Purchasing Services and Supplies

Supplies including microscope slides, sample preparation tools, acetone, triacetin, copper specimen grids, filters, acids and dispersion oils are to be ordered through the corporate

purchasing department. This allows for company-wide control and standardization of consumable supplies.

**A.4.7 Service to the Customer**
No additional requirements. See § 4.7 of QMS Manual.

**A.4.8 Complaints**
No additional requirements. See § 4.8 of QMS Manual.

**A.4.9 Control of Nonconforming Testing**
No additional requirements. See § 4.9 of QMS Manual.

**A.4.10 Improvement**
No additional requirements. See § 4.10 of QMS Manual.

**A.4.11 Corrective Action**
No additional requirements. See § 4.11 of QMS Manual.

**A.4.12 Preventive Action**
No additional requirements. See § 4.12 of QMS Manual.

**A.4.13 Control of Records**
No additional requirements. See § 4.13 of QMS Manual.

**A.4.14 Internal Audits**
No additional requirements. See § 4.14 of QMS Manual.

**A.4.15 Management Reviews**
No additional requirements. See § 4.15 of QMS Manual.

## A.5.0 Technical Requirements

**A.5.1 General**
No additional requirements. See § 5.0 of QMS Manual.

**A.5.2 Personnel**

***A.5.2.1 PCM***

The PCM training program follows a course that satisfies the requirements of a NIOSH 582 equivalent (582e) program. The laboratory manager is responsible for ensuring all PCM analysts receive proper training before performing analysis on client samples independently. The trainer may be a designee (i.e. a senior analyst) but must have completed the EMSL 582e training course within the past 5 years prior to performing training. The lab manager will draw on the candidate's previous training, if any. The candidate will receive sufficient in-house training following EMSL's PCM Training Checklist, to demonstrate proficiency and understanding in all related topics, to the laboratory manager's satisfaction.

EMSL PCM training follows specific policies as described below.

**A.5.2.1.1** Training Program – PCM

Theoretical

Trainees must read and understand the methods, procedures and policies related to the analysis. These include:

- NIOSH 7400 Method
- EMSL standard operating procedure for the analysis of fibers via PCM analysis
- Quality Management System (QMS) Manual
- (7) Presentations covering the EMSL-NIOSH 582e course material including:
    - Introduction to asbestos and the asbestos industry
    - Regulatory agencies/standards and compliance
    - Sampling (including types of cassettes, pumps, calibrations, strategies, safety)
    - PCM microscopy (including optics, scope, alignment, calibration, maintenance)
    - NIOSH Method 7400 (overview, history, calculations, etc.)
    - Sample receiving, handling and preparation
    - Sample analysis and reporting
    - Quality control (including introduction to statistics, ISO 17025 and counting statistics, proficiency programs)
    - Hands-on training with PCM scope alignments/calibrations and fiber counting
    - Written exam
- Reference material is contained in the NIOSH 582e folder on elink.

Demonstration

Working with the trainer, trainees must perform all steps involved with preparation and analysis. The hands-on tasks must include:

- Preparation of samples
- Calibration of the microscope
- Analysis of samples
- Calculation of results

Skills

An analyst-in-training must demonstrate proficiency in a number of ways. These include:

- Demonstration of accurate inter-analyst QC analysis on 95% of 50 real-time samples (see QC program for acceptance criteria; use QC report for PCM analysis)
- Generation of personal relative standard deviations ($S_r$) by the completion of 120 analyses on laboratory reference slides (20 analyses per slide, 40 analyses per range)
- Demonstration of accurate analysis on 80% of 10 past proficiency samples and success in generating data within the acceptable range as established by the agency(ies) statistical analysis

Records pertaining to the analyst's training, including the PCM training checklist, individual training records, bench sheets, etc., are maintained in the analyst's individual training binder.

#### A.5.2.1.2 Qualifications Checklist – PCM

Training is documented with a qualifications checklist. This checklist is completed by the trainer, then dated and initialed by both trainer and trainee. The checklist includes a general section and method specific section.

#### A.5.2.1.3 Qualifying an Analyst – PCM

All analysts must complete EMSL's training program (even if they have previously completed a NIOSH 582 equivalent training course).

Training checklists are maintained, recording all tasks that are completed. Demonstration of Capability (DOC) certifications are issued by the Quality Assurance Department once the training checklists are complete. Analysts are qualified to perform independent analysis when all requirements of the training checklist and the probationary periods (if applicable) are met. In addition, for AIHA-LAP accredited labs, the 20 day probation period must also be met (see A.5.2.4.3.)

#### A.5.2.1.4 Ongoing Training – PCM

Additional training is ongoing. For each PCM analytical method performed by EMSL, there is an associated training checklist to document successful training and sign-off date.

Also, if there are any updates to methodology or regulatory requirements, these are introduced at laboratory meetings or training sessions. During this meeting, the laboratory manager may review certain topics in PCM such as practical aspects of analyzing difficult samples, etc.

A record of this and other training will be documented on EMSL's Training Record forms and kept on file.

#### A.5.2.1.5 Analyst Performance

Analyst performance and qualifications are reviewed annually. Laboratories accredited by the AIHA-LLP must review analyst performance every 6 months. Recertification of Demonstration of Capability is complete when analysts have demonstrated continuing capability as discussed in the general section of this manual.

### *A.5.2.2 TEM Training and Qualification Requirements*

New analysts must complete the EMSL training program in TEM asbestos analysis in order to perform the analysis independently. Training includes:

- Understanding of asbestos regulations/aspects of the industry
- TEM theory, operation and calibration, including all support equipment
- Method review as applicable (AHERA, NIOSH 7402, etc.)

- Proficiency analysis as applicable
- Quality control re-analysis as applicable
- Completion of a TEM asbestos portfolio demonstrating morphology, chemistry (EDXA) and diffraction (SAED)

In all cases, previous training and experience is factored into this program. Completion of each phase of training is defined by the ability to demonstrate skills and knowledge to the satisfaction of the lab manager and QA manager. Each approved task is checked off on the analyst training log checklist. These training logs must be maintained even for those who have received prior training unless equivalent training records are transferred with the analyst.

#### A.5.2.2.1 TEM Theory

The lab manager or other trainer will draw on the candidate's previous training, if any. If not, the candidate will receive sufficient in-house training to demonstrate proficiency in these topics. Included are theory related to selected area electron diffraction, energy dispersive X-ray analysis, crystallography and TEM operation, and calibration.

#### A.5.2.2.2 Hands-on Practices – TEM

When the candidate has received sufficient training to analyze samples, he/she will work in the laboratory alongside an experienced analyst. Some analytical methods will specify requirements for inter-analyst QC analysis on the training checklist. In addition, for AIHA-LAP accredited labs, the 20 day probation period must also be met (See A.5.2.4.3.)

#### A.5.2.2.3 Proficiency Analysis – TEM

Where available and applicable, the trainee must perform analysis on one round of past proficiency samples, and succeed in generating data within the acceptable range as established by the agency(ies) statistical analysis.

#### A.5.2.2.4 Qualifications Checklist – TEM

Training is documented with a qualifications checklist. This checklist is completed by the trainer. The general TEM training checklist precedes subsequent method specific training checklists.

#### A.5.2.2.5 Qualifying an Analyst – TEM

All analysts must have completed the method specific and applicable pre-requisite general checklists to be qualified for analytical methods.

Training checklists, training records, analytical bench sheets, etc., are maintained to document each step of the training process. Demonstration of Capability (DOC) certifications are issued by the Quality Assurance Department once the training checklists are complete. Analysts are qualified to perform independent analysis when all requirements of the training checklist and any applicable probationary periods are met.

**A.5.2.2.6 Ongoing Training – TEM**

Additional training is ongoing. For each TEM analytical method performed by EMSL, there is a training checklist to document successful training and sign-off date.

Also, if there are any updates to methodology or regulatory requirements, these are introduced at laboratory meetings or training sessions. During this meeting, the laboratory manager may review certain topics in TEM such as practical aspects of analyzing difficult samples, etc.

A record of this and other training will be documented on EMSL's Training Record forms and kept on file.

**A.5.2.2.7 Analyst Performance**

Analyst performance and qualifications are reviewed annually. Laboratories accredited by the AIHA-LLP must review analyst performance every 6 months. Criteria for analysts to demonstrate continuing capability are discussed in the general section of this manual.

### *A.5.2.3 PLM Training and Qualification Requirements*

Analysts must complete the EMSL training program in PLM asbestos analysis in order to perform analysis independently. The level of training may be adjusted according to the candidate's work experience, academic background and prior training. Training consists of four phases:

1) Theory and operation of polarized light microscopy technique and asbestos identification (understanding of method and standard operating procedures)
2) Practical application of microscopy and analysis of samples
3) Proficiency analysis
4) Quality control

**A.5.2.3.1 Theory – PLM**

A basic understanding of the asbestos industry, microscopy and the crystallography of asbestos is the preliminary part of the training program. Areas covered include:

- Health effects/regulatory issues of asbestos
- Crystallography
- The polarized light microscope

**A.5.2.3.2 Practical Factors – PLM**

The trainee will work with an experienced analyst on polarized light microscopy techniques, sample preparation methods, the identification and quantitation of asbestos and non-asbestos materials found in bulk samples.

The trainee will analyze at least 50 field samples with an acceptable performance of accurate analysis on 94% of 50 real time samples (using the PLM QC program to determine acceptability).

**A.5.2.3.3 Proficiency Analysis – PLM**
Demonstration of accurate analysis on 80% of 10 past proficiency samples, and succeeding in generating data within the acceptable range established by the PT providers' statistical analysis.

**A.5.2.3.4 Qualifications Checklist – PLM**
Training is documented with a qualifications checklist. This checklist is completed by the trainer. The checklist is separated into a general section and the method specific training.

**A.5.2.3.5 Qualifying an Analyst – PLM**
All analysts must have completed the PLM training program.

Training checklists are maintained, recording all tasks that are completed. Demonstration of Capability (DOC) certifications are issued by the QA Department once the training checklists are complete. Analysts are qualified to perform independent analysis when all requirements of the training checklist and are met. In AIHA accredited laboratories, the 20 day probationary period requirement shall also be met (See Mod. A § A.5.2.4.3).

**A.5.2.3.6 Ongoing Training – PLM**
Additional training is ongoing. For each PLM analytical method performed by EMSL, there is a training checklist to document successful training and sign-off date.

Also, if there are any updates to methodology or regulatory requirements, these are introduced at laboratory meetings or training sessions. During this meeting, the laboratory manager may review certain topics in PLM such as practical aspects of analyzing difficult samples, etc.

A record of this and other training will be documented on EMSL's Training Record forms and kept on file

**A.5.2.3.7 Analyst Performance**
Analyst performance and qualifications are reviewed annually. Laboratories accredited by the AIHA-LLP must review analyst performance every six (6) months. Recertification is complete when analysts have demonstrated continuing capability as discussed in the general section of this manual.

### *A.5.2.4 Additional Personnel Requirements for Accredited Laboratories*

Summarized below are additional personnel requirements as established by AIHA-LAP and NYS ELAP. In addition, please seeError! Reference source not found. the end of this Module for additional personnel education and experience requirements by other accrediting authorities.

#### A.5.2.4.1 AIHA-LAP – Technical Manager

- Employee of laboratory with authority to provide day to day supervision of technical operations, with responsibility for ensuring accredited analysis are performed by qualified, trained personnel who are adequately supervised
- Bachelor's degree in an applicable physical or biological science (inorganic chemistry, environmental sciences, etc.)
- Present onsite at least 20 hours per week or 50% of operating hours (whichever is less) to address technical issues
- Three (3) years of non-academic analytical experience, of which at least two (2) years shall be in industrial hygiene analysis relevant to scope of AIHA-LAP accreditation
- A relevant post-graduate degree (MS or PhD) shall 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.

#### A.5.2.4.2 AIHA-LAP – Quality Manager

- BS degree in an applicable basic or applied science AND one (1) year non-academic analytical experience or quality control experience appropriate to the analysis performed
- OR, in lieu of a BS, four (4) years of non-academic analytical or quality control experience
- Documented training in statistics or laboratory quality assurance/quality control

**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 or equivalent course); or 3) relevant experience – documented examples of the level of quality assurance/quality control used in applicable work experience.

#### A.5.2.4.3 AIHA-LAP – Analysts for Accredited tests

- 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. Until this time, all analyses must be reviewed by the laboratory manager or senior analyst.
- For PCM analysts: Successful completion of the EMSL or other NIOSH

582 equivalent course.
**Note:** EMSL's 582 course has been reviewed by AIHA and found to be equivalent with the NIOSH course.

- 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.5.2.4.4 NVLAP – TEM Technical Supervisor**
The TEM technical supervisor(s) shall be qualified to conduct AEM studies, apply AEM to crystalline materials, and shall be proficient in the field of asbestos analysis including procedures for sample handling, preparation, analysis, storage, disposal, and contamination monitoring.

**A.5.2.4.5 NYS ELAP – Technical Director – PCM**

- Associate's Degree or two (2) years equivalent college study
- Formal course work in PCM analysis
- One (1) year experience under the supervision of an experienced analyst

**A.5.2.4.6 NYS ELAP – Technical Director – TEM**

- Bachelor's Degree
- Specialized course in TEM analysis
- One (1) year experience under supervision of an experienced analyst

**A.5.2.4.7 NYS ELAP – Technical Director – PLM**

- Associate's Degree or two (2) years equivalent college study
- Specialized course in PLM analysis
- One (1) year experience under the supervision of an experienced analyst

## A.5.3 Accommodation and Environmental Conditions

### *A.5.3.1 Laboratory Conditions*

**A.5.3.1.1 Temperature**
The rooms where analysis is performed should be held at normal temperature ranges. Reagents used in the analysis (refractive index oils, triacetin, etc.) shall not be kept in below freezing temperatures. Temperatures are recorded at the time of analysis when analyzing PLM samples due to the effect temperature has on the RI oils. Temperatures are recorded on the worksheet.

**A.5.3.1.2 Lighting**
Direct sunlight should be avoided when using the optical microscopes. Room lighting shall be provided which provides comfortable reading/writing conditions for the analysts. TEM analysis is performed in darkened conditions to provide ease while viewing the fluorescent screen.

**A.5.3.1.3 Ventilation**

Room ventilation must be provided so as to provide safe and comfortable conditions for the analyst.

**A.5.3.1.4 Location**
Air samples (PCM and TEM analysis) are not prepared in the same room where bulk sample preparation is performed.

### *A.5.3.2 Contamination*

**A.5.3.2.1 Contamination Management**
In addition to the procedures and policies discussed in the main section of this manual, specific steps to avoid contamination in the asbestos laboratory include:

- Bulk sample containers are opened and samples examined using the stereo microscope only in the hood.
- Only small numbers of active samples are kept near the hood. The sample containers are kept closed at all times. Inactive samples are stored in a suitable, out of the way area.
- Target containers, including samples, mounting reagents, microscope slides, and cover glasses, are opened one at a time as practical.
- Prepared slides are stored in a protected manner.
- Prepared TEM samples are stored in an indexed grid box.
- Surfaces are frequently wiped clean with moistened wipes.

**A.5.3.2.2 Ambient Air Monitoring**
On a quarterly schedule, air monitoring is performed in the laboratory. Ambient air samples shall be collected from each work area as well as other common areas such as Log In.

Sampling and analysis is performed according to the following requirements:

- Collection on 0.45 micron MCE filters (at least 1200 liters collected at no greater than 10 lpm)
- Collection during normal working hours to best monitor worker exposure
- Analysis is performed via TEM AHERA protocol.
- The action level is considered exceeded if any asbestos is detected.

If any asbestos is detected, a PCM analysis is performed (to coincide the data with health and safety (OSHA) limits) and the event is documented on a corrective action (CAR) form. The area is cleaned and additional samples are collected and analyzed by TEM AHERA to ensure the area is free of contamination. Cleaning and sampling is to continue until the sample results are negative for asbestos. Additional monitoring in addition to the regular quarterly samples may be needed to ensure corrective actions were effective.

**A.5.3.2.3 Resolution of Contamination in Lab Blanks**

If analyses of the blank samples indicate the possibility of contamination, the customer sample analysis is immediately halted. The area and tools are cleaned and another blank sample prepared and analyzed. If this second sample shows contamination, a complete investigation is conducted to determine the contamination source (acetone, triacetin, dispersion oils, preparation containers, etc.). A detailed flow chart for resolution of PLM and TEM contamination can be found in the appropriate SOPs.

## A.5.4 Test and Methods and Method Validation

### *A.5.4.1 List of Asbestos Specific SOPs*

The scopes of accreditation differ between branch labs. Accreditations and scopes are found in the EMSL websites – qualifications.

### *A.5.4.2 Estimation of Uncertainty of Measurement*

EMSL has established and applies procedures for the determination and reporting of uncertainty to customers. These procedures are summarized in the "Uncertainty Worksheet – Asbestos" and detailed in EMSL's PLM SOP, NIOSH 7400 SOP, and TEM AHERA SOPs.

### *A.5.4.3 Control of Data*

#### A.5.4.3.1 Data Review

As documented in the main QMS Manual § 5.4.3, data review is performed continuously by the laboratory management for irregularities or questionable results. Criteria for judging a result questionable will include deviation from prior data from the same sample, from another sample collected within the same homogenous area, etc. Any questionable result will be rechecked with other quality control samples.

#### A.5.4.3.2 Data Recording for TEM

EMSL is in the process of converting all worksheets to direct entry into our iL@b LIMS system. There are very specific requirements for the recording of information for the TEM analysis. Data recording should be done in a manner conducive to good record keeping. If data cannot be entered directly into the iL@b LIMS system, data should be recorded using either black or blue indelible ink on an original analytical worksheet. Data should not be recorded in lab notebooks or on scrap paper and then transposed to the analytical worksheet. If any correction on the data form needs to be made, it should be done using a dated and initialed single-line strikeout, and then the new data recorded next to the old data. The use of correction fluid is not permitted.

##### *A.5.4.3.2.1 Sample Preparation Data*

Sample preparation data is recorded on the internal chain of custody, the analytical worksheet, or directly into the LIMS system. The data recorded includes the following*:*

- Project identification number
- Sample identification number

- Customer identification
- Sample description
- Preparation date and analyst's ID
- Grid box identification
- Location of grid preparations in the grid box
- Effective filtration area and volume filtered (applicable for water, Chatfield, etc.)

Specific procedures for each sample type and criteria for acceptable final preparations can be found in the applicable Standard Operating Procedures for the method.

*A.5.4.3.2.2 Structure/Fiber Identification & Sizing (where applicable)*

The analytical worksheet or direct data entry system will contain at a minimum:

- SAED identification and negative number, if applicable
- EDXA spectra identification and printout or computer file ID, if applicable
- Structure's mineralogical identification (i.e., chrysotile, amphibole or non-asbestos)
- Structure classification (i.e., fiber, bundle, cluster, matrix)
- Structure size
- Quantitative totals of all structures / fibers identified as both asbestos and non-asbestos particles
- Analyst initials and date
- Sketch, if applicable

*A.5.4.3.2.3 SAED Indexing*

Images of SAED diffraction patterns are developed and measured to verify the pattern identity. Each analyst is required to have an on-screen identification accuracy of at least 80% as determined by indexing the negative. If any analyst falls below 80%, the analyst must not analyze customer samples until the laboratory manager has reviewed his/her data and determine the cause of the problems involved. Corrective actions will then be documented.

Chrysotile:
Suspected chrysotile diffraction patterns are examined for a 5.3 Å layer-line (row) spacing and for the correct orientation and d-spacings. If results differ by more than 5% of accepted values, identification of chrysotile is suspect.

Amphibole:
Suspected amphibole patterns are examined for a 5.3 Å layer-line spacing (if the pattern is of random orientation) or for Zone-Axis measurements if a zone axis pattern was obtained. If results differ

by more than 5% of accepted values, identification is suspect, particularly for amphiboles.

*A.5.4.3.2.4 EDXA Spectra Evaluation*

EDXA spectra can be evaluated either qualitatively, by comparing the sample spectra to spectra obtained from asbestos standards, or quantitatively, by calculating elemental concentrations using K-Factors obtained during EDXA calibration.

## A.5.5 Equipment

### *A.5.5.1 Equipment List*

Each EMSL asbestos laboratory maintains a variety of equipment suitable to their size and workload. Specific equipment requirements can be found in the analytical SOPs. Each lab maintains an inventory of equipment onsite which provides specific of their particular equipment. This list is available for review upon request.

### *A.5.5.2 PCM Verification of Instrument Verifications*

**A.5.5.2.1 Stage Micrometer**

See § 5.6 of main QMS Manual.

**A.5.5.2.2 Microscope Alignments**

*Daily* (each analyst to perform daily or prior to analysis):

- Phase Ring Alignment - adjust to concentric
- Contamination control - clean microscope and work area

*Weekly* (each analyst to perform weekly)

- HSE/NPL Test Slide – block 3 must be visible, 4-5 partially or completely visible, 6-7 invisible
- Some labs use the HSE/UPO slides. These slides are available in different resolutions and acceptable line visibility is as follow:
  - HSE/ULO Red - Lines 1- 4, must be visible, line 5 partially visible, lines 6-7 invisible
  - HSE/ULO Green - Lines 1-5 visible, line 6 partially visible, line 7 invisible

*Monthly*

- Measurement of Walton-Beckett Graticule: Diameter must be 100 µm

**Note:** For labs complying with the TNI (NELAC) Standard, this must be checked and recalculated once per day by each analyst using the scope.

- Mechanical Counting Aids (clickers) must be verified for accuracy monthly. The clicker must read 100 on the 100$^{th}$ count.

*First time use (new, newly received or repaired microscope)*

- Phase Ring Alignment - adjust to concentric

- Contamination control - clean microscope and work area
- HSE/NPL or HSE/UPO acceptability as listed above
- Walton Beckett Graticule: Diameter must be 100 µm

**A.5.5.2.3 Analyst Calibration**

*Daily* (each analyst, each day prior to performing PCM analysis):

- Standard reference slide (past proficiency test slide or other well characterized real world samples with targets and acceptance limits)

**A.5.5.2.4 Hood Check**

Quarterly, measure and record flow rate of PCM hoods with anenometer following the *EMSL Hood Maintenance and Calibration SOP* for acceptance criteria.

### *A.5.5.3 TEM Instrument Verifications*

**A.5.5.3.1 Alignment**

Proper alignment of a transmission electron microscope is imperative in order to provide quality data. Each microscope will be checked for alignment daily, before first use, and thereafter during the day as the analyst deems necessary, or due to deteriorating conditions during analysis. If the microscope cannot be brought into alignment, the microscope should be serviced. The microscope is then realigned for use. Any service performed on the microscope is recorded in the equipment maintenance log.

**A.5.5.3.2 Magnification**

It is imperative to know accurate image magnifications for the sizing of asbestos, both on the TEM phosphor screen and negative. In order to achieve, this, the magnification will be calibrated monthly, both on phosphor screen and negative, using a carbon replica standard of 2,160 lines/mm at both 20,000 X and 10,000 X nominal magnifications. If the calibrated magnifications do not fall within acceptable limits of 2SD < 5% cumulative mean, the calibration should be checked for accuracy.

If the calculated magnification differs significantly from the target magnification and no other sources of error can be found (e.g., calculation errors, non-eucentricity), the microscope should be serviced and realigned, and magnification recalibrated.

Magnifications will be charted over time to indicate any trends or problems. The variation of calibration data points, (defined as 2X the standard deviation of the past measurements to date) must be <5% of the mean. This data is tracked and managed in the EMSL QC program.

Any service is documented in the equipment maintenance log.

**A.5.5.3.3 Verification of Measuring Aids**

It is important to determine the exact size of the 0.5 and 5 micron measuring aids on the phosphor screen (either circles or two perpendicular lines, depending on manufacturer). This is easily measured, but requires a one-time procedure of removing the viewing glass and measuring directly on the phosphor screen with a fine ruler. Be careful not to scratch the phosphor coating. Armed with these measurements and the most current magnification calibration results, one can calculate their exact size at the magnification used for analysis. (These markings need to be recalibrated every time the phosphor screen is recoated.)

For asbestos in water analysis, additional calibration of 1.0 and 10 µm aids at 10,000x is also required.

**A.5.5.3.4 Camera Constant**

In order to index or measure selected area electron diffraction (SAED) patterns, an accurate camera constant must be obtained. For this purpose, the on screen and on negative camera constants will be calculated monthly (provided measurements have been stable over time; see below) using a gold-coated TEM grid. Camera constants shall not fall outside 2xSD <5% cumulative mean (the average calibrated camera constant to date). In the event the camera constant measurement indicates change outside these limits, sources of variability should be investigated and the calibration frequency should be increased to weekly for a period of four weeks.

If sources of error have been investigated, but the measured result differs significantly from the target value, the microscope should be serviced and realigned and the camera constant recalculated. Any service is documented in the equipment maintenance log.

Camera constant calibrations will be charted over time to indicate any trends or problems. The variation of calibration data points, (defined as 2X the standard deviation of the past measurements to date) must be <5% of the mean. This data is tracked and managed in the EMSL QC program.

If sufficient data has been collected which indicates confidence in the stability of measurements, frequency of camera constant calibration can be adjusted to monthly. If values fall outside the acceptance range based on statistical evaluation, frequency is increased to weekly for approximately four weeks until stability of measurements is obtained again. Stability is determined by evaluating the data collected from weekly measurements over a period of 6 months (24 data points), using the above mentioned criteria.

**Note:** For those instruments utilized for water calibration, frequency must be maintained at a weekly rate during the period of time analysis is performed.

**A.5.5.3.5 Chrysotile Beam Dose**
Low beam dose verifies the TEM can generate SAED diffraction patterns obtained from single chrysotile fibrils without damaging the fibril. A fibril having a diameter ≤ 0.5 µm, and length greater than, or equal to 1 µm, must produce a SAED pattern visible for a minimum of 15 seconds. Low beam dose must be demonstrated quarterly with 9 of 10 SAED patterns obtained from NIST traceable chrysotile fibrils. The electron micrograph of both the chrysotile fibril image and the SAED pattern obtained from that fibril must be maintained.

When proper dose levels are achieved, the parameters of the microscope settings are documented and known by each operator. These settings include:

- Condenser aperture
- Spot size
- Accelerated voltage
- Beam current

**A.5.5.3.6 Spot Diameter**
The beam size (at crossover) normally used for EDXA elemental analysis is calibrated quarterly to verify a spot diameter of less than 250 nm. An electron micrograph verifying this calibration must be recorded quarterly.

Diameter measurements will be charted over time to indicate any trends or problems. The variation of spot diameters, defined as 2X the standard deviation of the past measurements to date, must be <25% of the mean. This data is tracked and managed in the EMSL QC program.

**A.5.5.3.7 Mapping Areas with Abnormal Spectra**
The sections of the grid specimen holder, which may produce abnormal spectra, must be known to all analysts. Using a known standard reference material, such as NIST 2063a or 1866 Amosite, the areas producing abnormal spectra are recorded. This way determination can be made as to the regions that should be avoided in routine analysis.

**A.5.5.3.8 EDXA Detector**
The following lists the proper calibration and monitoring of performance for the EDXA detector.

The TEM's Energy Dispersive X-Ray Analyzer (EDXA) is checked daily at the Al and Cu peak center line measuring, 1.48 keV and 8.04 keV respectively, within ±0.01 keV.

To assure low energy detection of the EDXA system, a resolvable Na kα peak must be measured using the NIST SRM 1866 crocidolite standard. These measurements must meet the criteria:

$I_B > 3(2I_B^b)^{1/2}$ where:
$I_B$ = Integrated peak intensity, background subtracted
$I_B^b$ = Integrated background intensity

Print a hard copy of the spectrum, sign, date, and file. It is strongly recommended to save spectrum on the hard drive. This must be done quarterly.

Using a single fibril of chrysotile from NIST SRM 1866, 1876a, or 1876b, the detector must be capable of clearly resolving the Mg and Si peaks. Hard copies of the spectrum are printed, signed, dated, and filed. This must be done quarterly.

The detector resolution is measured using a Mn source to verify the Mn kα peak has a resolution of less than, or equal to 175 eV, at full width half maximum. Resolution measurements are tracked over time to indicate any trends or problems. The value of the sum measurements and the variation (defined as 2X the standard deviation of the past measurements to date) must not exceed 180 eV. Print a hard copy of the spectrum, sign, date, and file. This must be done on a semi-annual basis.

For labs complying with the TNI (NELAC) standard, this frequency shall be quarterly.

Elemental K-Factors for Mg, Ca, and Fe relative to Si are calculated using NIST SRM 2063a or equivalent as a standard. The Mg to Fe ratio is also calculated using NIST SRM 2063a or equivalent as a standard. Elemental K-Factors for Na and Al to relative to Si are calculated from an Albite standard. These two sets of standards for K-Factor determination should be done at the same time (preferably the same day.) The following are some of the pass-fail criteria (see *Asbestos QC SOP* for complete listing):

Mg:Si – 1.0 - 2.0
Ca:Si – 1.0 - 1.75
Fe:Si – 1.0 - 2.0
Mg:Fe – 1.5 or less
Na:Si – 1.0 – 4.0
Al:Si – 1.0 – 1.75

Print a hard copy of the spectrum, sign, date, and file. It is strongly recommended to save spectrum on the hard drive. This must be done on a semi-annual basis.

**A.5.5.3.9 Plasma Asher**
Although the AHERA method specifies 10% ashing, it allows for etching less than this amount if 10% generates a texture that negatively affects structure

counting. EMSL has evaluated the ashing procedures, and found that in most cases, 5% removal produces better preparations with lower fiber loss and better intact carbon films.

The low temperature plasma asher is calibrated quarterly to provide the calculated time needed to remove 5% of the collapsed mixed cellulose ester filter. This is performed gravimetrically and is charted against time.

Any service is documented in the equipment maintenance log.

#### A.5.5.3.10 Grid Opening Measurement

TEM 200 mesh locator grids are to be measured using light microscopy. Upon the receipt of each batch (10 vials of 100 grids), 2 grids per vial are removed and measured at the rate of 20 grid openings per grid, for a total of 400 grid openings measured. A total of 2% of the grid lot is measured to determine the average grid opening area in $mm^2$.

For labs meeting the TNI Standard, uniformity of the grid opening area measurements across all 400 grids is also tracked for acceptability.

Precision of the measuring system itself is tracked and documented. TEM Grids used for NIOSH 7402 analysis have a reduced measurement frequency of 20 grid openings per batch of 1000 grids.

See also *EMSL Grid Measurement SOP*.

#### A.5.5.3.11 Muffle Furnace

The high temperature muffle furnace is verified quarterly at three different temperatures in the temperature range of 450° to 520°C. A high temperature thermometer should be immersed in a sand bath for temperature readings. Record date, target temperature, measured temperature and initials of technician. Any service performed is recorded in the equipment maintenance log.

#### A.5.5.3.12 Effective Filtration Area

An accurate result in any procedure requiring filtration depends upon an accurate measurement of the effective filter area (EFA). Prior to use on actual samples, the EFA of all filter funnel apparatus should be determined and documented. The EFA of disposable filter funnels needs to be documented using the "Effective Filtration Area Log" form at a rate of 2 per lot. This information should be stored in the laboratory files for future reference.

#### A.5.5.3.13 Analytical Balance & Weights

See §§s 5.5.3 & 5.6.2 of main QMS Manual.

### *A.5.5.4 PLM Equipment Verifications*

Verification procedures must be followed prior to the analysis of samples to ensure results of analysis reflect true and accurate data. The following summarizes the type and frequency of calibration required. Details on the performance of these functions are found in the PLM SOP.

**A.5.5.4.1 Microscope**

*Daily* (each analyst to perform daily, prior to analysis):

- Center stage or objectives, and center and align condenser lens
- Align polars (full extinction)
- Crosshair alignment fixed in polarizer's privileged direction

**A.5.5.4.2 Analyst**

*Daily*

- Standard reference sample (may be actual sample material or pre-mounted slides from old proficiencies that are mounted in the proper RI medium that allow the recording of all the optical properties, including the refractive index of the asbestos fiber)
- Contamination check with fine grained, reagent grade salt

*Monthly*

- Check standard Amosite mount for proper dispersion color / wavelengths.

**A.5.5.4.3 Verification of Balance**

See § 5.5.3 of main QMS Manual.

**A.5.5.4.4 RI Oil Calibration**

RI oils are calibrated quarterly and every time a new bottle is open. If the date of last calibration on the bottle is greater than three months, then the oil needs to be verified before use.

For NIOSH 9002 samples, RI Oils must be calibrated weekly or on next use.

See *EMSL RI Calibration SOP*.

**A.5.5.4.5 Muffle Furnace Checks**

Muffle furnace temperature are checked quarterly at three different temperatures in the temperature range of 450° to 520°C.

### A.5.6 Measurement Traceability

The results of analytical measurements are traceable to recognized standards. These standards include:

- SRM 1876b (or equivalent, i.e., NVLAP PT round)
- SRM 1867 or 1867a
- SRM 2063a
- SRM 1866 (a or b)

- Calibration using NIST certified or NIST traceable support equipment (thermometers, balance weights, stage micrometer, etc.)
- Consensus standards such as past proficiency testing samples

### A.5.7 Sampling

For purposes of this manual, sampling is defined as the procedures involved with the splitting of samples for distribution to other laboratories and the preparation of samples for analysis (sub-sample preparation).

Samples are not split prior to shipment to another laboratory for regular customer analysis (as in sub-contracting). Where samples are shipped for quality control purposes, the originating laboratory may choose to retain a portion of the sample for internal quality control purposes. Care must be taken when splitting the sample to ensure the sample is split as evenly as possible. **Note***:* The splitting of samples for QC is optional and at the discretion of the laboratory manager.

Sub-sample preparation procedures are detailed in the *EMSL PLM SOP*.

### A.5.8 Handling of Test and Calibration Items

#### *A.5.8.1 Sample Acceptance Criteria: General*

Whenever samples fail to meet sample acceptance criteria, the chain of custody and/or other project records shall be appropriately commented, and the customer shall be contacted immediately. If the customer requests the analysis to continue, the laboratory shall ensure that all correspondence is clearly documented. Sample acceptance criteria include, but are not limited to, the lists included in the following sections.

#### *A.5.8.2 Sample Acceptance Criteria: PCM*

Samples accepted for PCM analysis must comply with a number of specific conditions. Examples of situations where samples might be judged unacceptable include:

- Bulk samples packaged with the PCM cassettes
- Cassettes packaged with Styrofoam 'peanuts'
- Tops missing from the cassette (or disassembled)
- Sample numbers on the COC don't match sample numbers on the sample, or are otherwise unidentifiable
- Wet filter

See also *EMSL PCM NIOSH 7400 SOP* for additional criteria (if any).

#### *A.5.8.3 Sample Acceptance Criteria: TEM*

Samples accepted for TEM analysis must comply with a number of conditions. Samples are judged unacceptable under any of the following circumstances:

- Air cassettes submitted with bulk samples in the same package
- Air cassettes packaged with Styrofoam 'peanuts'
- Air cassettes submitted with missing tops (or disassembled)
- Air cassettes submitted with wet filters
- Water samples submitted with preservative added

- Sample numbers on the COC don't match sample numbers on the sample, or are otherwise unidentifiable
- Insufficient amount of sample submitted
- More than one non-layered sample in container ('Baggie')

See also individual *EMSL TEM Method SOPs* for additional criteria (if any).

#### *A.5.8.4 Sample Acceptance Criteria: PLM*

Samples accepted for PLM analysis must comply with a number of conditions. Samples are judged unacceptable under any of the following circumstances:

- Insufficient amount of sample submitted
- Sample containers open
- Sample numbers on the COC don't match sample numbers on the sample, or are otherwise unidentifiable.
- Samples submitted in damaged sample containers
- Evidence of cross contamination

See also the EMSL PLM SOPs for additional criteria (if any).

#### *A.5.8.5 Sample Storage & Disposal*

| Sample Type | Standard Retention Time | Storage Conditions |
|---|---|---|
| Bulk and Air Asbestos | 60 days | Stored in Ziplock bags; Air & Bulk not stored together |
| TEM Grids (AHERA) | 3 Years | Stored in indexed grid boxes |
| TEM Grids (NYS NOBs) | 3 Years | Stored in indexed grid boxes |
| TEM NOB Grids (non-NY) | 60 days | Stored in indexed grid boxes |
| Water grids filtrate of sample/ petri dishes may be stored also) | 3 Years | Grids are stored in indexed grid boxes If filters are also stored; the filters used for prep are stored in individual 50 mm Petri dishes |
| Microvac & wipe Petri dishes and grids | 1 Year | Grids are stored in indexed grid boxes The filters used for prep are stored in individual 50 mm Petri dishes |

Following analysis, all bulk and air samples are placed in Ziplock bags and held for at least 60 days from the final report date, unless otherwise requested by the customer. TEM grids analyzed for AHERA compliance are held for three (3) years. Grids for microvac and wipes are held for only one (1) year. Samples containing $\geq$1% asbestos are discarded through a licensed hazardous waste removal company. A copy of the waste manifest is stored in the laboratory files.

Air samples shall not be stored with bulk samples, containers must be secure, and storage boxes should be placed in an area void of any possibility of damage.

After analysis is complete, the filtrate of water samples may optionally be stored on the filters used for preparation in individual 50 mm petri dishes. The prepared grids are stored in grid boxes assigned specifically to be stored for three (3) years. The petri dishes and grids are stored for three (3) years from the date of the final report.

If requested, samples will be returned to the customer.

## A.5.9 Assuring the Quality of Test and Calibration Results

### *A.5.9.1 Monthly QC Data Report to Management*

In addition to the Quarterly Report required by all departments, the asbestos quality control coordinator or quality assurance coordinator (or lab manager) shall compile a monthly report of quality control data by the 15th of each month for submission to the corporate QA Department.

The laboratory manager will prepare the monthly report highlighting the following:

- Summary of all QC activities
- Results of investigations of any QC outlier results
- Report of any problems, discrepancies encountered

This Monthly QC Report is forwarded to the QA Department for review, to the attention of MonthlyQA@emsl.com. The report is reviewed for compliance, and pertinent feedback is sent to the lab manager.

The QA review encompasses;

- Completeness and timeliness of the report
- Amount of analytical QC performed,
- Other QC frequencies being met
- Correct usage of spreadsheets and bench sheets
- Periodic trend analysis
- Whether outliers are addressed by the lab



### *A.5.9.2 Trend Analysis of QC Data – PLM, TEM, PCM*

Calibration and Standard data for the Asbestos Department will be charted over time in order to evaluate analyst and laboratory performance. This data shall be reviewed by the laboratory and/or the Quality Assurance Department. Statistically relevant trends should trigger an evaluation.

Items being monitored:

- Failures exceeding the Control Limits (3s)
- Two consecutive points above or below the Warning Limits (2s)
- Seven (7) consecutive data points on either side of the mean
- Seven (7) consecutive points moving in the same direction

Trend analysis shall be documented along with conclusions. One on one discussion and training/re-calibration of the analyst with the manager or other senior analyst oftentimes is successful. Actions taken as a result may be documented as preventive actions unless a failure has occurred. Failures should be documented with a corrective action record. Trend evaluations shall be included in quality reports submitted to the laboratory manager and corporate Quality Assurance Department.

### *A.5.9.3 Selection of Quality Control Samples*

The selection of samples for re-analysis is as random as possible. Original (first) results should not be known when the second analysis is performed.

Samples chosen are typically random, however, samples may be selectively chosen if there is a problematic or unique sample where a re-analysis may provide important information about that sample.

For PLM sample selection, also must strive to meet the requirement that 30% of QC samples fall between 1%-10%.

#### A.5.9.3.1 *Single Analyst Laboratories*

Laboratories that have a single analyst will be unable to perform in-house, inter-analyst QC analysis. Therefore, the percentage of intra-analyst QC analysis, as well as inter-lab QC analysis should be increased to maintain the overall 10% QC requirement.

### *A.5.9.4 PCM Quality Control*

The laboratory manager will determine how QC testing is implemented, either on a frequency basis (e.g., after the analyses of every ten samples), or by a percentage of sample volume.

The Quality Assurance Department will inspect the results of QC testing on a regular basis, and provide the necessary support and directives to the laboratory manager to ensure the QC program is properly executed.

The laboratory manager (or manager's designee) of each laboratory is responsible for implementing the day-to-day QC testing, and ensuring the correct types of testing occur at the appropriate frequencies. The laboratory manager is also responsible for ensuring complete records of QC testing are maintained.

#### A.5.9.4.1 Intra-Analyst – PCM

The original analyst re-analyzes the same sample. An attempt should be made to perform the analysis as 'blindly' as possible. Data is evaluated using the PCM QC program spreadsheet.

#### A.5.9.4.2 Reference Samples – PCM

Known standards are analyzed as if they were actual customer samples.

At the start of each day, the analyst will be given a standard reference slide at random for analysis. Results of this count are then compared to the standard reference value calculated by the proficiency program, which will determine whether the results are within accepted limits. Slides are rotated to ensure all slides are analyzed at approximately the same frequency over a period of time.

**Note:** The analyst may not analyze customer samples until she/he has counted a reference slide. Results must be within acceptance criteria before analysis of customer samples can begin.

**A.5.9.4.3 Statistical Analysis of PCM QC Data**

Copies of data produced in intra-analyst testing will be submitted to the laboratory quality assurance or quality control coordinator or laboratory manager. The quality assurance or quality control coordinator or lab manager then carries out statistical analysis, using the EMSL monthly quality control report Excel spreadsheet. Procedures for this analysis, data collection, and data evaluation are described below. This program covers the QC requirements for airborne fiber counting. It addresses the requirements as defined in NIOSH Method 7400.



The principle objectives of the QC program are as follows:

- To determine analyst CV values over the ranges of fiber concentrations to assist in identifying random intra-analyst errors
- To verify that results of analysis are precise within the 95% confidence level as measured using the determined analyst CV
- To verify that an analyst's results are accurate as measured against a general consensus result (past PAT samples and well characterized customer samples)

The program has three principle methods used to satisfy our objectives. These include:

1) Intra-analyst re-analysis of 10% of the samples
2) Routine re-analysis of Standard Reference slides
3) Inter-laboratory round robins

Data collected from the analysis of the same reference sample will be used to calculate analyst CV values, from all three (3) required ranges (5-20, >20-50, >50-100 fibers in 100 fields). The CV is updated as data is collected from daily reference slide analysis. Calculations are updated as 20 data points are collected.

The system will use these CV values to determine the acceptance of each set of sample replicates that are run daily. The system uses statistical calculations following those referenced in the NIOSH 7400 Method, Issue 2, dated August 1994, and is based on paired difference statistic. When a re-analysis does not

agree with the original, all other samples in that set are to be re-analyzed. Test the new analysis with the original analysis for the entire batch and discard any failed data. Report results from those sample sets that fall within the control limits only.

*A.5.9.4.3.1 Calculations*

**Standard Deviation:**

A standard deviation is calculated for each set of data (minimum 20 data points/range) generated for each applicable range of fiber concentration using the formula:

$$\sigma = \sqrt{\frac{\sum(x - \bar{x})^2}{n}}$$

Where: $\sigma =$ standard deviation

$x =$ Data point (f/mm$^2$)

$\bar{x} =$ Average of data points (f/mm$^2$)

$n =$ Number of data points

**Coefficient of Variation** (also referred to as relative standard deviation $S_r$)

With the standard deviation calculated,

CV = $\frac{\sigma}{\bar{x}}$

**Paired Difference Analysis (for pass / fail):**

Each pair of replicate counts is compared using the paired difference quality test as follows:

$$|\sqrt{x_1} - \sqrt{x_2}| > 2.77 \times mean \times (0.5 \times CV)$$

Where:

$X_1$ = Original fiber count (f/mm$^2$)

$X_2$ = Replicate (f/mm$^2$)

Mean = Average of the square roots of the two fiber counts (f/mm$^2$)

CV = Coefficient of variation (as established for each analyst, each fiber range)

**Note:** This calculation represents the rejection/ acceptance criteria for replicate data.

*A.5.9.4.3.2 Control Charts*

The replicate data is plotted on a control chart as a graphic tool designed to monitor the analyst's precision performance. It graphs the

comparison with the difference of the square roots of the paired data with the acceptable limit calculated, using the quality test explained above. The program normalizes these numbers to 1 and uses the following control limits.

- 1 = UCL (upper control limit) - 0.65 = UWL (upper warning limit)
- -1 = LCL (lower control limit) - 0.65 = LWL (lower warning limit)

Data is generated and charted to monitor and measure both individual analysts and overall laboratory performance. The control charts allow for monitoring trends in the analytical processes, which may affect the quality of data. These control charts contribute to the criteria for determining validity of the data, and monitors any bias that an analyst and/or laboratory may be experiencing.

### *A.5.9.5 TEM Quality Control*

The following section describes the type and frequency of quality control analysis performed by the TEM laboratory. The frequency and type of quality control analyses are dictated by the analytical procedures and regulatory agencies.

Quality control data is tracked and managed in the EMSL quality control program.

The laboratory enters QC data into Excel worksheets, which then provides information including:

- Acceptance/rejection of replicate and duplicate data
- Performance trends
- Upper and lower control limits
- Acceptance/rejection of calibration measurements
- Record of QC and calibration measurements and frequencies
- Contamination events
- Corrective actions
- Control charts

#### A.5.9.5.1 Standards Analysis

SRM 1876b or equivalent must be analyzed once each year per analyst when applicable.

The laboratory and analysts must obtain mean analytical results on SRM 1876b (or equivalent) so that trimmed mean values fall within 80% of the lower limit, and 110% of the upper limit, as published on the certificate.

Where the SRM 1876b is unavailable (as of the date of this revision of the QMS Manual, NIST has chosen to discontinue this SRM), EMSL will use NVLAP or New York State ELAP past proficiency testing samples as the reference standard. The results are evaluated using the criteria established by the PT provider.

**Note2:** Standard analysis of NIST 1876 or substitute is not applicable for TEM Bulk (NOB) samples; they have their own standards detailed in Section A.5.9.6.4.3.

**A.5.9.5.2 AHERA, EPA Level I, II & III**

*A.5.9.5.2.1* *Intra-Analyst Same Grid Opening Re-analysis*

At least 2% (1/50 samples analyzed) of analyses are re-analyzed by the same analyst counting the same grid openings. QC samples are to be analyzed without prior knowledge of results where possible.

The measure of variance is calculated using the formula:
**R = |(A-B)/((A+B)/2)|**

Where:

R = the measure of variance for the analysis
A = the value of the first analysis in structures
B = the value of the second analysis in structures

Measures of variance (R) are recorded and plotted over time to determine trends and problems in analyses.

The Pass/Fail criteria for the QC analyses are as follows:

- < 5 structures = ± 1 structure
- 5-20 structures = ± 2 structures
- >20 structures = ± 3 structures

A failure based on the above criteria will result in a verified analysis in order to determine the cause of the problem. A cumulative record of False Positives, False Negatives, and True Positives based on the verified analysis are kept for each analyst.

*A.5.9.5.2.2* *Inter-Analyst Same Grid Opening Re-analysis*

At least 4% (1/25) of analyses are re-analyzed by a different analyst counting the same grid openings.

The measure of variance is calculated using the formula

**R = (A-B)/((A+B)/2)**

Where:

R = the measure of variance for the analysis
A = the value of the first analysis in structures
B = the value of the second analysis in structures

Measures of variance (R) are recorded and plotted over time to determine trends and problems in analysis.

The Pass/Fail criteria for the QC analyses are as follows:

- < 5 structures = ± 1 structure
- 5-20 structures = ± 2 structures
- 20 structures = ± 3 structures

A failure based on the above criteria will result in a verified analysis in order to determine the cause of the problem. A cumulative record of False Positives, False Negatives, and True Positives based on the verified analysis are kept for each analyst.

*A.5.9.5.2.3 Same Prep Different Grid Opening Analysis*

Sample re-analysis of different grid openings is performed to monitor and evaluate the method in regard to loading deposition uniformity on the filter. Re-analyses are analyzed at a frequency of 1 in 100 samples; QC analyses should be split relatively evenly between intra-and inter-analyst analyses.

Poissonian statistics are applied to re-analysis data. QC samples are to be analyzed without prior knowledge of results where possible. The analysis is accepted when:

$$|(A-B)| \leq 1.5 \times ((A+B)/2)^{1/2}$$

Where:

A = the value of the first analysis in structures
B = the value of the second analysis in structures

*A.5.9.5.2.4 Sample Re-Preparations*

Same sample re-preparations are performed to monitor any possible discrepancies that may occur in the implementation of the overall method. This includes sampling, preparation and analysis. Re-preparations are analyzed at a frequency of 1 in 100 samples and should be split relatively evenly between intra- and inter-analyst analyses.

Poissonian statistics are applied to re-preparation data. QC samples are to be analyzed without prior knowledge of results, where possible. The analysis is accepted when:

$$|(A-B)| \leq 2.0 \times ((A+B)/2)^{1/2}$$

Where:

A = the value of the first analysis in structures
B = the value of the second analysis in structures

*A.5.9.5.2.5 Verified Analysis*

At least 1/100-grid openings analyzed are re-analyzed by at least one other analyst using a verified analysis technique. Structures within a

grid opening are either sketched or plotted, and their locations are verified. 20% of the samples used for verified analysis must contain between 6-40 structures/grid opening (approximately 1,000 – 5,000 asbestos structures per $mm^2$), with the exception of verified analysis used to resolve discrepancies.

Analysts-in-training are required to perform a greater amount of verified analysis as seen fit by the laboratory manager and as dictated by the analyst's QC results.

*A.5.9.5.2.6 Laboratory Blank Sample Analysis*

Prep one blank per batch or 10% of total (whichever is greater), and after cleaning or servicing the preparation area. At minimum at least 1 blank must be prepped with each batch processed.

Analyze 1 per 100 filter analyses for MCE (mixed cellulose ester) filters, and 1 per 25 filter analyses for PC (polycarbonate) filters. Store all prepared grids (even if not analyzed) and record blank data on appropriate forms. Blank QC analysis is made part of the overall 10% quality assurance analysis.

For labs complying with the TNI (NELAC) standard and/or NYS ELAP, lab blanks must be analyzed at a rate of 1 per 20 samples analyzed. Criteria for the maximum allowable contamination levels for laboratory blanks are:

*MCE Filters*:

- Cumulative average of < 5 structures/$mm^2$ , AND
- A single preparation level of < 15 structures/$mm^2$

*PC Filters*:

- Cumulative average of 18 structures/$mm^2$ , AND
- Any single preparation level of 53 structures/$mm^2$



**A.5.9.5.3 ASTM Dust Microvac and Wipe Sample Analysis**

Quality control for this method specifies 10% of total analyses (excluding blanks) and requires following the QC procedures as recommended by NVLAP/NIST and ASTM. The re-analysis must be performed on second grid preparations from the final filter.

*A.5.9.5.3.1 Re-analysis*

At least 10% of analyses are re-analyzed on different grid openings of different preparations. QC samples are to be analyzed without prior knowledge of results, where possible.

The analysis is accepted when:

$$|(A-B)| \leq 2.0 \times ((A+B)/2)^{1/2}$$

Where:

A = the value of the first analysis in structures
B = the value of the second analysis in structures

For any failure of the above criteria, re-analysis is required in order to determine the cause of the problem.

*A.5.9.5.3.2 Process Blanks*

One (1) process blank is prepped and analyzed along with each sample set.

**A.5.9.5.4 TEM EPA NOB, NYS ELAP 198.4 & Chatfield VAT**

*A.5.9.5.4.1 Intra-Analyst Re-analysis*

At least 2% of analyses are re-analyzed by the same analyst. QC samples are to be analyzed without prior knowledge of results, where possible.

Measures of variance are calculated using the formula:

$$R = |(A-B)/((A+B)/2)|$$

Where:

R = the measure of variance for the analysis
A = the value of the first analysis in % asbestos
B = the value of the second analysis in % asbestos

Measures of variance are recorded and plotted over time to determine trends and problems in analyses.

The Pass/Fail criteria for the QC analyses are as follows:

- $R \leq 1$ – PASS
- $R > 1$ – FAIL
- Incorrect Asbestos ID – FAIL
- Asbestos missed during analysis (false negative) – FAIL
- Asbestos incorrectly identified to be present in a negative sample (false positive) – FAIL

A failure based on the above criteria will result in re-analysis to determine the cause of the problem. If this should fail to resolve the problem, the sample will be re-prepped (a complete gravimetric reduction) and again re-analyzed by the initial analyst.

R values are maintained, and R control charts updated monthly along with results for, and resolutions of failures.

*A.5.9.5.4.2 Inter-Analyst Re-analysis*

At least 7% of analyses are re-analyzed by a different analyst; the sample is re-prepped, including ashing and acid dissolution. QC samples are to be analyzed without prior knowledge of results, where possible.

Measures of variance are calculated using the formula:

**R = (A-B)/((A+B)/2)**

*Where:*

R = the measure of variance for the analysis
A = the value of the first analysis in % asbestos
B = the value of the second analysis in % asbestos

R-values are recorded and plotted over time to determine trends and problems in analyses.

The Pass/Fail criteria for the QC analyses are as follows:

- R > 1 or R< -1 – FAIL
- Incorrect Asbestos ID – FAIL
- Asbestos missed during analysis (false negative) – FAIL
- Asbestos incorrectly identified to be present in a negative sample (false positive) – FAIL

A failure based on the above criteria will result in re-analysis to determine the cause of the discrepancy. If this should fail to resolve the problem, the sample will be re-prepped (complete gravimetric reduction) and again re-analyzed by the initial analyst.

R-values are maintained and R control charts updated monthly, along with reasons for, and resolutions of failures.

*A.5.9.5.4.3 Standard Analysis*

At least 1% of analyses are prepared of reference standards or consensus standards. Results are charted to determine analyst as well as laboratory precision and accuracy.

*A.5.9.5.4.4 Blank Analysis*

At 5% (1/20) of sample volume, a known negative floor tile is prepped and analyzed for % weight recovery and contamination. If asbestos is detected, the source of contamination must be traced and problem resolved.

**A.5.9.5.5 EPA 100.2**

Quality control follows TNI guidelines:

- Total number of QA samples and blanks must be at least 10% of total

sample workload.

- There are no inherent inter- or intra-analyst QC requirements, but good practice would include both.

*A.5.9.5.5.1 Verified QC Analysis*

At least 5% of samples must be analyzed by the analysis of the same grid openings in a verified format, including sketches and verification of all discrepancies. Verified QC is evaluated by NISTIR 5351, and analysts must maintain at least ≥ 80% True Positives, ≤ 10% False Positives, and ≤ 20% False Negatives for cumulative verified analysis from analysis of verified samples and standards.

*A.5.9.5.5.2 Verified Standard Analysis*

Standard samples are analyzed at a rate of 1% of total analyses. Standard samples must be created from all six NIST (NIST 1866 & 1867) standard asbestos types, with final preparations containing 1-20 asbestos fibers (>10µm long) per grid opening.

Standards should be re-analyzed using verified analysis of the same grid openings, including sketches and verification of all discrepancies. Verified QC is evaluated by NISTIR 5351, and analysts must maintain at least ≥ 80% True Positives, ≤ 10% False Positives, and ≤ 20% False Negatives for cumulative verified analysis from analysis of verified samples and standards.

*A.5.9.5.5.3 Replicate QC*

At least 1% of samples should be re-analyzed by the analysis of different grid openings of the same preparation used for analysis.

The analysis is accepted when;

$$\mathbf{|(A-B)| \leq 1.5 \times ((A+B)/2)^{1/2}}$$

Where:

A = the value of the first analysis in structures
B = the value of the second analysis in structures

For any failure of the above criteria, re-analysis is required in order to determine the cause of the problem.

**Note:** Definition for Replicate QC above is specific to water methodologies.

*A.5.9.5.5.4 Duplicate QC*

At least 1% of samples should be re-analyzed by re-preparing the sample by re-filtration, re-preparation, and re-analysis of the same volume aliquot as used for the original analysis.

For the analysis of different preparations, the analysis is accepted when:

$$|(A-B)| \leq 2.0 \times ((A+B)/2)^{1/2}$$

Where:

A = the value of the first analysis in structures
B = the value of the second analysis in structures

For any failure of the above criteria, re-analysis is required in order to determine the cause of the problem.

**Note:** Definition for Duplicate QC above is specific to water methodologies.

*A.5.9.5.5.5 Blanks*

A process blank is filtered, prepared and analyzed before each batch of samples. The process blank must meet a contamination level of ≤ 0.01MFL for fibers > 10µm long.

### *A.5.9.6 PLM Quality Control*

#### A.5.9.6.1 Intra-Analyst Re-analysis – PLM

*A.5.9.6.1.1 Frequency*

At least 2% of analyses are re-prepared and re-analyzed by the same analyst. A full analysis is performed (re-prepared, optical properties recorded, etc.).

*A.5.9.6.1.2 Statistical Analysis*

Measures of variance are calculated using the formula

$$R = |(A-B)/((A+B)/2)|$$

Where :

R = the measure of variance for the analysis
A = the value of the first analysis in %
B = the value of the second analysis in %

Measures of variance are recorded and plotted over time to determine trends and problems in analyses.

The Pass/Fail criteria for the QC analyses are as follows:

- R ≤ 1 – PASS
- R > 1 – FAIL
- Incorrect Asbestos ID – FAIL

- Asbestos missed during analysis (false negative) – FAIL
- Asbestos incorrectly identified to be present in a negative sample (False positive) – FAIL

For any failure of the above criteria, the cause of the failure is identified and corrected.

**Inter-Analyst Re-analysis – PLM**

*A.5.9.6.1.3 Frequency*

At least 7% of analyses are re-analyzed by a different analyst; the sample is re-prepped from the original sample.

*A.5.9.6.1.4 Statistical Analysis*

Measures of variance are calculated using the formula

$$R = (A-B)/((A+B)/2)$$

Where:

R = the measure of variance for the analysis
A = the value of the first analysis in %
B = the value of the second analysis in %

R-values are recorded and plotted over time to determine trends and problems in analyses.

The Pass/Fail criteria for the QC analyses are as follows:

- $-1 \leq R \leq 1$ – PASS
- $R > 1$ and $R < -1$ – FAIL
- Incorrect Asbestos ID – FAIL
- Asbestos missed during analysis (false negative) – FAIL
- Asbestos incorrectly identified to be present in a negative sample (False positive) –FAIL

For any failure of the above criteria, the cause of the failure is identified and corrected.

**A.5.9.6.2 Reference Samples – PLM**

*A.5.9.6.2.1 Frequency*

Past proficiencies and known standards made with SRM 1866 and 1867 are analyzed. These standards are used to both calibrate and evaluate the performance of the analyst.

The standards are used to:

- Calibrate analyst visual estimation technique

- Verify the analyst's ability to correctly measure optical properties. Optical properties recorded must be within the acceptance criteria established by NIST.

At least 1% of analyses are to be quantitative standards repeatedly submitted and quantified.

*A.5.9.6.2.2 Statistical Analysis*

Results are quantified and charted to determine analyst, as well as laboratory precision and accuracy, using the following formula for percent recovery.

**%R = (A/F) x 100**

Where:

%R = percent recovery
A = the analytical result
F = the formulated standard weight

**A.5.9.6.3 EMSL PLM Consensus Program**

In addition to the EMSL standard quality control program, a separate quality analysis program is used which provides for an additional check on analyst performance.

Following the sampling strategies documented in the AHERA regulations, sample batches are generally submitted to our laboratories in sets collected from homogenous areas. Samples are typically collected in sets of 2, 3, 5 or 7 depending on the total area sampled. These sets should contain samples of identical type.

Collection of samples in this way helps to minimize the chance for false negative results whether the source is from sampling error, non-homogeneity or lab error.

The homogeneous makeup of these sets provides the lab with an opportunity to provide an extra layer of quality control. If the analysis of each homogenous group is split between two different analysts, a check on each analysis is achieved without any additional analytical effort.

See *EMSL PLM Consensus Analysis Program SOP* for additional information and procedure.

**A.5.9.6.4 Blanks**

For friable samples, at least one blank should be processed daily; this should entail the preparation of a known negative material (salt) to slide using all tools and oils to be used for analysis that day.

For NOB samples, a known negative floor tile is prepped and analyzed for % weight recovery and contamination.

For labs complying with the TNI (NELAC) Standard and NYS DOH ELAP 198.1, a similar check is made with every 20 uses of a piece of homogenization equipment.

For all blanks, if asbestos is detected, the source of contamination must be traced and problem resolved.

### *A.5.9.7 Proficiency Testing*

#### A.5.9.7.1 General

In addition to the policies and procedures mentioned in the general section of this manual;

For NVLAP PT rounds, all analysts (full and part-time) participate in proficiency testing rounds (all analysts need not participate prior to returning the results to the PT provider, but all analysts must participate without prior knowledge of the testing results at a later date, where applicable).

For all other PT rounds, where laboratories have multiple analysts, only one analyst is to analyze the sample for reporting to the PT provider. Once results have been scored, then all other analysts may analyze, but are not required to analyze the sample, and their results are compared to the official results from the PT provider. The samples are scheduled for analysis similar to routine client samples.

For all PT Rounds

- Analyses are not contracted out to another laboratory
- The laboratory keeps and uses proficiency testing materials as in-house instructional materials, unless otherwise directed.
- A single result is reported back to the PT provider by the laboratory, unless otherwise specified in the PT instructions.
- Procedures and calculations (if any) are documented as to how a single result is determined.
- Results from multiple analyses are not averaged.
- If analyzed by multiple analysts, test results are used for inter-analyst comparisons.
- Corrective actions are taken and documented for problems indicated by proficiency testing.
- Test results, when applicable, are used in determining accuracy and precision for participating analyst.
- When analyzed by multiple analysts, each analyst should analyze and record sample results independently as part of the lab's internal QC system.

**A.5.9.7.2 PCM Proficiency Testing**
EMSL laboratories will participate in proficiency testing programs, where accredited.

EMSL asbestos laboratories participate in the following programs at the discretion of the QA manager:

- AIHA-PAT Programs Industrial Hygiene Proficiency Analytical Testing program (IHPAT)
- Mandatory proficiency testing administered by state agencies as applicable
- AIHA Registry Programs Asbestos Analyst Registry (AAR)

Proficiency samples from these programs are to be run as normal laboratory samples, except where agency policy dictates additional requirements.

**A.5.9.7.3 TEM Proficiency Testing**
EMSL TEM asbestos laboratories participate in the following programs:

- Mandatory proficiency testing administered by the NYS ELAP
- The mandatory testing administered by the NIST (National Institute for Standards and Technology) National Voluntary Laboratory Approval Program (NVLAP)

Proficiency samples are to be run as normal laboratory samples, except where agency policy dictates additional requirements.

All analysts, including those analyzing in a NVLAP sub-facility, participate in the analysis of the NVLAP proficiency samples.

**A.5.9.7.4 PLM Proficiency Testing**
EMSL PLM asbestos laboratories participate in the following programs:

- Mandatory proficiency testing administered by the NYS ELAP
- The mandatory testing administered by the NIST (National Institute for Standards and Technology) National Voluntary Laboratory Approval Program (NVLAP)

Proficiency samples are to be run as normal laboratory samples, except where agency policy dictates additional requirements.

All analysts, including those analyzing in a NVLAP sub-facility, participate in the analysis of the NVLAP proficiency samples.

### *A.5.9.8 Round Robin Program*
In addition to the intra-laboratory QC, laboratories participate in an inter-lab exchange among other EMSL laboratories and select laboratories outside of the EMSL network. The

TEM, PLM and PCM programs are managed by the corporate Quality Assurance Department.

The EMSL asbestos Inter-laboratory program is documented in the *EMSL Inter-Laboratory Sample Exchange – Asbestos – SOP*.

The compiled results of the most recent inter-lab exchange shall remain posted in the laboratory for review by analysts.

### A.5.10 Reporting the Results

EMSL's LIMS reports are designed to meet method specific reporting requirements, as well as those applicable regulatory agencies such as AIHA-LAP, NVLAP, A2LA, CALA, TNI, and State Regulatory Agencies. EMSL has defined the requirements for referencing accreditation in reports, advertising and promotional materials in the EMSL Referencing Accreditation – Advertising Policy SOP (QA-SOP-310).

# A.6.0 Revision History

Previous revision histories are available from the QA Department on request.

| Revision | Date | Changes |
|---|---|---|
| 19.2 | 6/1/17 | Appendix A, Attachment 1, TEM Calibration Frequencies Semi-Annually: Added allowable usage of BIR1G Standards and changed requirement of 10 runs to multiple runs.<br>Section A.5.9.1: Specified the Monthly Report is submitted to MonthlyQA@emsl.com and pertinent comments from the review will be forwarded to the lab manager. |
| 19.1 | 3/16/17 | Section A.5.5.3.8 and TEM Calibration Frequency Daily: Changed the Al-Cu acceptance criteria from +/-0.2 keV to +/- 0.1 keV.<br>Section A.5.5.3.11: Removed requirement for +/-5% of target from muffle furnace temperature verification. |
| 19 | 12/16/16 | Removed revision histories for Revisions 15 through 18.<br>A.2.0: Updated dates for AIHA Policy Modules , NVLAP 150 Handbook, A2LA<br>A.4.5: For EMSL lab qualifications see EMSL website.<br>A.5.2.1.1: PCM training changed to include the addition of the NIOSH 582e program into all analysts' training.<br>A.5.2.1.4, A.5.2.1.5: Reorganized PCM ongoing training, Analyst Performance sections<br>A.5.2.2.2: Updated requirements for inter-analyst hands-on training<br>A.5.2.2.5: Revised wording to better explain TEM general and specific training<br>A.5.2.2.6, A.5.2.6.7: Reorganized TEM ongoing training and Analyst Performance section<br>A.5.2.3.6, A.5.2.3.6.7: Reorganized PLM ongoing training, Analyst Performance sections<br>A.5.2.4.5, 6 and 7: Reorganized sections to follow Module A format - PCM, TEM, PLM<br>A.5.5.2.2: Added mechanical counting aid calibration criteria<br>A.5.5.3.5: Clarified dimensions of fibril for beam dose<br>A.5.8.2, A.5.8.3: Added sample acceptance criteria<br>A.5.8.5: Added Non-New York NOB storage and disposal details<br>A.5.9.2: Clarified and reorganized trend analysis/monitoring<br>A.5.9.2: Reworded selection of QC (removing requirement for PLM)<br>A.5.9.4.2, A.5.9.4.3: Removed requirement reference slides should be from PAT rounds<br>A.5.9.4.3.1: Revised PCM QC calculation to read 2.77 (from 2.8)<br>A.5.9.5.2.2: Removed "single analyst" instructions from this section (addressed in Section A.5.9.3.1)<br>A.5.9.5.2.6: Added requirement in lab blank section and moved NYS ELAP requirement to NELAC TNI requirements<br>A.5.9.5.3: Revised requirement for dust microvac and wipe to comply with methods<br>A.5.9.5.4.4, A.5.9.6.4: Removed reference to 10% recovery on residue for NOB blanks<br>A.5.9.7.1: Clarified general policy on proficiency testing<br>A.5.10.1: Removed reference to use of NVLAP accreditation/logo (reference SOP)<br>Attachments QC- Frequency: Removed requirement for 10% residue recovery, revised PCM Phase Shift Detection slide resolution requirements, reworded to note PCM reference slide must pass prior to reading daily, changed TEM air ash to 5% removal |

# Module A
# Attachment 1: QC Frequencies

## POLARIZED LIGHT MICROSCOPY
## PHASE CONTRAST MICROSCOPY
## TRANSMISSION ELECTRON MICROSCOPY

# PLM CALIBRATION FREQUENCIES

## DAILY

**PLM SCOPE ALIGNMENT**
Record date, check off for rotation centering, condenser & objective alignment, axial illumination full extinction and crosshair alignment fixed in polarizer's privileged direction.

**CONTAMINATION CHECK**
Clean microscope and work area daily and all equipment after each use. Prep and analyze a fine grained reagent grade salt sample as a check for asbestos contamination.

## EACH DAY OF USE

**ANALYTICAL BALANCE VERIFICATION**
Analytical balance should be checked as per the *Balance Calibration Verification SOP*.

## MONTHLY

**AMOSITE DISPERSION COLORS**
Check Amosite Standard for proper dispersion color wavelengths. Record DS color wavelengths, date and the analyst performing the calibration.

## QUARTERLY

**RI OILS**
RI oil calibration to ± 0.004 using certified refractive index solids. Record date, nominal refractive index, measured refractive index, temperature and analyst's initials. All RI oils are calibrated when bottle is opened for first use. In addition, calibrate on next use if an oil has not been used in three (3) months.

**MUFFLE FURNACE TEMPERATURE CHECK**
Muffle furnace should be verified using three-point calibration covering 450, 485 and 520 degrees Celsius. Thermometer should be immersed in sand bath. Record date, target temperatures, measured temperatures, and analyst's initials.

**AIR MONITORING**
Ambient air samples should be collected (0.45 micron MCE cassettes, at least 1200 liters collected at not greater than 10 lpm) from each work area and analyzed by TEM using AHERA rules.

**HOOD CALIBRATION**
Measure and record flow rate of hoods with Anenometer as per the *EMSL Hood Maintenance and Calibration SOP*.

## ANNUALLY

**THERMOMETERS**
Thermometers used for measurement of ambient air temperature shall be verified annually as per § 5.6.2 of the main QMS Manual and the *EMSL Thermometer Calibration Verification SOP*.

**ANALYTICAL BALANCE**
The analytical balance shall be calibrated annually by an outside calibration firm accredited to ISO 17025.

**WORKING WEIGHTS**
Weights used for routine measurements in the lab shall be verified annually as per § 5.6.2 of the main QMS Manual and the *EMSL Working Weight Verification SOP*.

## 5 YEARS

**REFERENCE WEIGHTS**
Weights (used only for calibration of working weights) must be calibrated by an ISO 17025 accredited calibration service to NIST-traceable source every five (5) years as per § 5.6.2 of main QMS Manual.

# PLM FRIABLE QC ANALYSIS FREQUENCIES

**STANDARD REFERENCE SAMPLE ANALYSIS (Daily)**
One standard reference sample (known percentages) should be analyzed daily to determine precision and accuracy. Record date, analyst, type and percentage of asbestos present, as determined by calibrated visual estimation. A full analysis documented completely on a standard PLM bench sheet is required for each sample for each analyst.

In the event an analyst analyzes over 100 samples per day consistently, after the 100$^{th}$ sample, another reference sample must be analyzed for that day.

**INTER-ANALYST RE-ANALYSIS (1/15 Friable Samples and 1/15 Non Friable Samples)**
1/15 samples should be analyzed by different analyst (inter analyst) and R-values calculated R = {(A-B)/((A+B)/2)}. Analysis fails if R > 1 or < -1, misidentification of Asbestos occurs, or ACM vs. Non-ACM. A full analysis documented completely on a standard PLM bench sheet is required for each sample for each analyst.

**INTRA-ANALYST RE-ANALYSIS (1/50 Friable and 1/50 Non Friable Samples)**
1/50 samples should be re-analyzed by the same analyst (intra-analyst) and R-values calculated R = {(A-B)/((A+B)/2)}. Analysis fails if R > 1 or < -1, misidentification of Asbestos occurs, or ACM vs. Non-ACM. A full analysis documented completely on a standard PLM bench sheet is required for each sample for each analyst.

**ROUND ROBIN (2 times/year)**
All analysts should participate. Record dates of analysis, in-house analyst signatures, all results, and reasons for and resolution of disagreements.

A full analysis documented completely on a standard PLM bench sheet is required for each sample for each analyst.

# PLM NOB QC ANALYSIS FREQUENCIES

**STANDARD REFERENCE SAMPLE (1/100 Samples)**
At least 1 out of 100 samples shall be a verified quantitative standard that has been routinely resubmitted to determine analyst's precision and accuracy.

**INTER-ANALYST RE-ANALYSIS (1/15 Non-Friable Samples)**
1/15 samples should be analyzed by different analyst (inter analyst) and R-values calculated R = {(A-B)/(A+B)/2}. Analysis fails if R > 1 or < -1, misidentification of Asbestos occurs, or ACM vs. Non-ACM. A full gravimetric prep and analysis documented completely on a standard PLM bench sheet is required for each sample for each analyst.

**INTRA-ANALYST RE-ANALYSIS (1/50 Non-Friable Samples)**
1/50 samples should be reprepped and re-analyzed by the same analyst (intra-analyst) and R-values calculated R = {(A-B)/(A+B)/2}. Analysis fails if R > 1 or < -1, misidentification of Asbestos occurs, or ACM vs. Non-ACM. A full analysis documented completely on a standard PLM bench sheet is required for each sample for each analyst.

**NOB PREPARATION CHECK (1/20 NOB Samples)**

A known negative floor tile is routinely re-submitted. These samples must go through the full preparation and analysis regimen and then be analyzed for asbestos contamination and residue recovery (% wt.). If asbestos is detected, the source of contamination must be traced and problem resolved to prevent recurrence.

**ROUND ROBIN (2 times/year)**
All analysts should participate. Record dates of analysis, in-house analyst signatures, all results, and reasons for and resolution of disagreements.

A full analysis documented completely on a standard PLM bench sheet is required for each sample for each analyst.

# PCM CALIBRATION FREQUENCIES

## DAILY

**ALIGNMENT**
Check alignment (phase rings) and illumination. Record date and analyst's initials.

**CONTAMINATION CONTROL**
Clean microscope and work area daily and all equipment after each use.

**MICROSCOPE FIELD AREA MEASUREMENT** (For TNI/NELAC labs)
Once per day, each analyst using the scope shall check the diameter of the Walton-Beckett Graticule. Measurement must equal 100 microns. Record diameter, PASS / FAIL, corrective action if necessary, date and the analyst's initials.

Other labs shall complete this monthly.

## WEEKLY

**PHASE SHIFT DETECTION**

The HSE / NPL Slide contains seven (7) blocks of grooves. Block 3 must be visible. Blocks 4 and 5 must be partially or completely visible. The last two sets of lines (6-7) should be invisible. Record the highest block visible, PASS / FAIL, corrective action if necessary, date and the analyst's initials.

For HSE/UPO Slides, if used:

HSE/ULO Red - The microscope should be able to completely resolve the first 4 sets of lines (1-4); the next set of lines (5) should be partially visible. The microscope should not be able to see the last two sets (6-7) of lines at all; they should be invisible.

HSE/ULO Green - The microscope should be able to completely resolve the first five (5) sets of lines (1-5); the next set of lines (6) should be partially visible. The microscope should not be able to see the last set (7) of lines at all; they should be invisible.



## MONTHLY

**MICROSCOPE FIELD AREA MEASUREMENT**
Check the diameter of the Walton-Beckett Graticule. It must equal 100 microns. Record diameter, PASS / FAIL, corrective action if necessary, date and the analyst's initials.

For Labs complying w/ TNI (NELAC) Standard, this should be increased to each time a different analyst uses the scope.

**MECHANICAL COUNTER**
Mechanical counter accuracy is documented by counting to 100 while clicking the counter with each count. The clicker must read 100 on the 100th count. Record date, PASS/FAIL and analyst's initials.

## QUARTERLY

**AIR MONITORING**

Ambient air samples should be collected from each work area. The samples should be collected on 0.45 micron MCE filters (at least 1200 liters collected at no greater than 10 lpm) and analyzed by TEM using AHERA rules.

**HOOD CALIBRATION**

Measure and record flow rate of PCM hoods with anemometer as per the *EMSL Hood Maintenance and Calibration SOP*. Record date, flow rate and the analyst's initials. See SOP for acceptance criteria.

## PRIOR TO FIRST USE

**STAGE MICROMETER**

The stage micrometer in use at laboratory should be calibrated to ISO 17025 standards initially prior to first use, and if damaged, as per § 5.6.2 of the main QMS Manual.

# PCM QC ANALYSIS FREQUENCIES

**DAILY REFERENCE SLIDE**
Each analyst must successfully analyze at least one Reference Slide daily (taken from low, medium and high fiber count pool randomly) prior to reading samples, in order to generate a CV for each analyst and for the lab. Record fiber counts and date for each analyst.

**INTRA-ANALYST RE-ANALYSIS (1/10 Samples)**
Perform blind intra-analyst recounts on 10% of filters counted. Record analyst, date, Reference #, Sample #, initial and QC fiber counts, density, PASS / FAIL, and comments / corrective actions.

**ROUND ROBIN (2 times/year)**
All analysts should participate. Record dates of analysis, in-house analyst signatures, all results, and reasons for, and resolution of disagreements.

A full analysis documented completely on a standard PCM bench sheet is required for each sample for each analyst.

Samples are chosen from previously analyzed customer samples or past proficiency testing samples.

# TEM CALIBRATION FREQUENCIES

## DAILY

**Al, Cu CALIBRATION**
Collect spectrum on Al, Cu grid. Peaks should be centered at 1.48 and 8.04, both to within ± 0.01 keV. If not, then calibration of the instrument is necessary. Record date, analyst and results. It is strongly recommended to save spectrum on the hard drive.

**MICROSCOPE ALIGNMENT**
Check off after each step. Record date and analyst's initials.

**CONTAMINATION CONTROL**
Clean work area and equipment after each sample.

**SAED (as needed)**
Index diffraction patterns to verify a cumulative 80% accuracy rating for each analyst.

## EACH DAY OF USE

**ANALYTICAL BALANCE**
Analytical balance should be checked in all applicable ranges as per § 5.5.3.1 of main QMS Manual and *EMSL Balance Calibration Verification SOP*. See SOP for acceptance criteria.

## WEEKLY

**CAMERA CONSTANT**
Record camera length and camera constant as per the *TEM CAL Worksheet*. Produce negative, measurement results from photo and screen (+/- 5% change acceptable), and screen diameter that corresponds to 5.3 Angstroms.

**Note:** If sufficient data has been collected which indicates confidence in the stability of measurements, frequency of camera constant calibration can be adjusted to monthly.

**Note:** For those instruments utilized for water calibration, frequency must be maintained at a weekly rate during the period of time analysis is performed.

## MONTHLY

**MAGNIFICATION CALIBRATION**
Calibration of screen and negative (± 5% change acceptable). Produce negative, calculations and results and conversion factors for the small and large circles on the phosphor screen.

## QUARTERLY

**BEAM DOSE**
ED patterns of 9 out of 10 NIST SRM chrysotile fibrils (max. diameter 0.05 microns) must remain visible for 15 seconds. Record one fibril image and one photo of ED pattern.

**ASHER CALIBRATION**
Determine ash time to etch 5% of collapsed filter. Chart ash time versus date.

**SPOT SIZE MEASUREMENT**
Crossovers at spot size 3; take picture and record actual diameter.

**Na SENSITIVITY**
Resolvable (statistically significant) Na K alpha peaks from NIST SRM 1866 crocidolite. Produce calculations, dated and signed spectra AND photo of fibril. It is strongly recommended to save spectrum on the hard drive.

**CHRYSOTILE FIBRIL SENSITIVITY**
Check for resolvable Mg and Si peaks from single fibril from either NIST SRM 1866 or 1876b chrysotile. Produce dated and signed spectra. It is strongly recommended to save spectrum on the hard drive.

**AIR MONITORING**
Ambient air samples should be collected from each work area on 0.45 micron MCE filters and analyzed by TEM AHERA.

**X-RAY DETECTOR RESOLUTION** (For TNI/NELAP labs)
FWHM of Mn peak: Collect 2000 FS counts in the Mn peak (or more, but be consistent each time). Resolution must be <175 eV. Produce dated and signed spectra. It is strongly recommended to save spectrum on the hard drive.

**Note:** For other labs, this must be done semi-annually.



## SEMI - ANNUALLY

**X-RAY DETECTOR RESOLUTION**:
FWHM of Mn peak: Collect 2000 FS counts in the Mn peak (or more, but be consistent each time). Resolution must be <175 eV. Produce dated and signed spectra. It is strongly recommended to save spectrum on the hard drive.

**Note:** For labs complying with the TNI (NELAC) Standard, this frequency must be quarterly.

**K FACTORS:**
USING Various Standards (SRM 2063 , Albite and/or BIR1G): Collect 10,000 integral or 2000 FS counts in the Si peak (or more, but be consistent each time). Multiple spectra are acquired and various sensitivity factors are calculated, each with their own PASS/FAIL criteria. Produce dated & signed hard copy of the spectra, or optionally, it is strongly recommended to save the spectrum as a digital file.

**GRID OPENING AREA**: Twenty grid opening areas from twenty grids (400 total) must be calculated for **each lot of 1,000** grids. Record date, lot number, analyst and average GSO.

## ANNUALLY

**ANALYTICAL BALANCE**
The analytical balance shall be calibrated by an external calibration service accredited to ISO 17025 annually as per § 5.5.3 of the main QMS Manual and *EMSL Balance Calibration Verification SOP*.

**WORKING WEIGHTS**
Weights used for routine measurements in the lab shall be verified annually as per § 5.6.2 of the main QMS Manual and the *EMSL Working Weight Verification SOP*.

## 5 YEARS

**REFERENCE WEIGHTS**
Weights (used only for calibration of working weights) must be calibrated by an ISO 17025 accredited calibration service to NIST-traceable source every 5 years as per § 5.6.2 of main QMS Manual.

# TEM AIR QC ANALYSIS FREQUENCIES

**INTRA-ANALYST SAME GRID OPENING REANALYSES (1/50 Air Samples)**
This re-analysis used to determine the analyst's precision. Calculate R values where R=[(A-B)/(A+B)/2]. Chart R values and PASS/FAIL results for each analyst and for lab in the following four ranges of asbestos structures only:

| RANGE of MEAN of RECOUNT | PASS/FAIL CRITERIA |
|---|---|
| <5 Structures | ±1 Structure |
| 5-20 Structures | ±2 Structures |
| >20 Structures | ±3 Structures |

Verified analysis is required to resolve failures. Record sample, grid(s) and grid opening(s) analyzed date(s) of analyses, analyst's signature, both results, R-value reasons for and resolutions of disagreements. A cumulative record of true positives, false positives, and false negatives is maintained for each analyst.

**INTER-ANALYST SAME GRID OPENING REANALYSES (1/25 AIR Samples)**
This re-analysis of the same grid openings is used to determine both the laboratory's overall precision and to detect bias of the various analysts. Calculate R values where R=[(A-B)/(A+B)/2]. Chart R values and PASS/FAIL results for each analyst and for lab in the following four ranges of asbestos structures only:

| RANGE of MEAN of RECOUNT | PASS/FAIL CRITERIA |
|---|---|
| <5 Structures | ±1 Structure |
| 5-20 Structures | ±2 Structures |
| >20 Structures | ±3 Structures |

For any failures, verified analysis is required. Record sample, grid(s) and grid openings) analyzed date(s) of analysis, signatures, both results, R-value, reason(s) for analysts and resolution(s) of disagreement(s). A cumulative record of true positives, false positive and false negatives is maintained for each analyst.

**SAME GRID/DIFFERENT OPENING RE-ANALYSIS (1/100)**
Inter-analyst analysis – 0.5 in 100 samples
Intra-analyst analysis – 0.5 in 100 samples

**SAME SAMPLE REPREPARATIONS (1/100)**
Inter-analyst analysis – 0.5 in 100 samples
Intra-analyst analysis – 0.5 in 100 samples

**VERIFIED ANALYSES (1/100 Grid Openings Analyzed)**

20% of the samples used must contain between 6-40 structures/grid opening (approximately 1,000-5,000 asbestos structures/$mm^2$. Previous customer samples, NYS ELAP proficiency

samples, NVLAP proficiency samples and NIST SRM 1876b samples may be used). Record results from each analyst, date(s) of analysis, acceptability (within appropriate guidelines), reason(s) for and resolution(s) of disagreements, analyst's signature(s). A cumulative record of true positives, false positives and false negatives is maintained for each analyst. Maintain a separate record for NIST SRM 1876b or equivalent analysis.

**INTER-LAB ROUND ROBIN ANALYSES (1/200 G.O.) (2 times/year)**

Use asbestos samples that cover a range from less than 100 to more than 2,000 structures/sq. mm. This re-analysis of the same grid opening must be by VERIFIED ANALYSIS. Record sample, grid and grid opening identification, date(s) of analysis, in-house analyst's signature, all results reasons for and resolutions of disagreements. Analysts must attain an average accuracy of $\geq$ 80% True Positives, $\leq$ 20% False Negatives, and $\leq$10% False Positives to maintain verified status.

**LABORATORY BLANK (AIR Samples)**

Prep 1 blank per series or 10% of daily total; Analyze 1 per 100 filter analyses for MCE (mixed cellulose ester) filters, and 1 per 25 filter analyses for PC (polycarbonate) filters. For those laboratories meeting TNI or NYS ELAP requirements, analysis frequency is 5%.

# TEM NOB QC ANALYSIS FREQUENCIES

**INTRA ANALYST - NEW PREP (1/50 NOB Samples Re-analyzed by Same Analyst)**
This re-analysis is used to determine the analyst's precision and check the laboratory's preparation technique. Calculate R-values for each pair. R= |(A-B)/[(A+B)/2]|. Analysis fails if R >1, misidentification of Asbestos occurs, or ACM vs. Non-ACM. Record sample, dates of analyses, analyst's initials, both results, R-values, causes and corrective actions for failures. R control charts updated monthly for each analyst's precision. Also check recovery (% Wt.) of both sample preparations. Failure requires a third prep be made (all steps checked by lab supervisor) to determine correct recovery. Record dates of all preparations, weights, technician initials, and reasons for and resolutions of failures.

**INTER ANALYST - NEW PREP (1/15 NOB Samples Re-analyzed by Different Analyst)**
This re-analysis is used to determine laboratory precision and to check the laboratory's preparation technique. Calculate R-values for each pair of analyses. R= (A-B)/[(A+B)/2]. Analysis fails if R >1 or < -1, misidentification of Asbestos occurs, of ACM vs. Non-ACM. Record sample, dates of analysis, analyst's initials, both results, R-values, reasons for and resolutions for failures. R control charts updated for each analyst's precision.

Also check residue recovery (% Wt.) of both sample preparations. In case of failure, request a third prep be made (all steps checked by lab supervisor) to determine correct recovery. Record dates of all preparations, weights, technician initials, and reasons for and resolutions of failures.

**INTER-LAB ANALYSIS (2 times/year) ROUND ROBIN**
Samples analyzed for asbestos contents and residue recovery (% Wt.). Record asbestos type(s), percentage(s), analyst(s), and dates of analyses. Track misclassifications (false positive, false negative) and misidentification of asbestos types and residue recovery (% Wt.).

**NOB PREPARATION CHECK (1/20 NOB Samples)**
A known negative floor tile sample is prepped and analyzed for asbestos contamination and residue recovery (%Wt.). These samples must go through the full preparation and analysis regimen. If asbestos is detected, the source of contamination must be traced and problem resolved to prevent recurrence.

**STANDARD REFERENCE SAMPLE (1/100 Samples)**
At least 1 out of 100 samples shall be a verified quantitative standard that has been routinely resubmitted to determine analyst's precision and accuracy.

# Module A

# Attachment 2: Additional Qualification Requirements

## Additional Qualification Requirements by Accrediting Authorities for ASBESTOS

| Accred. Agency | Lab Manager | | QA Manager | | Analyst | |
|---|---|---|---|---|---|---|
| | Required Degree | Required Lab Exp | Required Degree | Required Lab Exp | Required Degree | Required Lab Exp |
| AIHA-LAP | **(Tech. Manager)**<br>B.S. – applicable physical or biological science | **(Tech. Manager)**<br>- 3 years of relevant non-academic analytical experience. A minimum of 2 years in IH within the Scope of Accreditation and 1 year from other lab analytical procedures.<br>- M.S. or Ph.D. is equivalent to 1 year of work experience. | **(Quality Manager)**<br>B.S. – applicable basic or applied science | **(Quality Manager)**<br>1 year of non-academic analytical or QC experience appropriate to types of analysis performed. Or in lieu of a degree – 4 years of non-academic analytical or QC experience.<br>- Documented training in statistics or laboratory quality assurance/quality control. | **(Analyst)**<br>B.S. in chemistry or related science<br>**(Technician)**<br>No degree is necessary | - Both Analysts and Technicians shall complete a training course (in-house is acceptable) for applicable analysis.<br>- Both Analysts and Technicians need to demonstrate capability through SRMs, PT or QC samples. Re-certification every 6 months<br>- Both Analysts and Technicians shall have 20 business days of hands-on experience before independent analysis on customer samples. |
| NYS ELAP<br><br>TNI standard (July 2011) | **(Tech. Director) TEM**<br>B.S. degree and specialized courses in use of instrument<br>**PLM**<br>A.S. degree and specialized courses in use of instrument or 2 years of equiv. college study or formal coursework<br>**PCM**<br>A.S. degree or 2 years of equiv. college study or formal coursework | **(Tech. Director) TEM**<br>1 year experience under supervision in use of instrument<br>**PLM**<br>1 year experience under supervision in use of instrument<br>**PCM**<br>1 year experience under supervision in use of instrument | | | | |
| CA ELAP | **(Lab Director)**<br>B.S. degree in applicable science | **(Lab Director)**<br>3 years experience<br>M.S. substituted for 1 year of exp.<br>Ph.D. substituted for 2 years of exp. | **(Principal Analyst)**<br>B.S. – applicable basic or applied science | **(Principal Analyst)**<br>6 months experience in analysis.<br>Completion of training course | | |

| Accred. Agency | Lab Manager | | QA Manager | | Analyst | |
|---|---|---|---|---|---|---|
| | Required Degree | Required Lab Exp | Required Degree | Required Lab Exp | Required Degree | Required Lab Exp |
| FL DOH<br><br>TNI standard (July 2011) | **(Tech. Director) TEM**<br>B.S. degree and specialized courses in use of instrument<br>**PLM**<br>A.S. degree and specialized courses in use of instrument or 2 years of equiv. college study or formal coursework<br>**PCM**<br>A.S. degree or 2 years of equiv. college study or formal coursework | **(Tech. Director) TEM**<br>1 year experience under supervision in use of instrument<br>**PLM**<br>1 year experience under supervision in use of instrument<br>**PCM**<br>1 year experience under supervision in use of instrument | | | | |
| NJ DEP | **(Lab Manager)**<br>B.S. degree or<br>A.A. degree or<br>No degree | **(Lab Manager)**<br>B.S. – 1 year experience<br>A.A. – 3 years experience<br>None – 5 years experience | **(QA Officer)**<br>B.S. degree or<br>A.A. degree or<br>No degree | **QA Officer)**<br>B.S. – 1 year experience<br>A.A. – 3 years experience<br>None – 5 years experience | **(Operators)**<br>B.S. degree or<br>A.A. degree or<br>No degree | **(Operators)**<br>B.S. – 1 year experience<br>A.A. – 3 years experience<br>None – 5 years experience<br>**TEM**<br>Completion of formal training course |
| Louisiana LADEQ | **(Lab Tech Director)**<br>B.S. degree in science or 4 years equiv. experience | **(Lab Tech Director)**<br>Minimum of 2 years in environmental analysis. | **(QA Manager)**<br>B.S. degree in science or 4 years equiv. experience | **(QA Manager)**<br>Minimum of 2 years in environmental analysis. | **(Supervisors)**<br>B.S. degree or 4 years experience<br>**(Instrument Operators)**<br>H.S. diploma and completion of in-house training course | **(Supervisors)**<br>Minimum of 1 year experience<br>**(Instrument Operators)**<br>6 months experience and passing PT results. |
| Pennsylvania DEP<br><br>TNI Standard (July 2011) | **(Lab Supervisor) TEM**<br>B.S. degree and specialized courses in use of instrument<br>**PLM**<br>A.S. degree and specialized courses in use of instrument or 2 years of equiv. college study or formal coursework<br>**PCM**<br>A.S. degree or 2 years of equiv. college study or formal coursework | **(Lab Supervisor) TEM**<br>1 year experience under supervision in use of instrument<br>**PLM**<br>1 year experience under supervision in use of instrument<br>**PCM**<br>1 year experience under supervision in use of instrument | | | | |

| Accred. Agency | Lab Manager | | QA Manager | | Analyst | |
|---|---|---|---|---|---|---|
| | Required Degree | Required Lab Exp | Required Degree | Required Lab Exp | Required Degree | Required Lab Exp |
| Texas<br>TCEQ<br><br>TNI standard<br>(July 2011) | **(Tech. Director) TEM**<br>B.S. degree and specialized courses in use of instrument<br>**PLM**<br>A.S. degree and specialized courses in use of instrument or 2 years of equiv. college study or formal coursework<br>**PCM**<br>A.S. degree or 2 years of equiv. college study or formal coursework | **(Tech. Director) TEM**<br>1 year experience under supervision in use of instrument<br>**PLM**<br>1 year experience under supervision in use of instrument<br>**PCM**<br>1 year experience under supervision in use of instrument | | | | |

# Quality Assurance Manual Cover Page

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# Title Page:

# Quality Assurance Manual

# Approval Signatures

Laboratory Director – Crystal Pollock — Date: 5/19/2017

Quality Manager – Lisa Stafford — Date: May 18, 2017

Technical Manager, Dioxins, LCMS, Inorganics
Robert Hrabak — Date: 5/18/17

Manager, Volatiles, Semivolatiles, Organic/Dioxin Prep
Koroush Vaziri — Date: 5/18/17

## SECTION 2. TABLE OF CONTENTS

# LIST OF TABLES



# LIST OF FIGURES

# LIST OF APPENDICES



# REFERENCED CORPORATE SOPs AND POLICIES

| SOP / Policy Reference | Title |
|---|---|
| CA-I-P-002 | Electronic Reporting and Signature Policy |
| CA-I-S-006 | Software Testing, Validation and Verification |
| CW-I-P-006 | Computer Systems Account and Naming Policy |
| CW-I-P-007 | Computer Systems Password Policy |
| CA-L-P-002 | Contract Compliance Policy |
| CA-Q-M-002 | Corporate Quality Management Plan |
| CA-Q-S-001 | Solvent and Acid Lot Testing and Approval |
| CA-Q-S-002 | Acceptable Manual Integration Practices |
| CA-Q-S-006 | Detection Limits |
| CA-Q-S-009 | Root Cause Analysis |
| CA-T-P-001 | Qualified Products List |
| CW-E-M-001 | Corporate Environmental Health & Safety Manual |
| CW-F-P-002 | Company-Wide Authorization Matrix |
| CW-F-P-004 | Procurement and Contracts Policy |
| CW-F-S-007 | Capital Expenditure, Controlled Purchase Requests and Fixed Asset Capitalization |
| CW-L-P-004 | Ethics Policy |
| CW-L-S-002 | Internal Investigation s |
| CW-L-S-004 | Subcontracting |
| CW-Q-S-001 | Corporate Document Control and Archiving |
| CW-Q-S-002 | Writing a Standard Operating Procedure (SOPs) |
| CW-Q-S-003 | Internal Auditing |
| CW-Q-S-004 | Management Systems Review |
| CW-Q-S-005 | Data Recalls |
| CA-C-S-001 | Work Sharing Process |

# REFERENCED LABORATORY SOPs

| SOP Reference | Title |
|---|---|
| WS-PQA-013 | Procedures to Address Customer Complaints |
| WS-QA-0050 | Management of Change |
| WS-QA-0009 | Document Archiving |
| WS-QA-0022 | Employee Orientation and Training |
| WS-QA-0021 | Preparation and Management of Standard Operating Procedures |
| WS-QA-0006 | Method Detection Limits (MDL) and Instrument Detection Limits (IDL) |
| WS-PQA-0011 | Manual Integration Documentation Procedures |
| WS-PQA-003 | Quality Control Program |
| WS-QA-0018 | Subsampling and Compositing of Samples |
| WS-QA-0003 | Sample Receipt and Procedures |
| WS-QA-0035 | Statistical Process Control / Control Chart |
| WS-EHS-0001 | Waste Disposal |

# SECTION 3. INTRODUCTION, SCOPE AND APPLICABILITY

## 3.1 Introduction and Compliance References

TestAmerica Sacramento'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, 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.

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.*
- *U.S. Department of Defense, Quality Systems Manual for Environmental Laboratories, Version 4.2, October 2010.*
- *U.S. Department of Denfense (DoD)/Department of Energy (DOE) Consolidated Quality Systems Manual (QSM) for Environmental Laboratories, Version 5.0, July 2013.*
- *U.S. Department of Denfense (DoD)/Department of Energy (DOE) Consolidated Quality Systems Manual (QSM) for Environmental Laboratories, Version 5.1, 2017.*
- Federal Register, 40 CFR Parts 136, 141, 172, 173, 178, 179 and 261.
- *Manual for the Certification of Laboratories Analyzing Drinking Water (EPA 815-R-05-004, January 2005)*
- *Statement of Work for Inorganics & Organics Analysis, SOM, ISM, DLF and CBC, current versions, USEPA Contract Laboratory Program Multi-media, Multi-concentration.*
- APHA, *Standard Methods for the Examination of Water and Wastewater,* 18th Edition, 19th, 20th, 21st, and on-line Editions.
- *U.S. Department of Energy Order 414.1B, Quality Assurance, Approved April 29, 2004.*
- *U.S. Department of Energy Order 414.1C, Quality Assurance, June 17, 2005.*

- *U.S. Department of Energy Order 414.1D, Quality Assurance, April 25, 2011*
- Nuclear Regulatory Commission (NRC) Quality Assurance Requirements.
- Marine Protection, Research, and Sanctuaries Act (MPRSA).
- Toxic Substances Control Act (TSCA).

## 3.2 Terms and Definitions

A Quality Assurance Program is a company-wide system designed to ensure that data produced by 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, tissue and soils. The Quality Assurance Program contains specific procedures and methods to test samples of differing matrices for chemical, physical and biological parameters. The Program also contains guidelines on maintaining documentation of analytical 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 and the Quality Assurance (QA) Manager. In some cases, QAPPs and DQOs may specify less stringent requirements. The Laboratory Director and the QA Manager must determine if it is in the lab's best interest to follow the less stringent requirements.

## 3.4 Management of the Manual

### 3.4.1 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 itself is reviewed annually by senior laboratory management to assure that it reflects current practices and meets the requirements of the laboratory's clients and regulators as well as the CQMP. Occasionally, the manual may need changes in order to meet new or changing regulations and operations. The QA Manager will review the changes in the normal course of business and incorporate changes into revised sections of the document. All updates will be reviewed by the senior laboratory management staff. The laboratory updates and approves such changes according to our Document Control & Updating procedures (refer to SOP No. WS-QA-0021).

# SECTION 4. MANAGEMENT REQUIREMENTS

## 4.1 Overview

TestAmerica Sacramento 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., Chief Executive Officer, Executive VP Operations, Corporate Quality, etc.). The laboratory operational and support staff work under the direction of the Laboratory Director. The organizational structure for both Corporate & TestAmerica Sacramento 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 CQMP. This manual is specific to the operations of TestAmerica's Sacramento laboratory.

### 4.2.2 President and Chief Executive Officer (CEO)

The President and CEO is a member of the Board of Directors and is ultimately responsible for the quality and performance of all TestAmerica facilities. The President and CEO establishes the overall quality standard and data integrity program for the Analytical Business, providing the necessary leadership and resources to assure that the standard and integrity program are met.

### 4.2.3 Senior Vice President of Operations (SVPO)

The COO reports directly to the President and CEO of TestAmerica. The SVPO~~VPO~~ oversees the operations of all TestAmerica. The VP's of Operations report directly to the SVPO.

### 4.2.4 Vice President of Operations

Each VP of Operations reports directly to the Senior VP of Operations and is a part of the Executive Committee. Each VP of Operations is responsible for the overall administrative and operational management of their respective laboratories. The VP's responsibilities include allocation of personnel and resources, long-term planning, goal setting, and achieving the financial, business, and quality objectives of TestAmerica. The VP's ensure timely compliance with Corporate Management directives, policies, and management systems reviews. The VP's are also responsible for restricting any laboratory from performing analyses that cannot be consistently and successfully performed to meet the standards set forth in this manual.

### 4.2.5 Vice President of Quality and Environmental Health and Safety (VP-QA/EHS)

The Vice President (VP) of QA/EHS reports directly to the President and CEO. With the aid of the Executive Committee, Laboratory Directors, Quality Directors, Safety Manager, EH&S Coordinators and QA Managers, the VP-QA/EHS has the responsibility for the establishment, general overview and Corporate maintenance of the Quality Assurance and EH&S Programs within TestAmerica. Additional responsibilities include:

- Review of QA/QC and EHS aspects of Corporate SOPs & Policies, national projects and expansions or changes in services.
- Work with various organizations outside of TestAmerica to further the development of quality standards and represent TestAmerica at various trade meetings.
- Preparation of a monthly report that includes quality metrics across the analytical laboratories and a summary of any quality related initiatives and issues.
- Preparation of a monthly report that includes EH&S metrics across the analytical laboratories and a summary of any EH&S related initiatives and issues.
- Work with various organizations outside of TestAmerica to further the development of quality standards and represent TestAmerica at various trade meetings.
- With the assistance of the Corporate Senior Management Teams and the EHS Directors, development and implementation of the TestAmerica Environmental, Health and Safety Program.

### 4.2.6 Vice President of Client Service

The VP of Client Services leads the Client Service Organization (CSO) and is responsible for client satisfaction, driving operational excellence and improving client responsiveness. The VP provides direction to the Client Service Directors, Programs Managers and Project Managers.

### 4.2.7 Quality Assessment Director

The Quality Assessment Director reports to the VP-QA/EHS. The Quality Assessment Director has QA oversight of laboratories; responsible for the internal audit system, schedule and procedure; monitors laboratory internal audit findings; identifies common laboratory weaknesses; and monitors corrective action closures. Together with the Quality Compliance Director, the Quality Systems Director, and the VP-QA/EHS, the Quality Assessment Director has the responsibility for the establishment, general overview and maintenance of the Analytical Quality Assurance Program within TestAmerica.

### 4.2.8 Quality Compliance Director

The Quality Compliance Director reports to the VP-QA/EHS. The Quality Compliance Director has QA oversight of laboratories; monitors and communicates DoD / DoE requirements; develops corporate tools for ensuring and improving compliance; develops corporate assessment tools; identifies common laboratory weaknesses; and monitors corrective action closures. Together with the Quality Assessment Director, Quality Systems Director and the VP-QA/EHS, the Quality Compliance Director has the responsibility for the establishment, general overview and maintenance of the Analytical Quality Assurance Program within TestAmerica.

### 4.2.9 Quality Systems Director

The Quality Systems Director reports to the VP-QA/EHS. The Quality Systems Director has QA oversight of laboratories; develops quality policies, procedures and management tools; monitors and communicates regulatory and certification requirements; identifies common laboratory weaknesses; and monitors corrective action closures. Together with the Quality Assessment Director, Quality Compliance Director and the VP-QA/EHS, the Quality Systems Director has the responsibility for the establishment, general overview and maintenance of the Analytical Quality Assurance Program within TestAmerica.

### 4.2.10 Quality Information Manager

The Quality Information Manager is responsible for managing all company official documents (e.g., Policies, Procedures, Work Instructions), the company's accreditation database, intranet websites, external laboratory subcontracting, regulatory limits for clients on the company's TotalAccess website; internal and external client support for various company groups (e.g., Client Services, EH&S, Legal, IT, Sales) for both quality and operational functions. The Quality Information Manager reports to the VP-QA/EHS; and works alongside the Quality Assessment, Quality Compliance and Quality System Directors and EHS Managers to support both the Analytical Quality Assurance and EHS Programs within TestAmerica.

### 4.2.11 Technical Services Director

The Technical Services Director is responsible for establishing, implementing and communicating TestAmerica's Analytical Business's Technical Policies, SOPs, and Manuals. Other responsibilities include conducting technical assessments as required, acting as a technical resource in national contracts review, coordinating new technologies, establishing best practices, advising staff on technology advances, innovations, and applications.

### 4.2.12 Ethics and Compliance Officers (ECOs)

TestAmerica has designated two senior members of the Corporate staff to fulfill the role of Ethics and Compliance Officer (ECO) – Corporate Counsel & VP of Human Resources and the VP-QA/EHS. Each ECO acts as a back-up to the other ECO and both are involved when data investigations occur. Each ECO has a direct line of communication to the entire senior Corporate and lab management staff.

The ECOs ensure that the organization distributes the data integrity and ethical practices policies to all employees and ensures annual trainings and orientation of new hires to the ethics program and its policies. The ECO is responsible for establishing a mechanism to foster employee reporting of incidents of illegal, unethical, or improper practices in a safe and confidential environment.

The ECOs monitor and audit procedures to determine compliance with policies and to make recommendations for policy enhancements to the President and CEO, VPOs, Laboratory Director or other appropriate individuals within the laboratory. The ECO will assist the laboratory QA Manager in the coordination of internal auditing of ethical policy related activities and processes within the laboratory, in conjunction with the laboratories regular internal auditing function.

The ECOs will also participate in investigations of alleged violations of policies and work with the appropriate internal departments to investigate misconduct, remedy the situation, and prevent recurrence of any such activity.

### 4.2.13 Chief Information Officer (CIO)

The CIO is responsible for establishing, implementing and communicating TestAmerica's Information Technology (IT) Policies, SOPs and Manuals. Other responsibilities include coordinating new technologies, development of electronic communication tools such as TestAmerica's intranet and internet sites, ensuring data security and documentation of software, ensuring compliance with the NELAC standard, and assistance in establishing, updating, and maintaining Laboratory Information Management Systems (LIMS) at the various TestAmerica facilities.

### 4.2.14 Environmental Health and Safety Managers (Corporate)

The EHS Managers report directly to the VP-QA/EHS. The EHS Managers are responsible for the development and implementation of the TestAmerica Environmental, Health and Safety program. Responsibilities include:

- Consolidation and tracking all safety and health-related information and reports for the company, and managing compliance activities for TestAmerica locations.
- Coordination/preparation of the corporate Environmental, Health and Safety Manual Template that is used by each laboratory to prepare its own laboratory-specific Safety Manual/ CHP.
- Preparation of information and training materials for laboratory EHS Coordinators.
- Assistance in the internal and external coordination of employee exposure and medical monitoring programs to insure compliance with applicable safety and health regulations.
- Serving as Department of Transportation (D.O.T.) focal point and providing technical assistance to location management.
- Serving as Hazardous Waste Management main contact and providing technical assistance to location management.

### 4.2.15 Laboratory Director

TestAmerica Sacramento's Laboratory Director is responsible for the overall quality, safety, financial, technical, human resource and service performance of the laboratory and reports to their respective VP of Operations. The Laboratory Director provides the resources necessary to implement and maintain an effective and comprehensive Quality Assurance and Data Integrity Program.

Specific responsibilities include, but are not limited to:

- Providing one or more technical managers for the appropriate fields of testing. If the Technical Manager is absent for a period of time exceeding 15 calendar days, the Laboratory Director must designate another full time staff member meeting the qualifications of the Technical Manager to temporarily perform this function. If the absence

exceeds 65 consecutive calendar days, the primary accrediting authority must be notified in writing.

- Ensuring that all analysts and supervisors have the appropriate education and training to properly carry out the duties assigned to them and ensures that this training has been documented.
- Ensuring that personnel are free from any commercial, financial and other undue pressures which might adversely affect the quality of their work.
- Ensuring TestAmerica's human resource policies are adhered to and maintained.
- Ensuring that sufficient numbers of qualified personnel are employed to supervise and perform the work of the laboratory.
- Ensuring 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.
- Reviewing and approving all SOPs prior to their implementation and ensures all approved SOPs are implemented and adhered to.
- Pursuing and maintaining appropriate laboratory certification and contract approvals.
- Supporting ISO 17025 requirements.
- Supporting DoD/DOE ELAP requirements.
- Supporting The NELAC Institute (TNI) Standard requirements
- Ensuring client specific reporting and quality control requirements are met.
- Directing the management team, consisting of the QA Manager, the Operations Manager, the EH&S Coordinator and the Office Manager as direct reports.

### 4.2.16 Quality Assurance (QA) Manager or Designee

The QA Manager has responsibility and authority to ensure the continuous implementation of the quality system. The QA Manager reports directly to the Laboratory Director and their Corporate Quality Director. This person 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. This person has documented training and/or experience in QA/QC procedures and the laboratory's Quality System. The QA Manager directs the activities of the QA officers to accomplish specific responsibilities, which include, but are not limited to:

- Serving as the focal point for QA/QC in the laboratory.
- Having functions independent from laboratory operations for which he/she has quality assurance oversight.
- Maintaining and updating the QAM.

- Monitoring and evaluating laboratory certifications; scheduling proficiency testing samples.
- Monitoring and communicating regulatory changes that may affect the laboratory to management.
- Training and advising the laboratory staff on quality assurance/quality control procedures that are pertinent to their daily activities.
- Having a general knowledge of the analytical test methods for which data audit/review is performed (and/or having the means of getting this information when needed).
- Arranging for or conducting internal audits on quality systems and the technical operation.
- Maintaining records of all ethics-related training, including the type and proof of attendance.
- Maintaining, improving, and evaluating the corrective action database and the corrective and preventive action systems.
- Notifying laboratory management of deficiencies in the quality system and ensuring corrective action is taken. Procedures that do not meet the standards set forth in the QAM or laboratory SOPs shall be investigated following procedures outlined in Section 12 and if deemed necessary may be temporarily suspended during the investigation.
- Objectively monitoring standards of performance in quality control and quality assurance without outside (e.g., managerial) influence.
- Coordinating document control of SOPs, MDLs, control limits, and miscellaneous forms and information.
- Reviewing external audit reports and data validation requests.
- Following-up with audits to ensure client QAPP requirements are met.
- Establishing reporting schedule and preparation of various quality reports for the Laboratory Director, clients and/or Corporate QA.
- Developing suggestions and recommendations to improve quality systems.
- Researching current state and federal requirements and guidelines.
- Directing the QA team to enable communication and to distribute duties and responsibilities.
- Ensuring communication with laboratory staff and monitoring standards of performance to ensure that systems are in place to produce the level of quality as defined in this document.
- Evaluating of the thoroughness and effectiveness of training.
- Assuring compliance with ISO 17025.
- Assuring compliance with DoD/DOE ELAP.
- Assuring compliance with The NELAC Institute (TNI) Standard.

### 4.2.17 Technical Manager (Manager of Operations) or Designee

The Technical Manager(s) (noted as Manager of Operations on the organizational chart) 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 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:

- Exercising day-to-day supervision of laboratory operations for the appropriate field of accreditation and reporting of results. Coordinating, writing, and 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 versus demonstrated versus first run yield) utilization.

- Reviewing and approving, with input from the QA Manager, proposals from marketing, in accordance with an established procedure for the review of requests and contracts. This procedure addresses the adequate definition of methods to be used for analysis and any limitations, the laboratory's capability and resources, the client's expectations. Differences are resolved before the contract is signed and work begins. A system documenting any significant changes is maintained, as well as pertinent discussions with the client regarding their requirements or the results of the analyses during the performance of the contract. All work subcontracted by the laboratory must be approved by the client. Any deviations from the contract must be disclosed to the client. Once the work has begun, any amendments to the contract must be discussed with the client and so documented.

- Monitoring the validity of the analyses performed and data generated in the laboratory. This activity begins with reviewing and supporting all new business contracts, insuring data quality, analyzing internal and external non-conformances to identify root cause issues and implementing the resulting corrective and preventive actions, facilitating the data review process (training, development, and accountability at the bench), and providing technical and troubleshooting expertise on routine and unusual or complex problems.

- Providing 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.

- Enhancing efficiency and improving quality through technical advances and improved LIMS utilization. Capital forecasting and instrument life cycle planning for second generation methods and instruments as well as asset inventory management.

- Coordinating sample management from "cradle to grave," insuring that no time is lost in locating samples.

- Scheduling all QA/QC-related requirements for compliance, e.g., MDLs, etc.

- Directing department personnel to communicate quality, technical, personnel, and instrumental issues for a consistent team approach.

- Complying with ISO 17025, The NELAC Institute (TNI) Standard, DoD/DOE ELAP and the various QC programs implemented at the Sacramento laboratory.

### 4.2.18 Client Services Manager

The CSM reports directly to the Client Service Director (Western Region) and indirectly to the Laboratory Director. The CSM serves as the interface between the laboratory's Project Management team, technical departments, and clients. The CSM shall:

- Oversee training and growth of the Project Management team.
- Act as technical liaison for the Project Management team.
- Provide human resource management support to the Project Management team.
- Assist PMs with responses to client inquiries or with resolutions to problems or complaints.
- Ensure that client specifications, when known, are met by communicating project and QA requirements to the laboratory.
- Notify Department Managers or supervisors of incoming projects and sample delivery schedules.
- Discuss with client any project-related problems, resolve service issues, and coordinate technical details with the laboratory staff.
- Monitor the status of projects in-house to ensure timely and accurate delivery of reports.
- Prepare price quotes or project bids.

### 4.2.19 Manager of Project Managers

The Manager of Project Management reports to the Regional Client Services 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 duties of this position are outlined below:

- Managing technical training and growth of the Project Management team
- Serving as technical liaison for the Project Management team
- Providing human resource management of the Project Management team
- Ensuring that clients receive the proper sampling supplies
- Overseeing response to client inquiries concerning sample status
- Assisting clients regarding the resolution of problems concerning COC
- Ensuring that client specifications, when known, are met by communicating project and quality assurance requirements to the laboratory
- Notifying the supervisors of incoming projects and sample delivery schedules
- Being accountable to clients for communicating sample progress in daily status meeting with agreed-upon due dates

- Discussing with clients any project-related problems, resolving service issues, and coordinating technical details with the laboratory staff
- Providing information to staff with respect to specific quotes, sample log-in review, and final report completeness
- Monitoring the status of all data package projects in-house to ensure timely and accurate delivery of reports
- Informing clients of data package-related problems and resolve service issues
- Coordinating requests for sample containers and other services (data packages)

### 4.2.20 Project Manager

Project Managers are a liaison between the laboratory's clients and the analytical staff. They report directly to the Manager of Program Management. The Project Managers have signature authority for final reports, and review project data packages for completeness and compliance with client needs and quality requirements.
The Project Manager's responsibilities include:

- Ensuring client specifications are met by communicating project and quality assurance requirements to the laboratory
- Notifying laboratory personnel of incoming projects and sample delivery schedules
- Monitoring the status of all projects in-house to ensure timely delivery of reports
- Informing clients of project-related problems, resolving service issues and coordinating technical issues with the laboratory staff
- Coordinating client requests for sample containers and other services
- Scheduling sample pick-ups from client offices or project sites and notifying the laboratory staff of incoming samples
- Coordinating subcontract work
- Assisting clients in procuring the proper sampling supplies
- Responding to client inquiries concerning sample status
- Assisting clients with resolution of problems concerning Chains-of-Custody
- Invoicing completed data packages
- Generating credit or debit invoices to ensure proper payment

### 4.2.21 Project Administrator

The Project Administrator reports to the Manager of Project Management and designated Project Manager. The Project Administrator assists the Project Manager in servicing the client's needs and communicating those needs to the laboratory. The Project Administrator's responsibilities include:

- Collating data reports, expanded deliverables, and electronic data deliverables (EDDs) for delivery to clients.
- Writing case narratives accompanying data packages to communicate anomalies to clients
- Coordinating client requests for sample containers and other services
- Assisting clients in procuring the proper sampling supplies
- Assisting Project Managers in changing compound lists, TAT, and other LIMS set up tasks.
- Monitoring report due dates for timely delivery
- Invoicing completed data packages
- Generating credit or debit invoices to ensure proper payment

### 4.2.22 Department Manager, Team Leader, or Supervisor

Department Managers report directly to the Operations Manager. They supervise the daily activities of analysis within a given laboratory area, and either oversee the review and approval, or perform the review and approval of all analytical data within that area.

Specific responsibilities include, but are not limited to:

- Exercising day-to-day supervision of laboratory operations for the appropriate field of accreditation and reporting of results.
- Ensuring that analysts in their department adhere to applicable SOPs and the QA Manual.
- Coordinating the writing and reviewing of documentation for all test methods, i.e., SOPs, with regard to quality, integrity, regulatory requirements and optimum and efficient production techniques, and subsequent analyst training and interpretation of the SOPs for implementation and unusual project samples.
- Monitoring the validity of the analyses performed and data generated in the laboratory. This activity includes 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.
- Providing 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.
- Enhancing efficiency and improving quality through technical advances, improved LIMS utilization, capital forecasting and instrument life cycle planning for second generation methods and instruments as well as asset inventory management.
- Scheduling all QA/QC-related requirements for compliance, e.g., MDLs, etc.
- Coordinating audit responses with the QA Manager.

- Complying with ISO 17025, The NELAC Institute (TNI) Standard, DoD/DOE QSM and the various QC programs implemented at the Sacramento laboratory.
- Participating in the selection, training (familiarization with SOP, QC, Safety and computer systems), developing performance objectives and standards of performance, appraising (measurement of objectives), scheduling, counseling, disciplining, and motivating analysts and documenting these activities in accordance with systems developed by the QA and Human Resources Departments.
- Evaluating staffing sufficiency and overtime needs.
- Encouraging the development of analysts to become cross-trained in various methods and/or operate multiple instruments efficiently while performing maintenance and documentation, self-supervise, and function as a department team.
- Providing guidance to analysts in resolving problems encountered daily during sample prep/analysis in conjunction with the Operations Manager, and/or QA Manager. Each is responsible for 100% of the data review and documentation, non-conformance and corrective actions, the timely and accurate completion of performance evaluation samples and MDLs, for his/her department.
- Ensuring all logbooks are maintained, current, reviewed, and properly labeled or archived.
- Reporting all non-conformance conditions to the QA Manager, Operations Manager, and/or Laboratory Director.
- Ensuring that preventive maintenance is performed on instrumentation as detailed in the QA Manual or SOPs. He/She has responsibility for developing and implementing a system for preventive maintenance, troubleshooting, and repairing or arranging for repair of instruments.
- Maintaining adequate and valid inventory of reagents, standards, spare parts, and other relevant resources required to perform daily analysis.
- Achieving optimum turnaround time on analyses and compliance with holding times.
- Conducting efficiency and cost control evaluations on an ongoing basis to determine optimization of labor, supplies, overtime, first-run yield, capacity (designed vs. demonstrated), second- and third-generation production techniques/instruments, and long term needs for budgetary planning.

### 4.2.23 Analyst

Analysts report to their respective Department Managers. They perform sample analyses and generate analytical data in accordance with documented procedures.

The responsibilities of the analysts are listed below:

- Collecting and preparing materials and supplies for the laboratory
- Retrieving samples from Sample Control for analysis

- Performing sample preparation 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.
- Documenting standard and sample preparation, sample matrix effects, and any observed non-conformance on worklists, benchsheets, lab notebooks and/or the Non-Conformance Database.
- Reporting all non-conformance situations, sample preparation problems, matrix problems and QC failures, which might affect the reliability of the data, to their supervisor, the Technical Manager, and/or the QA Manager or member of QA staff.
- Performing 100% review of the data generated prior to entering and submitting for secondary level review.
- Suggesting method improvements to their supervisor, the Technical Manager, and the QA Manager. These improvements, if approved, will be incorporated. Providing 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.
- Working cohesively as a team member in their department to achieve the goals of accurate results, optimum turnaround time, cost effectiveness, cleanliness, complete documentation, and personal knowledge of environmental analysis.

### 4.2.24 Sample Custodian

The Sample Custodian ensures the implementation of proper sample receipt procedures, including maintaining chain-of-custody. The Sample Custodian logs samples into the LIMS and ensures that all samples are stored appropriately. Duties for the Sample Custodian include the following:

- Receiving and unloading samples or consignments in accordance with DOT regulations
- Verifying samples against the Chain of Custody (COC)
- Logging samples into the LIMS to assign a lot number for tracking purposes, and notifying Project Managers of any irregularities with the sample shipment.
- Labeling samples with lot number assigned and deliver the samples to the appropriate labs for analysis daily
- Monitoring freezer and cooler temperatures daily to confirm that the readings are within SOP guidelines
- Shipping all subcontracted samples to designated lab in accordance with DOT regulations as needed.

### 4.2.25 Quality Assurance Staff

The Quality Assurance staff members report to the QA manager. They have responsibility and authority to ensure the continuous implementation of the quality system based on ISO 17025, through involvement in the following activities:

- Assisting the QA Manager in performing the annual internal laboratory audits, compiling the evaluation, and coordinating the development of an action plan to address any deficiency identified.
- Facilitating external audits, coordinating with the QA Manager and Laboratory Staff to address any deficiencies noted at the time of the audit and subsequently presented in the final audit report.
- Assisting the QA Manager in the preparation of new SOPs and in the maintenance of existing SOPs, coordinating annual reviews and updates.
- Managing the performance testing (PT) studies, coordinating follow-up studies for failed analytes, and working with QA Manager and Laboratory Staff to complete needed corrective action reports.
- Serving as a project manager for proficiency testing samples and other QC samples.
- Reviewing and maintaining personnel training records.
- Assisting the QA Manager and Project Management Group in the review of program plans for consistency with organizational and contractual requirements. Summarize and convey to appropriate personnel anomalies or inconsistencies observed in the review process.
- Managing certifications and accreditations.
- Monitoring for compliance with the following QA Metrics: Temperature Monitoring of refrigeration units; thermometer verifications and calibrations; balance verifications and callibrations; and Eppendorf/pipette calibrations.
- Periodically checking the proper use and review of logbooks.
- Assisting in the technical review of data packages which require QA review.
- Assisting the QA Manager in maintaining the laboratory's reference data to keep it current and accurate.
- Preparing certification applications for states as directed by QA Manager.
- Reviewing and maintaining personnel training records.
- Performing document control maintenance.
- Assisting departments in generating MDL spreadsheets and calculations, reviewing MDL studies submitted to QA.
- Assisting in control limit generation.
- Ensuring maintenance of records archives.

- Maintaining historical indices for all technical records including SOPs, QC records, laboratory data, etc.
- Assisting the QA Manager in meeting the responsibilities of the QA Department as described in laboratory policies and SOPs.

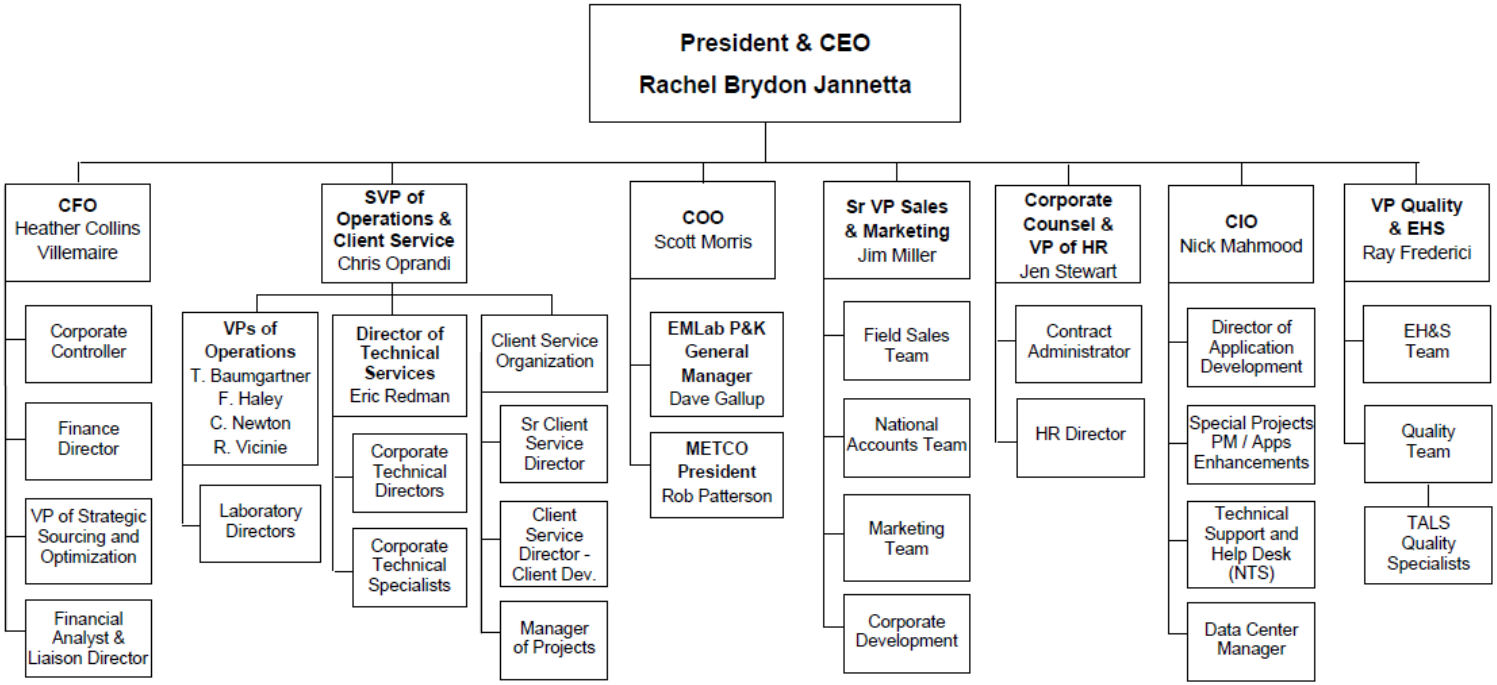
### 4.3 Deputies

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

| Key Personnel | Deputy |
|---|---|
| Crystal Pollock - Laboratory Director | Robert Hrabak - Technical Director, Manager of Dioxins, LCMS & Inorganics |
| Lisa Stafford - Quality Assurance Manager | Russell Evans - Quality Assurance Staff<br><br>Crystal Pollock - Laboratory Director |
| Robert Hrabak - Technical Director, Manager of Dioxins, LCMS & Inorganics | Crystal Pollock - Laboratory Director |
| Koroush Vaziri – Manager of Volatiles, Semivolatiles, & Organic/Dioxin Prep | Robert Hrabak - Technical Director, Manager of Dioxins, LCMS & Inorganics<br><br>Crystal Pollock - Laboratory Director |
| Jill Kellmann - Manager of Project Management | David Herbert - Client Relations Manager (Corporate) |
| Joe Schairer - EHS Coordinator | Richard Kester - Hazardous Materials Specialist |

**Figure 4-1. Corporate and Laboratory Organization Charts**

*All organizational charts are current as of the date noted. Contact the laboratory for the most recent organizational chart.*

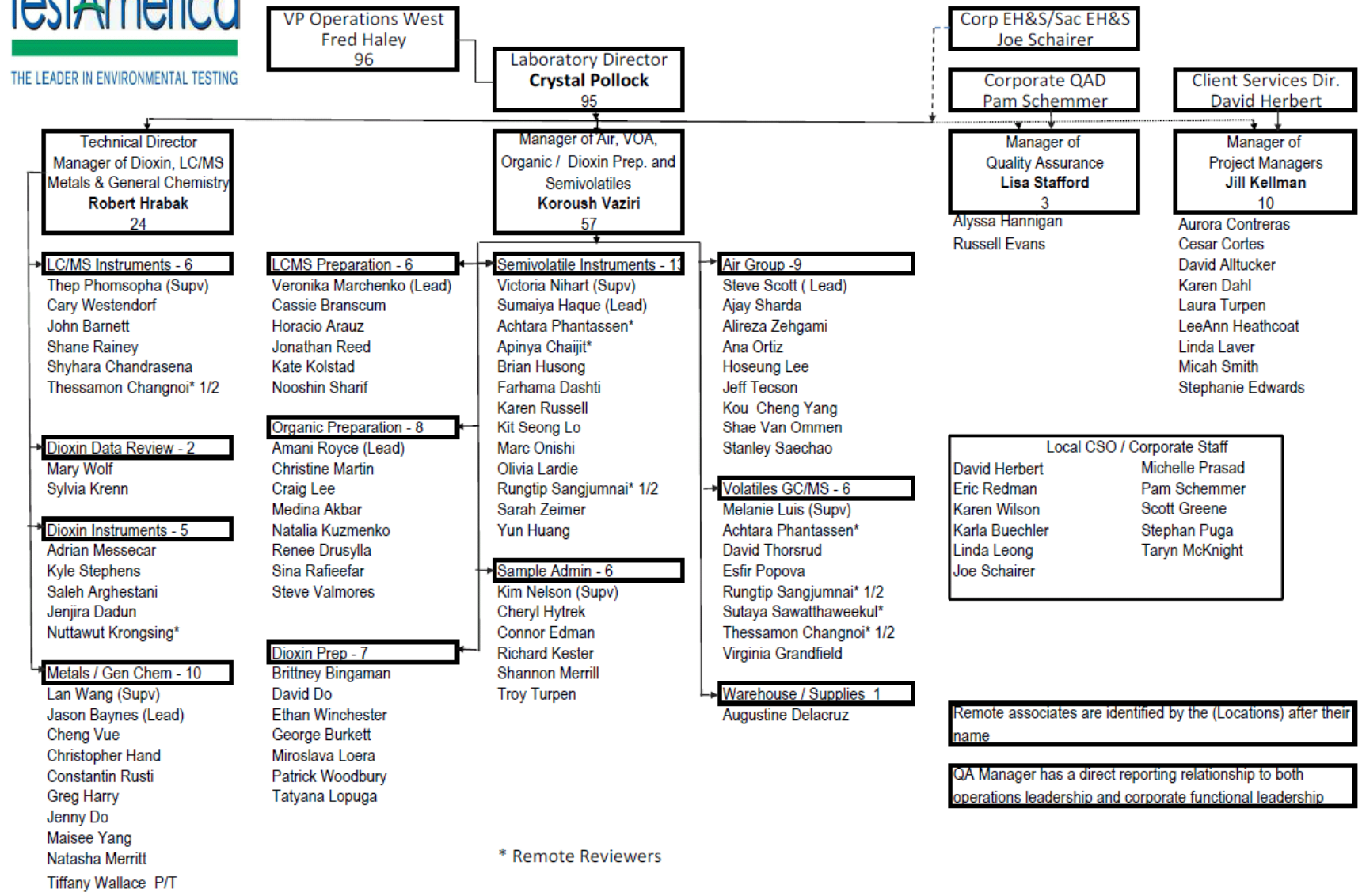





February 27 2017

TestAmerica
THE LEADER IN ENVIRONMENTAL TESTING

# Sacramento Laboratory Organization



# 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 characterizes the sites or materials that have been sampled. Quality Assurance is generally understood to be more comprehensive than Quality Control. Quality Assurance can be defined as the integrated system of activities that ensures that a product or service meets defined standards.

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

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

Historically, laboratories have described their QC objectives in terms of precision, accuracy, representativeness, comparability, completeness, selectivity and sensitivity (PARCCSS).

### 5.4.1 Precision

The laboratory objective for precision is to meet the performance for precision demonstrated for the methods on similar samples and to meet data quality objectives of the EPA and/or other regulatory programs. Precision is defined as the degree of reproducibility of measurements under a given set of analytical conditions (exclusive of field sampling variability). Precision is documented on the basis of replicate analysis, usually duplicate or matrix spike (MS) duplicate samples.

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

### 5.4.7 Sensitivity

Sensitivity refers to the amount of analyte necessary to produce a detector response that can be reliably detected (Method Detection Limit) or quantified (Reporting Limit).

## 5.5 Criteria for Quality Indicators

The laboratory maintains Reference Data in the LIMS that summarizes the precision and accuracy acceptability limits for performed analyses. This data includes an effective date, is updated each time new limits are generated and is managed by the laboratory's QA department. Unless otherwise noted, limits within these tables are laboratory generated. Some acceptability limits are derived from US EPA methods when they are required. Where US EPA method limits are not required, the laboratory has developed limits from evaluation of data from similar matrices. The criteria for development of control limits is contained in SOP WS-QA-0035, "Statistical Process Control / Control Chart" and Section 24.

## 5.6 Statistical Quality Control

Statistically-derived precision and accuracy limits are required by selected methods (such as SW-846) and programs. The laboratory routinely utilizes statistically-derived limits to evaluate method performance and determine when corrective action is appropriate. The analysts are instructed to use the current limits in the laboratory (dated and approved by the Technical Manager and QA Manager) and entered into the Laboratory Information Management System (LIMS). The Quality Assurance department maintains an archive of all limits used within the laboratory. If a method defines the QC limits, the method limits are used.

If a method requires the generation of historical limits, the lab develops such limits from recent data in the QC database of the LIMS following the guidelines described in SOP WS-QA-0035, "Statistical Process Control / Control Chart" and 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

As the QC limits are calculated, QC charts are generated showing warning and control limits for the purpose of evaluating trends. The QA Manager evaluates these to determine if adjustments need to be made or for corrective actions to methods. All findings are documented and kept on file. Control charts are generated according to laboratory SOP No. SOP WS-QA-0035, "Statistical Process Control / Control Chart"

## 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 No. WS-QA-0021, "Preparation and Management of Standard Operating Procedures".

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

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

### 6.2 Document Approval and Issue

The pertinent elements of a document control system for each document include a unique document title and number, 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. In order to develop a new document, a manager submits an electronic draft to the QA Department for suggestions and approval before use. Upon approval QA personnel add the identifying version information to the document and retains 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 two years (annually for documents applicable to drinking water and DoD/DOE programs), and revised as appropriate. Changes to documents occur when a procedural change warrants.

## 6.3 Procedures for Document Control Policy

For changes to the QA Manual, refer to SOP No. WS-QA-0021, "Preparation and Management of Standard Operating Procedures". Uncontrolled copies must not be used within the laboratory. Previous revisions and back-up data are stored by the QA department. Electronic copies are stored on the QA share on the local server for the applicable revision, and are accessible using the laboratory's Intranet.

For changes to SOPs, refer to SOP No. CW-Q-S-002, Writing a Standard Operating Procedure SOP and SOP No. WS-QA-0021, Preparation and Management of Standard Operating Procedures". The SOP identified above also defines the process of changes to SOPs.

Forms, worksheets, work instructions and information are organized by department in the QA office. There is a table of contents. Electronic versions are kept on a hard drive in the QA department; hard copies are kept in QA files. The procedure for the care of these documents is in SOP No. WS-QA-0021, "Preparation and Management of Standard Operating Procedures".

## 6.4 Obsolete Documents

All invalid or obsolete documents are removed, or otherwise prevented from unintended use. The laboratory has specific procedures as described above to accomplish this. In general, obsolete documents are collected from employees according to distribution lists and are marked obsolete on the cover or destroyed. At least one copy of the obsolete document is archived according to SOP No. WS-QA-0021, Preparation and Management of Standard Operating Procedures.

# 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. The PM will also get approval by the Laboratory Director to commit to delivery schedules that are shorter than the published standard turnaround times (TATs). The Laboratory Director updates these TATs on a routine basis, and it is the responsibility of CSMs and PMs to review them prior to making commitments for the laboratory.

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 Client Relationship Manager or Proposal Team, who will decide which lab will receive the work based on the scope of work and other requirements, including certification, testing methodology, and available capacity to perform the work. The contract review process is outlined in TestAmerica's Corporate SOP No. CA-L-P-002**,** Contract Compliance Policy.

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

- Contract Administrator
- VP of Operations
- Client Relations Manager
- Laboratory Project Manager
- Laboratory and/or Corporate Technical Managers / Directors
- Laboratory and/or Corporate Information Technology Managers/Directors
- Account Executives
- Laboratory and/or Corporate Quality
- Laboratory and/or Corporate Environmental Health and Safety Managers/Directors
- The Laboratory Director reviews the formal laboratory quote and makes final acceptance for their facility.

The Sales Director, Contracts Administrator, Account Executive, or Proposal Coordinator 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 Contracts Department maintains copies of all signed contracts. TestAmerica Sacramento's Customer Service Organization maintains copies of all signed contracts on the computer network for reference locally.

### 7.3 Documentation

Appropriate records are maintained for every contract or work request. All stages of the contract review process are documented and include records of any significant changes. These records are archived by client and project in a restricted network folder accessible to laboratory department managers, project managers, and senior managers.

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. Each Laboratory Project Manager keeps a phone log of conversations with the client. In addition, all conversations involving notification of important information, or actions directed by the client are documented with a follow up e-mail and archived in the contracts folder or the SDG documentation and case narrative. Instances include change in scope, alterations to the requests listed on a chain of custody, directions to proceed in the event of a non-conformance, and any other conversation that changes the direction of a COC or contract.

#### 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, a PM is assigned 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. Quality Assurance Project Plans, if submitted by the client, will be evaluated per policy WS-PQA-0018.

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

Prior to work on a new project, the dissemination of project information and/or project opening meetings may occur to discuss schedules and unique aspects of the project. Items to be discussed may include the project technical profile, turnaround times, holding times, methods, analyte lists, reporting limits, deliverables, sample hazards, or other special requirements. The PM introduces new projects to the laboratory staff through project kick-off meetings or to the supervisory staff during production meetings. These meetings provide direction to the laboratory staff in order to maximize production and client satisfaction, while maintaining quality. In addition, project notes may be associated with each sample batch as a reminder upon sample receipt and analytical processing.

During the project, any change that may occur within an active project is agreed upon between the client/regulatory agency and the PM/laboratory. These changes (e.g., use of a non-standard method or modification of a method) and approvals must be documented prior to implementation. Documentation pertains to any document, e.g., letter, e-mail, variance, contract addendum, which has been signed by both parties.

Such changes are updated to the Quality Assurance Summary (QAS) and introduced to the managers at these meetings. The laboratory staff is then introduced to the modified requirements via the PM or the individual laboratory Technical 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". This topic is discussed in Section 7.

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

- Reasonable access for our clients or their representatives to the relevant areas of the laboratory for the witnessing of tests performed for the client.
- Assist client-specified third party data validators as specified in the client's contract.
- Allow the client access to supplemental information that pertains 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.

Any member of the laboratory's senior staff or any of the laboratory's identified technical experts is 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 SOPs on Subcontracting Procedures (CW-L-S-004).

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 TNI/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 accredited work where required.

Project Managers (PMs), Client Service Managers (CSM), or Account Executives (AE) for the Export Lab (TestAmerica laboratory that transfers samples to another laboratory) are responsible for obtaining client approval prior to subcontracting 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. Standard TestAmerica Terms and Conditions include the flexibility to subcontract samples within the TestAmerica laboratories. Therefore, additional advance notification to clients for intra-laboratory subcontracting is not necessary unless specifically required by a client contract.

**Note:** In addition to the client, some regulating agencies (e.g., USDA) or contracts (e.g., DoD and DOE projects) may require notification prior to placing such work. Documentation of approval is stored electronically in the quote folder within SACSALES share on a local laboratory server.

### 8.2 Qualifying and Monitoring Subcontractors

Whenever a PM or Client Services Manager 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:

- Subcontractors specified by the client - In these circumstances, the client assumes responsibility for the quality of the data generated from the use of a subcontractor.

- Subcontractors reviewed by TestAmerica – Firms which have been reviewed by the company and are known to meet standards for accreditations (e.g., State, TNI and DoD/DOE); technical specifications; legal and financial information.

A listing of vendors is available on the TestAmerica intranet site.

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).

**8.2.1** When the potential sub-contract laboratory has not been previously approved, PMs or CSMs may nominate a laboratory as a subcontractor based on need. The decision to nominate a laboratory must be approved by the Client Relations Manager (CRM) or Laboratory Director. The CRM or Laboratory Director requests that the QA Manager begin the process of approving the subcontract laboratory as outlined in Corporate SOP No. CW-L-S-004, Subcontracting.

Once the appropriate accreditation and legal information is received by the laboratory, it is evaluated for acceptability (where applicable) and forwarded to the Corporate Quality Information Manager (QIM) for review. After the Corporate QIM reviews the documents for completeness, the information is forwarded to the Finance Department for formal signature and contracting with the laboratory. The approved vendor will be added to the approved subcontractor list on the intranet site and the finance group is concurrently notified for JD Edwards.

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 subcontractors on our approved list can only be recommended to the extent that we would use them.

## 8.3 Oversight and Reporting

**8.3.1** The status and performance of qualified subcontractors will be monitored by the Corporate Quality department. Any problems identified will be brought to the attention of TestAmerica's Corporate Finance, Legal and 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. CSO personnel 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 CSO Personnel, Laboratory Directors, QA Managers and Sales Personnel.

Prior to initially sending samples to the subcontracted laboratory, the PM confirms their certification status to determine if it's current and scope-inclusive. The information is stored electronically in the quote folder within the SACSALES share on a local laboratory server.

**8.3.2** For continued use of a subcontractor, verification of certification is placed upon the subcontractor for the defined project. Samples are subcontracted under Chain of Custody with the program defined as 'Accreditation Required' and the following statement for verification upon sample receipt:

**Note:** Since laboratory accreditations are subject to change, TestAmerica Laboratories, Inc. places the ownership of method, analyte & accreditation compliance upon our subcontract laboratories. This sample shipment is forwarded under Chain of Custody. If the laboratory does not currently maintain accreditation in the State of Origin listed above for analytes/tests/matrix being analyzed, the samples must be shipped back to the TestAmerica laboratory or other instructions will be provided. Any changes to accreditation status should be brought to TestAmerica Laboratories, Inc. attention immediately. If all requested accreditations are current to date, return the signed Chain of Custody attesting to said compliance to TestAmerica Laboratories, Inc.

For TestAmerica laboratories, certifications can be viewed on the company's TotalAccess Database.

**8.3.3** All subcontracted samples must be accompanied by a TestAmerica Chain of Custody (COC). A copy of the original COC sent by the client must be available in TALS for 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 accredited work must be identified in the subcontractor's report as appropriate. If TNI accreditation is not required, the report does not need to include this information.

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

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

## 8.4 Contingency Planning

The full qualification of a subcontractor may be waived 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 time.

The use of any emergency subcontractor will require the PM to complete a JDE New Vendor Add Form in order to process payment to the vendor and add them to TALS. This form requires the user to define the subcontractor's category/s of testing and the reason for testing.

# 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 Controlled Purchase Requests and Fixed Asset Capitalization Procedure, SOP No. CW-F-S-007.

Contracts will be signed in accordance with TestAmerica's Company-Wide 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, consumables, 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. Approval information for the solvents and acids tested under SOP CA-Q-S-001 is stored on the TestAmerica Sharepoint, under Solvent Approvals. A master list of all tested materials, as well as the certificates of analysis for the materials, is stored in the same location.

### 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. Many items used routinely are pre-qualified and placed into the on-site consignment system.

For items not available from the consignment system or items that are not used routinely, an order is placed in the JDE ordering system. Only personnel trained in the ordering program JDE may place orders using the program. All relevant information, including quantity, must be entered. Only approved vendors may be used. A vendor must be approved by corporate to be on the approved vendor list in JDE. The Laboratory Director or designee approves all orders placed in JDE.

### 9.3.2 Receiving

It is the responsibility of the purchasing manager to receive the shipment. For items received for the on-site consignment system, the purchasing manager verifies that the material received meets the quality level specified. This is documented by stamping the packing slip with "Received" and the date. For materials that are outside of the on-site consignment systems, it is the responsibility of the analyst who ordered the materials to document the date materials were received. Once the ordered reagents or materials are received, the analyst compares the information on the label or packaging to the original order to ensure that the purchase meets the quality level specified. This is documented through the addition of the received date and initials to the information present on the daily order log.

The purchasing manager verifies the lot numbers of received solvents and acids against the pre-approval lists. If a received material is listed as unapproved, or is not listed, it is sequestered and returned to the vendor. Alternatively, the laboratory may test the material for the intended use, and if it is acceptable, document the approval on the approval list. Records of any testing performed locally are maintained on the shared "public" folder on the computer network.

Materials may not be released for use in the laboratory until they have been inspected, verified as suitable for use, and the inspection/verification has been documented.

Safety Data Sheets (SDSs) are available online through the Company's intranet website. Anyone may review these for relevant information on the safe handling and emergency precautions of on-site chemicals.

### 9.3.3 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 the grade of reagent.

Chemicals must not be used past the manufacturer's expiration date and must not be used past the expiration time noted in a method SOP. If expiration dates are not provided, the laboratory may contact the manufacturer to determine an expiration date.

The laboratory assumes a five year expiration date on inorganic dry chemicals unless noted otherwise by the manufacturer or by the reference source method. Chemicals should not be used past the manufacturer's or SOP's expiration date unless 'verified' (refer to bullet 3 below). See laboratory SOP No. WS-QA-0017, "Standards and Reagent Preparation and Quality Control Check Procedures", for standard verification procedures.)

- 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 on the shared public folder on the computer network.

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 would 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. For the automated “tank farm” in use through most of the laboratory, the minimum total pressure at which the system switches to the next bank of tanks is 250 psig. The quality of the gases must meet method or manufacturer specification or be of a grade that does not cause any analytical interference.

Water used in the preparation of standards or reagents must have a specific conductivity of less than 1- µmho/cm (or specific resistivity of greater than 1.0 megohm-cm) at 25$^{o}$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 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. See laboratory SOP No. WS-QA-0017, “Standards and Reagent Preparation and Quality Control Check Procedures”, for standard QC procedures.

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.

Each laboratory section maintains records of manufacturer’s certification and traceability statements on the network. These records include date of receipt, lot number (when

applicable), and expiration date (when applicable). Furthermore, certificates of analysis for standards are scanned and attached to the preparation record in the LIMS. Incorporation of the item into the record indicates that the analyst has compared the new certificate with the previous one for the same purpose and that no difference is noted, unless approved and so documented by the Technical Manager or QA Manager.

### 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 Manager and/or the Laboratory Director. If they agree with the request, the procedures outlined in TestAmerica's Corporate Policy No. CA-T-P-001, Qualified Products List, are followed. A decision is made as to which piece of equipment can best satisfy the 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 QA Department. Software certificates supplied by the vendors are filed with the LIMS Administrator. The manufacturer's operation manual is retained at the bench and inventoried in the master document list.

## 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 Managers. The service providers that perform the services are approved by the Technical Manager.

Analytical balances are serviced and calibrated annually in accordance with SOP WS-QA-0041, Calibration and Calibration Check of Balances. The calibration and maintenance services are performed on-site, and the balances are returned to use immediately following successful calibration. When the calibration certificates are received (usually within two weeks of the service), they are reviewed, and documentation of the review is filed with the certificates. If the calibration was unsuccessful, the balance is immediately removed from service and segregated pending either further maintenance or disposal.

Calibration services for support equipment such as thermometers, weight sets, autopipettors, etc, are obtained from vendors with current and valid ISO 17025 accreditation for calibration of

the specific piece of equipment. Prior to utilizing the vendor's services, the vendor's accreditation status is verified. Once the equipment has been calibrated, the calibration certificates are reviewed by the QA department, and documentation of the review is filed with the calibration certificates. The equipment is then returned to service within the laboratory.

## 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 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 Technical Services 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 is documented following laboratory policy WS-PQA-013, Procedure to Address Customer Complaints.

### 10.2 External Complaints

An employee that receives a complaint initiates the complaint resolution process by first documenting the complaint according to laboratory policy WS-PQA-013, Procedure to Address Customer Complaints.

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 supervisor for resolution. The supervisor may elect to discuss it with the Technical Manager or have a representative contact the client to decide on a logical course of action. Once an approach is agreed upon, the analyst documents it using the laboratories corrective action system described in Section 12. This information can then be supplied to the client in the form of a footnote or a case narrative with the report.

Project Management may encounter situations where a client may request that a special procedure be applied to a sample that is not standard lab practice. Based on a technical evaluation, the lab may accept or opt to reject the request based on technical or ethical merit. An example might be the need to report a compound that the lab does not normally report. The lab would not have validated the method for this compound following the procedures in Section 19. The client may request that the compound be reported based only on the calibration. Such a request would need to be approved by the Technical Manager 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

Under certain circumstances, the Laboratory Director, a Technical 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, and the

Technical 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), Exec. Director of Quality & EHS 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, Executive VP of Operations, VP of Operations, and the Quality Directors have the authority and responsibility to halt work, withhold final reports, or suspend an analysis for due cause as well as authorize the resumption of work.

### 11.3 Evaluation of Significance and Actions Taken

For each nonconforming issue reported, an evaluation of its significance and the level of management involvement needed is made. This includes reviewing its impact on the final data, whether or not it is an isolated or systematic issue, and how it relates to any special client requirements.
When the laboratory discovers that erroneous or biased data may have been reported to clients or regulatory agencies, the procedures described in the corporate SOP CW-Q-S-005, Data Recalls, must be followed.
During investigation and correction of situations involving alleged incidents of misconduct or violation of the company's ethics policy, the procedures described in the corporate SOP CW-L-S-002, Internal Investigations, must be followed.

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, on a monthly basis, the QA Department evaluates non-conformances to determine if any nonconforming work has been repeated multiple times. If so, the laboratory's corrective action process may be followed.

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

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

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

The Laboratory Director shall arrange for the appropriate personnel to meet with the QA Manager as needed. This meeting shall be held to confirm that there is a problem, that

suspension/restriction of the method is required and will be concluded with a discussion of the steps necessary to bring the method/target or test fully back on line. In some cases, that may not be necessary if all appropriate personnel have already agreed there is a problem and there is agreement on the steps needed to bring the method, target or test fully back on line.

The QA Manager will also initiate a corrective action report as described in Section 12 if one has not already been started. A copy of any meeting notes and agreed upon steps should be faxed or e-mailed by the laboratory to the appropriate VP of Operations 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 Manager/Director, QA 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. Corrective actions are documented using Non-Conformance Memos (NCM) and 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 Non-Conformance Memo (NCM)** - is used to document the following types of corrective actions:

- Deviations from an established procedure or SOP
- QC outside of limits (non-matrix related)
- Isolated reporting / calculation errors
- Client complaints
- Discrepancies in materials / goods received vs. manufacturer packing slips.

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

- Questionable trends that are found in the review of NCMs.
- Issues found while reviewing NCMs that warrant further investigation.
- Internal and external audit findings.
- Failed or unacceptable PT results.
- Corrective actions that cross multiple departments in the laboratory.
- Systematic reporting / calculation errors
- Client complaints

- Data recall investigations
- Identified poor process or method performance trends
- Excessive revised reports
- Health and Safety violations

This 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. An NCM or CAR must be initiated, someone is assigned to investigate the issue and the event is investigated for cause. Laboratory SOP No. WS-QA-0023, Nonconformance and Corrective Action System, provides some general guidelines on determining responsibility for assessment.
- The cause analysis step is the key to the process as a long term corrective action cannot be determined until the cause is determined.
- If the cause is not readily obvious, the Technical 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 NCM or CAR is used for this documentation.

### 12.3.3 Root Cause Analysis

Root Cause Analysis is a class of problem solving (investigative) methods aimed at identifying the basic or causal factor(s) that underlie variation in performance or the occurrence of a significant failure. The root cause may be buried under seemingly innocuous events, many steps preceding the perceived failure. At first glance, the immediate response is typically directed at a symptom and not the cause. Typically, root cause analysis would be best with three or more incidents to triangulate a weakness. Corporate SOP Root Cause Analysis (No. CA-Q-S-009) describes the procedure.

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 the 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 Technical Manager and QA Manager are responsible to ensure that the corrective action taken was effective.
- Ineffective actions are documented and re-evaluated until acceptable resolution is achieved. Technical Managers are accountable to the Laboratory Director to ensure final acceptable resolution is achieved and documented appropriately.
- Each NCM and CAR is entered into a database for tracking purposes and these are periodically reviewed to ensure that the corrective actions have taken effect.
- TestAmerica laboratories began using the Incident/Corrective Action Tracker (iCAT) database developed by the company in 2015. (Previously, a local database [name of local system here] served this purpose.) An incident is an event triggering the need for one or more corrective actions as distinct from a corrective action, a potential deficiency stemming from an incident that requires investigation and possibly fixing. The database is independent of TALS, available to all local and corporate managers, and capable of notifying and tracking multiple corrective actions per event, dates, and personnel. iCAT allows associated document upload, categorization (such as, external/internal audit, client service concerns, data quality issues, proficiency testing, etc.), and trend analysis. Refer to Figure 12-1.
- The QA Manager reviews monthly NCMs and CARs for trends. Highlights are included in the QA monthly report (refer to Section 16). If a significant trend develops that adversely affects quality, an audit of the area is performed and corrective action implemented.
- Any out-of-control situations that are not addressed acceptably at the laboratory level may be reported to the Corporate Quality Director by the QA Manager, indicating the nature of the 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 an NCM or CAR.

Table 12-1 includes examples of general technical corrective actions. For specific criteria and corrective actions, refer to the analytical methods or specific method SOPs. The laboratory may also 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 SOP also provides general guidance on how a data set should be treated if associated QC measurements are unacceptable. Specific procedures are included in Method SOPs, Work Instructions, QAM Sections 19 and 20. All corrective actions are reviewed monthly, at a minimum, by the QA Manager and highlights are included in the QA monthly report.

To the extent possible, samples shall be reported only if all quality control measures are acceptable. If the deficiency does not impair the usability of the results, data will be reported with an appropriate data qualifier and/or the deficiency will be noted in the case narrative. Where sample results may be impaired, the Project Manager is notified by an NCM and appropriate corrective action (e.g., reanalysis) is taken and documented.

## 12.5 Basic Corrections

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

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

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**

TestAmerica
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**Sacramento**
**Corrective Action Report**

**Title: <Enter Title Here>**

**Reference:** <tracking information>

**Initiated by:** **Date:**

**Responsible Party:** **Date:** <date report submitted>

**Description of Problem:**
[enter text to briefly explain how the problem was discovered, who discovered it and when, and what work, if any, is affected]

**Investigation Planned or Completed:**
[enter text to briefly what was examined to determine the extent of the problem, when the investigation was conducted, what was the proximate cause(s), and what were the root causes. The key is to demonstrate that the investigation is comprehensive]

**Root Cause Analysis**
[True root cause analysis should involve multiple layers of questioning]
*Examples:*
*- Why did this problem occur?*
*- What weaknesses are indicated by this problem?*
*- What Quality Systems mechanisms are in place that should have prevented this problem from occurring?*
*- Is this issue acute or chronic?*
*- Are changes needed to existing SOPs to correct this problem and prevent its recurrence?*
*- Are other departments affected by this issue?*

**Corrective Action Plan**
[Based on the Root Cause Analysis outlined above, what action items need to be completed to correct this deficiency, and prevent its recurrence?]
*Examples:*
*- Identify impacted lots*
*- Revise results/reports*
*- Initiate formal Data Recall*
*- Revise SOP*
*- Re-train staff*

**QA Monitoring of Corrective Action Status**
[If an anomalous or isolated event, and no further action required, this section may be omitted. Otherwise, note the need for a routine follow-up assessment and the associated details (responsible party, due date, documentation necessary), or the need to add to the internal audit checklist for reassessment at a later date.

Closed by: _____ _____
<Name, title> Date

**Table 12-1. Example – General Corrective Action Procedures**

| QC Activity *(Individual Responsible for Initiation/Assessment)* | Acceptance Criteria | Recommended Corrective Action |
|---|---|---|
| Initial Instrument Blank<br><br>*(Analyst)* | - Instrument response <MDL. | - Prepare another blank.<br>- If same response, determine cause of contamination: reagents, environment, instrument equipment failure, etc. |
| Initial Calibration Standards<br><br>*(Analyst, Technical Manager(s))* | - Correlation coefficient > 0.99 or standard concentration value.<br>- % Recovery within acceptance range.<br>- See details in Method SOP. | - Reanalyze standards.<br>- If still unacceptable, remake standards and recalibrate instrument. |
| Independent Calibration Verification (Second Source)<br><br>*(Analyst, Technical Manager(s))* | - % Recovery within control limits. | - Remake and reanalyze standard.<br>- If still unacceptable, then remake calibration standards or use new primary standards and recalibrate instrument. |
| Continuing Calibration Standards<br><br>*(Analyst, Data Reviewer)* | % Recovery within control limits. | - Reanalyze standard.<br>- If still unacceptable, then recalibrate and rerun affected samples. |
| Matrix Spike / Matrix Spike Duplicate (MS/MSD)<br><br>*(Analyst, Data Reviewer)* | - % Recovery within limits documented in the LIMS or Project QAPP. | - 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.<br>- If the LCS is within acceptable limits the batch is acceptable.<br>- The results of the duplicates, matrix spikes and the LCS are reported with the data set.<br>- 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 (LCS)<br><br>*(Analyst, Data Reviewer)* | - % Recovery within limits documented in the LIMS or Project QAPP. | - Batch must be re-prepared and re-analyzed. This includes any allowable marginal exceedance.<br>When not using marginal exceedances, the following exceptions apply:<br>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;<br>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.<br><br>**Note:** If there is insufficient sample or the holding time cannot be met, contact client and report with flags. |
| Surrogates<br><br>*(Analyst, Data Reviewer)* | - % Recovery within limits of method or within three standard deviations of the historical mean. | - Individual sample must be repeated. Place comment in LIMS.<br>- Surrogate results outside criteria shall be reported with qualifiers. |
| Method Blank (MB)<br><br>*(Analyst, Data Reviewer)* | < Reporting Limit [1] | - Reanalyze blank.<br>- If still positive, determine source of contamination. If necessary, reprocess (i.e. digest or extract) entire sample batch. Report blank results.<br>- Qualify the result(s) if the concentration of a targeted analyte in the MB is at or above the reporting limit AND is > 1/10 of the amount measured in the sample. |
| Proficiency Testing (PT) Samples<br><br>*(QA Manager, Technical Manager(s))* | - Criteria supplied by PT Supplier. | - Any failures or warnings must be investigated for cause. Failures may result in the need to repeat a PT sample to show the problem is corrected. |
| Internal / External Audits<br><br>*(QA Manager, Technical Manager(s), Laboratory Director)* | - Defined in Quality System documentation such as SOPs, QAM, etc.. | - Non-conformances must be investigated through CAR system and necessary corrections must be made. |

| QC Activity *(Individual Responsible for Initiation/Assessment)* | Acceptance Criteria | Recommended Corrective Action |
|---|---|---|
| Reporting / Calculation Errors<br><br>(Depends on issue – possible individuals include: Analysts, Data Reviewers, Project Managers, Technical 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. |
| Client Complaints<br><br>*(Project Managers, Lab Director/Manager, Sales and Marketing)* | - | - Corrective action is determined by the type of complaint. For example, a complaint regarding an incorrect address on a report will result in the report being corrected and then follow-up must be performed on the reasons the address was incorrect (e.g., database needs to be updated). |
| QA Monthly Report (Refer to Section 16 for an example)<br><br>(QA Manager, Lab Director/Manager, *Technical Manager(s)*) | - 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<br><br>(Safety Officer, Lab Director/Manager, *Technical Manager(s)*) | - Environmental Health and Safety (EHS) Manual. | - Non-conformance is investigated and corrected through CAR system. |

**Note:**
1. Except as noted below for certain compounds, the method blank should be below the detection limit. Concentrations up to five times the reporting limit will be allowed for the ubiquitous laboratory and reagent contaminants as defined in policy WS-PQA-003 **provided** they appear at similar levels in the reagent blank and samples. The ubiquitous contaminants include: methylene chloride, toluene, acetone, 2-butanone, phthalates and octachlorodibenzodioxin. This allowance presumes that the detection limit is significantly below any regulatory limit to which the data are to be compared and that blank subtraction will not occur. For benzene and ethylene dibromide (EDB) and other analytes for which regulatory limits are extremely close to the detection limit, the method blank must be below the method detection limit.

## 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, the laboratory continually strives to improve customer service and client satisfaction through continuous improvements to laboratory systems.

Opportunities for improvement may be discovered through any of the following:

- review of the monthly QA Metrics Report,
- trending NCMs,
- review of control charts and QC results,
- trending proficiency testing (PT) results,
- performance of management system reviews,
- trending client complaints,
- review of processing operations, or
- staff observations.

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. The metrics report is reviewed monthly by the laboratory management, Corporate QA and TestAmerica's Executive Committee. These metrics are used ~~to~~ in evaluating the management and quality system performance on an ongoing basis and provide a tool for identifying areas for improvement.

Items identified as continuous improvement opportunities to the management system may be issued as goals from the annual management systems review, recommendations from internal audits, white papers, Lesson Learned, Technical Services audit report, Technical Best Practices, or as Corporate or management initiatives.
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 and non-conformances provides a valuable mechanism for identifying preventive action opportunities.

**13.1.1** The following elements are part of a preventive action/process improvement system:

- Identification of an opportunity for preventive action or process improvement.
- Process for the preventive action or improvement.
- Define the measurements of the effectiveness of the process once undertaken.
- Execution of the preventive action or improvement.
- Evaluation of the plan using the defined measurements.
- Verification of the effectiveness of the preventive action or improvement.
- Close-Out by documenting any permanent changes to the Quality System as a result of the Preventive Action or Process Improvement. Documentation of Preventive Action/Process Improvement is incorporated into the monthly QA reports, corrective action process and management review.

**13.1.2** Any Preventive Actions/Process Improvement 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.

## 13.2 Management of Change

The Management of Change process is designed to manage significant events and changes that occur within the laboratory. Through these procedures, the potential risks inherent with a new event or change are identified and evaluated. The risks are minimized or eliminated through pre-planning and the development of preventive measures. The types of changes covered under this system include: Facility Changes, Major Accreditation Changes, Addition or Deletion to Division's Capabilities or Instrumentation, Key Personnel Changes, Laboratory Information Management System (LIMS) changes. This process is discussed in further detail in WS-QA-0050, Management of Change Procedures.

## 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. More detailed information on retention of specific records is provided in CW-L-P-001, Records Retention Policy and CW-L-WI-001, TestAmerica Records Retention/Storage Schedule. Quality records are maintained by the QA department in a database or in specific folders on the local QA share on a corporate server, which is backed up as part of the regular 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 Department Managers.

**Table 14-1. Record Index[1]**

| | **Record Types [1]:** | **Retention Time:** |
|---|---|---|
| **Technical Records** | - Raw Data<br>- Logbooks[2]<br>- Standards<br>- Certificates<br>- Analytical Records<br>- MDLs/IDLs/DOCs<br>- Lab Reports | 5 Years from analytical report issue* |
| **Official Documents** | - Quality Assurance Manual (QAM)<br>- Work Instructions<br>- Policies<br>- SOPs<br>- Policy Memorandums<br>- Manuals<br>- Published Methods | Indefinitely |
| **QA Records** | - Certifications<br>- Method and Software Validation / Verification Data | Indefinitely |
| **QA Records** | - Internal & External Audits/Responses<br>- Corrective/Preventive Actions<br>- Management Reviews<br>- Data Investigation | 5 Years from archival*<br><br>**Data Investigation:** 5 years or the life of the affected raw data storage whichever is greater (beyond 5 years if ongoing project or pending investigation) |

| | Record Types [1]: | Retention Time: |
|---|---|---|
| **Project Records** | - Sample Receipt & COC Documentation<br>- Contracts and Amendments<br>- Correspondence<br>- QAPP<br>- SAP<br>- Telephone Logbooks<br>- Lab Reports | 5 Years from analytical report issue* |
| **Administrative Records** | Finance and Business Operations | Refer to CW-L-WI-001 |
| | EH&S Manual, Permits | Indefinitely |
| | Disposal Records (Add Permits?) | Indefinitely |
| | Employee Handbook | Indefinitely |
| | Personnel files, Employee Signature & Initials, Administrative Training Records (e.g., Ethics) | Refer to HR Manual. All HR documents have different retention times. |
| | Administrative Policies | Indefinitely |
| | Technical Training Records | 7 years |
| | Legal Records | Indefinitely |
| | HR Records | Refer to CW-L-WI-001 |
| | IT Records | Refer to CW-L-WI-001 |
| | Corporate Governance Records | Refer to CW-L-WI-001 |
| | Sales & Marketing | 5 years |
| | Real Estate | Indefinitely |

[1] Record Types encompass hardcopy and electronic records.
[2] 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. 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**.** Logs are maintained in each storage box to note removal and return of records. Records are maintained for a minimum of five years unless otherwise specified by a client or regulatory requirement.

For raw data and project records, record retention shall be calculated from the date the project report is issued. For other records, such as Controlled Documents, QA, or Administrative Records, the retention time is calculated from the date the record is formally retired. Records related to the programs listed in Table 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**

| Program | [1]Retention Requirement |
|---|---|
| Drinking Water – All States | 10 years (lab reports and raw data)<br>10 years - Radiochemistry (project records) |
| Drinking Water Lead and Copper Rule | 12 years (project records) |
| Commonwealth of MA – All environmental data 310 CMR 42.14 | 10 years |
| FIFRA – 40 CFR Part 160 | Retain for life of research or marketing permit for pesticides regulated by EPA |
| Housing and Urban Development (HUD) Environmental Lead Testing | 10 years |
| Alaska | 10 years |
| Louisiana – All | 10 years |
| Michigan Department of Environmental Quality – all environmental data | 10 years |
| Navy Facilities Engineering Service Center (NFESC) | 10 years |
| Ohio VAP | 10 years and State contacted prior to disposal |
| TSCA - 40 CFR Part 792 | 10 years after publication of final test rule or negotiated test agreement |
| OSHA | 30 years |

[1]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 and WS-PQA-017 for more information.

**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.) Refer to SOP WS-QA-0009, Document Archiving. Instrument data is stored by project, except for inorganics and calibration data. Inorganics and calibration data is stored sequentially by instrument as appropriate. 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 re-constructing an analytical sequence. Where an analysis is performed without an instrument, bound logbooks or bench sheets are used to record and file data. Standard and reagent information is recorded in logbooks or entered into the LIMS for each method as required.

- Changes to hardcopy records shall follow the procedures outlined in Section 12 and 19. Changes to electronic records in LIMS or instrument data are recorded in audit trails.

- The reason for a signature or initials on a document is clearly indicated in the records such as "sampled by," "prepared by," "reviewed by", or "analyzed by".

- All generated data except those that are generated by automated data collection systems, are recorded directly, promptly and legibly in permanent dark ink.

- Hard copy data may be scanned into PDF format for record storage as long as the scanning process can be verified in order to ensure that no data is lost and the data files and storage media must be tested to verify the laboratory's ability to retrieve the information prior to the destruction of the hard copy that was scanned. The procedure for this verification can be found in SOP WS-QA-0009.

- Also refer to Section 19.14.1 'Computer and Electronic Data Related Requirements'**.**

## 14.2 Technical and Analytical Records

**14.2.1** The laboratory retains records of original observations, derived data and sufficient information to establish an audit trail, calibration records, staff records and a copy of each analytical report issued, for a minimum of five years unless otherwise specified by a client or regulatory requirement. The records for each analysis shall contain sufficient information to enable the analysis to be repeated under conditions as close as possible to the original. The records shall include the identity of laboratory personnel responsible for the sampling, performance of each analysis and reviewing 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, on a benchsheet or in the LIMS.
- Instrumentation identification and instrument operating conditions/parameters. Operating conditions/parameters are typically recorded in instrument maintenance logs where available.
- analysis type**;**
- all manual calculations and manual integrations**;**
- analyst's or operator's initials/signature**;**
- sample preparation including cleanup, separation protocols, 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**.**

**14.2.4** All logbooks used during receipt, preparation, storage, analysis, and reporting of samples or monitoring of support equipment shall undergo a documented supervisory or peer review on a monthly basis.

## 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 a logbook or in the LIMS. Records are considered archived when noted as such in the records management system (a.k.a., document control.)

### 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. CW-Q-S-003. 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 |
| QA Technical Audits | Joint responsibility:<br>a) QA Manager or designee<br>b) Technical Manager or Designee<br>(Refer to CW-Q-S-003) | Technical Audits Frequency:<br>50% of methods annually |
| SOP Method Compliance | Joint responsibility:<br>a) QA Manager or designee<br>c) Technical Manager or Designee<br>(Refer to CW-Q-S-003) | SOP Compliance Review Frequency<br>• Minimum of every two years.<br>• Annually for all methods and administrative SOPs relating to DoD/DOE programs. |
| 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 and Chrom AuditMiner) 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 Manager or qualified designee at least every two years. (Annually for methods and administrative SOPs related to DoD/DOE programs.) 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 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: Soil, Water Supply, Water Pollution, Air, and round-robin studies for sediments and biological materials. When available for parameters tested by the laboratory, the laboratory will also participate in the DOE administered MAPEP program.

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 within the 2009 TNI standards.

## 15.3 Audit Findings

Audit findings are documented using the corrective action process and database. The laboratory's corrective action responses for both types of audits may include action plans that could not be completed within a predefined timeframe. In these instances, a completion date must 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 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, their Quality Director as well as the VP of Operations. 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, VP of Operations, or Corporate QA may request that additional information be added to the report.

On a monthly basis, Corporate QA compiles information from all the monthly laboratory reports. The Corporate Quality Directors prepare a report that includes a compilation of all metrics and notable information and concerns regarding the QA programs within the laboratories. The report also includes a listing of new regulations that may potentially impact the laboratories. This report is presented to the Senior Management Team and General Managers.

### 16.2 Annual Management Review

The senior lab management team (Laboratory Director, Technical Managers, and QA Manager) conducts a review annually of its quality systems and LIMS to ensure its continuing suitability and effectiveness in meeting client and regulatory requirements and to introduce any necessary changes or improvements. It will also provide a platform for defining 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 cannot be solved by the lab and report them to Corporate IT.

This management systems review (Corporate SOP No. CW-Q-S-004 & Work Instruction No. CW-Q-WI-003) 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, Executive VP of Operations, VP of Client & Technical Services, VPs of Operations and Quality Directors receive a monthly report from the Exec Director of Quality & EHS summarizing any current data integrity or data recall investigations. The VPs of Operations 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. Selection of qualified candidates for laboratory employment begins with documentation of minimum education, training, and experience prerequisites needed to perform the prescribed task. Minimum education and training requirements for TestAmerica employees are outlined in job descriptions and are generally summarized for analytical staff in the table below.

The laboratory maintains job descriptions for all personnel who manage, perform or verify work affecting the quality of the environmental testing the laboratory performs. Job Descriptions are located on the TestAmerica intranet site's Human Resources web-page (Also see Section 4 for position descriptions/responsibilities).

Experience and specialized training are occasionally accepted in lieu of a college degree (basic lab skills such as using a balance, colony counting, aseptic or quantitation techniques, etc., are also considered).

As a general rule for analytical staff:

| Specialty | Education | Experience |
|---|---|---|
| Extractions, Digestions, some electrode methods (pH, Conductivity, 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, 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<br>Or<br>5 years of prior analytical experience |
| Technical Managers – **General** | Bachelors Degree in an applied science or engineering with 24 semester hours in chemistry<br><br>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 Managers – **Wet Chem** only (no advanced instrumentation) | Associates degree in an applied science or engineering or 2 years of college with 16 semester hours in chemistry | And 2 years relevant experience |

When an analyst does not meet these requirements, they can perform a task under the direct supervision of a qualified analyst, peer reviewer or Technical Manager, and are considered an analyst in training. The person supervising an analyst in training is accountable for the quality of the analytical data and must review and approve data and associated corrective actions.

### 17.3 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. Below are examples of various areas of required employee training:

| Required Training | Time Frame | Employee Type |
|---|---|---|
| Environmental Health & Safety | Prior to lab work | All |
| Ethics – New Hires | 1 week of hire | All |
| Ethics – Comprehensive | 90 days of hire | All |
| Data Integrity | 30 days of hire | Technical and PMs |
| Quality Assurance | 90 days of hire | All |
| Ethics – Comprehensive Refresher | Annually | All |
| Initial Demonstration of Capability (DOC) | Prior to unsupervised method performance | Technical |

The laboratory maintains records of relevant authorization/competence, education, professional qualifications, training, skills and experience of technical personnel (including contracted personnel) as well as the date that approval/authorization was given. These records are kept on file at the laboratory. Also refer to "Demonstration of Capability" in Section 19.

The training of technical staff is kept up to date by:

- Each employee must have documentation in their training file that they have read, understood and agreed to follow the most recent version of the laboratory QA Manual and SOPs in their area of responsibility. This documentation is updated as SOPs are updated.
- Documentation from any training courses or workshops on specific equipment, analytical techniques or other relevant topics are maintained in their training file.
- Documentation of proficiency (refer to Section 19).
- An Ethics Agreement signed by each staff member (renewed each year) and evidence of annual ethics training.
- A Confidentiality Agreement signed by each staff member signed at the time of employment.
- Human Resources maintains documentation and attestation forms on employment status & records; benefit programs; timekeeping/payroll; and employee conduct (e.g., ethics violations). This information is maintained in the employee's secured personnel file.

Evidence of successful training could include such items as:

- Adequate documentation of training within operational areas, including one-on-one technical training for individual technologies, and particularly for people cross-trained.
- Analyst knowledge to refer to QA Manual for quality issues.
- Analysts following SOPs, i.e., practice matches SOPs.
- Analysts regularly communicate to supervisors and QA if SOPs need revision, rather than waiting for auditors to find problems.

Further details of the laboratory's training program are described in the Laboratory Training SOP (WS-QA-0022, Employee Orientation 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 66,000 $ft^2$ secure laboratory facility with controlled access and designed to accommodate an efficient workflow and to provide a safe and comfortable work environment for employees. All visitors sign in and are escorted by laboratory personnel. Access is controlled by various measures.

The laboratory is equipped with structural safety features. Each employee is familiar with the location, use, and capabilities of general and specialized safety features associated with their workplace. The laboratory provides and requires the use of protective equipment including safety glasses, protective clothing, gloves, etc., OSHA and other regulatory agency guidelines regarding required amounts of bench and fume hood space, lighting, ventilation (temperature and humidity controlled), access, and safety equipment are met or exceeded.

Traffic flow through sample preparation and analysis areas is minimized to reduce the likelihood of contamination. Adequate floor space and bench top area is provided to allow unencumbered sample preparation and analysis space. Sufficient space is also provided for storage of reagents and media, glassware, and portable equipment. Ample space is also provided for refrigerated sample storage before analysis and archival storage of samples after analysis. Laboratory HVAC and deionized water systems are designed to minimize potential trace contaminants.

The laboratory is separated into specific areas for sample receiving, sample preparation, volatile organic sample analysis, non-volatile organic sample analysis, inorganic sample analysis, and administrative functions.

### 18.2 Environment

Laboratory accommodation, test areas, energy sources, lighting are adequate to facilitate proper performance of tests. The facility is equipped with heating, ventilation, and air conditioning (HVAC) systems appropriate to the needs of environmental testing performed at this laboratory.

The environment in which these activities are undertaken does not invalidate the results or adversely affect the required accuracy of any measurements.

The laboratory provides for the effective monitoring, control and recording of environmental conditions that may affect the results of environmental tests as required by the relevant specifications, methods, and procedures. Such environmental conditions include humidity, voltage, temperature, and vibration levels in the laboratory. In the event of a power outage, the laboratory can be equipped with a back up power supply for sample storage, as detailed in SOP No. WS-QA-0005, Temperature Monitoring and Corrective Action for Refrigerators and Freezers.

When any of the method or regulatory required environmental conditions change to a point where they may adversely affect test results, analytical testing will be discontinued until the environmental conditions are returned to the required levels.

Environmental conditions of the facility housing the computer network and LIMS are regulated to protect against raw data loss.

### 18.3 Work Areas

There is effective separation between neighboring areas when the activities therein are incompatible with each other. Examples include:

- Volatile organic chemical handling areas, including sample preparation and waste disposal, and volatile organic chemical analysis areas.

Access to and use of all areas affecting the quality of analytical testing is defined and controlled by secure access to the laboratory building as described below in the Building Security section.

Adequate measures are taken to ensure good housekeeping in the laboratory and to ensure that any contamination does not adversely affect data quality. These measures include regular cleaning to control dirt and dust within the laboratory. Work areas are available to ensure an unencumbered work area. Work areas include:

- Access and entryways to the laboratory.
- Sample receipt areas.
- Sample storage areas.
- Chemical and waste storage areas.
- Data handling and storage areas.
- Sample processing areas.
- Sample analysis areas.

### 18.4 Floor Plan

A floor plan can be found in Appendix 1.

### 18.5 Building Security

Building keys and alarm codes are distributed to employees as necessary.

Employees wear photographic identification name cards while on the premises.

Visitors to the laboratory sign in and out in a visitor's logbook. A visitor is defined as any person who visits the laboratory who is not an employee of the laboratory. In addition to signing into the laboratory, the Environmental, Health and Safety Manual contains requirements for visitors and vendors. There are specific safety forms that must be reviewed and signed. Visitors (with the exception of company employees) are escorted by laboratory personnel at all times, or the location of the visitor is noted in the visitor's logbook.

# SECTION 19. TEST METHODS AND METHOD VALIDATION

## 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 in the laboratory.

- All SOPs contain a revision number, effective date, and appropriate approval signatures. Controlled copies are available to all staff.
- Procedures for writing an SOP are incorporated by reference to TestAmerica's Corporate SOP entitled 'Writing a Standard Operating Procedure', No. CW-Q-S-002 or the laboratory's SOP WS-QA-0021 (Preparation and Management of Standard Operating Procedures).
- SOPs are reviewed at a minimum of every 2 years (annually for Drinking Water and DoD/DOE 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

Routine analytical services are performed using standard EPA-approved methodology. In some cases, modification of standard approved methods may be necessary to provide accurate analyses of particularly complex matrices. When the use of specific methods for sample analysis is mandated through project or regulatory requirements, only those methods shall be used.

When clients do not specify the method to be used or methods are not required, the methods used will be clearly validated and documented in an SOP and available to clients and/or the end user of the data.

The analytical methods used by the laboratory are those currently accepted and approved by the U. S. EPA and the state or territory from which the samples were collected. Reference methods include:

- *Compendium of Methods for the Determination of Toxic Organic Compounds in Ambient Air, US EPA, January 1996.*
- *Guidelines Establishing Test Procedures for the Analysis of Pollutants Under the Clean Water Act, Analysis and Sampling Procedures; 40 CFR Part 136 as amended by Method Update Rule; May 18, 2012.*
- *Methods for Chemical Analysis of Water and Wastes, EPA 600 (4-79-020), 1983.*
- *Methods for the Determination of Inorganic Substances in Environmental Samples, EPA-600/R-93/100, August 1993.*
- *Methods for the Determination of Metals in Environmental Samples, EPA/600/4-91/010, June 1991. Supplement I: EPA-600/R-94/111, May 1994.*
- *Methods for the Determination of Organic Compounds in Drinking Water, EPA-600/4-88-039, December 1988, Revised, July 1991, Supplement I, EPA-600-4-90-020, July 1990, Supplement II, EPA-600/R-92-129, August 1992. Supplement III EPA/600/R-95/131 - August 1995 (EPA 500 Series) (EPA 500 Series methods)*
- *Technical Notes on Drinking Water Methods, EPA-600/R94-173, October 1994*
- *NIOSH Manual of Analytical Methods, 4th ed., August 1994.*
- *Statement of Work for Inorganics & Organics Analysis, SOM, DLM, CBC, and ISM, current versions, USEPA Contract Laboratory Program Multi-media, Multi-concentration.*

- *Standard Methods for the Examination of Water and Wastewater, 18th/19th /20th/ on-line edition; Eaton, A.D. Clesceri, L.S. Greenberg, A.E. Eds; American Water Works Association, Water Pollution Control Federation, American Public Health Association: Washington, D.C.*
- *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; Final Update V, August 2015..*
- *Annual Book of ASTM Standards, American Society for Testing & Materials (ASTM), Philadelphia, PA.*
- *National Status and Trends Program, National Oceanographic and Atmospheric Administration, Volume I-IV, 1985-1994.*
- *Manual for the Certification of Laboratories Analyzing Drinking Water (EPA 815-R-05-004, January 2005)*
- *Code of Federal Regulations (CFR) 40, Parts 136, 141, 172, 173, 178, 179 and 261*
- *Underground Storage Tanks Procedures Manual, State of Alaska Department of Environmental Conservation, Division of Spill Prevention and Response Contaminated Sites Program, November 7, 2002*
- *Tri-Regional Board Staff Recommendations for Preliminary Investigation and Evaluation of Underground Tank Sites, North Coast Regional Water Quality Control Board, San Francisco Bay Regional Water Quality Control Board and Central Valley Regional Water Quality Control Board, August 10, 1990*
- *Analytical Methods for Petroleum Hydrocarbons, Washington State Department of Ecology, June 1997*
- *Compendium of Methods for the Determination of Air Pollutants in Indoor Air, (EPA 600/4-90-10, April 1990)*
- *Compendium of Methods for the Determination of Inorganic Compounds in Ambient Air, (EPA 625/R-96/010a, June 1999*
- *Methods for Determining Emissions of Toxic Air Contaminants from Stationary Sources, Stationary Source Test Methods, Volume 3, California Air Resources Board*
- *Leaking Underground Fuel Tank Guidance Manual, September 2012, California State Water Resources Control Board*

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 (DOC, Lab SOP # WS-QA-0022) is performed whenever there is a change in instrument type (e.g., new instrumentation), matrix, method or personnel (e.g., analyst hasn't performed the test within the last 12 months).

**Note:** The laboratory shall have a DOC for all analytes included in the methods that the laboratory performs, and proficiency DOCs for each analyst shall include all analytes that the laboratory routinely performs. Addition of non-routine analytes does not require new DOCs for all analysts if those analysts are already qualified for routine analytes tested using identical chemistry and instrument conditions.

The initial demonstration of capability must be thoroughly documented and approved by the Technical Manager and QA Manager prior to independently analyzing client samples. All associated documentation must be retained in accordance with the laboratories 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 client request is documented and the lab informs the client of its procedure for working with unusual compounds. The final report must be footnoted: *Reporting Limit based on the low standard of the calibration curve.*

### 19.4.3 Initial Demonstration of Capability (IDOC) Procedures

**19.4.3.1** The spiking standard used must be prepared independently from those used in instrument calibration.

**19.4.3.2** The analyte(s) shall be diluted in a volume of clean matrix sufficient to prepare four aliquots at the concentration specified by a method or the laboratory SOP. If the concentration is unspecified, the routine LCS spike level may be used.

**19.4.3.3** At least four aliquots shall be prepared (including any applicable clean-up procedures) and analyzed according to the test method (either concurrently or over a period of days).

**19.4.3.4** Using all of the results, calculate the mean recovery in the appropriate reporting units and the standard deviations for each parameter of interest.

**19.4.3.5** When it is not possible to determine the mean and standard deviations, such as for presence, absence and logarithmic values, the laboratory will assess performance against criteria described in the Method SOP.

**19.4.3.6** Compare the information obtained above to the corresponding acceptance criteria for precision and accuracy in the test method (if applicable) or in laboratory generated acceptance criteria (LCS or interim criteria) if there is no mandatory criteria established. If any one of the parameters do not meet the acceptance criteria, the performance is unacceptable for that parameter.

**19.4.3.7** When one or more of the tested parameters fail at least one of the acceptance criteria, the analyst must proceed according to either option listed below:

- Locate and correct the source of the problem and repeat the test for all parameters of interest beginning with 19.4.3.3 above.
- Beginning with 19.4.3.3 above, repeat the test for all parameters that failed to meet criteria. Repeated failure, however, will confirm a general problem with the measurement system. If this occurs, locate and correct the source of the problem and repeat the test for all compounds of interest beginning with 19.4.3.1 above.

Note: Results of successive LCS analyses can be used to fulfill the DOC requirement.

A certification statement (refer to Figure 19-1 as an example) shall be used to document the completion of each initial demonstration of capability. A copy of the certification is archived in the analyst's training folder.

Methods on line prior to the effective date of this Section shall be updated to the procedures outlined above as new analysts perform their demonstration of capability. A copy of the new record will replace that which was used for documentation in the past. At a minimum, the precision and accuracy of four mid-level laboratory control samples must have been compared to the laboratory's quality control acceptance limits.

In accordance with Arizona Administrative Code R9-14-616.5f, documentation of each analyst's performance of proficiency testing, as applicable, will be maintained in the training record.

## 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. Generally, the analyst prepares at least seven replicates of solution spiked at one to five times the estimated method detection limit (most often at the lowest standard in the calibration curve) into the applicable matrix with all the analytes of interest. Each of these aliquots is extracted (including any applicable clean-up procedures) and analyzed in the same manner as the samples. Where possible, the seven replicates should be analyzed over 2-4 days to provide a more realistic MDL.

Refer to the Corporate SOP No. CW-Q-S-006 or the laboratory's SOP No. WS-QA-0006 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 MDL but without sample preparation, or by the analysis of 10 instrument blanks and calculating 3 x the absolute value of the standard deviation.

If IDL is > than the MDL, it may be used as the reported MDL.

## 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. The analytes must be qualitatively identified or see SOP No. WS-QA-0006 for other options. If the MDL does not verify, then the lab will not report to the MDL, or redevelop their MDL or use the level where qualitative identification is established. MDLs must be verified at least annually.

For DoD ELAP certified methods, and methods utilized in support of DOE programs: Once the MDL is determined, it must be verified on each instrument used for the given method. TestAmerica defines the DoD/DOE QSM Detection Limit (DL) as being equal to the MDL. TestAmerica also defines the DoD/DOE QSM Limit of Detection (LOD) as being equal to the lowest concentration standard that successfully verifies the MDL, also referred to as the MDLV standard. MDL and MDLV standards are extracted/digested and analyzed through the entire analytical process. The MDL and MDLV determinations do 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 MDLV standard is not successful, then the laboratory will redevelop their MDL or perform and pass two consecutive MDLVs at a higher concentration and set the LOD at the higher concentration. Initial and quarterly verification is required for all methods listed in the laboratory's DoD ELAP Scope of Accreditation or utilized in support of DOE programs. Refer to the laboratory SOP WS-QA-0006, Method Detection Limits (MDL) and Instrument Detection Limits (IDL) for further details.

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.

For DoD ELAP certified methods and methods utilized in support of DOE programs: The laboratory quantitation limit is equivalent to the DoD/DOE Limit of Quantitation (LOQ), which is at a concentration equal to or greater than the lowest non-zero calibration standard. The DoD/DOE QSM requires the laboratory to perform an initial characterization of the bias and precision at the LOQ and quarterly LOQ verifications thereafter. If the quarterly verification results are not consistent with the three-standard deviation confidence limits established initially, then the bias and precision will be reevaluated and clients contacted for any on-going projects. For DoD/DOE projects, TestAmerica makes a distinction between the Reporting Limit (RL) and the LOQ. The RL is a level at or above the LOQ that is used for specific project reporting purposes, as agreed to between the laboratory and the client. The RL cannot be lower than the LOQ concentration, but may be higher.

## 19.10 Retention Time Windows

Most organic analyses and some inorganic analyses use chromatography techniques for qualitative and quantitative determinations. For every chromatography analysis or as specific in the reference method, each analyte will have a specific time of elution from the column to the detector. This is known as the analyte's retention time. The variance in the expected time of elution is defined as the retention time window. As the key to analyte identification in chromatography, retention time windows must be established on every column for every analyte used for that method. These records are kept with the files associated with an instrument for later quantitation of the analytes. Complete details are available in the laboratory SOPs.

## 19.11 Evaluation of Selectivity

The laboratory evaluates selectivity by following the checks within the applicable analytical methods, which include mass spectral tuning, second column confirmation, ICP interelement interference checks, chromatography retention time windows, sample blanks, spectrochemical, atomic absorption 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": 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 uncertainties at approximately the 99% confidence level with a coverage factor of k = 3. As an example, for a reported result of 1.0 mg/L with an LCS recovery range of 50 to 150%, the estimated uncertainty in the result would be 1.0 +/- 0.5 mg/L.

**19.12.5** In the case where a well recognized test method specifies limits to the values of major sources of uncertainty of measurement (e.g., 524.2, 525, etc.) and specifies the form of presentation of calculated results, no further discussion of uncertainty is required.

## 19.13 Sample Reanalysis Guidelines

Because there is a certain level of uncertainty with any analytical measurement, a sample 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 ± 1 reporting limit for samples ≤ 5x the reporting limit, the original analysis will be reported. At the client's request, both results may be reported on the same report but not on two separate reports.

- If the reanalysis does not agree (as defined above) with the original result, then the laboratory will investigate the discrepancy. If a problem is uncovered then the re-analysis will be repeated correctly. If no problem is uncovered then the laboratory will consult with the client to decide on actions needed.

- 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 Nos. CW-I-P-006, "Computer Systems Account and Naming Policy", CW-I-P-007, "Computer Systems Password Policy and CA-I-S-006, "Software Testing, Validation and Verification." The laboratory is currently running the TestAmerica Laboratory Information Management System ("TALS") which is a custom in-house developed LIMS system that has been highly customized to meet the needs of the laboratory. It is referred to as LIMS for the remainder of this section. The LIMS utilizes Sequel Server which is an industry standard relational database platform. It is referred to as Database for the remainder of this section.

**19.14.1.1** **Maintain the Database Integrity:** Assurance that data is reliable and accurate through data verification (review) procedures, password-protecting access, anti-virus protections, 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).

Manual integration of peaks will be documented and reviewed and the raw data will be flagged in accordance with the TestAmerica Corporate SOP No. CA-Q-S-002, *Acceptable Manual Integration Practices* and WS-PQA-011, Manual Integration Documentation Procedures.

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 (µg/L) for liquids and milligrams per kilogram (mg/kg) or micrograms per kilogram (µg/kg) for solids. For values greater than 10,000 mg/L, results can be reported in percent, i.e., 10,000 mg/L = 1**%.** Units are defined in each lab SOP.

**19.14.2.3** In reporting, the analyst or the instrument output records the raw data result using values of known certainty plus one uncertain digit. If final calculations are performed external to LIMS, the results should be entered in LIMS with at least three significant figures. In general, 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 may print a copy of what has been entered to check for errors. This printout and the instrument's data file of calibrations, concentrations, retention times, chromatograms, and mass spectra, if applicable, are retained within Chrom or the LIMS, based on the type of data.

### 19.14.3 Logbook / Worksheet Use Guidelines

Logbooks and worksheets are filled out 'real time' and have enough information on them to trace the events of the applicable analysis/task. (e.g. calibrations, standards, analyst, sample

ID, date, time on short holding time tests, temperatures when applicable, calculations are traceable, etc.)

- Corrections are made following the procedures outlined in Section 12.
- Logbooks are controlled by the QA department. A record is maintained of all logbooks in the lab.
- Unused portions of pages must be "Z'd" out, signed and dated.
- Worksheets are created with the approval of the Technical 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 several SOPs (WS-PQA-003, "Quality Control Program"***,*** WS-PQA-012, "Technical Data Review Requirements***",*** WS-PM-0004, "Final Report Assembly and Third Level Data Review") to ensure that reported data are free from calculation and transcription errors, that QC parameters have been reviewed and evaluated before data is reported. The laboratory also has an SOP discussing Manual Integrations to ensure the authenticity of the data (WS-PQA-0011, "Manual Integration Documentation and Practices"). The general review concepts are discussed below, more specific information can be found in the SOPs.

**19.14.4.1** Log-In Review - The data review process starts at the sample receipt stage. Sample control personnel review chain-of-custody forms and project instructions from the project management group. This is the basis of the sample information and analytical instructions entered into the LIMS. The log-in instructions are reviewed by the personnel entering the information, and a second level review is conducted by the project management staff.

**19.14.4.2** First Level Data Review - The next level of data review occurs with the analysts. As data are generated, analysts review their work to ensure that the results meet project and SOP requirements. First level reviews include inspection of all raw data (e.g., instrument output for continuous analyzers, chromatograms, spectra, and manual integrations), evaluation of calibration/calibration verification data in the day's analytical run, evaluation of QC data, and reliability of sample results. The analyst transfers data into LIMS, data qualifiers are added as needed. All first level reviews are documented.

**19.14.4.3** Second Level Data Review – All analytical data are subject to review by a second qualified analyst or supervisor. Second level reviews include inspection of all raw data (e.g., instrument output, chromatograms, and spectra) including 100% of data associated with any changes made by the primary analyst, such as manual integrations or reassignment of peaks to different analytes, or elimination of false negative analytes. The second review also includes evaluation of initial calibration/calibration verification data in the day's analytical run, evaluation of QC data, reliability of sample results, qualifiers and NCM narratives. Manual calculations are checked in second level review. All second level reviews are documented. 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

**19.14.4.4** Unacceptable analytical results may require reanalysis of the samples. Any problems are brought to the attention of the Laboratory Director, Project Manager, Quality Director/Manager, Technical Manager, or Supervisor for further investigation. Corrective action is initiated whenever necessary.

**19.14.4.5** The results are then entered or directly transferred into the computer database and a hard copy (or .pdf) is printed for the client.

**19.14.4.6** 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 the COC is followed, cover letters/ narratives are present, flags are appropriate, and project specific requirements are met. The project manager may also evaluate the validity of results for different test methods given expected chemical relationships.

**19.14.4.7** Any project that requires a data package is subject to a tertiary data review for transcription errors and acceptable quality control requirements. The Project Manager then signs the final report. The accounting personnel also check the report for any clerical or invoicing errors. When complete, the report is sent out to the client.

**19.14.4.8** 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 our internal SOP No. WS-PQA-0011, entitled “Manual Integration Documentation and Practices”.

**19.14.5.1** The analyst must adjust baseline or the area of a peak in some situations, for example when two compounds are not adequately resolved or when a peak shoulder needs to be separated from the peak of interest. The analyst must use professional judgment and common sense to determine when manual integrating is required. Analysts are encouraged to ask for assistance from a senior analyst or manager when in doubt.

**19.14.5.2** Analysts shall not increase or decrease peak areas for the sole purpose of achieving acceptable QC recoveries that would have otherwise been unacceptable. The intentional recording or reporting of incorrect information (or the intentional omission of correct information) is against company principles and policy and is grounds for immediate termination.

**19.14.5.3** Client samples, performance evaluation samples, and quality control samples are all treated equally when determining whether or not a peak area or baseline should be manually adjusted.

**19.14.5.4** All manual integrations receive a second level review. Manual integrations must be indicated on an expanded scale “after” chromatograms such that the integration performed can be easily evaluated during data review. Expanded scale “before” chromatograms are also required for all manual integrations on QC parameters (calibrations, calibration verifications, laboratory control samples, internal standards, surrogates, etc.) unless the laboratory has another documented corporate approved procedure in place that can demonstrate an active process for detection and deterrence of improper integration practices. Instrument operators must assure that all manual integration documentation identifies the analyst, the date and the reason for the integration.

**Figure 19-1. Example - Demonstration of Capability Documentation**

## Analyst Demonstration of Capability

TestAmerica Sacramento

Victoria Nihart

4/24/2017

| | |
|---|---|
| **Preparation Method(s):** | 3535 |
| **Analytical Method(s):** | GCMSMS_NDMA |
| **Matrix:** | Water |
| **Method Description:** | Nitrosamines by Isotope Dilution and GC/CI/MS/MS |
| **Preparation SOP No:** | WS-IDP-0020 Rev. 3.3 |
| **Analytical SOP No:** | WS-MS-0012 Rev. 2.1 |

We, the undersigned, CERTIFY that:

1. The analyst identified above, using the cited test method with the specifications in the cited SOP, which is in use at this facility for the analysis of samples under the laboratory's Quality Assurance Plan, has completed the Demonstration of Capability (DOC).
2. The test method(s) was performed by the analyst identified on this certificate.
3. A copy of test method(s) and laboratory SOPs are available for all personnel on-site.
4. The data associated with the demonstration of capability are true, accurate, complete and self-explanatory.
5. All raw data necessary to reconstruct and validate these analyses have been retained at the facility. The associated information is organized and available for review.

| | | |
|---|---|---|
| **Technical Director** | **Signature** | **Date** 5/3/17 |
| **Quality Assurance Officer** | **Signature** | **Date** 5/3/17 |

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## Analyst Demonstration of Capability

### ANALYST DEMONSTRATION OF CAPABILITY

**Method** GCMSMS_NDMA **Laboratory:** TestAmerica Sacramento
**Method Desc:** Nitrosamines by Isotope Dilution and GC/CI/MS/MS
**Analyst:** Victoria Nihart **Limit Group:** MSS - NDMA - Water - QC

**Current Limits** — Recovery / Precision
**Demonstration of Capability** — Recovery / Precision

**N-Nitrosodimethylamine**
All values within Control limits
**Analysis Dates:** 6/1/2016 to 6/3/2016

| LCL | UCL | Std Dev | Units |
|---|---|---|---|
| | | 20 | % |

| Mean | Std Dev | Units | | Amount | Amount/RL |
|---|---|---|---|---|---|
| 90.09 | 8.53983 | % | Pass | 2.0 | 1 |

| Laboratory ID | Anal Date | Batch | Smp | Analyst | Prep Analyst | Result | Units | Spike Amount | RL | % Rec | | In Rec Limits? |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| LLCS 320-110927/3-A | 06/01/2016 | 112005 | 17 | Nihart, Victoria M | Mantri, Anil | 1.618 | ng/L | 2.0 | 2.0 | 81 | | Pass |
| LLCSD 320-110927/4-A | 06/01/2016 | 112005 | 18 | Nihart, Victoria M | Mantri, Anil | 1.962 | ng/L | 2.0 | 2.0 | 98 | 19 | Pass |
| LLCS 320-111960/3-A | 06/03/2016 | 112484 | 4 | Nihart, Victoria M | Kuzmenko, Natalia | 1.931 | ng/L | 2.0 | 2.0 | 97 | | Pass |
| LLCSD 320-111960/4-A | 06/03/2016 | 112484 | 5 | Nihart, Victoria M | Kuzmenko, Natalia | 1.695 | ng/L | 2.0 | 2.0 | 85 | 13 | Pass |

**N-Nitrosodimethylamine-d6**
All values within Control limits
**Analysis Dates:** 6/1/2016 to 6/3/2016

| LCL | UCL | Std Dev | Units |
|---|---|---|---|
| 25 | 150 | | % |

| Mean | Std Dev | Units | | Amount |
|---|---|---|---|---|
| 75.77 | 7.50265 | % | Pass | 100.0 |

| Laboratory ID | Anal Date | Batch | Smp | Analyst | Prep Analyst | Result | Units | Spike Amount | % Rec | | In Rec Limits? |
|---|---|---|---|---|---|---|---|---|---|---|---|
| LLCS 320-110927/3-A | 06/01/2016 | 112005 | 17 | Nihart, Victoria M | Mantri, Anil | 80.88 | ng/L | 100.0 | 81 | | Pass |
| LLCSD 320-110927/4-A | 06/01/2016 | 112005 | 18 | Nihart, Victoria M | Mantri, Anil | 81.20 | ng/L | 100.0 | 81 | 0.4 | Pass |
| LLCS 320-111960/3-A | 06/03/2016 | 112484 | 4 | Nihart, Victoria M | Kuzmenko, Natalia | 65.13 | ng/L | 100.0 | 65 | | Pass |
| LLCSD 320-111960/4-A | 06/03/2016 | 112484 | 5 | Nihart, Victoria M | Kuzmenko, Natalia | 75.88 | ng/L | 100.0 | 76 | 15 | Pass |

Precision = standard deviation of percent recoveries of spiked control samples.

4/24/2017

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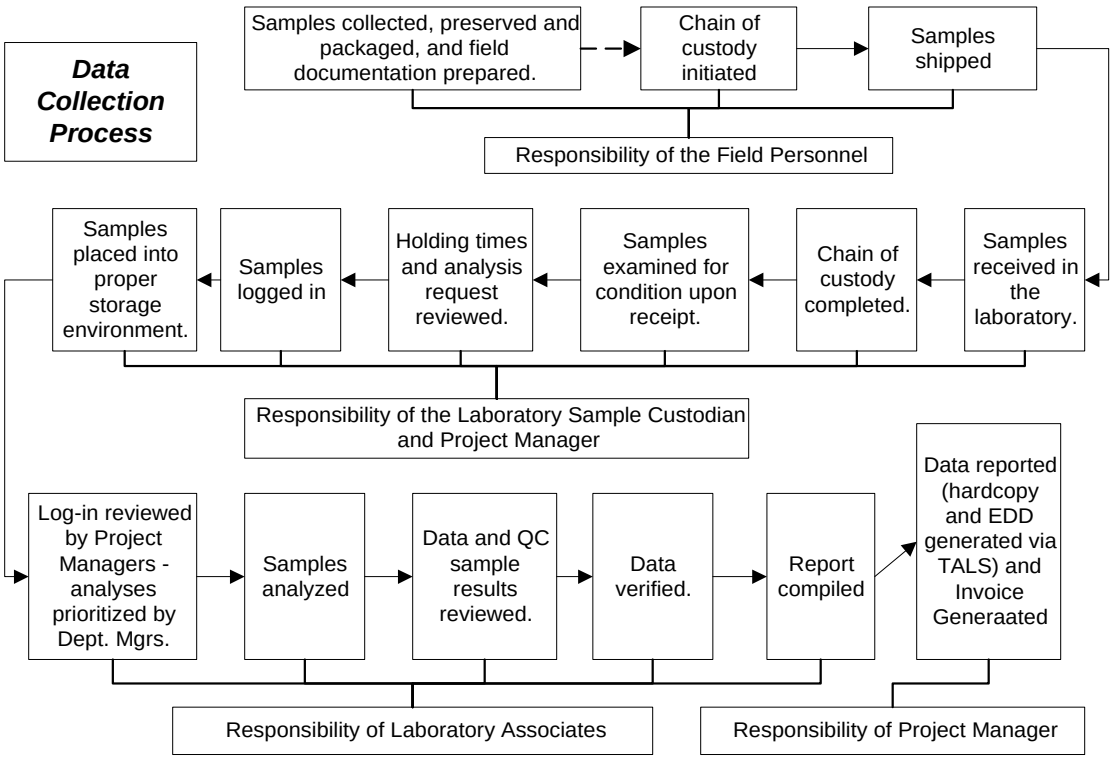
**Figure 19-2. Example: Work Flow**



# 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 laboratory SOPs. A list of laboratory instrumentation is presented in Table 20-1.

Equipment is only operated by authorized and trained personnel. Manufacturers' instructions for equipment use are readily accessible to all appropriate laboratory personnel.

## 20.2 Preventive Maintenance

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 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.

At a minimum, if an instrument is sent out for service or transferred to another facility, it must be recalibrated and the laboratory MDL verified (using an MDLv) 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, 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 an ISO 17025 qualified service representative, who supplies the laboratory with a certificate that identifies traceability of the calibration to the NIST standards.

All of this information is recorded in logs, and the recalibration/recertification certificates are kept on file. See SOP No. WS-QA-0041, "Calibration and Calibration Check of Balances" for more details.

### 20.3.2 pH, Conductivity, and Turbidity Meters

The pH meters used in the laboratory are accurate to ± 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 their logs.

Consult pH and Conductivity, and Turbidity SOPs for further information.

### 20.3.3 Thermometers

All thermometers are calibrated on an annual basis with a NIST-traceable thermometer at temperatures bracketing the range of use.

- If the temperature measuring device is used over a range of 10℃ or less, then a single point verification within the range of use is acceptable;
- If the temperature measuring device is used over a range of greater than 10℃, then the verification must bracket the range of use.

IR thermometers, digital probes and thermocouples are calibrated quarterly**.** IR Thermometers should be calibrated over the full range of use, including ambient, iced (4 degrees) and frozen (0 to -5 degrees), per the Drinking Water Manual.

The digital NIST thermometer is recalibrated every five years by an approved outside service and the provided certificate of traceability is kept on file. Alternately a new NIST thermometer with certificate of traceability from the manufacturer may be purchased. The NIST thermometer(s) have increments of 1 degree (0.5 degree or less increments are required for drinking water microbiological laboratories), and have ranges applicable to method and certification requirements. The NIST traceable thermometer is used for no other purpose than to calibrate other thermometers.

All of this information is documented in logbooks. Monitoring method-specific temperatures, including heating blocks, water baths, and ovens, is documented in method-specific logbooks. More information on this subject can be found in the SOP No. WS-QA-0016, "Thermometer Calibration."

### 20.3.4 Refrigerators/Freezer Units, Waterbaths, Ovens and Incubators

The temperatures of all refrigerator units and freezers used for sample storage are monitored 7 days a week; and each working day for units used for standard storage.

Ovens and water baths are monitored on days of use. Drying oven temperature must be recorded before and at the end of use. For example, an oven used for moisture determination must have its temperature recorded at the start and end of the drying process. Temperature must be ± 5% of set temperature for DoD/DOEwork.

All of this equipment has a unique identification number, and is assigned a unique thermometer for monitoring.

Sample storage refrigerator temperatures are kept at > 0ºC and ≤ 6 ºC.

Specific temperature settings/ranges for other refrigerators, ovens, and water baths can be found in method specific SOPs.

All of this information is documented in Daily Temperature Logbooks and method-specific logbooks.

### 20.3.5 Autopipettors, Dilutors, and Syringes

Mechanical volumetric dispensing devices including burettes (except Class A Glassware 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 applied to the device stating that it is not calibrated. Any device not regularly verified cannot be used for any quantitative measurements. See SOP WS-QA-0004, “Maintenance and Calibration Check of Fixed and Adjustable Volume Autopipettors, Autodispensers and Volumetric Containers”.

Micro-syringes are purchased from Hamilton Company. Each syringe is traceable to NIST. The laboratory keeps on file an “Accuracy and Precision Statement of Conformance” from Hamilton attesting established accuracy. The laboratory also assigns a unique ID# to each syringe. The delivery volume of each syringe is verified gravimetrically before initial use.

### 20.3.6 Autoclaves

Autoclaves used for sample digestion are capable of maintaining conditions of 15 psi at 120ºC for 15 minutes. The temperature of the autoclave is verified quarterly.

## 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 however, the annual requirement does not apply to Isotope Dilution methods.

### 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 and ICPMS methods which define the working range with periodic linear dynamic range studies, rather than through the range of concentrations of daily calibration 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). 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. 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 each daily as specified in the laboratory method SOPs in accordance with the referenced analytical methods and in the 2009 TNI Standard. The process of calibration verification applies to both external standard and internal standard calibration techniques, as well as to linear and non-linear calibration models. Initial calibration verification is with a standard source secondary (second source standard) to the calibration standards, but continuing calibration verifications may use the same source standards as the calibration curve.

**Note:** The process of calibration verification referred to 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.

**Note:** If an internal standard calibration is being used (basically GCMS) then bracketing standards are not required, only daily verifications are needed. (Exception: Some QC programs, such as the DoD/DOE QSM Version 5, require bracketing standards with internal standard calibration). The results from these verification standards must meet the calibration verification criteria and the retention time criteria (if applicable).

Generally, the initial calibrations must be verified at the beginning of each 12-hour analytical shift during which samples are analyzed. (Some methods may specify more or less frequent verifications). The 12-hour analytical shift begins with the injection of the calibration verification standard (or the MS tuning standard in MS methods). The shift ends after the completion of the analysis of the last sample, QC, or standard that can be injected within 12 hours of the beginning of the shift.

A continuing instrument calibration verification (CCV) must be repeated at the beginning and, for methods that have quantitation by external calibration models, at the end of each analytical batch. Some methods have more frequent CCV requirements - see specific SOPs. Most inorganic methods require the CCV to be analyzed after ever 10 samples or injections, including matrix or batch QC samples.

**Note:** If an internal standard calibration is being used (basically GCMS) then bracketing standards are not required, only daily verifications are needed. The results from these verification standards must meet the calibration verification criteria and the retention time criteria (if applicable).

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 unacceptable calibration verification may be fully useable under the following special conditions and reported based upon discussion and approval of the client:

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.

- When the acceptance criteria for the calibration verification are exceeded high, i.e., high bias, and there are associated samples that are non-detects, then those non-detects may be reported. Otherwise, the samples affected by the unacceptable calibration verification shall be reanalyzed after a new calibration curve has been established, evaluated and accepted.

- When the acceptance criteria for the calibration verification are exceeded low, i.e., low bias, those sample results may be reported if they exceed a maximum regulatory limit/decision

level. Otherwise, the samples affected by the unacceptable verification shall be reanalyzed after a new calibration curve has been established, evaluated and accepted. Alternatively, a reporting limit standard may be analyzed to demonstrate that the laboratory can still support non-detects at their reporting limit.

## 20.5 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. Guidelines for evaluating and reporting TICs are in the specific laboratory SOPs.

**Note:** If the TIC compound is not part of the client target analyte list but is calibrated by the laboratory and is 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. Example: Instrumentation List**

| Instrument Type | Number in Use |
|---|---|
| Autoanalyzer | 1 |
| Autotitrator | 1 |
| Cold-Vapor Analyzers | 1 |
| GC/HRMS | 6 |
| GC/MS - Semivolatiles | 6 |
| GC/MS - Volatiles | 5 |
| GC/MS – Volatile Air | 5 |
| GC/MS/MS | 1 |
| GC-ECD/ECD | 7 |
| GC-FID/FID | 2 |
| GC-FID | 1 |
| GC-FPD | 1 |
| GC-TCD/TCD | 1 |
| HPLC | 5 |
| HPLC/MS/MS | 5 |
| ICP | 1 |
| ICP/MS | 1 |
| Ion Chromatograph | 3 |
| Spectrometer | 1 |

**Table 20-2. Example: Schedule of Routine Maintenance**

| INSTRUMENT | MAINTENANCE | FREQUENCY |
|---|---|---|
| APCI/ESI LC/MS/MS | Change pump seals.<br>Change in-line filters in autosampler (HPLC).<br>Check/replace in-line frit if excessive pressure or poor performance.<br>Replace column if no change following in-line frit change.<br>Clean corona needle.<br>Replace sample inlet tube in APCI (10.1 cm).<br>Replace fused silica tube in ESI interface.<br>Clean lenses.<br>Clean skimmer.<br>Ballast rough pump 30 minutes. | As Needed |
| | Check solvent reservoirs for sufficient level of solvent.<br>Verify that pump is primed, operating pulse free.<br>Check needle wash reservoir for sufficient solvent.<br>Verify capillary heater temperature functioning.<br>Verify vaporizer heater temperature.<br>Verify rough pump oil levels.<br>Verify turbo-pump functioning.<br>Verify nitrogen pressure for auxiliary and sheath gasses.<br>Verify that corona and multiplier are functioning. | Daily(2) |
| | Replace rough-pump oil (4-6 months).<br>Replace oil mist and odor elements.<br>Replace activated alumina filter if applicable. | Semi-Annually |
| | Vacuum system components including fans and fan covers.<br>Clean/replace fan filters, if applicable. | Annually |
| HIGH PRESSURE LIQUID CHROMATOGRAPH(1) | Replace columns when peak shape and resolution indicate that chromatographic performance of column is below method requirements.<br>Rinse flow cell with 1N nitric acid if dirty flow cell.<br>Change pump seals when flow becomes inconsistent.<br>Backflush column if applicable.<br>Change in-line filters for solvents. | As Needed |
| | Check level of solution in reservoirs. If adding, verify that solvent is from the same source. If changing, rinse delivery lines to prevent contamination of the new solvent.<br>Check gas supply if applicable.<br>Flush with an appropriate solvent to remove all bubbles.<br>Pre-filter all samples. | Daily(2) |
| | Change pump seals. | Every 6-9 Months |

| INSTRUMENT | MAINTENANCE | FREQUENCY |
|---|---|---|
| GAS CHROMATOGRAPH(1) | Replace septum.<br>Clean injector port<br>Cut off front portion of capillary columns. Replace column if this fails to restore column performance or when column performance (e.g. peak tailing, poor resolution, high backgrounds, etc.) indicates it is required.<br>Change glass wool plug in injection port and/or replace injection port liner when front portion of capillary column is removed.<br>Replace or repair flow controller if constant gas flow cannot be maintained.<br>Detectors: clean when baseline indicates contamination or when response is low.<br>FID: clean/replace jet, replace igniter.<br>ECD: follow manufacturers suggested maintenance schedule<br>PID: Clean lamp window or replace. Replace seals.<br>Replace fuse.<br>Reactivate external carrier gas dryers.<br>HP 7673 Autosampler: replace syringe, fill wash bottle, dispose of waste bottle contents.<br>Check inlets, septa. | As Needed |
| | Check for sufficient supply of carrier and detector gases. Check for correct column flow and/or inlet pressures.<br>Check temperatures of injectors and detectors. Verify temperature programs.<br>Check baseline level.<br>Inspect chromatogram to verify symmetrical peak shape and adequate resolution between closely eluting peaks. | Daily(2) |
| | Oxidation and Reduction Catalysts: Perform leak checks. Replace/condition when poor response is observed. | Quarterly |
| | ECD: perform wipe test. | Semi-Annually |
| PURGE AND TRAP SYSTEMS | Change trap.<br>Check purge flow.<br>Flush lines (after foaming sample).<br>Periodic leak checks (when replace traps/spargers)<br>Replace/condition traps and/or spargers (when poor response or disappearance of reactive or poorly trapped compounds), clean sample lines, valves (if they become contaminated), and clean or replace glassware/spargers.<br>Bake trap as needed to correct for high background.<br>Change trap whenever loss of sensitivity, or erratic response or failing resolution is observed.<br>Purge & trap autosamplers: leak check system, clean sample lines, valves. | As Needed |
| | Bake out trap & analyze primers (as needed) prior to commencing analysis. | Daily(2) |
| GAS CHROMATOGRAPHY/LOW-RESOLUTION MASS SPECTROMETER(1) | Replace septum.<br>Clean injector port.<br>Cut off front portion of capillary columns. Replace column if this fails to restore column performance or when column performance (e.g. peak tailing, poor resolution, high backgrounds, etc.) indicates it is required.<br>Replace injection port liner when front portion of capillary column is removed.<br>Check level of oil in mechanical pumps and diffusion pump if vacuum is insufficient. Add oil if needed. | As Needed |

| INSTRUMENT | MAINTENANCE | FREQUENCY |
|---|---|---|
| | Replace electron multiplier when the tuning voltage approaches the maximum and/or when sensitivity falls below required levels.<br>Clean Source, including all ceramics and lenses - the source cleaning is indicated by a variety of symptoms including inability of the analyst to tune the instrument to specifications, poor response, and high background contamination.<br>Replace filaments when both filaments burn out or performance indicates need for replacement.<br>Check mass calibration (PFTBA or FC-43).<br>Check ion source and analyzer (clean, replace parts as needed).<br>Check vacuum, relays, gas pressures and flows.<br>Change oil in the mechanical rough pump.<br>Relubricate the turbomolecular pump-bearing wick.<br>HP 7673 Autosampler: Replace syringe. | |
| | Check for sufficient gas supply. Check for correct column flow and/or inlet pressure.<br>Check temperatures of injector, detector.<br>Verify temperature programs.<br>Check inlets, septa.<br>Check baseline level.<br>Check values of lens voltages, electron multiplier, and relative abundance and mass assignments of the calibration compounds.<br>Inspect chromatogram to verify symmetrical peak shape and adequate resolution between closely eluting peaks.<br>Autosampler: fill wash bottle, dispose of waste bottle contents.<br>Air Autosampler: Check for proper operation. Leak check system. | Daily(2) |
| | Replace the exhaust filters on the mechanical rough pump every 1-2 years. | Annually |
| GAS CHROMATOGRAPHY/HIGH-RESOLUTION MASS SPECTROMETER(1) | Full Bake-Out.<br>Change oil in rotary pump.<br>Change oil in diffusion pump. Replace o-rings.<br>Solvent rinse the flight tube.<br>Clean the first field free region.<br>Check detector voltages.<br>Clean and dust connectors, etc on the outside of the instrument.<br>Check the vacuum: ~5 x. $10^{-7}$ MBAR on both analyzer ion gauges, and ~5 x $10^{-6}$ MBAR on the source, with no helium flowing.<br>Check isolation valve for leaks, correct if needed.<br>Check for thermal trip by taking the magnet to maximum current, and verify that the coolant flow is acceptable.<br>Replace septum.<br>Clean injector port.<br>Cut off front portion of capillary columns. Replace column if this fails to restore column performance or when column performance (e.g. peak tailing, poor resolution, high backgrounds, etc.) indicates it is required.<br>Replace injection port liner when front portion of capillary column is removed.<br>Clean Source, including all ceramics and lenses - the source cleaning is indicated by a variety of symptoms | As Needed |

| INSTRUMENT | MAINTENANCE | FREQUENCY |
|---|---|---|
| | including inability of the analyst to tune the instrument to specifications, poor response, and high background contamination.<br>Replace filaments when performance indicates need for replacement. | |
| | Check resolution sensitivity.<br>Check stability.<br>Check for sufficient gas supply. Check for correct column flow and/or inlet pressure.<br>Check temperatures of injector, detector.<br>Verify temperature programs.<br>Check inlets, septa.<br>Check baseline level.<br>Check values of lens voltages, electron multiplier, and relative abundance and mass assignments of the calibration compounds.<br>Inspect chromatogram to verify symmetrical peak shape and adequate resolution between closely eluting peaks. | Daily(2) |
| COLD VAPOR ATOMIC ABSORPTION (LEEMAN PS 200) (1) | Change pump tubing.<br>Check/change Hg lamp.<br>Clean optical cell.<br>Change drying tube.<br>Grease pump. | As Needed |
| | Check sample tip for clogs.<br>Check drying tube.<br>Check pump tubing/drain tubing.<br>Check gas pressure.<br>Check liquid/gas separator.<br>Check tubing. | Daily(2) |
| INDUCTIVELY COUPLED ARGON PLASMA/MASS SPECTROMETRY (ICAP/MS)(1) | Check electronic settings for optimum sensitivity: resolution, mass calibration, ion optics.<br>Measure quartz torch for proper alignment when removed and cleaned.<br>Clean spray chamber and nebulizer.<br>Clean all filters and fans.<br>Check chiller coolant level.<br>Check and drain oil mist eliminator on roughing pumps. | As Needed |
| | Check sample waste container level.<br>Check quartz torch condition.<br>Check RF coil.<br>Check peristaltic pump: proper roller pressure, sample introduction tubing, correct pump rotation, condition of drain tubing.<br>Check condition of sampler and skimmer cones.<br>Check oil level of roughing pumps. | Daily(2) |
| | Replace oil in roughing pumps. | Every 2-3 Months |
| ICP(1) | Check that argon feed pressure is 80-120 psi.<br>Check that chiller coolant pressure is 45-80 psig, no leaks.<br>Check purge and shear gasses. Nitrogen purge gas pressure 40-120 psig, compressed air shear gas pressure 80-120 psig.<br>Check radial purge and axial windows for deposits.<br>Check that nebulizer is not clogged.<br>Check that capillary tubing is clean and in good condition.<br>Check that peristaltic pump windings are secure.<br>Check that exhaust vent is operational<br>Check that torch, glassware, aerosol injector tube are clean. | Daily(2) |

| INSTRUMENT | MAINTENANCE | FREQUENCY |
|---|---|---|
| | Clean plasma torch assembly to remove accumulated deposits.<br>Check RF coil.<br>Clean nebulizer and drain chamber; keep free flowing to maintain optimum performance.<br>Clean filters on back of power unit to remove dust.<br>Replace when needed:<br>peristaltic pump tubing.<br>sample capillary tubing.<br>autosampler sipper probe.<br>Check performance with manganese.<br>Check O-rings.<br>Clean/lubricate pump rollers | Monthly or As Needed |
| | Check chiller coolant filter. (may require more or less frequently) | Semi-Annually |
| | Notify manufacturer service engineer for scheduled preventive maintenance service. | Annually |
| ION CHROMATOGRAPH(1) | Clean micromembrane suppressor when decreases in sensitivity are observed.<br>Check fuses when power problems occur.<br>Change column when peak shape and resolution deteriorate or when retention time shortening indicates that exchange sites have become deactivated.<br>De-gas pump head when flow is erratic.<br>Check all air and liquid lines for discoloration and crimping, if indicated.<br>Check/change bed supports guard and analytical columns, if indicated. | As Needed |
| | Check plumbing/leaks.<br>Check eluent level.<br>Check gases.<br>Check pump pressure.<br>Check conductivity meter. | Daily(2) |
| | Check pump heads for leaks.<br>Check filter (inlet). | Weekly |
| | Change pump seals.<br>Change injection valve.<br>Clean conductivity cell.<br>Check conductivity cell for calibration. | Annually |
| ALPKEM COLORIMETRIC AUTO ANALYZER(1) | Prepare fresh reagents.<br>Replace tubing. (About every 100 hours of use) | As Needed |
| | Check detector. Make sure there are no trapped bubbles in detector cell.<br>Check Valves<br>Check peristaltic tubing.<br>Check sampler. | Daily(2) |
| | Clean pump, and XYZ Sampler. | Weekly |
| | Lubricate pump roller. | Monthly |
| | Clean pump rollers with steel wool and lubricate. | Semi-Annually |
| CHEMICAL OXYGEN DEMAND (COD) REACTOR(1) | Electronics serviced. | As Needed |
| | Check temperature with NIST reference thermometer. | Annually |
| AUTO TITRATOR(1) | Electronics serviced. | As Needed |
| | Calibrate with check standards. | Daily(2) (When Used) |

| INSTRUMENT | MAINTENANCE | FREQUENCY |
|---|---|---|
| | Inspect electrodes daily, clean as needed.<br>Inspect electrode proper levels of filling solutions daily, fill as needed.<br>Clean probe, each use.<br>Prime buret<br>Check rinse water reservoir. | |
| CONDUCTANCE METER(1) | Electronics serviced.<br>Replace batteries | As Needed |
| SPECTROPHOTOMETER(1) | Replace lamp.<br>Replace fuse. | As Needed |
| | Check instrument manual.<br>Perform wavelength calibration.<br>Replace lamp annually or when erratic response is observed. | Annually |
| PH METER(1) | Clean electrode.<br>Refill reference electrode. | As Needed |
| | Inspect electrode. Verify electrodes are properly connected and filled.<br>Inspect electrode proper levels of filling solutions.<br>Make sure electrode is stored in buffer. | Daily(2) |
| TURBIDIMETER(1) | Electronics serviced. | As Needed |
| | Clean instrument housing. | Monthly |
| DIGESTION BLOCK | Check temperature with NIST thermometer. | Annually |
| SONICATOR (1) | Replace probe tip.<br>Disassemble and clean sonicator probe tips.<br>Tune sonicator assembly (if recommended by manufacturer) | As Needed |
| | Inspect probe tips for inconsistencies (etching/pitting). | Daily(2) (When Used) |
| ANALYTICAL/TOP LOADING BALANCES(1) | Check using ASTM Class 3 weights once daily or before use.<br>Clean pan and weighing compartment. | Daily(2) |
| REFRIGERATORS/WALK-IN COOLERS(1) | Manufacturer cleaning and calibration. | Annually |
| | Refrigerant system and electronics serviced. | As Needed |
| | Temperatures checked and logged. | Daily(2) |
| OVENS(1) | Electronics serviced. | As Needed |
| | Temperatures checked and logged. | Daily(2) |
| ZYMARK PE WORKSTATION | Change O-rings whenever there are visible leaks or poor sealing on the SPE columns.<br>Sample lines are clean after samples have been extracted by SPE with a program “Clean Sample Lines” with methanol followed by water. Occasionally for a more rigorous cleaning, or after a highly contaminated sample, a mixture of methanol/DCM at 50:50 may be used in place of methanol, follow by methanol, then water (never use acetone).<br>Syringe pump may be primed using a program “Prime Solvent Lines” whenever air bubbles are suspected in the lines from running out of solvents and whenever solvents are changed.<br>Syringe pump in good condition – replace if showing signs of wear or suspected of poor performance.<br>Sample pumps may be re-calibrated whenever major | As Needed |

| INSTRUMENT | MAINTENANCE | FREQUENCY |
|---|---|---|
| | repairs are performed, or whenever the pumps are suspected to be out of calibration. Follow manufacturer's procedure for re-calibrating the sample pumps. For method 8330, the pump loads 1050 mL of sample on the SPE. It should used up the whole sample bottle (quart bottles and 1-L bottles). | |
| SONICATION WATER BATH(1) | If the water bath is dirty, empty and refill with tap water. A couple drops of anti-bacterial solution may be added to inhibit the growth of bacteria in the water.<br>The water level in the sonication batch should be about 1.2 to 1 inch from the top while in operation. Do not allow sonication batch to operate with water bath at lower levels. If the level is low, add more water, if the levels is too high, remove water to the proper level. | As Needed |

***Footnotes to Preventive Maintenance Tables***

(1) Refer to manufacturer's instructions for each instrument to identify and perform maintenance operations.

(2) Daily checks and verifications are performed prior to instrument startup and are not documented in maintenance logs unless problems are noted.

(3) Where there are differences between this table and the tables present in method SOPs, the table in the method SOP should be followed.

## 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) or another accreditation organization that is a signatory to a MRA (Mutual Recognition Arrangement) of one or more of the following cooperations – ILAC (International al Laboratory Accreditation Cooperation) or APLC (Asia-Pacific Laboratory Accreditation Cooperation). A calibration certificate and scope of accreditation is kept on file at the laboratory. Refer to Section 21 for calibration of weights and thermometers.

The calibration laboratory's policy for achieving measurement traceability is defined and includes the subsequent elements of uncertainty.

The uncertainty calculations of the calibration laboratory are supported by uncertainty budgets and are represented by expanded uncertainties typically using a coverage factor of k=2 to approximate the 95% confidence level. This explanation accompanies the measurement result and the associated uncertainty.

The tolerance uncertainty ratio (TUR) is calculated using the expanded uncertainty of the measurement, not the collective uncertainty of the measurement standards. A statement to this effect accompanies the TUR along with the coverage factor and confidence level.

The calibration report or certificate submitted to TestAmerica Sacramento contains, in a well designed format, a traceability statement, the conditions under which the calibrations were made in the context of any potential influence, a compliance statement with an identified metrological specification and the pertinent clauses, a clearly identified record of the quantities and functional test results before and after re-calibration, and no recommendation on the calibration interval. Opinions and interpretations of results are presented along with the basis

upon which they were made and identified as such. The report may be submitted by facsimile or other electronic means as long as the requirements of the International Standard are achieved. If significant amendments are made to a calibration certificate, a supplemental certificate for the serial-number-specified piece of equipment is so identified. When a new certificate is offered, it uniquely identifies and references the one it replaces. All calibration reports are filed in the QA Office.

The calibration laboratory supports in-house calibration systems: documented procedures for in-house calibrations, evidence by a report, certificate, or sticker, for an appropriate amount of time; training records of calibration personnel; certificates from accreditation services demonstrating traceability to national or international standards of measurement; procedures for evaluating measurement uncertainty; timely and documented recalibration of reference standards. When subcontracting to a calibration laboratory, TestAmerica Sacramento does not use a firm who subcontracts the work.

An external certified service engineer services laboratory balances on an annual basis. This service is documented on each balance with a signed and dated certification sticker. Balance calibrations are checked each day of use. All mercury thermometers are calibrated annually against a traceable reference thermometer. Temperature readings of ovens, refrigerators, and incubators are checked on each day of use.

### 21.3 Reference Standards / Materials

Reference standards/materials, where commercially available, are traceable to certified reference materials. Commercially prepared reference standards are purchased from vendors that are accredited to ISO Guide 34 and ISO/IEC Guide 17025. All reference standards from commercial vendors shall be accompanied with a certificate that includes at least the following information:

- Manufacturer
- Analytes or parameters calibrated
- Identification or lot number
- Calibration method
- Concentration with associated uncertainties
- Purity

.

If a standard cannot be purchased from a vendor that supplies a Certificate of Analysis, the purity of the standard is documented by analysis. The receipt of all reference standards must be documented. Reference standards are labeled with a unique Standard Identification Number and expiration date. All documentation received with the reference standard is retained as a QC record and references the Standard Identification Number.

All reference, primary and working standards/materials, whether commercially purchased or laboratory prepared, must be checked regularly to ensure that the variability of the standard or material from the ‘true’ value does not exceed method requirements. The accuracy of calibration standards is checked by comparison with a standard from a second source. In cases where a second standard manufacturer is not available, a vendor certified different lot is acceptable for use as a second source. For unique situations, such as air analysis where no other source or lot is available, a standard made by a different analyst would be considered a

second source. The appropriate Quality Control (QC) criteria for specific standards are defined in laboratory SOPs. In most cases, the analysis of an Initial Calibration Verification (ICV) or LCS (where there is no sample preparation) is used as the second source confirmation. These checks are generally performed as an integral part of the analysis method (e.g. calibration checks, laboratory control samples).

All standards and materials must be stored and handled according to method or manufacturer's requirements in order to prevent contamination or deterioration. Refer to the Corporate Environmental Health & Safety Manual or laboratory SOPs. For safety requirements, please refer to method SOPs and the laboratory Environmental Health and Safety Manual.

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 Certificate of Analysis or Purity must be retained, stored appropriately, and readily available for use and inspection. These records are scanned and retained on the local server. Records must be kept of the date of receipt and date of expiration of standards, reagents and reference materials. In addition, records of preparation of laboratory standards, reagents, and reference materials must be retained, stored appropriately, and be readily available for use and inspection. For detailed information on documentation and labeling, please refer to method specific SOPs and SOP No. WS-QA-0017, "Standards and Reagents and Quality Control Check Procedures".

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 (for 1613B dioxin/furan analyses the purity must be 98% or corrections must be made). Blended gas standard cylinders use a nominal concentration if the certified value is within +/-15%, otherwise the certified values is used for the canister concentration.

**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 or standards logbook.

- Standard ID
- Description of Standard

- Department
- Preparer's name
- Final volume and number of vials prepared
- Solvent type and lot number
- Preparation Date
- Expiration Date
- Standard source type (stock or daughter)
- Standard type (spike, surrogate, other)
- Parent standard ID (if applicable)
- Parent Standard Analyte Concentration (if applicable)
- Parent Standard Amount used (if applicable)
- Component Analytes
- Final concentration of each analyte
- Comment box (text field)

Records are maintained electronically or in logbooks for standard and reference material preparation. These records show the traceability to purchased stocks or neat compounds. These records also include method of preparation, date of preparation, expiration date and preparer's name or initials. Preparation procedures are provided in the Method SOPs.

**21.4.2** All standards, reagents, and reference materials must be clearly labeled with a minimum of the following information:

- Expiration Date (include prep date for reagents)
- Standard ID (from the preparation logbook)
- 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 in the SDS section of OASIS.

**21.4.3** In addition, the following information may be helpful:

- 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 preparation/analytical batch records.

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 does not provide sampling services. The laboratory's responsibility in the sample collection process lies in supplying the sampler with the necessary coolers, reagent water, sample containers, preservatives, sample labels, custody seals, COC forms, ice, and packing materials required to properly preserve, pack, and ship samples to the laboratory

## 22.2 Sampling Containers

The laboratory offers clean sampling containers for use by clients. These containers are obtained from reputable container manufacturers and meet EPA specifications as required. Certificates of cleanliness for bottles and preservatives are provided by the supplier and are maintained at the laboratory. Alternatively, the certificate may be maintained by the supplier and available to the laboratory on-line.

### 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:

- Hydrochloric Acid – Reagent ACS (Certified VOA Free) or equivalent
- Methanol – Purge and Trap grade
- Nitric Acid – Instra-Analyzed or equivalent
- Sodium Bisulfate – ACS Grade or equivalent
- Sodium Hydroxide – Instra-Analyzed or equivalent
- Sulfuric Acid – Instra-Analyzed or equivalent
- Sodium Thiosulfate – ACS Grade or equivalent

## 22.3 Definition of Holding Time

The date and time of sampling documented on the COC form establishes the day and time zero. As a general rule, when the maximum allowable holding time is expressed in "days" (e.g., 14 days, 28 days), the holding time is based on calendar day measured. Holding times expressed in "hours" (e.g., 6 hours, 24 hours, etc.) are measured from date and time zero. Holding times for analysis include any necessary reanalysis. However, there are some programs and regulators, which determine holding time compliance based on the date and specific time of analysis compared to the time of sampling regardless of how long the holding time is.

## 22.4 Sampling Containers, Preservation Requirements, Holding Times

The preservation and holding time criteria specified in the laboratory SOPs are derived from the source documents for the methods. 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 subsample or aliquot of the sample provided for analysis.

Analysts should handle each sample as if it is potentially dangerous. At a minimum, safety glasses, gloves, and lab coats must be worn when preparing aliquots for analysis.

Guidelines on taking sample aliquots & subsampling are located SOP Nos. WS-QA-0018, “Subsampling and Compositing of Samples (Method ASTM D 6323-98**)**” and WS-QA-0028, “Incremental Sampling Methodology of Soils and Sediments”.

## 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.

### 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 log-in by date; it lists all receipts each date.

### 23.1.2 Legal / Evidentiary Chain-of-Custody

If samples are identified for legal/evidentiary purposes on the COC, legal COCs will be generated per the Manual for Certification of Laboratories Analyzing Drinking Water, Fifth Edition, January 2005, Appendix A, and SOP No. WS-QA-0003, "Sample Receipt and Procedures".

## 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 and in SOP No. WS-QA-0003, "Sample Receipt and Procedures".

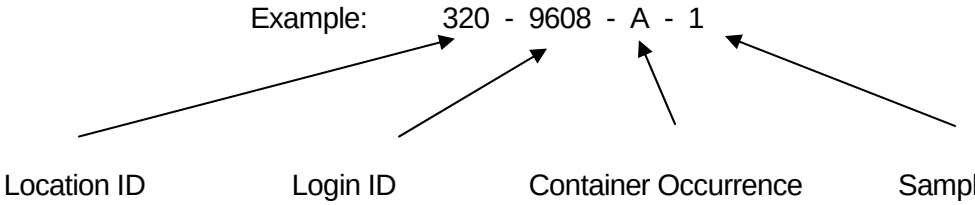
### 23.2.1 Laboratory Receipt

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 the lot receipt checklist and within the non-conformance program 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. Laboratory receipt procedures are described in more detail in SOP No. WS-QA-0003.

#### 23.2.1.1 Unique Sample Identification

All samples that are processed through the laboratory receive a unique sample identification to ensure that there can be no confusion regarding the identity of such samples at anytime. This system includes identification for all samples, subsamples and subsequent extracts and/or digestates.

The laboratory assigns a unique identification (e.g., Sample ID) code to each sample container received at the laboratory. This Primary ID is made up of the following information (consisting of 4 components):




The above example states that TestAmerica Sacramento Laboratory (Location 320) is the receiving laboratory. Login ID is 9608 (unique to a particular client/job occurrence). The container code indicates it is the first container ("A") of Sample #1.

If the primary container goes through a prep step that creates a "new" container, then the new container is considered secondary and gets another ID. An example of this being a client sample in a 1-Liter amber bottle is sent through a Liquid/Liquid Extraction and an extraction vial is created from this step. The vial would be a SECONDARY container. The secondary ID has 5 components.

Example: 320 - 9608 - A - 1 - **A** ←——**Secondary Container Occurrence**

Example: 320-9608-A-1-A would indicate the PRIMARY container listed above that went through a step that created the 1st occurrence of a Secondary container.

With this system, a client sample can literally be tracked throughout the laboratory in every step from receipt to disposal.

## 23.3 Sample Acceptance Policy

The laboratory has a written sample acceptance policy (Figure 23-2) that clearly outlines the circumstances under which samples shall be accepted or rejected. These include:

- a COC filled out completely;
- samples must be properly labeled;
- proper sample containers with adequate volume for the analysis (Sampling Guide) and necessary QC;
- samples must be preserved according to the requirements of the requested analytical method (Sampling Guide);
- sample holding times must be adhered to (Sampling Guide);
- the project manager will be notified if any sample is received in damaged condition.

Data from samples which do not meet these criteria are flagged and the nature of the variation from policy is defined.

**23.3.1** After inspecting the samples, the sample receiving personnel sign and date the COC form, make any necessary notes of the samples' conditions and store them in appropriate refrigerators or storage locations.

**23.3.2** Any deviations from these checks 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.

| Note: North Carolina requires that they be notified when samples are processed that do not meet sample acceptance criteria. |
|---|

Once sample acceptance is verified, the samples are logged into the LIMS according SOP No. WS-QA-0003.

## 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. In addition, samples to be analyzed for volatile organic parameters are stored in separate refrigerators designated for volatile organic parameters only. Samples are never to be stored with reagents, standards or materials that may create contamination.

To ensure the integrity of the samples during storage, refrigerator blanks are maintained in the volatile sample refrigerators and analyzed every two weeks.

Analysts and technicians retrieve the sample container allocated to their analysis from the designated refrigerator and place them on carts, analyze the sample, and return the remaining sample or empty container to the refrigerator from which it originally came. All unused portions of samples are returned to the secure sample control area. Empty sample containers are marked as “DIT” (destroyed in testing) on the sample receiving check out form and are disposed by the analytical staff. All samples are kept in the refrigerators for 30 days past invoicing, unless other arrangements have been made with the client.

Access to the laboratory is controlled such that sample storage need not be locked at all times unless a project specifically demands it. Samples are accessible to laboratory personnel only. Visitors to the laboratory are prohibited from entering the refrigerator and laboratory areas unless accompanied by an employee of TestAmerica.

## 23.5 Hazardous Samples and Foreign Soils

Foreign soil samples are sent out for incineration by a USDA-approved waste disposal facility.

## 23.6 Sample Shipping

In the event that the laboratory needs to ship samples, the samples are placed in a cooler with enough ice to ensure the samples remain just above freezing and at or below 6.0°C during transit. The samples are carefully surrounded by packing material to avoid breakage (yet maintain appropriate temperature). 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.

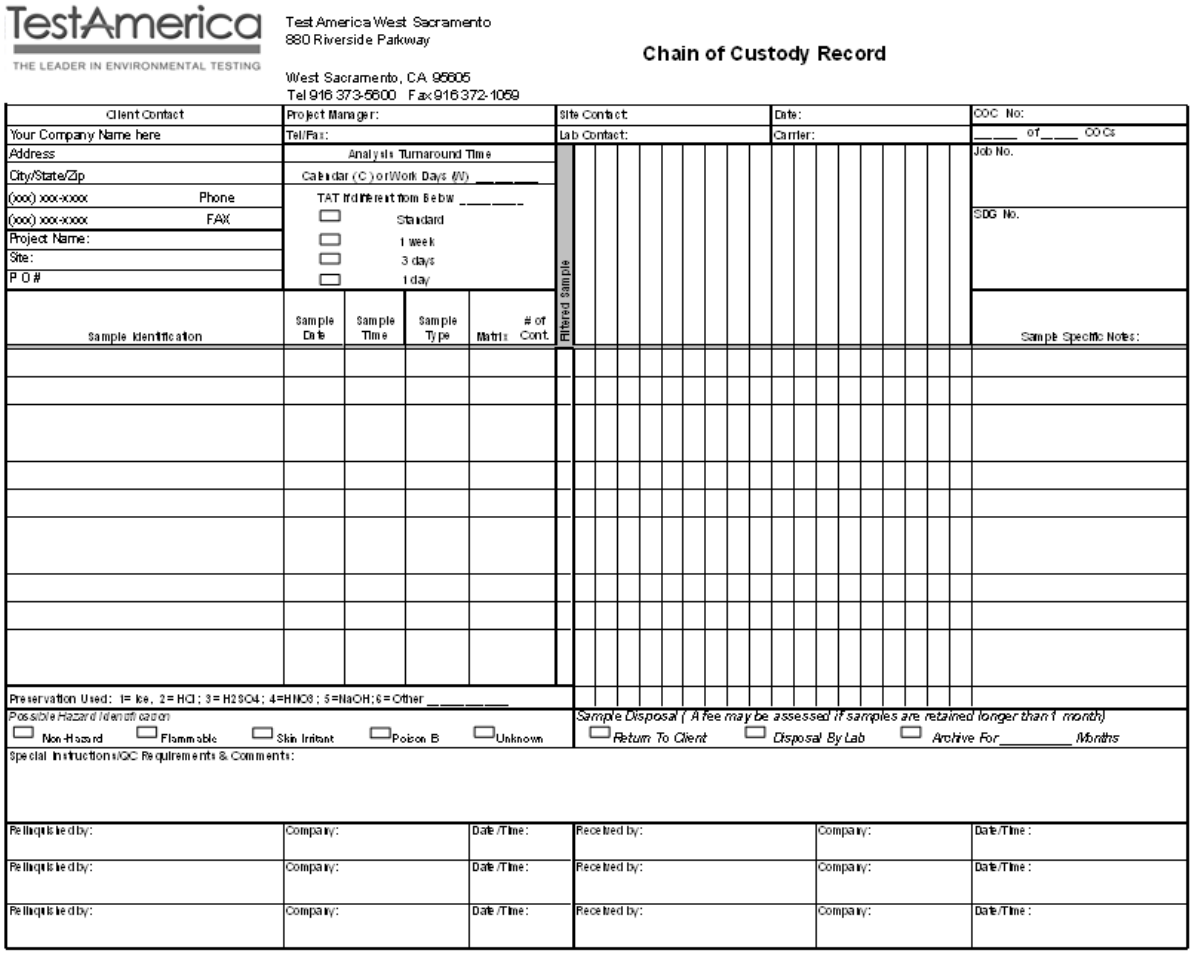
**Note:** If a client does not request trip blank analysis on the COC or other paperwork, the laboratory will not analyze the trip blanks that were supplied. However, in the interest of good client service, the laboratory will advise the client at the time of sample receipt that it was noted that they did not request analysis of the trip blank; and that the laboratory is providing the notification to verify that they are not inadvertently omitting a key part of regulatory compliance testing.

## 23.7 Sample Disposal

Samples should be retained for a minimum of 30 days after the project report is sent, however, provisions may be made for earlier disposal of samples once the holding time is exceeded. An exception is samples contained in laboratory-owned air sample canisters. These are held for a minimum of 24 hours after the project report is sent, prior to evacuating the canister and returning it to the equipment pool. 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: WS-EHS-001, "Waste Disposal"). 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.

All documentation and correspondence concerning the disposal decision process must be kept on file. Pertinent information includes the date of disposal, nature of disposal (such as sample depletion, hazardous waste facility disposal, return to client), names of individuals who conducted the arrangements and physically completed the task. The laboratory will remove or deface sample labels prior to disposal unless this is accomplished through the disposal method (e.g., samples are incinerated). A Waste Disposal Record should be completed.

**Figure 23-1. Example: Chain of Custody (COC)**



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West Sacramento, CA 95605
Tel 916 373-5600 Fax 916 372-1059

**Chain of Custody Record**

| Client Contact | Project Manager: | Site Contact: | Date: | COC No: |
|---|---|---|---|---|
| Your Company Name here | Tel/Fax: | Lab Contact: | Carrier: | ____ of ____ COCs |
| Address | Analysis Turnaround Time | | | Job No. |
| City/State/Zip | Calendar (C) or Work Days (W) _____ | | | |
| (xxx) xxx-xxxx Phone | TAT if different from Below _____ | | | SDG No. |
| (xxx) xxx-xxxx FAX | ☐ Standard | | | |
| Project Name: | ☐ 1 week | | | |
| Site: | ☐ 3 days | | | |
| P O # | ☐ 1 day | | | |

| Sample Identification | Sample Date | Sample Time | Sample Type | # of Cont. / Matrix | Filtered Sample | Sample Specific Notes: |
|---|---|---|---|---|---|---|
| | | | | | | |
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Preservation Used: 1= Ice, 2= HCl; 3= H2SO4; 4=HNO3; 5=NaOH; 6= Other _____

*Possible Hazard Identification*
☐ Non-Hazard ☐ Flammable ☐ Skin Irritant ☐ Poison B ☐ Unknown

*Sample Disposal ( A fee may be assessed if samples are retained longer than 1 month)*
☐ *Return To Client* ☐ *Disposal By Lab* ☐ *Archive For _____ Months*

Special Instructions/QC Requirements & Comments:

| Relinquished by: | Company: | Date/Time: | Received by: | Company: | Date/Time: |
|---|---|---|---|---|---|
| | | | | | |
| Relinquished by: | Company: | Date/Time: | Received by: | Company: | Date/Time: |
| | | | | | |
| Relinquished by: | Company: | Date/Time: | Received by: | Company: | Date/Time: |
| | | | | | |

**Figure 23-2. Example: Sample Acceptance Policy**

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SACRAMENTO LABORATORY
SAMPLE ACCEPTANCE POLICY
*(Effective 02/02/2015)*

Page 1 of 2

The TNI Standard and TestAmerica Sacramento have specific requirements under which all samples will be received by the laboratory for analysis. TestAmerica Sacramento will review your sample shipment against those requirements as listed below, and will communicate any discrepancies to you. Your project manager will assist you in the appropriate resolution of any issues related to sample receipt. Please contact your project manager with any questions.

VOA vials should be stored in controlled conditions. Exposure of trip blanks to temperature fluctuations is likely to cause development of bubbles in the trip blanks.

When completing the chain of custody form, please note that you must sign your name in the "relinquished by" box.

Requirements are as follows:

- Proper, full and complete documentation, which includes sample identification, the location, date and time of collection, the collector's name, the preservation type, the sample matrix type, the requested testing method, and any special remarks concerning the samples, shall be provided.
- Samples must be accompanied by written disclosure of the known or suspected presence of any hazardous substances, as defined by applicable federal or state law.
- Per State and/or Federal Regulation, the client is responsible to ensure that samples are shipped in accordance with DOT/IATA requirements, and that radioactive materials may only be delivered to licensed facilities. Any samples containing (or suspected to contain) Source, Byproduct, or Special Nuclear Material as defined by 10 CFR should be delivered directly to facilities licensed to handle such radioactive material. Natural material or ores containing naturally occurring radionuclides may be delivered to any TestAmerica facility or courier as long as the activity concentration of the material does not exceed 270 pCi/g alpha or 2700 pCi/g beta (49 CFR Part 173).
- Each sample shall be collected in the appropriate sample container and labeled with unique, durable and indelible identification.
- Drinking water samples for Method 1613B that may have residual chlorine must be checked and treated in the field, or collected in sodium thiosulfate preserved containers.
- Containers of water meant for perchlorate analysis should have adequate headspace to prevent anaerobic microbial degradation. A void approximately 1/3 of the container volume is sufficient.
- The samples shall arrive at the laboratory with adequate remaining holding time for the analyses requested.
- Sufficient sample volume must be available to perform the requested analyses.
- Received samples must not exhibit obvious signs of damage, contamination or inadequate preservation.
- Most analytical methods require chilling samples to 4° C (other than water samples for metals analysis). For these methods, the criteria are met if the samples are chilled to below 6° C and above freezing (0°C). For methods with other temperature criteria (e.g. some bacteriological methods require ≤ 10 °C), the samples must arrive within ± 2° C of the required temperature or within the method specified range.

    1. Samples that are delivered to the laboratory on the same day they are collected may not meet the requirements above. In these cases, the samples shall be considered acceptable if the samples were received on ice.
    2. If sample analysis is begun within fifteen (15) minutes of collection, thermal preservation is not required.
    3. Thermal preservation is not required in the field if the laboratory receives and refrigerates the sample within fifteen (15) minutes of collection.

- Chemical preservation (pH) will be verified prior to analysis and documented, either in sample control or at the analyst's level. 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.

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TestAmerica
THE LEADER IN ENVIRONMENTAL TESTING

## SACRAMENTO LABORATORY
## SAMPLE ACCEPTANCE POLICY

*(Effective 02/02/2015)*



- For samples undergoing chemical warfare degradate analysis, the sample must be screened for agent prior to shipment in accordance with appendix 10 of our Sample Receipt Procedure (WS-QA-0003).
- Samples containing mammalian tissue will not be accepted without prior coordination with a project manager. Additional conditions for receipt and handling of tissue are outlined in appendix 11 of our Sample Receipt Procedure (WS-QA-0003).
- Air canisters (SUMMA® and other brands) have additional requirements:
    - Never write or affix a label directly on a canister. A special tag is attached to each canister for this purpose.
    - Complete the Canister Field Data Record with the initial and final vacuum/pressure reading for each canister during sampling.
    - Close all valves completely prior to shipping or transporting.
    - Return canisters, filters, flow controllers, vacuum flow regulators, and any other supplied equipment must be returned even if they were not used. Pack equipment carefully to minimize in-transit damage Sampling equipment that is damaged, lost or not returned will be invoiced to the client at the replacement cost. Delayed return of equipment to the laboratory may result in additional rental charges.
    - Do not attempt to adjust or alter any equipment, as it may result in loss of sample integrity as well as equipment damage that may be invoiced to the client.

The laboratory will notify the client/Project Manager upon sample receipt if the samples fail to meet any of the above requirements.

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**Figure 23-3. Example: Cooler Receipt Form**

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Sacramento
Sample Receiving Notes

Job:_____

Tracking #_____

Use this form to record Sample Custody Seal, Cooler Custody Seal, Temperature & corrected Temperature & other observations. File in the job folder with the COC.

Notes:_____

Therm. ID: **AK-1** / **AK-2** / **HACCP** /Other_____

Ice_____ Wet_____ Dry_____ Other_____

Cooler Custody Seal: _____

Sample Custody Seal: _____

Cooler ID:_____

Temp: Observed_____

Corrected: _____

From: Temp Blank ☐ Sample ☐

NCM Filed: Yes ☐ No ☐

| | Yes | No | NA |
|---|---|---|---|
| Perchlorate has headspace? | ☐ | ☐ | ☐ |
| CoC is complete w/o discrepancies? | ☐ | ☐ | ☐ |
| Samples received within holding time? | ☐ | ☐ | ☐ |
| Sample preservatives verified? | ☐ | ☐ | ☐ |
| Cooler compromised/tampered with? | ☐ | ☐ | ☐ |
| Samples compromised/tampered with? | ☐ | ☐ | ☐ |
| COC and Samples w/o discrepancies? | ☐ | ☐ | ☐ |
| Sample containers have legible labels? | ☐ | ☐ | ☐ |
| Containers are not broken or leaking? | ☐ | ☐ | ☐ |
| Sample date/times are provided. | ☐ | ☐ | ☐ |
| Appropriate containers are used? | ☐ | ☐ | ☐ |
| Sample bottles are completely filled? | ☐ | ☐ | ☐ |
| Zero headspace?* | ☐ | ☐ | ☐ |
| Multiphasic samples are not present? | ☐ | ☐ | ☐ |

Initials:_____ Date:_____

*Containers requiring zero headspace have no headspace, or bubble < 6 mm (1/4")

*Q:\DOCUMENT-MANAGEMENT\FORMS\QA-812 REV. 1.3 SAMPLE RECEIVING NOTES 2017-02-27.DOC QA-812 RKE 02/27/2017*

TestAmerica
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# Bottle Lot Inventory

Lot ID:_____

| | 1 | 2 | 3 | 4 | 5 | 6 | 7 | 8 | 9 | 10 | 11 | 12 | 13 | 14 | 15 | 16 | 17 | 18 | 19 | 20 |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| VOA* | | | | | | | | | | | | | | | | | | | | |
| VOAh* | | | | | | | | | | | | | | | | | | | | |
| AGB | | | | | | | | | | | | | | | | | | | | |
| AGBs | | | | | | | | | | | | | | | | | | | | |
| 250AGB | | | | | | | | | | | | | | | | | | | | |
| 250AGBs | | | | | | | | | | | | | | | | | | | | |
| 250AGBn | | | | | | | | | | | | | | | | | | | | |
| 500AGB | | | | | | | | | | | | | | | | | | | | |
| ____AGJ | | | | | | | | | | | | | | | | | | | | |
| 500AGJ | | | | | | | | | | | | | | | | | | | | |
| 250AGJ | | | | | | | | | | | | | | | | | | | | |
| 125AGJ | | | | | | | | | | | | | | | | | | | | |
| ____CGJ | | | | | | | | | | | | | | | | | | | | |
| 500CGJ | | | | | | | | | | | | | | | | | | | | |
| 250CGJ | | | | | | | | | | | | | | | | | | | | |
| 125CGJ | | | | | | | | | | | | | | | | | | | | |
| PJ | | | | | | | | | | | | | | | | | | | | |
| PJn | | | | | | | | | | | | | | | | | | | | |
| 500PJ | | | | | | | | | | | | | | | | | | | | |
| 500PJn | | | | | | | | | | | | | | | | | | | | |
| 500PJna | | | | | | | | | | | | | | | | | | | | |
| 500PJzn/na | | | | | | | | | | | | | | | | | | | | |
| 250PJ | | | | | | | | | | | | | | | | | | | | |
| 250PJn | | | | | | | | | | | | | | | | | | | | |
| 250PJna | | | | | | | | | | | | | | | | | | | | |
| 250PJzn/na | | | | | | | | | | | | | | | | | | | | |
| Acetate Tube | | | | | | | | | | | | | | | | | | | | |
| _____"CT | | | | | | | | | | | | | | | | | | | | |
| Encore | | | | | | | | | | | | | | | | | | | | |
| Folder/filter | | | | | | | | | | | | | | | | | | | | |
| PUF | | | | | | | | | | | | | | | | | | | | |
| Petri/Filter | | | | | | | | | | | | | | | | | | | | |
| XAD Trap | | | | | | | | | | | | | | | | | | | | |
| Ziploc | | | | | | | | | | | | | | | | | | | | |
| | | | | | | | | | | | | | | | | | | | | |
| | 1 | 2 | 3 | 4 | 5 | 6 | 7 | 8 | 9 | 10 | 11 | 12 | 13 | 14 | 15 | 16 | 17 | 18 | 19 | 20 |

h = hydrochloric acid  s = sulfuric acid  na = sodium hydroxide  n = nitric acid  zn = zinc acetate

Number of VOAs with air bubbles present / total number of VOA's

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# 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 measurements (e.g. Blanks, Laboratory Control Samples (LCS), Matrix Spikes (MS), duplicates (DUP), surrogates, Internal Standards (IS)). These quality control checks are performed as required by the method or regulations to assess precision and accuracy. In addition to the routine process quality control samples, Proficiency Testing (PT) Samples (concentrations unknown to laboratory) are analyzed to help ensure laboratory performance.

## 24.2 Controls

Sample preparation or pre-treatment is commonly required before analysis. Typical preparation steps include homogenization, grinding, solvent extraction, sonication, acid digestion, 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) | are used to assess preparation and analysis for possible contamination during the preparation and processing steps. |
| | The specific frequency of use for method blanks during the analytical sequence is defined in the specific standard operating procedure for each analysis. Generally it is 1 for each batch of samples; not to exceed 20 environmental samples. |
| | The method blank is prepared from a clean matrix similar to that of the associated samples that is free from target analytes (e.g., Reagent water, Ottawa sand, glass beads, etc.) and is processed along with and under the same conditions as the associated samples.<br><br>The method blank goes through all of the steps of the process (including as necessary: filtration, clean-ups, etc.). |
| | 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. |

**Table 24-1. Example – Negative Controls**

| Control Type | Details |
|---|---|
| Trip Blank [1] | 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. |
| Field Blanks [1] | are sometimes used for specific projects by the field samplers. A field blank prepared in the field by filling a clean container with pure reagent water and appropriate preservative, if any, for the specific sampling activity being undertaken. (EPA OSWER) |
| Equipment Blanks [1] | 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) |
| 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 |

[1] When known, these field QC samples should not be selected for matrix QC as it does not provide information on the behavior of the target compounds in the field samples. Usually, the client sample ID will provide information to identify the field blanks with labels such as "FB", "EB", or "TB."

Evaluation criteria and corrective action for these controls are defined in the specific standard operating procedure for each analysis.

## 24.4 Positive Controls

Control samples (e.g., QC indicators) are analyzed with each batch of samples to evaluate data based upon (1) Method Performance (Laboratory Control Sample (LCS) or Blank Spike (BS)), which entails both the preparation and measurement steps; and (2) Matrix Effects (Matrix Spike (MS) (Matrix spikes are not applicable to air) or Sample Duplicate (MD, DUP), which evaluates field sampling accuracy, precision, representativeness, interferences, and the effect of the matrix on the method performed. Each regulatory program and each method within those programs specify the control samples that are prepared and/or analyzed with a specific batch

Note that frequency of control samples vary with specific regulatory, methodology and project specific criteria. Complete details on method control samples are as listed in each analytical SOP.

### 24.4.1 Method Performance Control - Laboratory Control Sample (LCS)

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.

The LCS is prepared from a clean matrix similar to that of the associated samples that is free from target analytes (for example: Reagent water, Ottawa sand, glass beads, etc.) and is processed along with and under the same conditions as the associated samples. The LCS is spiked with verified known amounts of analytes or is made of a material containing known and verified amounts of analytes, taken through all preparation and analysis steps along with the field samples. Where there is no preparation taken for an analysis (such as in aqueous

volatiles), or when all samples and standards undergo the same preparation and analysis process (such as Phosphorus), a calibration verification standard is reported as the LCS. In some instances where there is no practical clean solid matrix available, aqueous LCS's may be processed for solid matrices; final results may be calculated as mg/kg or ug/kg, assuming 100% solids and a weight equivalent to the aliquot used for the corresponding field samples, to facilitate comparison with the field samples.

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.).

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.

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 as they cover the range of all of the Aroclors. Specific Aroclors may be used by request on a project specific basis.

## 24.5 Sample Matrix Controls

**Table 24-3. 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 [1] | 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 [1] | 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[2] | 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 [1] | 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 [1] | 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. |

[1] See the specific analytical SOP for type and frequency of sample matrix control samples.

[2] LCSD's are normally not performed except when regulatory agencies or client specifications require them. The recoveries for the spiked duplicate samples must meet the same laboratory established recovery limits as the accuracy QC samples. If an LCSD is analyzed both the LCS and LCSD must meet the same recovery criteria and be included in the final report. The precision measurement is reported as "Relative Percent Difference" (RPD). Poor precision between duplicates (except LCS/LCSD) may indicate non-homogeneous matrix or sampling.

## 24.6 Acceptance Criteria (Control Limits)

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. Control limits are established per method (as opposed to per instrument) regardless of the number of instruments utilized.

Laboratory generated % Recovery acceptance (control) limits are generally established by taking ± 3 Standard Deviations (99% confidence level) from the average recovery of a minimum of 30 data points (more points are preferred, however, fewer (minimum of 20) may be used to establish tentative acceptance limits in select circumstances).

- 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%. Some specific methods or SOPs may allow for higher recoveries.

- The maximum acceptable RPD limit will be 35% for waters and 40% for soils. The minimum RPD limit is 10%.

- If either the high or low end of the control limit changes by ≤ 5% from previous, the control chart is visually inspected and, using professional judgment, they may be left unchanged if there is no affect on laboratory ability to meet the existing limits.

**24.6.1** 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. See SOP WS-QA-0035 for further details.

**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 and DoD/DOE work, there are an allowable number of random 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

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).

A discussion regarding MDLs, Limit of Detection (LOD) and Limit of Quantitation (LOQ) can be found in Section 19.

- Use of formulae to reduce data is discussed in the method SOPs and in Section 20.
- Selection of appropriate reagents and standards is included in Section 9 and 21.
- A discussion on selectivity of the test is included in Section 5.
- Constant and consistent test conditions are discussed in Section 18.
- 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 or prepared electronically on laboratory letterhead, reviewed, and signed by the appropriate project manager. At a minimum, the standard laboratory report shall contain the following information:

**25.2.1** A report title (e.g. Analytical Report for Samples) with a "sample results" column header.

**25.2.2** Each report cover page printed on company letterhead, which includes the laboratory name, address and telephone number.

**25.2.3** A unique identification of the report (e.g. work order number) and on each page an identification in order to ensure the page is recognized as part of the report and a clear identification of the end.

**Note:** Page numbers of report are represented as page # of ##, where the first number is the page number and the second is the total number of pages.

**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 an integral part of the report.
- Any additional addenda to the report must be treated in a similar fashion so it is a recognizable part of the report and cannot accidentally get separated from the report (e.g., Sampling information).

**25.2.5** The name and address of client and a project name/number, if applicable.

**25.2.6** Client project manager or other contact

**25.2.7** Description and unambiguous identification of the tested sample(s) including the client identification code.

**25.2.8** Date of receipt of sample, date and time of collection, and date(s) of test preparation and performance, and time of preparation or analysis if the required holding time for either activity is less than or equal to 72 hours.

**25.2.9** Date reported or date of revision, if applicable.

**25.2.10** Method of analysis including method code (EPA, Standard Methods, etc).

**25.2.11** Reporting limit.

**25.2.12** Method detection limits (if requested)

**25.2.13** Definition of Data qualifiers and reporting acronyms (e.g. ND).

**25.2.14** Sample results.

**25.2.15** QC data consisting of method blank, surrogate, LCS, and MS/MSD recoveries and control limits.

**25.2.16** Condition of samples at receipt including temperature. This may be accomplished in a narrative or by attaching sample login sheets (Refer to Sec. 25.2.4 – Item 3 regarding additional addenda).

**25.2.17** A statement expressing the validity of the results, that the source methodology was followed and all results were reviewed for error.

**25.2.18** A statement to the effect that the results relate only to the items tested and the sample as received by the laboratory.

**25.2.19** A statement that the report shall not be reproduced except in full, without prior express written approval by the laboratory coordinator.

**25.2.20** A signature and title of the person(s) accepting responsibility for the content of the report and date of issue. Authorized signatories are qualified Project Managers appointed by the Manager of Project Managers.

**25.2.21** 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.

**25.2.22** Where applicable, a narrative to the report that explains the issue(s) and corrective action(s) taken in the event that a specific accreditation or certification requirement was not met.

**25.2.23** When soil samples are analyzed, a specific identification as to whether soils are reported on a “wet weight” or “dry weight” basis.

**25.2.24** Appropriate laboratory certification number for the state of origin of the sample, if applicable.

**25.2.25** If only part of the report is provided to the client (client requests some results before all of it is complete), it must be clearly indicated on the report (e.g., preliminary report). A complete report must be sent once all of the work has been completed.

**25.2.26** Any non-TestAmerica subcontracted analysis results are provided as a separate report on the official letterhead of the subcontractor. All TestAmerica subcontracting is clearly identified on the report as to which laboratory performed a specific analysis.

**25.2.27** A clear statement notifying the client that non-accredited tests were performed and directing the client to the laboratory’s accreditation certificates of approval shall be provided when non-accredited tests are included in the report.

**25.2.28** A Certification Summary Report, where required, will document that, unless otherwise noted, all analytes tested and reported by the laboratory were covered by the noted certifications.

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 II is a report with the features described in Section 25.2 above, plus summary information, including results for the method blank reported to the laboratory MDL if required, percent recovery for laboratory control samples and matrix spike samples, and the RPD values for all MSD and sample duplicate analyses.
- Level III contains all the information supplied in Level II, but presented on the CLP-like summary forms, and relevant calibration information. No raw data is provided unless it is necessary to provide the relevant calibration information.
- Level IV is the same as Level III with the addition of all raw supporting data.

In addition to the various levels of QC packaging, the laboratory also provides reports in electronic deliverable form via e-mail, posting to an FTP site, or CD ROM. 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 in addition to the test report as described in section 25.2. When NELAP accreditation is required and both a test report and EDD are provided to the client, the official version of the test report will be the combined information of the report and the EDD. TestAmerica Sacramento offers a variety of EDD formats including Environmental Restoration Information Management System (ERPIMS), New Agency Standard (NAS), Format A, Excel, Dbase, GISKEY, and Text Files.

EDD specifications are submitted to the IT department by the PM for review and undergo the contract review process. 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, and a copy filed on the QA share of the local server.

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 No. CW-L-S-004).

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 stationery 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 to meet all requirements of this document and to include a cover letter.

## 25.7 Format of Reports

The format of reports is designed to accommodate each type of environmental test carried out and to minimize the possibility of misunderstanding or misuse.

## 25.8 Amendments to Test Reports

Corrections, additions, or deletions to reports are only made when justification arises through supplemental documentation. Justification is documented using the laboratory's corrective action system (refer to Section 12).

The revised report is retained on the Archive data server, as is the original report. The revised report is stored in the Archive data server under the sample number followed by "R". Every page will have the report generation date present, to prevent confusion between report versions.

When the report is re-issued, a notation of "Revision "with the revision number is placed on the cover/signature page of the report. The case narrative is updated *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 11/3/11 to include toluene in sample NQA1504 per client's request. This final report replaces the final report generated on 10/27/11.*

## 25.9 Policies on Client Requests for Amendments

### 25.9.1 Policy on Data Omissions or Reporting Limit Increases

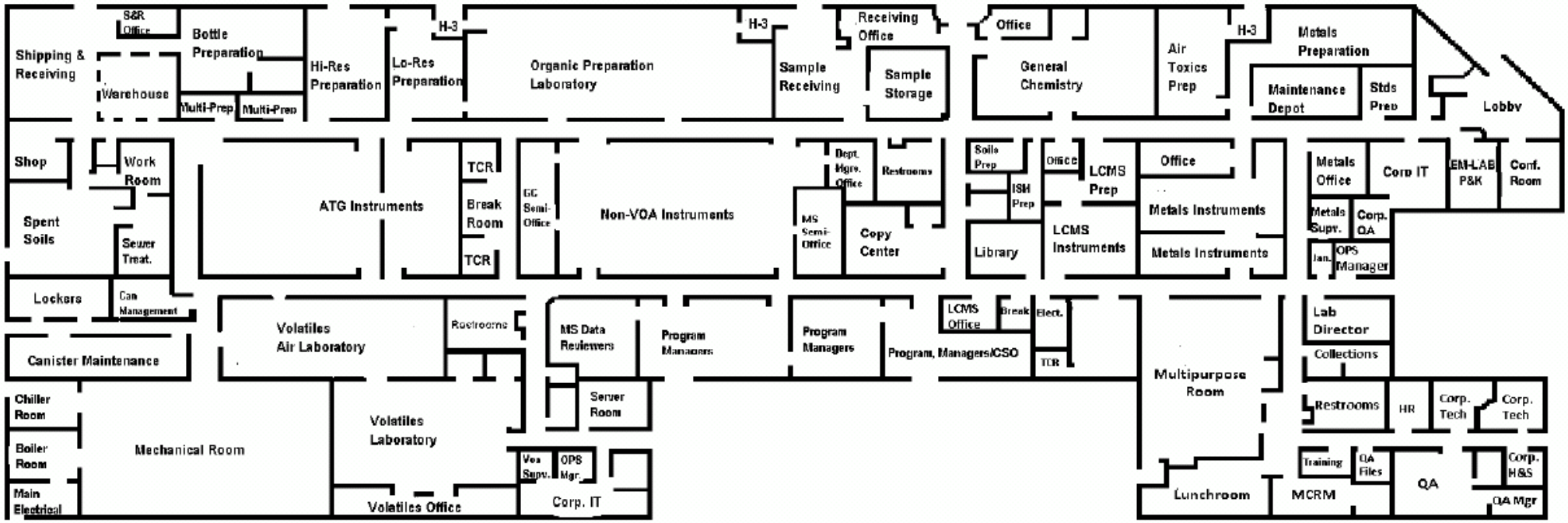
Fundamentally, our policy is simply to not omit previously reported results (including data qualifiers) or to not raise reporting limits and report sample results as ND. This policy has few exceptions. Exceptions are:

- 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 work order 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




| Facility Size | Square Feet |
|---|---|
| Total Area | 66.000 |
| Lab Area | 44,725 |
| Storage Area | 5200 |
| | **Linear Feet** |
| Bench Top | 3036 |
| Hoods | 464 |

## Appendix 2. Glossary/Acronyms (EL-V1M2 Sec. 3.1)

**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)

**Air Sample Bag:** A sampling container for air samples, commonly referred to as Flex-Film or Tedlar bag, in 1.0-L or 3.0-L volumes, that is constructed of proprietary material (E.G., SKC or ESS).

**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.

**Analytical Uncertainty:** A subset of Measurement Uncertainty that includes all laboratory activities performed as part of the analysis. (TNI)

**Anomaly:** A condition or event, other than a deficiency, that may affect the quality of the data, whether in the laboratory's control or not.

**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 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 Curve:** The mathematical relationship between the known values, such as concentrations, of a series of calibration standards and their instrument response. (TNI)

**Calibration Standard:** A substance or reference material used to calibrate an instrument (QAMS)

**Certified Reference Material (CRM):** A reference material accompanied by certificate, having a value, measurement uncertainty, and stated metrological traceability chain to a national metrology institute. (TNI)

**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 safeguarding identified CBI and to maintain all information identified as such in full confidentiality.

**Confirmation:** Verification of the identity of a component through the use of an approach with a different scientific principle from the original method. These may include, but are not limited to Second Column Confirmation; Alternate wavelength; Derivatization; Mass spectral interpretation; Alternative detectors or Additional Cleanup procedures. (TNI)

**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)

**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), whether in the laboratory's control or not.

**Demonstration of Capability:** A procedure to establish the ability of the analyst to generate analytical results of acceptable accuracy and precision. (TNI)

**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 filing a clean container with pure de-ionized water and appropriate preservative, if any, for the specific sampling activity being undertaken (EPA OSWER)

**Field of Accreditation:** Those matrix, technology/method, and analyte combinations for which the accreditation body offers accreditation.

**Holding Times:** The maximum time that samples may be held prior to analyses and still be considered valid or not compromised. (40 CFR Part 136)

**Internal Standard:** A known amount of standard added to a test portion of a sample 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 ± 100%. The IDL represents a range where qualitative detection occurs on a specific instrument. Quantitative results are not produced in this range.

**Laboratory Control Sample (however named, such as laboratory fortified blank, spiked blank, or QC check sample):** A sample matrix, free from the analytes of interest, spiked with verified known amounts of analytes or a material containing known and verified amounts of analytes, taken through all preparation and analysis steps 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.

**Least Squares Regression (1$^{st}$ 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)

**LOD Verification [a.k.a., MDL Verification]:** A processed QC sample in the matrix of interest, spiked with the analyte at no more than 3X the LOD for single analyte tests and 4X the LOD for multiple analyte tests and processed through the entire analytical procedure.

**Limit(s) of Quantitation (LOQ) [a.k.a., Reporting Limit]:** The minimum levels, concentrations, or quantities of a target variable (e.g., target analyte) that can be reported with a specified degree of confidence. (TNI)

**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 replicate matrix spike prepared and analyzed to obtain a measure of the precision of the recovery for each analyte.

**Method Blank:** A sample of a matrix similar to the batch of associated samples (when available) that is free from the analytes of interest and is processed simultaneously with and under the same conditions as samples through all steps of the analytical procedures, and in which no target analytes or interferences are present at concentrations that impact the analytical results for sample analyses.

**Method Detection Limit:** The minimum concentration of a substance (an analyte) that can be measured and reported with 99% confidence that the analyte concentration is greater than zero and is determined from analysis of a sample in a given matrix containing the analyte. (40 CFR Part 136, Appendix B)

**Negative Control:** Measures taken to ensure that a test, its components, or the environment do not cause undesired effects, or produce incorrect test results.

**Non-conformance:** An indication, judgment, or state of not having met the requirements of the relevant specifications, contract, or regulation.

**Observation:** A record of phenomena that (1) may assist in evaluation of the sample data; (2) may be of importance to the project manager and/or the client, and yet not at the time of the observation have any known effect on quality.

**Passivated Canister:** A sampling container for air samples; commonly referred to as a SUMMA canister, SilcoCan or T.O.-Can in 1.0, 1.8 6, or 15 L volumes.

1) SUMMA canister: A spherical stainless steel canister, of which the interior has been specially treated by a process (SUMMA passivation) that renders all surfaces inert to VOCs.

2) SilcoCan: A sampling canister manufactured by Restek Corporation using the Restek Silcosteel® process to coat the interior of the canister with fused silica, rendering it inactive to most VOCs.

3) T.O.-Can: A spherical stainless steel container (which is the equivalent of a SUMMA canister) that is manufactured by Restek using a proprietary electropolishing process and is extensively cleaned using an ultrasonic method that ensures a high-quality passivated surface that maintains the stability of VOCs during storage.

**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.

**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.

**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. (TNI)

**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 laboratory and is provided to test whether the 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. (TNI)

**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)

**Quality System 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 or Saline/Estuarine. 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 & Emissions:* Whole gas or vapor samples including those contained in flexible or rigid wall containers and the extracted concentrated analytes of interest from a gas or vapor that are collected with a sorbant tube, impinger solution, filter, or other device. (TNI)

**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 homogeneous and well established to be used for the calibration of an apparatus, the assessment of a measurement method, or for assigning values to materials. (TNI)

**Reference Standard:** Standard used for the calibration of working measurement standards in a given organization or a given location. (TNI)

**Sampling:** Activity related to obtaining a representative sample of the object of conformity assessment, according to a procedure.

**Second Order Polynomial Curve (Quadratic):** The $2^{nd}$ order curves are a mathematical calculation of a slightly curved line over two axis. The y axis represents the instrument response (or Response ratio) of a standard or sample and the x axis represents the concentration. The $2^{nd}$ order regression will generate a coefficient of determination (COD or $r^2$) that is a measure of the "goodness of fit" of the quadratic curvature the data. A value of 1.00 indicates a perfect fit. In order to be used for quantitative purposes, $r^2$ must be greater than or equal to 0.99.

**Selectivity:** 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. (TNI)

**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

**Technology:** A specific arrangement of analytical instruments, detection systems, and/or preparation techniques.

**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.

**Acronyms:**

A2LA – American Association for Laboratory Accreditation
ANSI – American National Standards Institute
ASQ – American Society for Quality
CAR – Corrective Action Report
CCB – Continuing Calibration Blank
CCV – Continuing Calibration Verification
CF – Calibration Factor
CFR – Code of Federal Regulations
COC – Chain of Custody
DOC – Demonstration of Capability
DQO – Data Quality Objectives
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
ICB – Initial Calibration Blank
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
LOD – Limit of Detection
LOQ – Limit of Quantitation
MDL – Method Detection Limit
MDLCK – MDL Check Standard
MDLV – MDL Verification Check Standard
MRL – Method Reporting Limit Check Standard
MS – Matrix Spike
MSD – Matrix Spike Duplicate
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
RF – Response Factor
RPD – Relative Percent Difference
RSD – Relative Standard Deviation
SD – Standard Deviation
SDS - Safety Data Sheet
SOP – Standard Operating Procedure
TAT – Turn-Around-Time
TALS – TestAmerica LIMS system
VOA – Volatiles
VOC – Volatile Organic Compound

## Appendix 3. Laboratory Certifications, Accreditations, Validations

TestAmerica Sacramento maintains accreditations, certifications, and approvals 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:

The certificates and accredited parameter lists are available, for each State/Program organization at www.testamericainc.com under Analytical Services Search – Certifications.

TestAmerica
THE LEADER IN ENVIRONMENTAL TESTING

### TestAmerica Certifications

| Laboratory | Program | Authority | Identification | Expiration Date |
|---|---|---|---|---|
| TestAmerica Sacramento | DoD ELAP | L-A-B | L2468 | 01/20/2018 |
| TestAmerica Sacramento | Federal | US Fish & Wildlife | LE148388-0 | 10/31/2017 |
| TestAmerica Sacramento | Federal | USDA | P330-11-00436 | 12/30/2017 |
| TestAmerica Sacramento | Federal | USEPA UCMR | CA00044 | 11/06/2018 |
| TestAmerica Sacramento | NELAP | Florida | E87570 | 06/30/2017 |
| TestAmerica Sacramento | NELAP | Illinois | 200060 | 03/17/2018 |
| TestAmerica Sacramento | NELAP | Kansas | E-10375 | 10/31/2017 |
| TestAmerica Sacramento | NELAP | Louisiana | 30612 | 06/30/2017 |
| TestAmerica Sacramento | NELAP | New Hampshire | 2997 | 04/18/2018 |
| TestAmerica Sacramento | NELAP | New Jersey | CA005 | 06/30/2017 |
| TestAmerica Sacramento | NELAP | New York | 11666 | 04/01/2018 |
| TestAmerica Sacramento | NELAP | Oregon | 4040 | 01/28/2018 |
| TestAmerica Sacramento | NELAP | Pennsylvania | 68-01272 | 03/31/2018 |
| TestAmerica Sacramento | NELAP | Texas | T104704399 | 07/31/2017 |
| TestAmerica Sacramento | NELAP | Utah | CA00044 | 02/28/2018 |
| TestAmerica Sacramento | NELAP | Virginia | 460278 | 03/14/2018 |
| TestAmerica Sacramento | State Program | Alaska (UST) | UST-055 | 12/18/2017 |
| TestAmerica Sacramento | State Program | Arizona | AZ0708 | 08/11/2017 |
| TestAmerica Sacramento | State Program | Arkansas DEQ | 88-0691 | 06/17/2018 |
| TestAmerica Sacramento | State Program | California | 2897 | 01/31/2018 |
| TestAmerica Sacramento | State Program | Colorado | CA00044 | 08/31/2017 |
| TestAmerica Sacramento | State Program | Connecticut | PH-0691 | 06/30/2017 |
| TestAmerica Sacramento | State Program | Hawaii | N/A | 01/29/2018 |
| TestAmerica Sacramento | State Program | Maine | CA0004 | 04/18/2018 |
| TestAmerica Sacramento | State Program | Michigan | 9947 | 01/31/2018 |
| TestAmerica Sacramento | State Program | Nevada | CA00044 | 07/31/2017 |
| TestAmerica Sacramento | State Program | Washington | C581 | 05/05/2018 |
| TestAmerica Sacramento | State Program | West Virginia (DW) | 9930C | 12/31/2017 |
| TestAmerica Sacramento | State Program | Wyoming | 8TMS-L | 01/29/2017 * |

## Appendix 4: Listing of Methods Performed

**Preparation Only Methods**

| Method | Aqueous | Solid | Waste | Biological | Air |
|---|---|---|---|---|---|
| **Organics** | | | | | |
| Calif. CAM-WET | X | X | X | | |
| EPA 1311 | X | X | X | | |
| EPA 3510C | X | | | | |
| EPA 3535 | X | | | | |
| EPA 3540B | | X | | | |
| EPA 3542 | | | | | X |
| EPA 3546 | | X | | | |
| EPA 3550B | | X | | X | |
| EPA 3580A | | | X | | |
| EPA 3600C | X | X | X | | |
| EPA 3620B | X | X | X | | |
| EPA 3630C | X | X | X | | |
| EPA 3640A | X | X | | X | |
| EPA 5030B | X | X | X | | |
| EPA 5035 | X | X | X | | |
| **Inorganics** | | | | | |
| Calif. CAM WET | X | X | X | | |
| EPA 1311 | X | X | X | | |
| EPA 1312 (E/W) | X | X | X | | |
| EPA 3005A | X | | | | |
| EPA 3010A | X | | | | |
| EPA 3050B | | X | X | X | |

**Organics Methods Performed**

| Parameter | Method | Aqueous | Solid | Waste | Biological | Air |
|---|---|---|---|---|---|---|
| **Volatile Organics** | SW846 8260B | X | X | X | | |
| | SW846 8260C | X | X | X | | |
| | EPA 624 | X | | | | |
| | TO-14A | | | | | X |
| | TO-15 | | | | | X |
| **Sulfur Containing Compounds** | EPA 15/16 | | | | | X |
| **Fixed Gases** | ASTM D1946 | | | | | X |
| | EPA 3C | | | | | X |
| **Base Neutrals and Acids (BNAs)** | SW846 8270C | X | X | X | X | |
| | SW846 8270D | X | X | X | X | |
| | EPA 625 | X | | | | |
| | TO-13A | | | | | X |
| | IP-7 | | | | | X |
| | EPA 23 | | | | | X |
| **Organochlorine Pesticides** | SW846 8081A | X | X | X | X | |
| | SW846 8081B | X | X | X | X | |
| | EPA 608 | X | | | | |
| | TO-4A | | | | | X |
| | TO-10A | | | | | X |
| **PCBs (Aroclors)** | EPA 8082 | X | X | X | X | |
| | EPA 8082A | X | X | X | X | |
| | EPA 608 | X | | | | |
| | TO-4A | | | | | X |
| | TO-10A | | | | | X |
| **PCB Congeners** | EPA 1668A | X | X | X | X | X |
| | EPA 1668C | X | X | X | X | X |
| **Petroleum Hydrocarbons** | EPA 8015B | X | X | X | | |
| | EPA 8015D | X | X | X | | |
| | CA LUFT | X | X | X | | |
| | AK101 | X | X | X | | |
| | Ak102 | X | X | X | | |
| | AK103 | X | X | X | | |
| | GRO/DRO | X | X | X | | |
| **Nitroaromatics and Nitroamines** | EPA 8330A | X | X | X | | |
| | EPA 8330B | X | X | X | | |
| | WS-LC-0010 | X | X | X | | |
| **Nitrosamines** | WS-MS-0012 | X | X | | | |

| Parameter | Method | Aqueous | Solid | Waste | Biological | Air |
|---|---|---|---|---|---|---|
| **PAHs** | EPA 8270C (SIM Isotope dilution) | X | X | X | X | X |
| | EPA 8270C (SIM) | X | X | X | | |
| | CARB 429 | X | X | X | X | X |
| | TO-13A | | | | | X |
| **1,4-Dioxane** | WS-MS-0010 | X | | | | |
| **Alkyl Phenols** | WS-MS-0013 | X | X | | X | |
| **Perfluorinated Compounds** | WS-LC-0025 | X | X | X | X | |
| | ISO 25101 | X | | | | |
| **(including PFOA/PFOS)** | EPA 537 | X | | | | |
| **Dioxins & Furans** | EPA 1613B | X | X | | | |
| | EPA 8290 | X | X | X | X | |
| | EPA 8290A | X | X | X | X | |
| | EPA 8280A | X | X | X | X | |
| | EPA 8280B | X | X | X | X | |
| | EPA 0023A | | | | | X |
| | EPA 23 | | | | | X |
| | TO-9 | | | | | X |

**Metals Methods Performed**

| Parameter | Methods | Aqueous | Solid | Waste | Biological | Air |
|---|---|---|---|---|---|---|
| **Trace Metals** | EPA 6010B | X | X | X | X | X |
| | EPA 6020 | X | X | X | X | X |
| | EPA 0060 | | | | | X |
| | EPA 200.7 | X | | | | |
| | EPA 200.8 | X | | | | |
| | EPA 12 | | | | | X |
| | CARB 12 | | | | | X |
| | EPA 29 | | | | | X |
| | CARB 436 | | | | | X |
| **Hardness** | SM 2340B | X | | | | |
| **Mercury** | EPA 7470A | X | | | | |
| | EPA 245.1 | X | | | | |
| | EPA 7471A | | X | X | X | X |
| | EPA 101A | | | | | X |
| | ASTM D6784-02 | | | | | X |
| | EPA 0060 | | | | | X |
| | EPA 29 | | | | | X |
| | CARB 436 | | | | | X |

**Inorganics Methods Performed**

| Parameter | Method | Aqueous | Solid | Waste | Biological | Air |
|---|---|---|---|---|---|---|
| **Alkalinity (Carbonate, Bicarbonate, Total)** | SM 2320B | X | | | | |
| **Bromide, Chloride, and Fluoride** | EPA 300.0 | X | | | | |
| | EPA 9056 | X | X | | | |
| | EPA 9057 | | | | | X |
| | EPA 26A | | | | | X |
| | CARB 421 | | | | | X |
| **Chromium, Hexavalent** | EPA 7196A | X | | | | |
| | EPA 0061 | | | | | X |
| | EPA 306 | | | | | X |
| | CARB 426 | | | | | X |
| **Conductivity** | EPA 9050A | X | | | | |
| | SM 2510 B | X | | | | |
| **Demand, Chemical Oxygen** | EPA 410.4 | X | | | | |
| **Moisture** | ASTM 2216 | | X | | | |
| **Nitrate** | EPA 353.2 | X | | | | |
| | EPA 300.0 | X | | | | |
| | EPA 9056 | X | X | | | |
| | CARB 421 | | | | | X |
| **Nitrate-Nitrite** | EPA 353.2 | X | | | | |
| **Nitrite** | EPA 353.2 | X | | | | |
| | EPA 300.0 | X | | | | |
| | EPA 9056 | X | X | | | |
| | CARB 421 | | | | | X |
| **Nitrocellulose** | EPA 353.2 | X | X | | | |
| | WS-WC-0050 | X | X | | | |
| **Orthophosphate** | EPA 300.0 | X | | | | |
| | EPA 9056 | X | X | | | |
| **Particulates in Air** | EPA 5 | | | | | X |
| | 40 CFR Part 50 | | | | | X |
| **Perchlorate** | EPA 314.0 | X | | | | |
| | EPA 331.0 | X | | | | |
| | EPA 6850 | X | X | | | |
| **pH** | SM 4500 H+ B | X | | | | |
| | EPA 150.2 | X | | | | |
| | EPA 9040A | X | | | | |
| | EPA 9041A | X | | | | |
| | EPA 9045C | | X | X | | |
| **Solids, Total** | SM 2540 B | X | | | | |
| **Solids, Total Dissolved** | SM 2540 C | X | | | | |

| | | | | | | |
|---|---|---|---|---|---|---|
| **Solids, Total Suspended** | SM 2540 D | X | | | | |
| **Sulfate** | EPA 300.0 | X | | | | |
| | EPA 9056 | X | X | | | |

## Appendix 5. Data Qualifiers

| Qualifier Organic | Qualifier Inorganic | Footnote |
|---|---|---|
| U | U | Analyte analyzed for but was not detected. |
| J | B | Estimated result. Result is less than RL. |
| E | I | Estimated result. Result concentration exceeds the calibration range. |
| B | J | Method blank contamination. The associated method blank contains the target analyte at a reportable level. |
| P | * | Relative percent difference (RPD) is outside stated control limits. |
| a | N | Spiked analyte recovery is outside stated control limits. |
| * | | Surrogate recovery is outside stated control limits. |
| PG | | The percent difference between the original and confirmation analyses is greater than 40%. |

# Quality Assurance Manual

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**Facility Distribution No.: 0** **Distributed To: See Electronic Distribution Sheet**

# Title Page:

## Quality Assurance Manual
## Approval Signatures

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| Technical Manager, (Volatiles) – Andrew Buettner | Date 5-10-17 |
| Manager of Project Management – Rhonda Ridenhower | Date 5-11-17 |

## SECTION 2. TABLE OF CONTENTS

# LIST OF TABLES



# LIST OF FIGURES

# LIST OF APPENDICES

## REFERENCED CORPORATE SOPs AND POLICIES

| SOP / Policy Reference | Title |
|---|---|
| CA-I-P-002 | Electronic Reporting and Signature Policy |
| CA-L-P-002 | Contract Compliance Policy |
| CW-L-S-004 | Subcontracting |
| CA-Q-M-002 | Corporate Quality Management Plan |
| CA-Q-S-001 | Solvent and Acid Lot Testing and Approval |
| CA-Q-S-002 | Acceptable Manual Integration Practices |
| CA-Q-S-006 | Detection Limits |
| CA-Q-S-009 | Root Cause Analysis |
| CA-T-P-001 | Qualified Products List |
| CW-E-M-001 | Corporate Environmental Health & Safety Manual |
| CW-F-P-002 | Company-Wide Authorization Matrix |
| CW-F-P-004 | Procurement and Contracts Policy |
| CW-F-S-007 | Capital Expenditure, Controlled Purchase Requests and Fixed Asset Capitalization |
| CW-L-P-004 | Ethics Policy |
| CW-L-S-002 | Internal Investigation |
| CW-Q-S-001 | Corporate Document Control and Archiving |
| CW-Q-S-002 | Writing a Standard Operating Procedure (SOPs) |
| CW-Q-S-003 | Internal Auditing |
| CW-Q-S-004 | Management Systems Review |
| CW-Q-S-005 | Data Recall Process |
| CA-C-S-001 | Work Sharing Process |

## REFERENCED LABORATORY SOPs

TestAmerica St. Louis Standard Operating Procedures are listed in Appendix 5.

# SECTION 3. INTRODUCTION, SCOPE AND APPLICABILITY

## 3.1 Introduction and Compliance References

TestAmerica St. Louis'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 U.S. Department of Energy Quality Systems for Analytical Services/U.S. Department of Defense Quality Systems Manual for Environmental Laboratories (QSM, current version), The NELAC Institute (TNI) Standard, dated 2009, Volume 1 Modules 2 and 4, 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.

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

- EPA 600/4-79-019, *Handbook for Analytical Quality Control in Water and Wastewater Laboratories,* EPA, March 1979.
- EPA Requirements for Quality Management Programs" (QA/R-2) (EPA/240/B-01/002, May 31, 2006).
- ANSI/ASQC, E4-1994, "Specifications and Guidelines for Quality Management Systems for Environmental Data Collection and Environmental Technology Programs" (American National Standard, January 5, 1995, or most recent version)
- *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, Final Update V, August 2015.*
- *U.S. Department of Defense/Department of Energy, Quality Systems Manual, Version 5.0, July 2013.*
- Federal Register, 40 CFR Parts 136, 141, 172, 173, 178, 179 and 261.
- APHA, *Standard Methods for the Examination of Water and Wastewater,* 18th Edition, 19th, 20th **and** 21st, and on-line Editions.
- *U.S. Department of Energy* Order 414.1B, *Quality Assurance*, Approved April 29, 2004.
- *U.S. Department of Energy* Order 414.1C, *Quality Assurance*, June 17, 2005.
- *U.S. Department of Energy Order 414.1D, Quality Assurance, Aril, 25, 2011.*
- *U.S. Department of Energy*, *Quality Systems for Analytical Services*, Revision 2.9, January 2012.
- Nuclear Regulatory Commission (NRC) Quality Assurance Requirements.
- Federal Register 10CFR 50 Appendix B
- Toxic Substances Control Act (TSCA).

- ASME NQA-1-2000 Quality Assurance Requirements for Nuclear Facility Applications (for nuclear safety related activities)
- ASME NQA-1-1994 Quality Assurance Requirements for Nuclear Facility Applications (for nuclear safety related activities)
- Federal Register 10CFR21 and 10CFR50.55e

### 3.2 Terms and Definitions

A Quality Assurance Program is a company-wide system designed to ensure that data produced by the laboratory conforms to the standards set by state and/or federal regulations. The program functions at the management level through company goals and management policies, and at the analytical level through Standard Operating Procedures (SOPs) and quality control. The TestAmerica program is designed to minimize systematic error, encourage constructive, documented problem solving, and provide a framework for continuous improvement within the organization. Refer to Appendix 2 for the Glossary/Acronyms.

### 3.3 Scope / Fields of Testing

The laboratory analyzes a broad range of environmental and industrial samples every month. Sample matrices vary among air, drinking water, effluent water, groundwater, hazardous waste, sludge and soils. The Quality Assurance Program contains specific procedures and methods to test samples of differing matrices for chemical and physical 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 on the www.testamericainc.com web site**.** 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, Technical Directors and the Quality Assurance (QA) Manager. In some cases, QAPPs and DQOs may specify less stringent requirements. The Laboratory Director and the QA Manager must determine if it is in the lab's best interest to follow the less stringent requirements.

### 3.4 Management of the Manual

#### 3.4.1 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 itself is reviewed **annually** by senior laboratory management to assure that it reflects current practices and meets the requirements of the laboratory's clients and regulators as well as the CQMP. Occasionally, the manual may need changes in order to meet new or changing regulations and operations. The QA Manager will review the changes in the normal course of business and incorporate changes into revised sections of the document. All updates will be reviewed by the senior laboratory management staff. The laboratory updates and approves such changes according to SOP ST-QA-0035, "Preparation and Management of Standard Operating Procedures".

## SECTION 4. MANAGEMENT REQUIREMENTS

### 4.1 Overview

TestAmerica St. Louis 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 and Chief Executive Officer (CEO), Chief Operating Officer (COO), Executive Vice President (VP) Operations, Corporate Quality, etc.). The laboratory operational and support staff work under the direction of the Laboratory Director. The organizational structure for both Corporate & TestAmerica St. Louis 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. More extensive job descriptions are maintained by laboratory management.

#### 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 CQMP. This manual is specific to the operations of TestAmerica's St. Louis laboratory.

#### 4.2.2 Laboratory Director (LD) or Designee

The St. Louis Laboratory Director is responsible for the overall quality, safety, financial, technical, human resource and service performance of the whole laboratory and reports to

his/her respective General Manager (GM). The Laboratory Director provides the resources necessary to implement and maintain an effective and comprehensive Quality Assurance and Data Integrity Program.

Specific Responsibilities include, but are not limited to:

- The Laboratory Director is responsible for maintaining positive operating margin to the company at the laboratory level and for meeting and exceeding the annual budget.
- Ensures that personnel are free from commercial, financial and other undue pressures which might adversely affect their quality of work
- Supervise all laboratory personnel and provide guidance and direction as needed.
- Ensure that sufficient numbers of qualified personnel are employed to supervise and perform the work of the laboratory.
- Responsible for ensuring compliance and integration of facility operation with corporate and regulatory policies and procedures.
- Ensures that appropriate corrective actions are taken to address issues identified by external and internal audits.
- The laboratory Director has signatory authority for the QAM, policies, SOPs and contracts (as defined by TestAmerica policy).

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

The QA Manager has responsibility and authority to ensure the continuous implementation, maintenance and improvement of the quality system.

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.
- Having functions independent from laboratory operations for which he/she has quality assurance oversight.
- Maintaining and updating the QAM.
- Monitoring and evaluating laboratory certifications; scheduling proficiency testing samples.
- Monitoring and communicating regulatory changes that may affect the laboratory to management.
- Training and advising the laboratory staff on quality assurance/quality control procedures that are pertinent to their daily activities.
- Have documented training and/or experience in QA/QC procedures and the laboratory's Quality System.

- Having a general knowledge of the analytical test methods for which data audit/review is performed (and/or having the means of getting this information when needed).
- Arranging for or conducting internal audits on quality systems and the technical operation.
- The laboratory QA Manager will maintain records of all ethics-related training, including the type and proof of attendance.
- Maintain, improve, and evaluate the corrective action database and the corrective and preventive action systems.
- Notifying laboratory management of deficiencies in the quality system and ensuring corrective action is taken. Procedures that do not meet the standards set forth in the QAM or laboratory SOPs shall be investigated following procedures outlined in Section 12 and if deemed necessary the procedures may be temporarily suspended during the investigation.
- Objectively monitor standards of performance in quality control and quality assurance without outside (e.g., managerial) influence.
- Coordinating of document control of SOPs, MDLs, control limits, and miscellaneous forms and information.
- Review a percentage of all final data reports for internal consistency. Review of Chain of Custody (COC), correspondence with the analytical request, batch QC status, completeness of any corrective action statements, 5% of calculations, format, holding time, sensibility and completeness of the project file contents.
- Review of external audit reports and data validation requests.
- Follow-up with audits to ensure client QAPP requirements are met.
- Establishment of reporting schedule and preparation of various quality reports for the Laboratory Director, clients and/or Corporate QA.
- Development of suggestions and recommendations to improve quality systems.
- Research of current state and federal requirements and guidelines.
- Captains the QA team to enable communication and to distribute duties and responsibilities.
- Ensuring Communication & monitoring standards of performance to ensure that systems are in place to produce the level of quality as defined in this document.
- Has final authority to accept or reject data and to stop work in progress in the event that procedures or practices compromise the validity and integrity of the analytical data.
- Evaluation of the thoroughness and effectiveness of training.
- **Compliance with ISO 17025** (where applicable)
- Providing Quality Systems training to all new personnel and ensuring that all personnel understand their contributions to the quality system.
- Evaluate the effectiveness of training.
- Has signatory authority over the QAM, SOPs and policies pertaining to QA/QC
- Compliance with the NELAC Standards (where applicable)
- Compliance with the QSM (where applicable)

### 4.2.4 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 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, and 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.
- Reviewing and approving, with input from the QA Manager, proposals from marketing, in accordance with an established procedure for the review of requests and contracts. This procedure addresses the adequate definition of methods to be used for analysis and any limitations, the laboratory's capability and resources, the client's expectations. Differences are resolved before the contract is signed and work begins. A system documenting any significant changes is maintained, as well as pertinent discussions with the client regarding their requirements or the results of the analyses during the performance of the contract. All work subcontracted by the laboratory must be approved by the client. Any deviations from the contract must be disclosed to the client. Once the work has begun, any amendments to the contract must be discussed with the client and so documented.
- Monitoring the validity of the analyses performed and data generated in the laboratory. This activity begins with reviewing and supporting all new business contracts, insuring data quality, analyzing internal and external non-conformances to identify root cause issues and implementing the resulting corrective and preventive actions, facilitating the data review process (training, development, and accountability at the bench), and providing technical and troubleshooting expertise on routine and unusual or complex problems.
- Providing 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.
- Enhancing efficiency and improving quality through technical advances and improved LIMS utilization. Capital forecasting and instrument life cycle planning for second generation methods and instruments as well as asset inventory management.
- Coordinating sample management from "cradle to grave," insuring that no time is lost in locating samples.
- Scheduling 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.

- Responsible for ensuring compliance with the NELAC Standards
- Compliance with ISO 17025 (where applicable)
- Compliance with the QSM (where applicable)

### 4.2.5 Technical Director

The Technical Director(s) report(s) directly to the Laboratory Director. The scope of responsibility ranges from the new hire process and existing technology through the on going training and development programs for existing analysts and second and third generation instrumentation.

Specific responsibilities include:

- Assists in coordinating, writing and reviewing SOPs.
- May assist in the review of proposals
- Solves day to day technical issues, provides technical training and guidance to staff, project managers, and clients.
- Investigates technical issues identified by QA, and directs evaluation of new methods.
- Responsible for ensuring compliance with the NELAC Standards
- Compliance with ISO 17025 (where applicable)
- Compliance with the QSM (where applicable)

### 4.2.6 Manager of Project Management/Customer Service Manager

In addition to filling the requirements of Project Manager for key accounts, he/she fulfills supervisory duties and responsibilities. As Manager, he supervises the Project Management staff, sets standards for and monitors productivity, manages the assignment of accounts and the daily workload and tracks and maintains information for various revenue reports. With the QA Manager, he determines acceptable corrective actions for the nonconformance occurring within his group, develops and reviews standard operating procedures for the group.

Additional responsibilities include:

- Has signatory authority for final reports.
- Training of the Project Management staff
- Notify supervisors of incoming projects and sample delivery schedules
- Coordinate requests for sample containers and sample pick-up/deliveries

### 4.2.7 Project Manager

- Coordinates and manages customers' projects through all phases of laboratory operations, ensuring fulfillment of TestAmerica's commitment to client requirements, error-free work, and on-time delivery.

- Responsible to ensure that clients get timely responses to status inquiries, resolutions to problems and the agreed upon deliverables
- Discusses with clients any project related problems, resolves service issues and coordinates technical details with the lab staff
- Responsible for staff familiarization with specific quotes, sample log-in review and final report accuracy and completeness
- Maintains communications with clients and Account Executives and serves as a liaison between clients and laboratory operations to meet client's needs.
- Works closely with business unit personnel to manage quotations and change orders for existing scopes of work.
- Generates narratives outlining project observations, QC excursions, and laboratory comments.
- Has signatory authority for final reports.

### 4.2.8 Department Manager/Supervisor

The Department Manager/Supervisor is responsible for the overall operations of a specific laboratory area.

These responsibilities include but are not limited to:

- Meeting client satisfaction goals, managing the human resources within the department, and ensuring health and safety and quality assurance plan compliance.
- Serves as a technical resource to department employees, as well as Project Managers, sales personnel, and clients.
- Make recommendations to laboratory management in regard to process improvements.
- Ensure analysts in their department adhere to applicable SOPs and the QAM.

### 4.2.9 Chemist/Analyst

- Laboratory analysts are responsible for the generation of data by preparing and analyzing samples according to written SOPs and client requirements.
- They are responsible for understanding the requirements in the QAM and the SOPs associated with their specific function.
- Perform the initial technical review of sample preparation information, calculations, qualitative identifications and raw data with the authority to stop, accept, or reject data based on compliance with self-defined QC criteria.
- The laboratory analyst also provides prompt documentation and notification to the Group Leader of problems or anomalies detected.
- Monitor, calibrate, and maintain standard laboratory equipment such as refrigerators, ovens, water systems, and pipettes, and instrumentation, as necessary.

### 4.2.10 Environmental Health and Safety Coordinator

- The Environmental Health and Safety Coordinator is responsible for administering the EH&S program that provides a safe, healthy working environment for all employees and the environment.
- Monitors all areas for unsafe conditions, acts, and potential hazards. Enforces environmental, health, and safety policies and procedures. Maintains regulatory compliance with local, state, and federal laws.
- Makes safety and health recommendations to laboratory management in conjunction with the facility safety committee.
- Develops and maintains the facility's health and safety and waste disposal procedures.
- Conduct ongoing, necessary safety training and conduct new employee safety orientation.
- Assist in developing and maintaining the Chemical Hygiene/Safety Manual.
- Administer dispersal of all Material Safety Data Sheet (MSDS) information.
- Perform regular chemical hygiene and housekeeping instruction.
- Give instruction on proper labeling and practice.
- Serve as chairman of the laboratory safety committee.
- Provide and train personnel on protective equipment.
- Oversee the inspection and maintenance of general safety equipment – fire extinguishers, safety showers, eyewash fountains, etc. and ensure prompt repairs as needed.
- Supervise and schedule fire drills and emergency evacuation drills.
- Determine what initial and subsequent exposure monitoring, if necessary to determine potential employee exposure to chemicals used in the laboratory.
- When determined necessary, conduct exposure monitoring assessments.
- Determine when a complaint of possible over-exposure is "reasonable" and should be referred for medical consultation.
- Assist in the internal and external coordination of the medical consultation/monitoring program conducted by TestAmerica's medical consultants.

### 4.2.11 Radiation Safety Officer (RSO)

- Under the direction of the Laboratory Director, implements the radiation protection program that, as a minimum, provides compliance with pertinent regulatory requirements, license provisions, and the Radiation Protection Program.
- Maintains direct access to the Laboratory Director on matters relating to radiological protection.
- Maintains sufficient organizational independence to review and evaluate activities involving the use of radioactive materials.
- Provides Authorized Users and radiation workers with the instruments, protective devices, dosimetry, training, and other items needed to perform their work in accordance with the radiological protection program elements.

- Maintains original copies of all St. Louis licenses/permits, including attachments and amendments, for radioactive materials.
- Directs program to monitor and control radioactive materials throughout the laboratory
- Conducts radiation safety training
- Maintains inventory of standards, tracers, and radiological samples
- Manages segregated area for storing radioactive and mixed wastes

### 4.3 Deputies

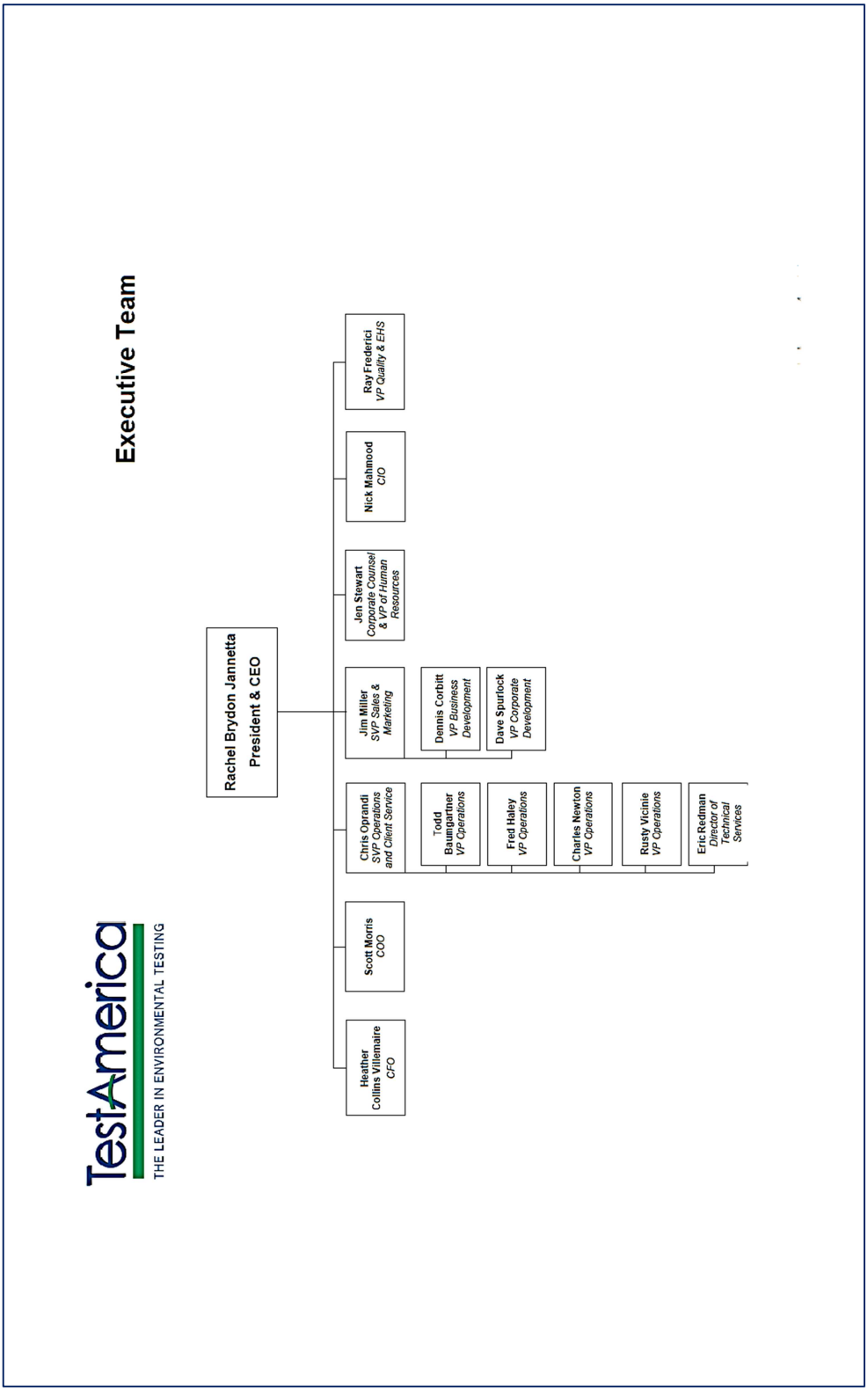
The following table defines who assumes the responsibilities of key personnel in their absence:

| Key Personnel | Deputy |
|---|---|
| Elaine Wild*<br>Laboratory Director | Andrew Buettner<br>Volatiles Manager |
| Kristen Ely<br>Quality Manager | Marti Ward<br>Quality Assurance Specialist |
| Cory Buffington*<br>Metals Technical Manager | Laura Johnson<br>Metals analyst |
| Jacob Boyd<br>Inorganics Technical Lead | Brandi Hayes<br>Inorganic Analyst |
| Sarah Bernsen *<br>Radiochemistry Prep Technical Manager<br><br>Rachel Muller*<br>Radiochemistry Analyst Technical Manager | Rachel Muller [Count Room Deputy]<br>Radiochemistry Analyst Manager<br><br>Sarah Bernsen [Prep Deputy]<br>Radiochemistry Prep Manager |
| Michael Ridenhower<br>EHS Coordinator/Radiation Safety Manager | Terry Romanko*<br>Technical Director |
| Rhonda Ridenhower<br>Manager of Project Management | Jayna Awalt<br>Project Manager |
| Dennis Konopka*<br>Extractable Organics Technical Manager | Andrew Buettner<br>Volatiles Manager |
| Andrew Buettner*<br>Volatile Organics Technical Manager | Dennis Konopka<br>Extractables Manager |
| Mark Minier<br>Organic Extractable Prep/Pre-prep Technical Manager | Kelli Agu<br>Organic Extraction Analyst |

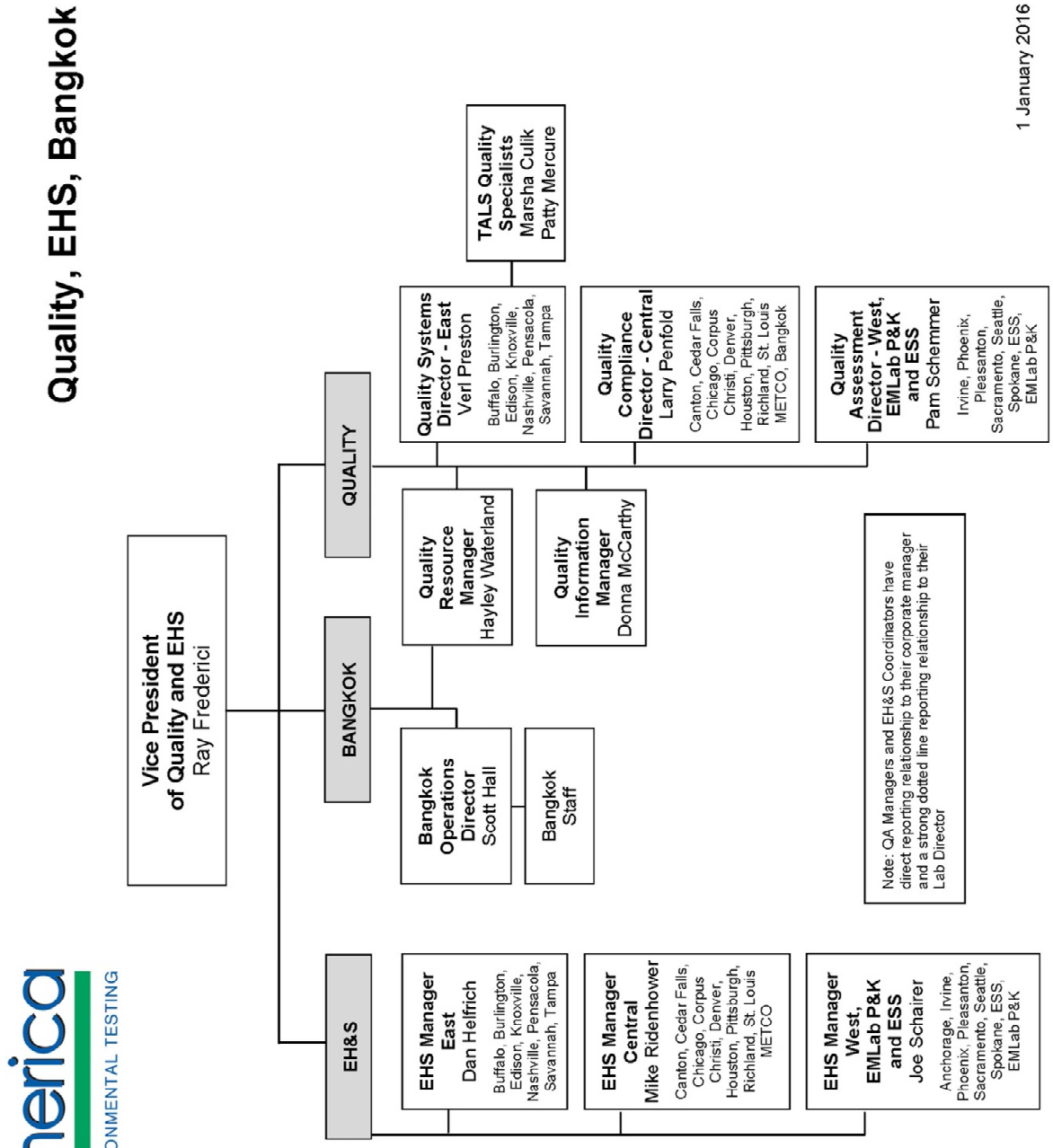
In the event that key Technical Managers are absent for a period exceeding 15 consecutive calendar days, the deputy will temporarily perform the absentee's functions. If the absence exceeds thirty-five consecutive calendar days, the primary accreditation body shall be notified in writing.

Technical Managers are designated with an asterisk (*).

**Figure 4-1. Corporate and Laboratory Organization Charts**



# Quality, EHS, Bangkok

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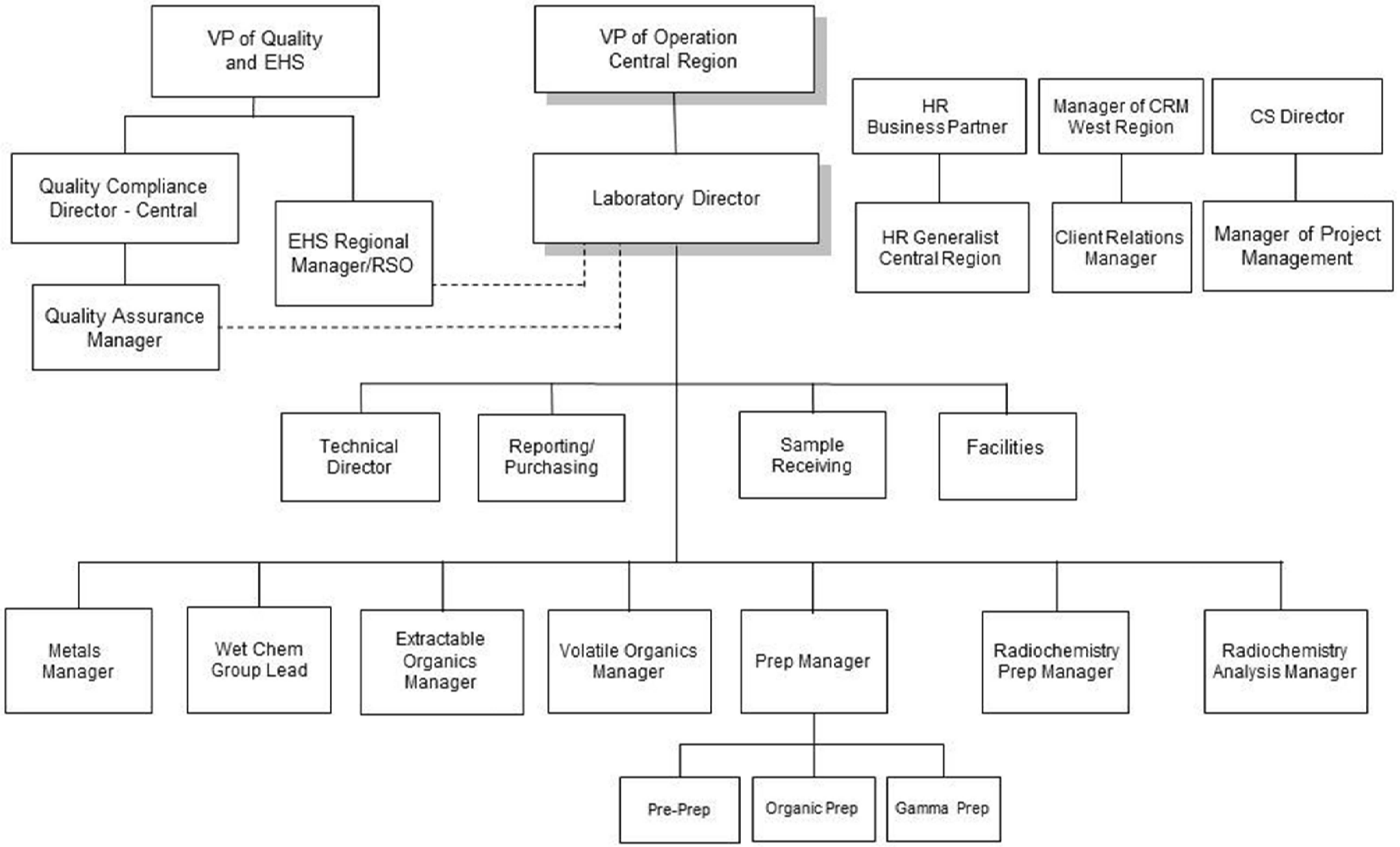
# St. Louis Laboratory Organization




**Note**: QA Manager and EH&S Manager have a direct reporting relationship to both operations leadership and corporate functional leadership.

# 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.
- ❖ To comply with the ISO/IEC 17025:2005(E) International Standard, the 2009 TNI Standard and to continually improve the effectiveness of the management system.
- ❖ TestAmerica St. Louis' policy includes compliance with the Department of Defense/Department of Energy QSM..

Every staff member at the laboratory plays an integral part in quality assurance and is held responsible and accountable for familiarizing themselves with the quality program documentation and implementing those policies and procedures to ensure 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-Q-S-005).
- 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
- Laboratory Waste Management Plan
- Laboratory Radiation Safety Program

#### 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).

### 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/Minimum Detectable Activity/Detection Limit) or quantified (Reporting Limit/Limit of Quantitation).

## 5.5 Criteria for Quality Indicators

The laboratory maintains quality limits reference data through the LIMS containing the precision and accuracy acceptability limits for performed analyses. This data is managed by the laboratory's QA department using the Control Chart app in LIMS. Printed and/or electronic copies of method specific QC limits are available upon request. Unless otherwise noted, limits are laboratory generated. Some acceptability limits are derived from US EPA methods when they are required. Where US EPA method limits are not required, the laboratory has developed limits from evaluation of data from similar matrices. Criteria for development of control limits are contained in SOP ST-QA-0014 and Section 24.

## 5.6 Statistical Quality Control

Statistically-derived precision and accuracy limits are required by selected methods (such as SW-846) and programs. The laboratory routinely utilizes statistically-derived limits to evaluate method performance and determine when corrective action is appropriate. The analysts are instructed to use the current limits in the laboratory (dated and approved by the QA Manager) and entered into the Laboratory Information Management System (LIMS). The Quality Assurance department maintains an archive of all limits used within the laboratory. If a method defines the QC limits, the method limits are used.

If a method requires the generation of historical limits, the lab develops such limits from recent data in the QC database of the LIMS following the guidelines described in Section 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

As the QC limits are calculated, QC charts are generated to show warning and control limits for the purpose of evaluating trends. The QA Manager evaluates these to determine if adjustments need to be made or for corrective actions to methods. All findings are documented and kept on file. See SOP ST-QA-0014 "Evaluation of Analytical Accuracy and Precision Through the Use of Control Charts".

### 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 No. ST-QA-0023, "Control of Records".

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

The laboratory maintains control of records for raw analytical data and supporting records such as audit reports and responses, logbooks, standard logs, training files, MDL studies, Proficiency Testing (PT) studies, certifications and related correspondence, validation requests 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. In order to develop a new document, a technical manager submits a draft to the QA Department for suggestions and approval before use. Upon approval, QA personnel add the identifying version

information to the document and retain 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 two years. When related to DoD (Department of Defense)/DOE (Department of Energy) work, the review will be done annually. Revisions are made as appropriate. Changes to documents occur when a procedural change warrants.

### 6.3 Procedures for Document Control Policy

For changes to the QA Manual, refer to SOP No. ST-QA-0035, "Preparation and Management of Standard Operating Procedures". Uncontrolled copies must not be used within the laboratory. Previous revisions and back-up data are stored by the QA department. Electronic copies are stored on the Public server in the QA folder.

For changes to SOPs, refer to SOP No. CW-Q-S-002, "Writing a Standard Operating Procedure SOP" and laboratory SOP No. ST-QA-0035, "Preparation and Management of Standard Operating Procedures".

Forms, worksheets, work instructions and information are organized electronically by department in the QA folder on the network server. There is an index. Hard copies are kept in QA files. In order to develop a new form, worksheet or work instruction, the user submits a draft to the QA Department and technical manager for suggestions, approval and validation (where required) before use. Upon approval, QA personnel add the identifying control information to the document. 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.

### 6.4 Obsolete Documents

All invalid or obsolete documents are removed, or otherwise prevented from unintended use. The laboratory has specific procedures as described above to accomplish this. In general, obsolete documents are collected from employees according to distribution lists and are marked obsolete on the cover or destroyed. At least one copy of the obsolete document is archived as described in Section 14.

## 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. SOP ST-PM-0001, "Project Setup and Quote", outlines the process at the TestAmerica St. Louis laboratory.

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 Sales Directors, who will decide which lab will receive the work based on the scope of work and other requirements, including certification, testing methodology, and available capacity to perform the work. The contract review process is outlined in TestAmerica's Corporate SOP No. CA-L-P-002**,** Contract Compliance Policy.

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

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

The Sales Director, Legal Contracts Director, Account Executive or local customer Service Manager or Project Manager 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. A copy is kept in the Project Management directory on the network server.

### 7.3 Documentation

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

The contract will be distributed to and maintained by the appropriate sales/marketing personnel and the Account Executive. A copy of the contract and formal quote will be filed with the laboratory PM and the Laboratory Director.

Records are maintained of pertinent discussions with a client relating to the client's requirements or the results of the work during the period of execution of the contract. The PM keeps a phone log or e-mail chain of conversations with the client.

### 7.3.1 Project-Specific Quality Planning

Communication of contract specific technical and QC criteria is an essential activity in ensuring the success of site specific testing programs. To achieve this goal, the laboratory assigns a PM to each client. It is the Project Manager'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.

Project Manager's are the primary client contact and they ensure resources are available to meet project requirements. Although Project Manager'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 is sufficient to perform work for the client's project. Project management is positioned between the client and laboratory resources.

Prior to work on a new project, the dissemination of project information and/or project opening meetings may occur to discuss schedules and unique aspects of the project. Items to be discussed may include the project technical profile, turnaround times, holding times, methods, analyte lists, reporting limits, deliverables, sample hazards, or other special requirements. The PM introduces new projects to the laboratory staff through project kick-off meetings or to the supervisory staff during production meetings. These meetings provide direction to the laboratory staff in order to maximize production and client satisfaction, while maintaining quality. In addition, a "Client Requirement Memo" may be associated with each sample lot as a reminder of special sample receipt instructions and analytical requirements.

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 may include letters, e-mails, variances and/or contract addendum.

Such changes are also communicated to the laboratory during production meetings. Such changes are updated to the Client Requirement Memo 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 Technical 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 17025 states that a laboratory "shall afford clients or their representative's 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 Managers/Directors 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 SOPs on Subcontracting Procedures (CW-L-S-004) and the Work Sharing Process (CA-C-S-001).

When outsourcing analytical services, the laboratory will assure, to the extent necessary, that the subcontract or work sharing laboratory maintains a program consistent with the requirements of this document, the requirements specified in NELAC/ISO 17025 and/or the client's Quality Assurance Project Plan (QAPP). All QC guidelines specific to the client's analytical program are transmitted to the subcontractor and agreed upon before sending the samples to the subcontract facility. Additionally, work requiring accreditation will be placed with an appropriately accredited laboratory. The laboratory performing the subcontracted work will be identified in the final report, as will non-NELAC accreditation work where required.

For Department of Defense/Department of Energy projects the subcontractor and/or Work Share laboratories used must have an established and documented laboratory quality system that complies with DoD/DOE QSM requirements. The subcontractor and/or Work Share laboratories are evaluated following the procedures outlined below. The subcontractor and/or Work Share laboratory must receive project-specific approval from the DoD/DOE client before any samples are analyzed.

The DoD QSM requirements for subcontracting:

1. Subcontractor laboratories must have an established laboratory quality system that complies with the QSM.
2. Subcontractor laboratories must be accredited by DoD or its designated representatives.
3. Subcontractor laboratories must receive project-specific approval from the DoD client before any samples are analyzed.
4. Subcontractor laboratories are subject to project-specific, on-site assessments by the DoD client or their designated representatives.

The DOE has the following requirements for subcontracting:

> "The laboratory shall not use any sub-tier laboratories or sub-clients (including those possessing the same or similar corporate name) for performance of work under this specification without written approval from the Procurement Representative. The laboratory using the sub-tier laboratory or sub-client shall document and is responsible for ensuring that such sub-client meets all of the requirements of this specification, including being available for client inspections and audits.
>
> Some clients may not allow any subcontracting to third party (sub-tier) laboratories. If this is the case, then this will be specifically noted in the site-specific contracts via Contracts, Task Orders, Laboratory Delivery Orders, etc."

Project Managers (PM), Customer Service Managers (CSM), or Account Executives (AE) 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), such as the DoD/DOE, require notification prior to placing such work.

## 8.2 Qualifying and Monitoring Subcontractors

Whenever a PM [or Account Executive (AE) or Client Relationship Manager, etc.] 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:

- Subcontractors specified by the client - In these circumstances, the client assumes responsibility for the quality of the data generated from the use of a subcontractor.
- Subcontractors reviewed by TestAmerica – Firms which have been reviewed by the company and are known to meet standards for accreditations (e.g., State, TNI and DoD/DOE); technical specifications; legal and financial information.

A listing of vendors is available on the TestAmerica intranet site.

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).

**8.2.1** When the potential sub-contract laboratory has not been previously approved, Account Executives or Project Managers may nominate a laboratory as a subcontractor based on need. The decision to nominate a laboratory must be approved by the Client Relations Manager (CRM) or Laboratory Director. The CRM or Laboratory Director requests that the QA Manager or PM begin the process of approving the subcontract laboratory as outlined in Corporate SOP No. CW-L-S-004, Subcontracting Procedures.

Once the appropriate accreditation and legal information is received by the laboratory, it is evaluated for acceptability (where applicable) and forwarded to the Corporate Quality Information Manager (QIM) for review. After the Corporate QIM reviews the documents for completeness, the information is forwarded to the Finance Department for formal signature and contracting with the laboratory. The approved vendor will be added to the approved subcontractor list on the intranet site and the finance group is concurrently notified for JD Edwards.

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 subcontractors on our approved list can only be recommended to the extent that we would use them.

## 8.3 Oversight and Reporting

**8.3.1** The status and performance of qualified subcontractors will be monitored by the Corporate Quality department. Any problems identified will be brought to the attention of TestAmerica's Corporate Finance, Legal and 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. CSO personnel 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 CSO Personnel, Laboratory Directors, QA Managers and Sales Personnel.

Prior to initially 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 within the project records.

**8.3.2** For continued use of a subcontractor, verification of certification is placed upon the subcontractor for the defined project. Samples are subcontracted under Chain of Custody with the program defined as 'Accreditation Required' and the following statement for verification upon sample receipt:

**Note:** Since laboratory accreditations are subject to change, TestAmerica Laboratories, Inc. places the ownership of method, analyte & accreditation compliance upon our subcontract laboratories. This sample shipment is forwarded under Chain of Custody. If the laboratory does not currently maintain accreditation in the State of Origin listed above for analytes/tests/matrix being analyzed, the samples must be shipped back to the TestAmerica laboratory or other instructions will be provided. Any changes to accreditation status should be brought to TestAmerica Laboratories, Inc. attention immediately. If all requested accreditations are current to date, return the signed Chain of Custody attesting to said compliance to TestAmerica Laboratories, Inc.

For TestAmerica laboratories, certifications can be viewed on the company's Total Access Database.

**8.3.3** All subcontracted samples must be accompanied by a TestAmerica Chain of Custody (COC). A copy of the original COC sent by the client must be available in TALS for all samples work shared 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, clients 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 accredited work must be identified in the subcontractor's report as appropriate. If TNI accreditation is not required, the report does not need to include this information.

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

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

## 8.4 Contingency Planning

With the exception of DoD/DOE and DOE programs the full qualification of a subcontractor may be waived 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 time.

The use of any emergency subcontractor will require the PM to complete a JDE New Vendor Add Form in order to process payment to the vendor and add them to TALS. This form requires the user to define the subcontractor's category/s of testing and the reason for testing.

# 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, consumables 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, laboratory SOP ST-QA-0037, "Procurement of Quality Related Items" and ST-QA0002, "Standard and Reagent Preparation". Approval information for the solvents and acids tested under SOP CA-Q-S-001 is stored on the TestAmerica SharePoint, under Solvent Approvals. A master list of all tested materials, as well as the certificates of analysis for the materials, is stored in the same location.

### 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 SOPs.

The procedure for purchasing/ordering quality related items can be found in the laboratory SOP ST-QA-0037, “Procurement of Quality Related Items”.

### 9.3.2 Receiving

It is the responsibility of the purchasing manager to receive the shipment. It is the responsibility of the analyst who ordered the materials to document the date materials where received. Once the ordered reagents or materials are received, the analyst compares the information on the label or packaging to the original order to ensure that the purchase meets the quality level specified. This is documented through the addition of the received date and initials to the information present on the daily order log.

The purchasing manager verifies the lot numbers of received solvents and acids against the pre-approval lists. If a received material is listed as unapproved, or is not listed, it is sequestered and returned to the vendor. Alternatively, the laboratory may test the material for the intended use, and if it is acceptable, document the approval on the approval list. Records of any testing performed locally are maintained on the shared “public” folder on the computer network.

Materials may not be released for use in the laboratory until they have been inspected, verified as suitable for use, and the inspection/verification has been documented.

Safety Data Sheets (SDS) are available online through the Company’s intranet website. Anyone may review these for relevant information on the safe handling and emergency precautions of on-site chemicals.

### 9.3.3 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. 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 manufacturer’s or SOPs expiration date.

- Standards can be re-verified and a new expiration date applied. See SOP ST-QA-0002, “Standard and Reagent Preparation”.

- 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.

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 3000 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.

Water used in the preparation of standards or reagents must have a specific conductivity of less than 1- µmho/cm (or specific resistivity of greater than 1.0 megohm-cm) at 25$^{o}$C. The specific resistivity is checked and recorded daily. The specific conductivity is checked and recorded monthly. If the water's specific resistivity or conductivity is greater than the specified limit, the Facility Manager and appropriate Technical 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 maintained in electronic files on the network server. 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. Standards and reference materials are stored separately from samples. Storage conditions are per the Corporate Environmental Health & Safety Manual (Corp. Doc. No. CW-E-M-001) and method SOPs or manufacturer instructions.

## 9.4 Purchase of Equipment / Instruments / Software

When a new piece of equipment is needed, either for additional capacity or for replacing inoperable equipment, the analyst or supervisor makes a supply request to the Laboratory Director. If they agree with the request, the procedures outlined in TestAmerica's Corporate Policy No. CA-T-P-001, Qualified Products List, is followed. A decision is made as to which

piece of equipment can best satisfy the requirements. The appropriate written requests are completed and purchasing places the order.

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

### 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 Managers. The service providers that perform the services are approved by the Technical Manager.

Analytical balances are serviced and calibrated annually in accordance with SOP ST-QA-0005. The calibration and maintenance services are performed on-site, and the balances are returned to use immediately following successful calibration. When the calibration certificates are received (usually within two weeks of the service), they are reviewed, and documentation of the review is filed with the certificates. If the calibration was unsuccessful, the balance is immediately removed from service and segregated pending either further maintenance or disposal.

Calibration services for support equipment such as thermometers, weight sets, autopipettors, etc, are obtained from vendors with current and valid ISO 17025 accreditation for calibration of the specific piece of equipment. Prior to utilizing the vendor's services, the vendor's accreditation status is verified. Once the equipment has been calibrated, the calibration certificates are reviewed by the QA department, and documentation of the review is filed with the calibration certificates. The equipment is then returned to service within the laboratory

### 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 J.D. 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 Technical Services 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 is documented in the laboratory's iCAT Database or LIMS NCM module.
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### 10.2 External Complaints

An employee that receives a complaint initiates the complaint resolution process by first documenting the complaint according to SOP ST-QA-0036 "Non-conformance Memorandum (NCM)/Validation Request and Corrective Action Processes".

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 supervisor for resolution. The supervisor may elect to discuss it with the QA Manager or Technical Director or have a representative contact the client to decide on a logical course of action. Once an approach is agreed upon, the analyst documents it using the laboratories corrective action system described in Section 12. This information can then be supplied to the client in the case narrative sent 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 Manager Director and QA Manager, documented and included in the project folder. Deviations **must** also be noted on the final report

with a statement that the compound is not reported in compliance with NELAC (or the analytical method) requirements and the reason. Data being reported to a non- NELAC state would need to note the change made to how the method is normally run.

### 11.2 Responsibilities and Authorities

Under certain circumstances, the Laboratory Director, a Technical 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, and the Technical Managers. The reporting of issues involving alleged violations of the company's Data Integrity or Manual Integration procedures must be conveyed to an ECO (e.g., the VP-QA/EHS) 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, VP of Operations and the Quality Directors have the authority and responsibility to halt work, withhold final reports, or suspend an analysis for due cause as well as authorize the resumption of work.

### 11.3 Evaluation of Significance and Actions Taken

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

Corporate SOP entitled Data Recalls (CW-Q-S-005) is the procedure to be followed when it is discovered that erroneous or biased data may have been reported to clients or regulatory agencies.

Corporate SOP entitled Internal Investigations (CW-L-S-002) is the procedure to be followed for investigation and correction of situations involved alleged incidents of misconduct or violation of the company's ethics policy.

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-Q-S-005.

### 11.4 Prevention of Non-Conforming Work

If it is determined that the nonconforming work could recur, further corrective actions must be made following the laboratory's corrective action system. Monthly 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 need to be followed.

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

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

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

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

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

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

Within 72 hours, the QA Manager will determine if compliance is now met and reports can be released, OR determine the plan of action to bring work into compliance, and release work. A team, with all principals involved (Laboratory Director, Technical Manager, Technical Director, QA 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. Corrective actions are documented using Non-Conformance Memos (NCM) and the iCAT database/ (refer to SOP ST-QA-0036).

For DOE, DoD and other programs where required, the client will be informed of proposed corrective actions.

### 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 Non-Conformance Memo (NCM)** - is used to document the following types of corrective actions:

- Deviations from an established procedure or SOP
- QC outside of limits (non-matrix related)
- Isolated reporting / calculation errors
- Discrepancies in materials / goods received vs. manufacturer packing slips.
- Questionable trends that are found in the review of NCMs.
- Issues found while reviewing NCMs that warrant further investigation.
- Internal and external audit findings
- Systematic reporting / calculation errors
- Client complaints
- Data recall investigations

- Identified poor process or method performance trends
- Excessive revised reports

**12.2.2 Corrective Action Tracker (iCAT)** - is used to document the following types of corrective actions:

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

Health and Safety violations are documented in the EH&S Quarterly Inspection Reports

This 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. An NCM or the iCAT database entry 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 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 NCM or iCAT entry 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. Corporate SOP Root Cause Analysis (No. CA-Q-S-009) describes the procedure.

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 Technical Manager and QA Manager are responsible to ensure that the corrective action taken was effective.
- Ineffective actions are documented and re-evaluated until acceptable resolution is achieved. Technical Managers are accountable to the Laboratory Director to ensure final acceptable resolution is achieved and documented appropriately.
- Each NCM and iCAT entry is entered into a database for tracking purposes and a monthly review of all corrective actions may be printed out for review to aid in ensuring that the corrective actions have taken effect.
- The QA Manager reviews monthly NCMs and Validation Requests 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.
- TestAmerica laboratories began using the Incident/Corrective Action Tracker (iCAT) database developed by the company in 2015. (Previously, a local database served this purpose.) An incident is an event triggering the need for one or more corrective actions as distinct from a corrective action, a potential deficiency stemming from an incident that requires investigation and possibly fixing. The database is independent of TALS, available

to all local and corporate managers, and capable of notifying and tracking multiple corrective actions per event, dates, and personnel. iCAT allows associated document upload, categorization (such as, external/internal audit, client service concerns, data quality issues, proficiency testing, etc.), and trend analysis.

- 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 an NCM or Validation Request.

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

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

To the extent possible, samples shall be reported only if all quality control measures are acceptable. If the deficiency does not impair the usability of the results, data will be reported with an appropriate data qualifier and/or the deficiency will be noted in the case narrative. Where sample results may be impaired, the Project Manager is notified by an NCM and appropriate corrective action (e.g., reanalysis) is taken and documented**.**

## 12.5 Basic Corrections

When mistakes occur in records, each mistake shall be crossed-out and 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.

**Table 12-1. Example – General Corrective Action Procedures**

| QC Activity *(Individual Responsible for Initiation/Assessment)* | Acceptance Criteria | Recommended Corrective Action |
|---|---|---|
| Initial Instrument Blank<br><br>*(Analyst)* | - Instrument response < RL. | - Prepare another blank.<br>- If same response, determine cause of contamination: reagents, environment, instrument equipment failure, etc... |
| Initial Calibration Standards<br><br>*(Analyst, Technical Manager(s))* | - Correlation coefficient > 0.99 or standard concentration value.<br>- % Recovery within acceptance range.<br>- See details in Method SOP. | - Reanalyze standards.<br>- If still unacceptable, remake standards and recalibrate instrument. |
| Independent Calibration Verification (Second Source)<br><br>*(Analyst, Technical Manager(s))* | - % Recovery within control limits. | - Remake and reanalyze standard.<br>- If still unacceptable, then remake calibration standards or use new primary standards and recalibrate instrument. |
| Continuing Calibration Standards<br><br>*(Analyst, Data Reviewer)* | % Recovery within control limits documented in QC Browser database | - reanalyze standard<br>-if still unacceptable, recalibrate and rerun affected samples |

| QC Activity *(Individual Responsible for Initiation/Assessment)* | Acceptance Criteria | Recommended Corrective Action |
|---|---|---|
| Matrix Spike / Matrix Spike Duplicate (MS/MSD)<br><br>*(Analyst, Data Reviewer)* | - % Recovery within limits documented in the LIMS | - 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.<br>- If the LCS is within acceptable limits the batch is acceptable.<br>- The results of the duplicates, matrix spikes and the LCS are reported with the data set.<br>- For matrix spike or duplicate results outside criteria the data for that sample shall be reported with qualifiers. |
| Laboratory Control Sample (LCS)<br><br>*(Analyst, Data Reviewer)* | - % Recovery within limits specified in the LIMS | - Batch must be re-prepared and re-analyzed. This includes any allowable marginal exceedance.<br>When not using marginal exceedances, the following exceptions apply:<br>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;<br>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.<br><br>**Note:** If there is insufficient sample or the holding time cannot be met, contact client and report with flags. |
| Surrogates<br><br>*(Analyst, Data Reviewer)* | - % Recovery within limits of method or within three standard deviations of the historical mean. | - Individual sample must be repeated. Place comment in LIMS.<br>- Surrogate results outside criteria shall be reported with qualifiers. |
| Method Blank (MB)<br><br>*(Analyst, Data Reviewer)* | < Reporting Limit [1] | - Reanalyze blank.<br>- If still positive, determine source of contamination. If necessary, reprocess (i.e. digest or extract) entire sample batch. Report blank results.<br>- Qualify the result(s) if the concentration of a targeted analyte in the MB is at or above the reporting limit AND is > 1/10 of the amount measured in the sample. |

| QC Activity *(Individual Responsible for Initiation/Assessment)* | Acceptance Criteria | Recommended Corrective Action |
|---|---|---|
| Proficiency Testing (PT) Samples<br><br>*(QA Manager, Technical Manager(s))* | - Criteria supplied by PT Supplier. | - Any failures or warnings must be investigated for cause. Failures may result in the need to repeat a PT sample to show the problem is corrected. |
| Internal / External Audits<br><br>*(QA Manager, Technical Manager(s) Laboratory Director)* | - Defined in Quality System documentation such as SOPs, QAM, etc... | - Non-conformances must be investigated through Validation system and necessary corrections must be made. |
| Reporting / Calculation Errors<br><br>(Depends on issue – possible individuals include: Analysts, Data Reviewers, Project Managers, Technical Managers, QA Manager, Corporate QA, Corporate Management) | - SOP CW-Q-S-005, Data Recall | - Corrective action is determined by type of error. Follow the procedures in SOP CW-L-S-002. |
| Client Complaints<br><br>*(Project Managers, Lab Director/Manager, Sales and Marketing)* | - | - Corrective action is determined by the type of complaint. For example, a complaint regarding an incorrect address on a report will result in the report being corrected and then follow-up must be performed on the reasons the address was incorrect (e.g., database needs to be updated). |
| QA Monthly Report (Refer to Section 16 for an example)<br><br>(QA Manager, Lab Director/Manager, *Technical Manager(s)*) | - QAM, SOPs. | - Corrective action is determined by the type of issue. For example, NCMs and Validations for the month are reviewed and possible trends are investigated. |
| Health and Safety Violation<br><br>(Safety Officer, Lab Director/Manager, *Technical Manager(s)*) | - Environmental Health and Safety (EHS) Manual. | - Non-conformance is investigated and corrected |

**Note:**
1. Except as noted below for certain compounds, the method blank should be below the detection limit. Concentrations up to five times the reporting limit will be allowed for the ubiquitous laboratory and reagent contaminants: methylene chloride, toluene, acetone, 2-butanone and phthalates **provided** they appear in similar levels in the reagent blank and samples. This allowance presumes that the detection limit is significantly below any regulatory limit to which the data are to be compared and that blank subtraction will not occur.

## 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 through any of the following:
- review of the monthly QA Metrics Report,
- trending NCMs,
- review of control charts and QC results,
- trending proficiency testing (PT) results,
- performance of management system reviews,
- trending client complaints,
- review of processing operations, or
- staff observations.

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.

## 13.2 Management of Change

The Management of Change process is designed to manage significant events and changes that occur within the laboratory. Through these procedures, the potential risks inherent with a new event or change are identified and evaluated. The risks are minimized or eliminated through pre-planning and the development of preventive measures. The types of changes covered under this system include: Facility Changes, Major Accreditation Changes, Addition or Deletion to Division's Capabilities or Instrumentation, Key Personnel Changes, Laboratory Information Management System (LIMS) changes.

TestAmerica St. Louis uses a series of spreadsheets and/or databases to track changes to major capabilities (e.g. equipment, accreditations, etc.). An equipment list is maintained by the QA department. Accreditations are maintained via the OASIS Total Access program on the TestAmerica intranet site.

## 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. More detailed information on retention of specific records is provided in CW-L-P-001, Records Retention Policy and CW-L-WI-001, TestAmerica Records Retention/Storage Schedule. Quality records are maintained by the QA department electronically, which are 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 in LIMS (raw data, analytical records, lab reports) and the QA Department (logbooks, standards, certificates, Quality documents).

**Table 14-1. Record Index[1]**

| | Record Types [1]: | Retention Time: |
|---|---|---|
| **Technical Records** | - Raw Data<br>- Logbooks[2]<br>- Standards<br>- Certificates<br>- Analytical Records<br>- MDLs/IDLs/DOCs<br>- Lab Reports | 5 Years from analytical report issue* |
| **Official Documents** | - Quality Assurance Manual (QAM)<br>- Work Instructions<br>- Policies<br>- SOPs<br>- Policy Memorandums<br>- Manuals | Indefinitely |
| **QA Records** | - Certifications<br>- Method & Software Validation / Verification Data | Indefinitely |
| **QA Records** | - Internal & External Audits/Responses<br>- Corrective/Preventive Actions<br>- Management Reviews<br>- Data Investigation | 5 Years from archival*<br><br>**Data Investigation:** 5 years or the life of the affected raw data storage whichever is greater (beyond 5 years if ongoing project or pending investigation) |

| | Record Types [1]: | Retention Time: |
|---|---|---|
| **Project Records** | - Sample Receipt & COC Documents<br>- Contracts and Amendments<br>- Correspondence<br>- QAPP<br>- SAP<br>- Telephone Logbooks<br>- Lab Reports | 5 Years from analytical report issue* |
| **Administrative Records** | Financial And Business Operations | Refer to CW-L-WI-001 |
| | EH&S Manual, Permits | Indefinitely |
| | Disposal Records | Indefinitely |
| | Employee Handbook | Indefinitely |
| | Personnel files, Employee Signature & Initials, Administrative Training Records (e.g., Ethics) | Refer to HR Manual |
| | Administrative Policies | Indefinitely |
| | Technical Training Records | 7 years |
| | Legal Records | Indefinitely |
| | HR Records | Refer to CW-L-WI-001 |
| | IT Records | Refer to CW-L-WI-001 |
| | Corporate Governance Records | Refer to CW-L-WI-001 |
| | Sales and Marketing | 5 years |
| | Real Estate | Indefinitely |

[1] Record Types encompass hardcopy and electronic records.
[2] Examples of Logbook types: Maintenance, Instrument Run, Preparation (standard and samples), Standard and Reagent Receipt, Archiving, Balance Calibration, Temperature (hardcopy or electronic records).
* Exceptions listed in Table 14-2.

**14.1.1** All records are stored and retained in such a way that they are secure and readily retrievable at the laboratory facility or an offsite location that provides a suitable environment to prevent damage or deterioration and to prevent loss**.** All records shall be protected against fire, theft, loss, environmental deterioration, and vermin. In the case of electronic records, electronic or magnetic sources, storage media are protected from deterioration caused by magnetic fields and/or electronic deterioration.

Access to the data is limited to laboratory and company employees and shall be documented with an access log**.** Whether on-site or off-site storage is used, logs are maintained in each storage box to note removal and return of records. Records are maintained for a minimum of five years unless otherwise specified by a client or regulatory requirement.

For raw data and project records, record retention shall be calculated from the date the project report is issued. For other records, such as Controlled Documents, QA, or Administrative Records, the retention time is calculated from the date the record is formally retired. Records related to the programs listed in Table 14-2 have lengthier retention requirements and are subject to the requirements in Section 14.1.2

### 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. For projects/programs that require a retention time longer than five years, the Project Manager notes the data retention requirement in the LIMS.

**Table 14-2. Example: Special Record Retention Requirements**

| Program | [1]Retention Requirement |
|---|---|
| Drinking Water – All States | 10 years (lab reports and raw data)<br>10 years - Radiochemistry (project records) |
| Drinking Water Lead and Copper Rule | 12 years (project records) |
| Commonwealth of MA – All environmental data 310 CMR 42.14 | 10 years |
| FIFRA – 40 CFR Part 160 | Retain for life of research or marketing permit for pesticides regulated by EPA |
| Housing and Urban Development (HUD) Environmental Lead Testing | 10 years |
| Alaska | 10 years |
| Louisiana – All | 10 years |
| Michigan Department of Environmental Quality – all environmental data | 10 years |
| Navy Facilities Engineering Service Center (NFESC) | 10 years |
| NY Potable Water NYCRR Part 55-2 | 10 years |
| Ohio VAP | 10 years and State contacted prior to disposal |
| TSCA - 40 CFR Part 792 | 10 years after publication of final test rule or negotiated test agreement |
| OSHA | 30 years |

[1]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 19.15.1 for more information.

**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 laboratory report. The chain of custody would indicate the name of the sampler. A log of names, initials and signatures for all individuals responsible for signing or initialing laboratory records is maintained in the Human Resources Department. If any sampling notes are provided with a work order, they are kept with the laboratory report.

- All information relating to the laboratory facilities equipment, analytical test methods, and related laboratory activities, such as sample receipt, sample preparation, or data verification are documented.

- The record keeping system facilitates the retrieval of all working files and archived records for inspection and verification purposes (e.g., set format for naming electronic files, set format for what is included with a given analytical data set). Instrument data is stored sequentially by instrument. A given day's analyses are maintained in the order of the analysis. Run logs are maintained for each instrument or method; a copy of each day's run long or instrument sequence is stored with the data to aid in re-constructing an analytical sequence. Where an analysis is performed without an instrument, bound logbooks or bench sheets are used to record and file data. Standard and reagent information is recorded in the Reagent Log in the LIMS and relevant printouts can be included in the data packages as needed.

- 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.

- Also refer to Section 19.15.1 'Computer and Electronic Data Related Requirements'**.**

## 14.2 Technical and Analytical Records

**14.2.1** The laboratory retains records of original observations, derived data and sufficient information to establish an audit trail, calibration records, staff records and a copy of each analytical report issued, for a minimum of five years unless otherwise specified by a client or regulatory requirement. The records for each analysis shall contain sufficient information to enable the analysis to be repeated under conditions as close as possible to the original. The records shall include the identity of laboratory personnel responsible for the performance of each analysis and reviewing results.

**14.2.2** Observations, data and calculations are recorded real-time and are identifiable to the specific task.

**14.2.3** Changes to hardcopy records shall follow the procedures outlined in Section 12 and 19. Changes to electronic records in LIMS or instrument data are recorded in audit trails. The essential information to be associated with analysis, such as strip charts, tabular printouts, computer data files, analytical notebooks, and run logs, include:

- laboratory sample ID code**;**
- Date of analysis; Time of Analysis is also required if the holding time is seventy-two (72) hours or less, or when time critical steps are included in the analysis (e.g., drying times, incubations, etc.); instrumental analyses have the date and time of analysis recorded as part of their general operations. Where a time critical step exists in an analysis, location for such a time is included as part of the documentation in a specific logbook or on a bench sheet.
- Instrumentation identification and instrument operating conditions/parameters. Operating conditions/parameters are typically recorded in instrument maintenance logs or posted on the instrument.
- 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.
- Chain of Custody protocols required by DOE and DoD

## 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 numbered sequentially. Within each logbook, pages are sequentially numbered. All data are recorded sequentially within a series of sequential notebooks. Bench sheets are filed sequentially. Standards are maintained in the Reagents Log Program in LIMS. Records are considered archived when moved off-site or are so labeled. Dual storage of these records is maintained by the IT Department during its daily and weekly back-ups of the laboratory network. These back-up tapes are stored off-site.

### 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. CW-Q-S-003. 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<br>QA Technical Audits | Joint responsibility:<br>a) QA Manager or designee<br>b) Technical Manager or Designee<br>(Refer to CA-Q-S-003) | Methods Audits Frequency:<br>50% of methods annually |
| SOP Method Compliance | Joint responsibility:<br>c) QA Manager or designee<br>d) Technical Manager or Designee<br>(Refer to CA-Q-S-003) | SOP Compliance Review Frequency:<br>• Every 2 years<br>• **100% of SOPs annually (DoD/DOE Labs)** |
| 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 applicable regulatory requirements |

### 15.1.1 Audit Planning/Reporting

An audit plan is developed to identify the scope of the audit, the time frame, the personnel involved, the activities to be included, reference documents (i.e. Methods, SOPs, Checklists, and Client Requirement Memos) and persons to be notified of results. The audit team is selected prior to the audit. The size of the team is dependent on the scope of the audit. The lead auditor organizes and directs the audit. The audit report is issued to the appropriate departments by the lead auditor in hardcopy or electronically. The audit report is signed or otherwise endorsed by the Lead Auditor. The report describes the scope of the audit, identifies auditors and persons contacted, summarizes results and describes all non-conformances found.

### 15.1.2 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.

### 15.1.3 QA Technical Audits

QA technical audits assess data authenticity and analyst integrity. These 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., Chrom AuditMiner) are used to identify unusual manipulations of the data deserving closer scrutiny. QA technical audits will include all methods within a two-year period. All analysts should be reviewed over the course of a two year period through at least one QA Technical Audit

### 15.1.4 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 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.

### 15.1.5 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.

### 15.1.6 Performance Testing

The laboratory participates semi-annually in performance audits conducted through the analysis of PT samples provided by a third party. The laboratory generally participates in the following types of PT studies: Drinking Water, Non-potable Water, Soil and Radiochemistry.

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 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 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, their Quality Director as well as the General Manager. All aspects of the QA system are reviewed to evaluate the suitability of policies and procedures. During the course of the year, the Laboratory Director, General Manager or Corporate QA may request that additional information be added to the report.

On a monthly basis, Corporate QA compiles information from all the monthly laboratory reports. The Corporate Quality Directors prepare a report that includes a compilation of all metrics and notable information and concerns regarding the QA programs within the laboratories. The report also includes a listing of new regulations that may potentially impact the laboratories. This report is presented to the Senior Management Team and General Managers.

### 16.2 Annual Management Review

The senior lab management team (Laboratory Director, Technical Director, Technical Managers, QA Manager, EH&S Manager and Radiation Safety Officer) 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 may be included in this meeting at the discretion of the Laboratory Director. The LIMS review consists of examining any audits, complaints or concerns that have been raised through the year that is related to the LIMS. The laboratory will summarize any critical findings that cannot be solved by the lab and report them to Corporate IT.

This management systems review (Corporate SOP No. CW-Q-S-004 & Work Instruction No. CW-Q-WI-003) 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
- Internal and External audit outcomes & corrective actions
- 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.
- Changes in the volume and type of work

- 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.
- Laboratory health and safety issues
- Radioactive materials management issues
    - Radiation Health and Safety
    - Radioactive hazardous waste management
    - Radioactive materials management

A report is generated by the QA Manager and management. The report is distributed to the appropriate General Manager and the Quality Director. The report includes, but is not limited to:

- The date of the review and the names and titles of participants.
- A reference to the existing data quality related documents and topics that were reviewed.
- Quality system or operational changes or improvements that will be made as a result of the review [e.g., an implementation schedule including assigned responsibilities for the changes].

Changes to the quality systems requiring update to the laboratory QA Manual shall be included in the next revision of the QA Manual. Quality system changes and improvements are incorporated into the laboratory's yearly goals.

### 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 Internal Investigations 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 President and CEO, Executive VP of Operations, VP of Client & Technical Services, VPs of Operations and Quality Directors receive a monthly report from the VP-QA/EHS summarizing any current data integrity or data recall investigations. The VPs of Operations 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.

Management is responsible for authorizing specific personnel to perform specific tests (i.e. environmental testing, issue reports, interpret data, operate equipment).

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.

The laboratory ensures that all personnel, including part time, temporary, contracted and administrative personnel, are trained in basic laboratory QA and safety programs.

Personnel dealing with sample receipt, radioactive waste management and materials shipping are trained in waste management, shipping and handling, and hazardous and/or radioactive materials control as appropriate.

## 17.2 Education and Experience Requirements for Technical Personnel

Selection of qualified candidates for laboratory employment begins with documentation of minimum education, training, and experience prerequisites needed to perform the prescribed task. Minimum education and training requirements for TestAmerica employees are outlined in job descriptions and are generally summarized for analytical staff in the table below.

The laboratory maintains job descriptions for all personnel who manage, perform or verify work affecting the quality of the environmental testing the laboratory performs. Job Descriptions are located on the TestAmerica intranet site's Human Resources web-page (Also see Section 4 for position descriptions/responsibilities).

Experience and specialized training are occasionally accepted in lieu of a college degree (basic lab skills such as using a balance, colony counting, aseptic or quantitation techniques, etc., are also considered).

As a general rule for analytical staff:

| Specialty | Education | Experience |
|---|---|---|
| Extractions, Digestions, some electrode methods (pH, DO, Redox, etc.), or Titrimetric and Gravimetric Analyses | H.S. Diploma | On the job training (OJT) |
| CVAA, Single component or short list Chromatography (e.g., Fuels, BTEX-GC, IC | A college degree in an applied science or 2 years of college and at least 1 year of college chemistry | Or 2 years prior analytical experience is required |
| ICP, ICPMS, Long List or complex chromatography (e.g., Pesticides, PCB, Herbicides, HPLC, etc.), GCMS | A college degree in an applied science or 2 years of college chemistry | or 5 years of prior analytical experience |
| Spectra Interpretation | A college degree in an applied science or 2 years of college chemistry | And 2 years relevant experience<br>Or<br>5 years of prior analytical experience |
| Technical Managers – General | Bachelors Degree in an applied science or engineering with 24 semester hours in chemistry<br><br>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 Managers – Wet Chemistry only (no advanced instrumentation) | Associates degree in an applied science or engineering or 2 years of college with 16 semester hours in chemistry | And 2 years relevant experience |

When an analyst does not meet these requirements, they can perform a task under the direct supervision of a qualified analyst, peer reviewers or Technical Manager, and are considered an analyst in training. The person supervising an analyst in training is accountable for the quality of the analytical data and must review and approve data and associated corrective actions.

### 17.3Training

The laboratory is committed to furthering the professional and technical development of employees at all levels. See the laboratory SOP ST-QA-0044 Training for additional information.

Orientation to the laboratory's policies and procedures, in-house method training, and employee attendance at outside training courses and conferences all contribute toward employee proficiency. Below are examples of various areas of required employee training:

| Required Training | Time Frame | Employee Type |
|---|---|---|
| Environmental Health & Safety | Prior to lab work | All |
| Ethics – New Hires | 1 week of hire | All |
| Ethics – Comprehensive | 90 days of hire | All |
| Data Integrity | 30 days of hire | Technical and PMs |
| Quality Assurance | 90 days of hire | All |
| Ethics – Comprehensive Refresher | Annually | All |
| Computer Security Awareness | Annually | All |
| Initial Demonstration of Capability (DOC) | Prior to unsupervised method performance | Technical |

The laboratory maintains records of relevant authorization/competence, education, professional qualifications, training, skills and experience of technical personnel (including contracted personnel) as well as the date that approval/authorization was given. These records are kept on file at the laboratory. Also refer to "Demonstration of Capability" in Section 19.

The following documentation must be on file at the laboratory for each employee:

- Ethics Training documentation
- Signed Ethics agreement
- Signed Confidentiality agreement
- TNI statement of qualification
- Copy of degree, if applicable
- New Employee Orientation checklist
- Safety Orientation checklist

In addition to items listed above, the following documentation is also included in the employee training record:

- Department training checklist
- Demonstration of Capability (IDOC/DOC)
- Manual Integration training, if applicable
- Annual evidence of continuing DOC (may be successful analysis of a blind sample on the specific test method, or a similar method or four successful LCS analyses.
- Specialty training as applicable

The training of technical staff is kept up to date by:

- Each employee must have documentation filed with the QA department 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 is 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 maintain documentation and attestation forms on employment status & records; benefit programs; timekeeping/payroll; and employee conduct (e.g., ethics violations). This information is maintained in the employee's secured personnel file.

Evidence of successful training could include such items as:

- Adequate documentation of training within operational areas, including one-on-one technical training for individual technologies, and particularly for people cross-trained.
- Analyst's knowledge to refer to QA Manual for quality issues.
- Analysts following SOPs, i.e., practice match SOPs.
- Analysts regularly communicate to supervisors and QA if SOPs need revision, rather than waiting for auditors to find problems.

### 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 quarterly refreshers 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 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. The Ethics Statement is re-signed annually.

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 52,000 $ft^2$ 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, radiological 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.

When any of the method or regulatory required environmental conditions change to a point where they may adversely affect test results, analytical testing will be discontinued until the environmental conditions are returned to the required levels.

Environmental conditions of the facility housing the computer network and LIMS are regulated to protect against raw data loss.

## 18.3 Work Areas

There is effective separation between neighboring areas when the activities therein are incompatible with each other. Examples include:

- Volatile organic chemical handling areas, including sample preparation and waste disposal, and volatile organic chemical analysis areas.
- Separate high and low level radiochemical preparation areas

Access to and use of all areas affecting the quality of analytical testing is defined and controlled by secure access to the laboratory building as described below in the Building Security section.

Adequate measures are taken to ensure good housekeeping in the laboratory and to ensure that any contamination does not adversely affect data quality. These measures include regular cleaning to control dirt and dust within the laboratory. Work areas are available to ensure an unencumbered work area. Work areas include:

- Access and entryways to the laboratory.
- Sample receipt areas.
- Sample storage areas.
- Chemical and waste storage areas.

- Data handling and storage areas.
- Sample processing areas.
- Sample analysis areas.

### 18.4 Floor Plan

A floor plan can be found in Appendix 1.

### 18.5 Building Security

Building keys are distributed to management as necessary. The Human Resources Manager maintains a list of all employees who have been issued keys. Electronic "swipe" cards are issued to all laboratory employees.

All visitors to the laboratory enter through the main entrance and sign in and out in a visitor's logbook. A visitor is defined as any person who visits the laboratory who is not an employee of the laboratory. In addition to signing into the laboratory, the Environmental, Health and Safety Manual contains requirements for visitors and vendors. There are specific safety forms that must be reviewed and signed. Visitors (with the exception of company employees) are given a visitor's badge and are escorted by laboratory personnel at all times. Vendors may be issued badges which state that escorts are not required. Visitors and vendors must sign out before leaving the premises.

Entry via the warehouse dock area is permitted for client sample delivery or material supply delivery, without Visitor Log sign-in. The Sample Control Department is responsible for the proper escorting of these visitors.

Vendors issued electronic swipe cards are not required to sign in or out. Visitors from other TestAmerica facilities, while required to sign the Visitor's log, may not require visitor badges.

At the laboratory's discretion, visitors may be asked to show photo identification.

## 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 in the laboratory.

- All SOPs contain a revision number, effective date, and appropriate approval signatures. Controlled copies are available to all staff.
- Procedures for writing an SOP are incorporated by reference to TestAmerica's Corporate SOP entitled 'Writing a Standard Operating Procedure', No. CW-Q-S-002 and the laboratory's SOP ST-QA-0035, "Preparation and Management of Standard Operating Procedures".
- SOPs are reviewed at a minimum of every 2 years (annually for Drinking Water and DoD/DOE SOPs), and where necessary, revised to ensure continuing suitability and compliance with applicable requirements.
- A listing of TestAmerica St. Louis' SOPs is included in Appendix 5.

## 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

Routine analytical services are performed using standard EPA-approved methodology. In some cases, modification of standard approved methods may be necessary to provide accurate analyses of particularly complex matrices. When the use of specific methods for sample analysis is mandated through project or regulatory requirements, only those methods shall be used.

When clients do not specify the method to be used or methods are not required, the methods used will be clearly validated and documented in an SOP and available to clients and/or the end user of the data.

The analytical methods used by the laboratory are those currently accepted and approved by the U. S. EPA and the state or territory from which the samples were collected. Reference methods include:

- *Prescribed Procedures for Measurement of Radioactivity in Drinking Water, EPA-600/4-80-032, August 1980.*
- *Eastern Environmental Radiation Facility Radiochemistry Procedures Manual, EPA, PB84-215581, June 1984.*
- *HASL-300 28th Edition, Environmental Measurements Laboratory (EML), 1997.*
- *Method 1664, Revision A: N-Hexane Extractable Material (HEM: Oil and Grease) and Silica Gel Treated N-Hexane Extractable Material (SGT-HEM): Non-polar Material by Extraction and Gravimetry, EPA-821-R-98-002, February 1999*
- *Guidelines Establishing Test Procedures for the Analysis of Pollutants Under the Clean Water Act, and Appendix A-C; 40 CFR Part 136, USEPA Office of Water. Revised as of July 1, 1995, Appendix A to Part 136 - Methods for Organic Chemical Analysis of Municipal and Industrial Wastewater (EPA 600 Series)*
- *Methods for Chemical Analysis of Water and Wastes, EPA 600 (4-79-020), 1983.*
- *Methods for the Determination of Inorganic Substances in Environmental Samples, EPA-600/R-93/100, August 1993.*
- *Methods for the Determination of Metals in Environmental Samples, EPA/600/4-91/010, June 1991. Supplement I: EPA-600/R-94/111, May 1994.*
- *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).*
- *Standard Methods for the Examination of Water and Wastewater, 18th/19th /20th/ on-line edition; Eaton, A.D. Clesceri, L.S. Greenberg, A.E. Eds; American Water Works Association, Water Pollution Control Federation, American Public Health Association: Washington, D.C.*
- *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, Final Update V, August 2015.*
- *Annual Book of ASTM Standards, American Society for Testing & Materials (ASTM), Philadelphia, PA.*
- *Code of Federal Regulations (CFR) 40, Parts 136, 141, 172, 173, 178, 179 and 261*

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 perform 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 is performed whenever there is a change in instrument type (e.g., new instrumentation), method or personnel.

The initial demonstration of capability must be thoroughly documented and approved by the Technical Manager and QA Manager prior to independently analyzing client samples. All associated documentation must be retained in accordance with the laboratories 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).

For tasks where spiking is not possible (prep techniques including but not limited to compositing, drying and grinding, sub-sampling) the initial demonstration of capability is documented in the analysts training record by the analyst and supervisor signing off on the relevant SOP on the department training checklist. The yearly review and the analyst's acknowledgement of revisions to the SOP serve as the continuing demonstration of capability.

**Note:** In some instances, a situation may arise where a client requests that an unusual analyte be reported using a method where this analyte is not normally reported. If the analyte is being reported for regulatory purposes, the method must meet all procedures outlined within this QA Manual (SOP, MDL, and Demonstration of Capability). If the client states that the information is not for regulatory purposes, the result may be reported as long as the following criteria are met:

- The instrument is calibrated for the analyte to be reported using the criteria for the method and ICV/CCV criteria are met (unless an ICV/CCV is not required by the method or criteria are per project DQOs).
- The laboratory's nominal or default reporting limit (RL) is equal to the quantitation limit (QL), must be at or above the lowest non-zero standard in the calibration curve and must be reliably determined. Project RLs are client specified reporting levels which may be higher than the QL. Results reported below the QL must be qualified as estimated values. Also see Section 19.6.1.3, Relationship of Limit of Detection (LOD) to Quantitation Limit (QL).
- The client request is documented and the lab informs the client of its procedure for working with unusual compounds. The final report must be footnoted.

**19.4.3 Initial Demonstration of Capability (IDOC) Procedures**

**19.4.3.1** The spiking standard used must be prepared independently from those used in instrument calibration.

**19.4.3.2** The analyte(s) shall be diluted in a volume of clean matrix sufficient to prepare four aliquots at the concentration specified by a method or the laboratory SOP.

**19.4.3.3** At least four aliquots shall be prepared (including any applicable clean-up procedures) and analyzed according to the test method (either concurrently or over a period of days).

**19.4.3.4** Using all of the results, calculate the mean recovery in the appropriate reporting units and the standard deviations for each parameter of interest.

**19.4.3.5** When it is not possible to determine the mean and standard deviations, such as for presence, absence and logarithmic values, the laboratory will assess performance against criteria described in the Method SOP.

**19.4.3.6** Compare the information obtained above to the corresponding acceptance criteria for precision and accuracy in the test method (if applicable) or in laboratory generated acceptance criteria (LCS or interim criteria) if there is no mandatory criteria established. If any one of the parameters do not meet the acceptance criteria, the performance is unacceptable for that parameter.

**19.4.3.7** When one or more of the tested parameters fail at least one of the acceptance criteria, the analyst must proceed according to either option listed below:

- Locate and correct the source of the problem and repeat the test for all parameters of interest beginning with 19.4.3.3 above.
- Beginning with 19.4.3.3 above, repeat the test for all parameters that failed to meet criteria. Repeated failure, however, may confirm a general problem with the measurement system. If this occurs, locate and correct the source of the problem and repeat the test for all compounds of interest beginning with 19.4.3.1 above.

Note: Results of successive LCS analyses can be used to fulfill the DOC requirement.

A certification statement (see Figure 19-1) shall be used to document the completion of each initial and continuing demonstration of capability. A copy of the certification is archived in the analyst's training folder.

## 19.5 Laboratory Developed Methods and Non-Standard Methods

Any new method developed by the laboratory must be fully defined in an SOP and validated by qualified personnel with adequate resources to perform the method. Method specifications and the relation to client requirements must be clearly conveyed to the client if the method is a non-standard method (not a published or routinely accepted method). The client must also be in agreement to the use of the non-standard method.

## 19.6 Validation of Methods

Validation is the confirmation by examination and the provision of objective evidence that the particular requirements for a specific intended use are fulfilled.

All non-standard methods, laboratory designed/developed methods, standard methods used outside of their scope, and major modifications to published methods must be validated to confirm they are fit for their intended use. The validation will be as extensive as necessary to meet the needs of the given application. The validation process may include one, or a combination of the following: calibration using known reference standards, comparison of results achieved with other methods, PT samples, etc. 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 a SOP, a 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 can be differentiated from blanks. 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. Generally, the analyst prepares at least seven replicates of solution spiked at one to five times the estimated method detection limit (most often at the lowest standard in the calibration curve) into the applicable matrix with all the analytes of interest. Each of these aliquots is extracted (including any applicable clean-up procedures) and analyzed in the same manner as the samples. Where possible, a minimum of seven replicates should be analyzed over 2-4 days to provide a more realistic MDL. To allow for some flexibility, this low level standard may be analyzed every batch or every week or some other frequency rather than doing the study all at once. In addition, a larger number of data points may be used if the appropriate t-value multiplier is used]

Refer to the Corporate SOP No. CA-Q-S-006 or the laboratory's SOP No. ST-QA-0016 "MDL/IDL, LOD/LOQ Determination", for details on the laboratory's MDL process.

### 19.8 Minimum Detectable Activity (MDA)/Minimum Detectable Concentration (MDC)

For radiochemical analyses, the MDA/MDC is determined based on normal factors and conditions which influence measurement. The MDA/MDC is used to evaluate the capability of a method relative to the required RLs. Sample size, count duration, tracer recovery, detector background and detector efficiency all contribute to determining the sample's MDA/MDC.

The Minimum Detectable Concentration (MDC) for a radionuclide by radiochemical measurement is determined from the blank/background variability associated with the appropriate detector, the detector efficiency, sample aliquot size and chemical yield. The background variability is proportional to the sample count time.

**NOTE:** The background variability is based on the analytical test and derived by: 1) using sample specific parameters, or 2) process blank specific parameters, or 3) by averaging the multiple MDCs derived in 1 or 2.

Matrix material is used whenever possible and is of a similar composition as the client samples.

The MDC is calculated for individual samples (depending on counting technique) using the formulas provided in Appendix 4. The MDC is expected to be less than the client required detection limit. Cesium-137 is the MDC analyte of interest for gamma evaluation.

If the sample MDC is greater than the client required detection limit (CRDL) or reporting limit (RL), the Data Reviewer shall examine the sample volume/weight, counting time, tracer yield and/or other relevant factors. The Data Reviewer shall decide the corrective action which may include reanalysis, recounting or data acceptance and document per laboratory procedure.

### 19.9 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 the MDL but without sample preparation, or by the analysis of 10 instrument blanks and calculating 3 times the absolute value of the standard deviation.

If IDL is > than the MDL, it may be used as the reported MDL.

## 19.10 Verification of Detection and Reporting Limits

Once the MDL is determined, it must be verified on each instrument used for the given method. TestAmerica defines the DoD/DOE QSM Detection Limit (DL) as being equal to the MDL. TestAmerica also defines the DoD/DOE QSM Limit of Detection (LOD) as being equal to the lowest concentration standard that successfully verifies the MDL, also referred to as the MDLV standard. MDL and MDLV standards are extracted/digested and analyzed through the entire analytical process. The MDL and MDLV determinations do 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 MDLV standard is not successful, then the laboratory will redevelop their MDL or perform and pass two consecutive MDLVs at a higher concentration and set the LOD at the higher concentration. Initial and quarterly verification is required for all methods listed in the laboratory's DoD ELAP Scope of Accreditation. Refer to the laboratory SOP ST-QA-0016, "MDL/IDL, LOD/LOQ Determination", for further details.

The laboratory quantitation limit is equivalent to the DoD/DOE Limit of Quantitation (LOQ), which is at a concentration equal to or greater than the lowest non-zero calibration standard. The DoD/DOE QSM requires the laboratory to perform an initial characterization of the bias and precision at the LOQ and quarterly LOQ verifications thereafter. If the quarterly verification results are not consistent with three-standard deviation confidence limits established initially, then the bias and precision will be reevaluated and clients contacted for any on-going projects where required. For DoD/DOE projects, TestAmerica makes a distinction between the Reporting Limit (RL) and the LOQ. The RL is a level at or above the LOQ that is used for specific project reporting purposes, as agreed to between the laboratory and the client. The RL cannot be lower than the LOQ concentration, but may be higher.

## 19.11 Retention Time Windows

Most organic analyses and some inorganic analyses use chromatography techniques for qualitative and quantitative determinations. For every chromatography analysis or as specific in the reference method, each analyte will have a specific time of elution from the column to the detector. This is known as the analytes 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.

## 19.12 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, fluorescence profiles, co-precipitation evaluations and specific electrode response factors.

## 19.13 Estimation of Uncertainty of Measurement

**19.13.1** 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 human factors, adequacy of analyte definition, sampling, matrix effects and interferences, climatic conditions, variances in weights, volumes, and standards, analytical procedure, and random variation. Some national accreditation organizations require the use of an "expanded uncertainty": the range within which the value of the measurand is believed to lie within at least a 95% confidence level with the coverage factor k=2.

**19.13.2** 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.

**19.13.3** 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.

**19.13.4** 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 uncertainties at approximately the 99% confidence level with a coverage factor of k = 3. As an example, for a reported result of 1.0 mg/L with a LCS recovery range of 50 to 150%, the estimated uncertainty in the result would be 1.0 ± 0.5 mg/L. This approach may be used for chemical analyses. For radiochemical uncertainty determination, see the calculations in Appendix 4.

**19.13.5** In the case where a well recognized test method specifies limits to the values of major sources of uncertainty of measurement (e.g., 524.2, 525, etc.) and specifies the form of presentation of calculated results, no further discussion of uncertainty is required.

## 19.14 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 Area Supervisor *or* Laboratory Director if unsure.

### 19.15 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.

### 19.15.1 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 laboratory SOPs ST-IS-0001 "Software Change Management", ST-IS-0002, "Software Testing, Verification and Validation", and ST-IS-0003, "Information Systems". The laboratory is currently running TALS which is a custom in-house developed laboratory information management 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 an industry standard relational database platform. It is referred to as Database for the remainder of this section.

**19.15.1.1 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.

**19.15.1.2** **Ensure Information Availability:** Protection against loss of information or service is ensured through scheduled back-ups, stable file server network architecture, and secure storage of media, line filter, Uninterruptible Power Supply (UPS), and maintaining older versions of software as revisions are implemented.

**19.15.1.3** **Maintain Confidentiality:** Ensure data confidentiality through physical access controls such as password protection or website access approval.

### 19.15.2 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 second level reviewer to confirm the accuracy of the manual entry(s).

Manual integration of peaks will be documented and reviewed and the raw data will be flagged in accordance with the TestAmerica Corporate SOP No. CA-Q-S-002, *Acceptable Manual Integration Practices"* and the laboratory SOP ST-QA-0040, "Manual Integration Procedure".

Analytical results are reduced to the appropriate concentration units as 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.

**19.15.2.1** All raw data must be retained in the reporting departments archive files. 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 (i.e. month/day/year). It must be easily identifiable who performed which tasks if multiple people were involved.

**19.15.2.2** In general, concentration results are reported in milligrams per liter (mg/L) or picocuries per liter (pCi/L) or micrograms per liter (µg/L) for liquids and milligrams per kilogram (mg/kg), micrograms per kilogram (µg/kg) or picocuries per gram (pCi/g) for solids. For values greater than 10,000 mg/L, results can be reported in percent, i.e., 10,000 mg/L = 1**%.**

**19.15.2.3** 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.

**19.15.2.4** 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.

**19.15.2.5** 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 reviews what has been entered to check for errors. If printed, the printout and the instrument's printout of calibrations, concentrations, retention times, chromatograms, and mass spectra, if applicable, are retained with the data file. Where possible, 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. For instruments without the capability of file storage the data is scanned to a pdf file and archived.

### 19.15.3 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.
- Logbooks have sequentially numbered pages.
- Unused portions of pages must be "Z'd" out, signed and dated.
- Worksheets are created with the approval of the QA Manager or Technical Manager at the facility. The QA Department controls all worksheets following the procedures in Section 6.

### 19.15.4 Review / Verification Procedures

Data review procedures are out lined in SOP ST-QA-0046 to ensure that reported data are free from calculation and transcription errors, that QC parameters have been reviewed and evaluated before data is reported. The laboratory also has an SOP discussing Manual Integrations to ensure the authenticity of the data (ST-QA-0040). The general review concepts are discussed below, more specific information can be found in the SOPs.

**19.15.4.1** 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 LIMS. The Sample Control Supervisor, or designee, reviews the transcription of the chain-of-custody forms and the inputted information. The

Project Managers perform final review of the chain-of-custody forms and inputted information.

**19.15.4.2** The next level of data review occurs with the Analysts. As results are generated, analysts review their work to ensure that the results generated meet QC requirements and relevant EPA methodologies. The Analysts transfer the data into the LIMS and add/review 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. One hundred percent of all manual integrations are reviewed. The review is documented on the chromatogram by the analyst responsible for the integration and on the Second Review Checklist by the peer reviewer. Manual integrations are also periodically 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

**19.15.4.3** Unacceptable analytical results may require reanalysis of the samples. Any problems are brought to the attention of the Laboratory Director, Project Manager, Quality Assurance Director/Manager, Technical Manager, or Supervisor for further investigation. Corrective action is initiated whenever necessary.

**19.15.4.4** The results are then entered or directly transferred into the computer database and a hard copy (or .pdf) is created for the client.

**19.15.4.5** 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.

**19.15.4.6** 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.

### 19.15.5 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 our internal SOP No. ST-QA-0040, entitled "Manual Integration Procedure".

**19.15.5.1** The analyst must adjust baseline or the area of a peak in some situations, for example when two compounds are not adequately resolved or when a peak shoulder needs to be separated from the peak of interest. The analyst must use professional judgment and common sense to determine when manual integrating is required. Analysts are encouraged to ask for assistance from a senior analyst or manager when in doubt.

**19.15.5.2** Analysts shall not increase or decrease peak areas for the sole purpose of achieving acceptable QC recoveries that would have otherwise been unacceptable. The intentional recording or reporting of incorrect information (or the intentional omission of correct information) is against company principals and policy and is grounds for immediate termination.

**19.15.5.3** Client samples, performance evaluation samples, and quality control samples are all treated equally when determining whether or not a peak area or baseline should be manually adjusted.

**19.15.5.4** All manual integrations receive a second level review. Manual integrations must be indicated on an expanded scale "after" chromatograms such that the integration performed can be easily evaluated during data review. Expanded scale "before" chromatograms are also required for all manual integrations done on samples, 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**

**Analyst Demonstration of Capability**

TestAmerica St. Louis

Analyst Name

M/DD/YYYY

**Preparation Method(s):**

**Analytical Method(s):**

**Matrix:** Solid/Water/Waste, etc...

**Method Description:**

**Preparation SOP No:** ST-XX-####

**Analytical SOP No:** ST-XX-####

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We, the undersigned, CERTIFY that:

1. The analyst identified above, using the cited test method with the specifications in the cited SOP, which is in use at this facility for the analysis of samples under the laboratory's Quality Assurance Plan, has completed the Demonstration of Capability (DOC).
2. The test method(s) was performed by the analyst identified on this certificate.
3. A copy of test method(s) and laboratory SOPs are available for all personnel on-site. **These documents have been reviewed by the analyst as part of this DOC.**
4. The data associated with the demonstration of capability are true, accurate, complete and self-explanatory.
5. All raw data necessary to reconstruct and validate these analyses have been retained at the facility. The associated information is organized and available for review.

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| | | |
|---|---|---|
| **Analyst** | **Signature** | **Date** |
| **Dept Supervisor** | **Signature** | **Date** |
| **QA Manager** | **Signature** | **Date** |

# 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 laboratory SOPs. A list of laboratory instrumentation is presented in Table 20-1.

Equipment is only operated by authorized and trained personnel. Manufacturer's instructions for equipment use are readily accessible to all appropriate laboratory personnel.

## 20.2 Preventive Maintenance

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 Manager to ensure that instrument maintenance logs are kept for all equipment in his/her department. Preventative maintenance procedures maybe/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. Folder pockets are used in some logbooks to store service receipts.

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. The instrument is "tagged-out" by the analyst who observed the issue, the department manager or the QA department. A non-conformance memo, or some other "tag", is posted on the affected instrument.

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 or MDL verification sample) 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. Calibration requirements for support can be found in Table 20-3.

#### 20.3.1 Weights and Balances

The accuracy of the balances used in the laboratory is checked every working day, before use. All balances are placed on stable counter tops.

Each balance is checked prior to initial serviceable use with at least two certified ASTM type 1 weights spanning its range of use (weights that have been calibrated to ASTM type 1 weights may also be used for daily verification). ASTM type 1 weights used only for calibration of other weights (and no other purpose) are inspected for corrosion, damage or nicks at least annually and if no damage is observed, they are calibrated at least every 5 years by an outside calibration laboratory. Any weights (including ASTM Type 1) used for daily balance checks or

other purposes are recalibrated/recertified annually to NIST standards (this may be done internally if laboratory maintains "calibration only" ASTM type 1 weights).
All balances are serviced annually by a qualified service representative, who supplies the laboratory with a certificate that identifies traceability of the calibration to the NIST standards.

All of this information is recorded in logs, and the recalibration/recertification certificates are kept on file.

Refer to SOP ST-QA-0005, "Calibration and Verification Procedures for Thermometers, Balances, Weights and Pipettes," for detailed information.

### 20.3.2 pH and Conductivity Meters

The pH meters used in the laboratory are accurate to ± 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.

Consult pH and Conductivity SOPs for further information.

### 20.3.3 Thermometers

All thermometers are calibrated on an annual basis with a NIST-traceable thermometer. IR thermometers, digital probes and thermocouples are calibrated quarterly**.**

- If the temperature measuring device is used over a range of 10℃ or less, then a single point verification within the range of use is acceptable;
- If the temperature measuring device is used over a range of greater than 10℃, then the verification must bracket the range of use.

The NIST thermometers are recalibrated every five years (unless thermometer has been exposed to temperature extremes or apparent separation of internal liquid) by an approved outside service and the provided certificate of traceability is kept on file. The NIST thermometer(s) have increments of 1 degree (0.5 degree or less increments are required for drinking water microbiological laboratories), and have ranges applicable to method and certification requirements. The NIST traceable thermometer is used for no other purpose than to calibrate other thermometers.

All of this information is documented in logbooks or filed in QA records. Monitoring of method-specific temperatures, including incubators, heating blocks, water baths, and ovens, is documented in method-specific logbooks. More information on this subject can be found in the SOP ST-QA-0005.

### 20.3.4 Refrigerators/Freezer Units, Water baths, Ovens and Incubators

The temperatures of all refrigerator units and freezers used for sample and standard storage are monitored each working day. (Sample storage is monitored 7 days a week for units storing DOE and/or DoD samples).

Ovens, water baths 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 > 0ºC and ≤ 6 ºC; freezers are kept below 10 ºC.

Specific temperature settings/ranges for other refrigerators, ovens water baths, and incubators can be found in method specific SOPs.

All of this information is documented in the Support Equipment Database..

### 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 applied to the device stating that it is non-critical  Any device not regularly verified cannot be used for any quantitative measurements.

Micro-syringes are purchased from Hamilton Company.  Each syringe is traceable to NIST.  The laboratory keeps on file an “Accuracy and Precision Statement of Conformance” from Hamilton attesting established accuracy.

## 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. Radiochemistry instrumentation calibrations are, at a minimum, verified annually..

### 20.4.1 Calibration Standards

Calibration standards are prepared using the procedures indicated in the Reagents and Standards section of the determinative method SOP. If a reference method does not specify the number of calibration standards, a minimum of 3 calibration points will be used.

Standards for instrument calibration are obtained from a variety of sources. When available, 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 exceptions to these rules ICP and ICPMS methods which define the working range with periodic linear dynamic range studies, rather than through the range of concentrations of daily calibration standards. This also does not apply to radiochemical methods.

All initial calibrations are verified with a standard obtained from a second source and traceable to a national standard, when available (or vendor certified different lot if a second source is not available). For unique situations, such as air analysis where no other source or lot is available, a standard made by a different analyst at a different time or a different preparation would be considered a second source. This verification occurs immediately after the calibration curve has been analyzed, and before the analysis of any samples.

#### 20.4.1.1 Calibration Verification (Organic/Inorganic)

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. The process of calibration verification applies to both external standard and internal standard calibration techniques, as well as to linear and non-linear calibration models. Initial calibration verification is with a standard source secondary (second source standard) to the calibration standards, but continuing calibration verifications may use the same source standards as the calibration curve.

**Note:** The process of calibration verification referred to 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 Standard.

All samples must be bracketed by periodic analyses of standards that meet the QC acceptance criteria (e.g., calibration and retention time). The frequency is found in the determinative methods or SOPs.

**Note:** If an internal standard calibration is being used then bracketing calibration verification standards are not required for some methods, only daily verifications are needed. The results from these verification standards must meet the calibration verification criteria and the retention time criteria (if applicable). The DoD/DOE QSM requires bracketing verification standards even when internal standards are used.

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 every 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 and 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 unacceptable calibration verification may be fully useable under the following special conditions and reported based upon discussion and approval of the client:

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 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 Appendix 4). 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.

- When the acceptance criteria for the calibration verification are exceeded high, i.e., high bias, and there are associated samples that are non-detects, then those non-detects may be reported. Otherwise, the samples affected by the unacceptable calibration verification shall be reanalyzed after a new calibration curve has been established, evaluated and accepted.

- When the acceptance criteria for the calibration verification are exceeded low, i.e., low bias, those sample results may be reported if they exceed a maximum regulatory limit/decision level. Otherwise, the samples affected by the unacceptable verification shall be reanalyzed after a new calibration curve has been established, evaluated and accepted. Alternatively, a reporting limit standard may be analyzed to demonstrate that the laboratory can still support non-detects at their reporting limit.

### 20.4.2 Radiochemical Calibrations

#### 20.4.2.1 CALIBRATION STANDARDS

Shelf life for stock radioactive standards shall not exceed 5 half-lives. Shelf life for stock solutions prepared in the laboratory from salts, metals or dilution from a parent solution shall be no greater than one year, unless stated otherwise on the calibration certificate from the manufacturer. Standards in the form of a soil, sealed sources, filter, plated sources and sealed epoxy Marinelli beakers do not always have an expiration date. After the 1 year shelf life of the stock solution has expired, it must be re-verified.

If the standard is not re-verified, the standard shall be removed or clearly designated as acceptable for qualitative purposes only.

The expiration date of the secondary standard shall not exceed the expiration date of the primary standard.

The accuracy of calibration standards is checked by comparison with a calibration verification standard from a second source. In cases where a second standard source is not available, a source from a different vendor is acceptable. All cases where this requirement cannot be met shall be documented with a nonconformance memo.

When a traceable standard is not available to use for calibration or verification activities, a non-traceable standard may be used if written client approval is obtained (when required).

Calibration standards are prepared using the appropriate procedures.

For each analyte of interest, prepare calibration standards at the minimum number of concentrations as stated in the analytical methods.

Standards for instrument calibration are obtained from a variety of sources. All radioactive standards are traceable to NIST whenever possible. Dilution standards are prepared from stock standards purchased from commercial suppliers. A standard log is maintained, containing concentration/activity, date of receipt, date of standard preparation, any dilutions made, lot number, supplier, type of solvent and a unique code number to identify the standard.

The frequency of calibration can be found in the laboratory's radiochemical methods and Table 20-4.

### 20.4.3 RADIOCHEMICAL CONTINUING INSTRUMENT CALIBRATION, VERIFICATION and RADIOCHEMICAL BACKGROUND MEASUREMENT

Performance checks shall be performed using appropriate check sources and monitored to ensure that the instruments are running properly and that detector response has not significantly changed. Background measurements are made according to the schedule on Table 20-4 and monitored to ensure that the laboratory maintains its capability to meet required data quality objectives.

### 20.4.4 RADIOCHEMICAL INSTRUMENT CONTAMINATION MONITORING

The laboratory radiochemical instrumentation SOPs specify the requirements for monitoring radiochemical instrumentation. The SOP specifies the monitoring frequencies and criteria for initiating corrective action.

## 20.5 Tentatively Identified Compounds (TIC) – GC/MS Analysis

For samples containing components not associated with the calibration standards, a library search may be made for the purpose of tentative identification. The necessity to perform this type of identification will be determined by the purpose of the analyses being conducted. Data system library search routines should not use normalization routines that would misrepresent the library or unknown spectra when compared to each other.

**Note:** If the TIC compound is not part of the client target analyte list but is calibrated by the laboratory and is both qualitatively and/or quantitatively identifiable, it should not be reported as a TIC. If the compound is reported on the same form as true TICs, it should be qualified and/or narrated that the reported compound is qualitatively and quantitatively (if verification in control) reported compared to a known standard that is in control (where applicable).

For example, the RCRA permit or waste delisting requirements may require the reporting of non-target analytes. Only after visual comparison of sample spectra with the nearest library searches may the analyst assign a tentative identification. See SOPs ST-MS-0001 and ST-MS-0002 for guidelines on making tentative identifications and reporting TICs.

### 20.6GC/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. Example: Instrumentation List**

| Equipment/ Instrument | Manufacturer | Model Number | Serial Number | Year(s) Put into Service | Condition When Received |
|---|---|---|---|---|---|
| GC/MS – "F" | Hewlett Packard | 5973 | DE00020247 | 1998 | NEW |
| GC/MS – "F" GC System | Hewlett Packard | 6890 | US80221392 | 1998 | NEW |
| GC/MS – "F" Concentrator | IO | Eclipse 4660 | D530466888P | 2002 | NEW |
| GC/MS – "F" Autosampler | Varian | Archon | 14613 | 2001 | NEW |
| GC/MS – "L" | Hewlett Packard | 5973 | CN10339019 | 2004 | NEW |
| GC/MS – "L" Concentrator | Teledyne Tekmar | Velocity XPT | US03346007 | 2004 | NEW |
| GC/MS – "L" Autosampler | Teledyne Tekmar | SOLATek 72 | US03349002 | 2004 | NEW |
| GC/MS – "M" | Hewlett Packard | 5973 | CN10412013 | 2004 | NEW |
| GC/MS – "M" Concentrator | Teledyne Tekmar | Velocity XPT | US0412001 | 2004 | NEW |
| GC/MS – "M" Autosampler | Teledyne Tekmar | SOLATek 72 | US04119003 | 2004 | NEW |
| GC/MS – "N" | Hewlett Packard | 5973 | CN10512032 | 2005 | NEW |
| GC/MS – "N" GC System | Hewlett Packard | 6890 | US44621325 | 2005 | NEW |
| GC/MS – "N" Concentrator | Tekmar/Dohrman n | Velocity XPT | US03247002 | 2009 | Used |
| GC/MS – "N" Autosampler | Teledyne Teckmar | Solatek 72 | US03100004 | 2009 | Used |
| GC/MS – "K | Hewlett Packard | 5973 | US81221525 | 1998 | NEW |

| Equipment/ Instrument | Manufacturer | Model Number | Serial Number | Year(s) Put into Service | Condition When Received |
|---|---|---|---|---|---|
| GC/MS – "K" GC System | Hewlett Packard | 6890 | US00022347 | 1998 | NEW |
| GC/MS – "K" Series Injector | Hewlett Packard | 7683 | CN31530345 | 1998 | NEW |
| GC/MS – "K" Autosampler | Hewlett Packard | G2614A | US83501656 | 1998 | NEW |
| GC/MS – "J" | Hewlett Packard | 5973 | US80321385 | 1998 | NEW |
| GC/MS – "J" GC System | Hewlett Packard | 6890 | US00021127 | 1998 | NEW |
| GC/MS – "J" Series Injector | Hewlett Packard | 7683 | US81801195 | 1998 | NEW |
| GC/MS – "J" Autosampler | Hewlett Packard | G2614A | US80600251 | 1998 | NEW |
| GC/MS – "I" | Hewlett Packard | 5973 | CN10514049 | 2005 | NEW |
| GC/MS – "I" GC System | Hewlett Packard | G2579A | US44621455 | 2005 | NEW |
| GC/MS – "I" Series Injector | Hewlett Packard | 7683 | CN51224243 | 2005 | NEW |
| GC/MS – "I" Autosampler | Hewlett Packard | G2614A | CN42229061 | 2005 | NEW |
| GC/MS – "X" | Agilent | 5973 | US10461280 | 2008 | NEW |
| GC/MS – "X" GC System | Agilent | 6890N | US10144027 | 2008 | NEW |
| GC/MS – "X" Series Injector | Tekmar | 7683 | US01330017 | 2008 | NEW |
| GC/MS – "X" Autosampler | IO | G2614A | 1411 | 2008 | NEW |
| GC/MS – "Z" | Hewlett Packard | 5973 | US80230105 | 2010 | Refurbished |
| GC/MS – "Z" GC System | Hewlett Packard | 6890 | US00009101 | 2010 | Refurbished |
| GC/MS – "Z" Concentrator | IO | Eclipse 4660 | E002466503P | 2010 | NEW |
| GC/MS – "Z" Autosampler | Varian | Archon | MS1003W019 | 2010 | NEW |
| LC/MS/MS – "R" Mass Spectrometer | Waters | Quattro Premier XE | VAB461 | 2006 | NEW |
| LC/MS/MS – "R" Liquid Chromatograph | Waters | Acquity PDA Detector | L05UPD807N | 2006 | NEW |
| LC/MS/MS – "R" Liquid Chromatograph | Waters | Acquity Sample Manager | 60UPS056M | 2006 | NEW |
| LC/MS/MS – "R" Liquid Chromatograph | Waters | Acquity Binary Solvent Man. | C06UPB008M | 2006 | NEW |
| LC/MS/MS – "T" Mass Spectrometer | Micromass | Ultima | VB280 | 2008 | NEW |

| Equipment/ Instrument | Manufacturer | Model Number | Serial Number | Year(s) Put into Service | Condition When Received |
|---|---|---|---|---|---|
| LC/MS/MS – "T" HPLC – "Q" ALS Therm | Hewlett Packard | G1330A | DE13201124 | 1999 | NEW |
| LC/MS/MS – "T" HPLC – "Q" Quat Pump | Hewlett Packard | G1311A | DE14916965 | 1999 | NEW |
| LC/MS/MS – "X" Liquid Chromatograph | Waters | Xevo | VBA453 | 2010 | NEW |
| LC/MS/MS – "X" Liquid Chromatograph | Waters | Acquity Sample Manager | H07UPB932M | 2010 | NEW |
| LC/MS/MS – "X" Liquid Chromatograph | Waters | Acquity Binary Solvent Manager | H07UPa802M | 2010 | NEW |
| GC – "L" | Hewlett Packard | 5890 | 2413A04451 | 1987 | NEW |
| GC – "L" Autosampler | Varian | Archon | 160098 | 2000 | NEW |
| GC – "L" Concentrator | Tekmar | LSC3000 | 93300001 | 1997 | NEW |
| GC – "K" | Agilent | 6890 | US00039258 | 2000 | NEW |
| GC – "K" Autosampler | Agilent | 7683 | US04709936 | 2000 | NEW |
| GC – "E" | Hewlett Packard | 6890 | US00011425 | 2000 | NEW |
| GC – "E" Autosampler | Hewlett Packard | 6890 | US71701354 | 2000 | NEW |
| GC – "M" | Agilent | 6890 | US10328036 | 2003 | NEW |
| GC – "M" Autosampler | Agilent | 7683 | CN32624339 | 2003 | NEW |
| GC – "O" | Agilent | 6890 | CN10422045 | 2004 | NEW |
| GC – "O" Autosampler | Agilent | 7683 | CN51132513 | 2004 | NEW |
| GC – "P" | Agilent | 6890N | CN10510018 | 2005 | NEW |
| GC – "P" Autosampler | Agilent | 7683 | CN51532846 | 2005 | NEW |
| GC – "W" | Hewlett Packard | 6890 | U5000Z9592 | 2016 | used |
| GC – W" Autosampler | Hewlett Packard | 7673 | 3108A2513 | 2016 | used |
| GC – "V" | Agilent | 6890 | US00008573 | 2009 | USED |
| GC – "V" (Auto Sampler) | Agilent | G1530A | US8090377 | 2009 | USED |
| HPLC – "N" | Hewlett Packard | G1329A | DE91603153 | 1999 | NEW |
| HPLC – "N" ALS Therm | Hewlett Packard | G1330A | DE82203165 | 1999 | NEW |
| HPLC – "N" COLCOM | Hewlett Packard | G1316A | DE91609858 | 1999 | NEW |
| HPLC – "N" DAD | Hewlett Packard | G1315A | DE91605478 | 1999 | NEW |
| HPLC – "N" Degasser | Hewlett Packard | G1322A | JP73016399 | 1999 | NEW |

| Equipment/ Instrument | Manufacturer | Model Number | Serial Number | Year(s) Put into Service | Condition When Received |
|---|---|---|---|---|---|
| HPLC – "N" Quat Pump | Hewlett Packard | G1311A | DE91605960 | 1999 | NEW |
| HPLC – "N" FLD | Hewlett Packard | G1321A | DE92001122 | 1999 | NEW |
| HPLC LCE (DAD) | Agilent | G1315D | DE64255811 | 2010 | USED |
| HPLC LCE (COL) | Agilent | G1316A | DE63065337 | 2010 | USED |
| HPLC LCE (Auto Sampler) | Agilent | G1329A | DE64764168 | 2010 | USED |
| HPLC LCE (Pump) | Agilent | G1311A | DE62962744 | 2010 | USED |
| GPC-1 | O-I Analytical | Autoprep 2000 | E427330254 | 2011 | NEW |
| ICP-MS – "6100" | Perkin Elmer | ELAN 6100 | 0859907 | 1999 | NEW |
| ICP-MS – "6100" Autosampler | Perkin Elmer | AS-91 | 4123 | 1999 | NEW |
| ICP-MS – "7500" | Agilent | 7500CX | JP82802890 | 2009 | NEW |
| ICP-MS – "7700" | Agilent | 7700 | JP10110271 | 2011 | NEW |
| ICP-MS – "9000" | Perkin Elmer | ELAN 9000 | P1000302 | 2013 | USED |
| ICP – "6500 Duel View" | Thermo Fisher | 6000 Series | 20105013 | 2011 | NEW |
| CVAA | Leeman Labs | Hydra AA 2 | 0035 | 2011 | NEW |
| IC – "S" Chromatography Oven | Dionex | LC30 | 98070139 | 2008 | NEW |
| IC – "S" Conductivity Detector | Dionex | CD20 | 99070231 | 2008 | NEW |
| IC – "S" Gradient Pump | Dionex | GP50 | 99070382 | 2008 | NEW |
| IC – "S" Autosampler | Dionex | AS40 | 00090205 | 2008 | NEW |
| IC – "2500" Chromatography Oven | Dionex | LC25 | 03120540 | 2004 | NEW |
| IC – "2500" Conductivity Detector | Dionex | CD25 | 03120540 | 2004 | NEW |
| IC – "2500" Gradient Pump | Dionex | GP50 | 03120633 | 2004 | NEW |
| IC – "2500" Autosampler | Dionex | AS40 | 07020461 | 2004 | NEW |
| IC – "1500" Ion Chromatography System | Dionex | ICS-1500 | 03080236 | 2008 | NEW |
| IC – "1500" Autosampler | Dionex | ASM-3 | 920937 | 2008 | NEW |
| TOX | Mitsubishi | 100 TOX | A7M00017 | 1999 | NEW |
| TOC | Shimadzu | TOC-VCPN | H51404635090 | 2010 | NEW |
| Solid Sample Module | Shimadzu | SSM-5000A | H52504700582NK | 2010 | NEW |

| Equipment/ Instrument | Manufacturer | Model Number | Serial Number | Year(s) Put into Service | Condition When Received |
|---|---|---|---|---|---|
| Discrete Analyzer | Systea | Easy Chem-Plus | 2002E100662 | 2010 | NEW |
| Discrete Analyzer | Systea | Easy Chem-Plus | 0901262 | 2010 | NEW |
| UV Spec 1 | Thermospectronic | Genysis | 3SGF211001 | 2003 | NEW |
| UV Spec 2 | Thermospectronic | Genysis | 3SGR172002 | 2013 | NEW |
| UV Spec | Shimadzu | UV-2401PC | A1083 (320053LP) | 2013 | USED |
| BOD | Man-Tech Associates | 04-227 | 270D3XB245 | 2003 | NEW |
| Ignitability Apparatus: Open Cup | Fisher | D-92 | 906N0014 | 1998 | NEW |
| Ignitability Apparatus: Closed Cup | Fisher | 162 | 1149 | 1992 | NEW |
| Multimeter | Thermo | 5 Star | B15814 | 2009 | NEW |
| Multimeter | Thermo | 5 Star | 015748 | 2009 | NEW |
| Alpha Spectrometer – “AV1 - AV24” “AV43 - AV122” “AV123 - AV226” “AV227 – AV247” | Ortec | Multi-Component | Multiple* | 1987-2011 | NEW |
| Gamma Spectrometer Intrinsic Germanium Detector “GE1 - GE10” “GE11 – GE19” | Tennelec / Ortec | Multi-Component | Multiple* | 1991-2011 | NEW |
| GFPC – “Protean” | Protean | MPC-9604 | 233126-BO<br>236534-BO<br>236532-BO<br>236533-BO | 2003 | NEW |
| GFPC – “Orange” | Protean | MPC-9604 | 08217155<br>08217156<br>08217154<br>08217153<br>10181186<br>10181187 | 2008-2010 | NEW |
| GFPC – “Blue” | Protean | MPC-9604 | 17069480<br>17069481<br>17069482 | 2017 | NEW |
| GFPC – “Purple” | Protean | MPC-9604 | 10181185<br>10181184<br>10029177<br>10029178<br>10029179<br>10029180 | 2010 | NEW |

| Equipment/ Instrument | Manufacturer | Model Number | Serial Number | Year(s) Put into Service | Condition When Received |
|---|---|---|---|---|---|
| GFPC “Green” | Tennelec | LB5100 | 31360 | 2000 | NEW |
| LSC – “3180”<br>Pink<br>Teal<br>Aqua<br>Brown | Packard | Tricarb 3180 | DG06095123<br>DG01117382<br>DG01117385<br>DG01117384<br>DG01117383 | 2009-2011 | NEW |
| LSC – “3170” | Packard | Tricarb 3170 | 429670/429774 | 2002 | NEW |

**Table 20-2. Example: Schedule of Routine Maintenance**

## Inductively Coupled Plasma

DAILY OR AS NEEDED - CHECK

- Gas supply
- Waste and rinse solution levels
- Droplet size (nebulizer)
- Replace orange/green tubing

WEEKLY
- Check water level in cool flow
- Nebulizer rinse
- Replace waste line
- Clean injector tip
- Check /Clean plasma torch assembly
- Replace sample tubing
- Clean spray chamber

MONTHLY
- Check /Clean air filter of power unit
- Clean fast autosampler valve and rotor

ANNUALLY
- Check vacuum system oil
- Check /Replace coolant water filter

## Inductively Coupled Plasma/Mass Spectrometer

DAILY OR AS NEEDED
- Check Waste and rinse water container levels
- Check/ Replace sample, internal and waste lines
- Clean cones (7500, 7700)
- Clean cone

WEEKLY
- Check /Clean interface cones
- Check Roughing pump oil level and color
- Replace Waste Tubing

MONTHLY
- Check /Change pump oil (6100)
- Check /Clean auto lens (6100)
- Clean torch & injector tip (6100)
- Clean auto lense (6100)
- Clean torch (7500, 7700)
- Move data set files (7500, 7700)

## Cold Vapor Automatic Analysis

DAILY OR AS NEEDED
- Check /Pump and drain tubing
- Check Gas pressure
- Instrument parameter check

WEEKLY
- Check /Change sample, reductant and draining tubings

MONTHLY
- Change/rinse tubing
- Check/change waste tubing

QUARTERLY
- Check /Change drying tube

## TOX

DAILY OR AS NEEDED
- Cell Performance Test
- Electrodes
- Cell Fluid, Dehydrating Fluid and Electrolyte
- Adsorption module (cleaned at end of use)

## TOC

DAILY OR AS NEEDED
- Air Supply and Gas Flow Rate (150mm)
- Humidifier
- A/LS Rinse Tank

MONTHLY

- Check /Inspect $SO_3$ scrubber – change if crystals at inlet are not white.
- Check /Inspect halogen scrubber – change if black color approaches outlet end.

ANNUALLY
- Check /Change $CO_2$ absorber

## Ion Chromatography

DAILY OR AS NEEDED
- Plumbing for leaks
- Gases and Pump Pressure
- Conductivity meter
- Fill eluent
- Column replacement

## UV Spec

DAILY OR AS NEEDED
- Rinse out Sample Cuvettes (after each use)

## Discrete Analyzer

DAILY
- Auto zero
- Perform rinse at completion of analysis
- Check DI water bottle/refill

## Alpha Spectrometer

DAILY
- Pulsars

MONTHLY
- Backgrounds
- Clean detectors
- Continuing calibration verifications

ANNUALLY
- Calibrations

## Gamma Spectrometer

DAILY
- Continuing calibration blank/continuing calibration verification

MONTHLY
- Clean/Long Backgrounds

ANNUALLY
- calibration checks

## Gas Flow Proportional Counting

DAILY OR AS NEEDED
- Gas level
- Calibration verifications

MONTHLY
- Clean/Long Backgrounds

ANNUALLY
- Calibrations

## Liquid Scintillation Counter

WEEKLY OR AS NEEDED
- Clean Fan

YEARLY
- Serviced by vendor

## Semi-volatile Gas Chromatography / Mass Spectrometer

DAILY OR AS NEEDED
- Gas supply, column flow and inlet pressure
- Fill solvent rinse vials
- Check /Injection Port Cleaning
- Check /Change Septum, injection port liner, and seals
- Check /Trim Column
- Check/replace injection syringe

ANNUALLY
- Check /Replace pump oil

AS NEEDED
- Replace column
- Clean ion source
- Replace multiplier
- Replace electronic circuit board
- Replace detector
- Replace transfer lines

## Volatile Gas Chromatography / Mass Spectrometer

DAILY OR AS NEEDED
- Gas supply, column flow and inlet pressure

QUARTERLY
- Check Trim Column
- Check/Change Trap

SEMI-ANNUALLY
- Check/Replace Column
- Check/Clean Source
- Check/Injection port maintenance

ANNUALLY
- Check/ Replace pump oil

## High Pressure Liquid Chromatograph (HPLC)

DAILY OR AS NEEDED
- Ensure column flow and pressure are correct
- Ensure HPLC solvents are sufficient to run

- Ensure proper DAD signals are on
- Visibly check for leaks

MONTHLY
- Check/Change Purge Valve Frit

SEMIANNUALLY
- Check/Change Guard Cartridge and Frit Cap

BIANNUALLY
- Check/Replace Column
- Check/Replace UV Source
- Check/Replace Visible Source
- Check/Replace pump seals

## Semi-Volatile Gas Chromatograph (Dual ECD)

DAILY OR AS NEEDED
- Ensure column flow and inlet pressure are correct
- Ensure temperature for oven, inlet(s), and detector(s) are correct
- Ensure solvent rinse vials are full
- Ensure injection syringe is secure in tower and plunger is engaged

MONTHLY
- Check/Replace injection port septum
- Visibly inspect injection port liner; replace if contaminated
- Check /Remove injection syringe and ensure plunger is free moving
- Check system for leaks (injection port, detector(s) and any column connectors)

SEMIANNUALLY
- Perform Radioactive leak test

## Semi-Volatile Gas Chromatograph (FID)

DAILY OR AS NEEDED
- Check gas supply, column flow, and inlet pressure
- Fill solvent rinse vials

MONTHLY
- Check/Replace septum, injection port liner and seals
- Check/ Trim Guard Column

SEMIANNUALLY
- Check/ Replace Column

## Volatile Gas Chromatograph

DAILY OR AS NEEDED
- Check gas supply, column flow and inlet pressure
- Change trap
- Trim column

SEMIANNUALLY

- Check/Replace Column
- Check/Injection port maintenance

ANNUALLY

- Check /Clean PID/FID

**Table 20-3 Example: Periodic Calibration**

| Instrument | Type of Calibration/ Number of Standards | Frequency | Acceptance Limits | Corrective Action |
|---|---|---|---|---|
| Analytical Balance | Accuracy determined using working weights that are annually checked against weights traceable to the International System of Units (SI) through a NMI.<br><br>Minimum of 2 standards bracketing the weight of interest.<br><br>Inspected and checked by ISO17025 accredited vendor annually. | Each day of use | ± 0.1% (QSM requires ± 0.1% or ±0.5 mg, whichever is greater) | Clean, check level, insure lack of drafts, and that unit is warmed up, recheck. If fails, call service. |
| Top Loading Balance | Accuracy determined using ISO17025-accredited NIST weights.<br><br>Minimum of 2 standards bracketing the weight of interest.<br><br>Inspected and checked by ISO17025 accredited vendor annually | Each day of use | ± 2.0% (QSM requires ± 2% or ±0.02 g, whichever is greater) | Clean. Replace. |
| ISO17025-accredited NIST Weights | Verification of standard mass using weights traceable to the International System of Units (SI) through a NMI | 5 years | Certificate of Calibration from ISO/IEC 17025 accredited calibration laboratory. | Replace. |
| NIST-Traceable Thermometer | Accuracy determined by ISO17025-accredited measurement laboratory. | 5 years | As per certificate. | Replace. |
| Thermometer | Against NIST-traceable thermometer | Yearly at appropriate temperature range for intended use | ± 1.0 °C | Replace |
| Digital thermometer | Against NIST-traceable thermometer | Quarterly | ± 1.0 °C | Replace |

| Instrument | Type of Calibration/ Number of Standards | Frequency | Acceptance Limits | Corrective Action |
|---|---|---|---|---|
| Refrigerator | Temperature checked using NIST-traceable thermometer. | Daily. If out of range, check again after several hours | 0 – 6 °C | Adjust. Repair. While waiting for repair, seal door, attach "Out of Service" sign, move items to functional unit. Notify supervisor. |
| Freezer | Temperature checked using NIST-traceable thermometer | Daily. If out of range, check again after several hours | <-10 °C | Adjust. Repair. While waiting for repair, seal door, attach "Out of Service" sign, move items to functional unit. Notify supervisor. |
| Oven | Temperature checked using NIST-traceable thermometer. | When in use. | 103 ± 2 °C (moisture determination) 180 ± 2°C (TDS) (DoD/DOE): ±5% of set temp) | Adjust. Replace. |
| Incubator | Temperature checked using NIST-traceable thermometer. | When in use. For microbiology, twice daily when in use. | BOD: 20 ± 1.0 °C | Adjust. Replace. |
| Water Bath | Temperature checked using NIST-traceable thermometer. | When in use. | ± 5 °C | Adjust. Replace. |
| Volumetric Dispensing Devices - pipettes | On delivery by weight. Using DI water, dispense into tared vessel. Record weight with device ID number.<br><br>Before first use: 10 replicate measurements with %RSD ≤ 1%. | Day of use 3 reps | ± 2% bias Precision RSD ≤ 1% | Adjust. Replace. |
| Non-volumetric labware (applicable only when measuring initial sample vol. or final extract/digest ate volume | Gravimetric – 10 reps before use | By lot before first use or upon evidence of deterioration | Bias: Mean within ± 3%of nominal volume Precision RSD ≤ 3% of stated value (based on 10 replicate measures) | replace |
| Volumetric glassware | The laboratory uses only Class A volumetric glassware. Calibration not required | N/A | Check for deterioration | Replace |

| Instrument | Type of Calibration/ Number of Standards | Frequency | Acceptance Limits | Corrective Action |
|---|---|---|---|---|
| Glass Microliter Syringes | None | Accuracy must be initially demonstrated if syringe was not received with a certificate attesting to established accuracy. | ± 1% | Not applicable. |
| Conductivity Meter | Cell impedance calibrated with three KCl standards. | Each use. | $r \geq 0.99$ | Recalibrate. |
| Deionized Water | Check in-line conductivity meter on system with conductivity meter in Inorganic Department. | Daily | <10 µmhos/$cm^2$ | Record on log. Report discrepancies to QA Department |

**Table 20-4 Radiochemistry Calibration, Verification & Background Criteria**

| Instrument | Calibration Procedure | Frequency of Calibration | Acceptance Criteria |
|---|---|---|---|
| Gamma Spectroscopy | Initial Calibration | Energy, FWHM and energy calibrations shall be established for the germanium spectroscopy systems **annually**, or when the calibration quality control check indicates an unacceptable change in the energy calibration parameters. | The curve should have eight calibration points used to determine the energy relationship of the calibration.<br>The calibration source must have radionuclides that "blanket" the intended range of calibration.<br>The energy difference should be less than 0.05% for all points or with 2 keV for calibration points.<br>Computed efficiency test for all points should have a percent difference less than 8%.<br>The FWHM must be less than 3.0 keV at 1332 keV. FWHM difference should be less than 8% for all points. |
| Gamma Spectroscopy | Initial Background | Background subtraction spectrum shall be established for the germanium spectroscopy systems **monthly**, or when the background quality control check indicates an unacceptable change in the daily background parameters, or as needed per client requirements. | Background count time is 12 hours. |
| Gamma Spectroscopy | Continuing | Daily Checks<br>The energy, resolution and efficiency calibrations for a detector shall be checked with its respective source each day that the germanium spectroscopy system is used.<br>The detector background shall be checked each day that the germanium spectroscopy system is used. | Calibration (efficiency, resolution, energy alignment, and background) quality control parameters will be found **not acceptable** if the result is outside the established limits (2σ to 3σ range) and marked as "action".<br>The Daily QC check may only be recounted once without corrective action. |
| Alpha Spectroscopy | Initial Calibration | Energy calibrations shall be established for the alpha spectroscopy systems **yearly**, or when the calibration quality control check indicates an unacceptable change in the energy calibration parameters.<br><br>Efficiency calibrations shall be established for the alpha spectroscopy systems **yearly**, or when the calibration quality control check indicates an unacceptable change in the efficiency calibration parameters. | Energy Calibrations shall be performed using at least three isotopes within the energy range of 3-6 meV. Final peak energy positions of all observed isotopes shall be within ± 40 keV of expected energy.<br>Efficiency should fall between 20 and 32%. |

| Instrument | Calibration Procedure | Frequency of Calibration | Acceptance Criteria |
|---|---|---|---|
| Alpha Spectroscopy | Initial Background | Background subtraction spectrum shall be established for the alpha spectroscopy systems **monthly**, or when the background quality control check indicates an unacceptable change in the daily background parameters. | Background count time is 960 minutes. |
| Alpha Spectroscopy | Continuing | Daily Checks<br>Routine pulser quality control verifications are to be performed each day of use.<br>The pulser energy, peak centroid, peak resolution, peak area quality control for a detector shall be checked each day that the alpha spectroscopy system is used. | Routine calibration, background and pulser quality control parameters using the "Boundary" out-of-range test will be found unacceptable if the value is outside reasonable parameter tolerance.<br>The routine quality control check should be rerun to determine the statistical significance of the errant parameter. |
| Gas Flow Proportional Counter | Initial Calibration | Mass attenuation alpha/beta curves should be performed on an **annual** basis, or when the calibration quality control check indicates an unacceptable change in the efficiency calibration parameters. | The efficiency calibration shall consist of at least seven single or dual sets of mass attenuated calibration standards. The standards shall have enough activity to generate at least 10,000 counts in 90 minutes of count time for the most highly attenuated source. The count rate shall not exceed 5,000 counts per second.<br><br>The coefficient of determination ($r^2$) shall be greater than or equal to 0.9. |
| Gas Flow Proportional Counter | Initial Background | Background established for the GFPC **monthly**, or when the background quality control check indicates an unacceptable change in the daily background parameters. | Backgrounds are counted for 1,000 minutes<br>Alpha < 0.2 counts per minute<br><br>Beta < 2.0 counts per minute |
| Gas Flow Proportional Counter | Continuing | Daily Checks<br>Efficiency check and background check | |

## 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 that are used to deliver volume critical measurements. 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) or another accreditation organization that is a signatory to a MRA (Mutual recognition Arrangement) of one or more of the following cooperation's – ILAC (International Laboratory Accreditation Cooperation) or APLAC (Asia-Pacific Laboratory Accreditation Cooperation).. A certificate and scope of accreditation is kept on file at the laboratory.

The calibration report or certificate submitted to TestAmerica St. Louis contains, in a well designed format, a traceability statement, the conditions under which the calibrations were made in the context of any potential influence, a compliance statement with an identified metrological specification and the pertinent clauses, a clearly identified record of the quantities and functional test results before and after re-calibration, and no recommendation on the calibration interval. All calibration reports are filed in the QA Office.

An external certified service engineer services laboratory balances on an annual basis. This service is documented on each balance with a signed and dated certification sticker. Balance calibrations are checked each day of use. All liquid thermometers are calibrated annually against a traceable reference thermometer. Temperature readings of ovens, refrigerators, and incubators are checked on each day of use.

## 21.3 Reference Standards / Materials

Reference standards/materials, where commercially available, are traceable to certified reference materials. Commercially prepared standard materials are purchased from vendors accredited by A2LA, NVLAP, and NIST 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 Reagents Log Identification Number generated by LIMS and an expiration date. All documentation received with the reference standard is retained as a QC record and references the Standards Log Standard Identification Number. Reference standards that are used in the radiochemical laboratory shall be obtained from NIST, or suppliers who participate in supplying NIST standards or NIST traceable radionuclides. When traceable standards are not available, written approval for use must be obtained from DoD/DOE clients.

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. Radiochemical standards must be verified prior to initial use. The accuracy of calibration standards is checked by comparison with a standard from a second source. In cases where a second standard manufacturer is not available, a vendor certified different lot is acceptable for use as a second source. For unique situations where no other source or lot is available, a standard made by a different analyst would be considered a second source. The appropriate Quality Control (QC) criteria for specific standards are defined in laboratory SOPs. In most cases, the analysis of an Initial Calibration Verification (ICV) or LCS (where there is no sample preparation) is used as the second source confirmation. These checks are generally performed as an integral part of the analysis method (e.g. calibration checks, laboratory control samples).

All standards and materials must be stored and handled according to method or manufacturer's requirements in order to prevent contamination or deterioration. Refer to the Corporate Environmental Health & Safety Manual and the analytical method SOPs "Standards and Reagents" section for additional details. Radiochemical standards and reference material are stored separately from samples and are protected in a controlled cabinet or refrigerator. For safety requirements, please refer to method SOPs and the laboratory Environmental Health and Safety Manual.

Standards and reference materials shall not be used after their expiration dates unless their reliability is verified by the laboratory. 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.] Purchased stock mixtures and reagents are labeled to indicate the date they are opened.

All manufacturer or vendor supplied Certificate of Analysis or Purity must be retained, stored appropriately, and readily available for use and inspection. These records are maintained in a

directory on the laboratory network drive. Records must be kept of the date of receipt and date of expiration of standards, reagents and reference materials. In addition, records of preparation of laboratory standards, reagents, and reference materials must be retained, stored appropriately, and be readily available for use and inspection. For detailed information on documentation and labeling, please refer to method specific SOPs and ST-QA-0002, "Standard and Reagent Preparation".

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, and are assigned a unique identification number. The following information is typically recorded in the electronic database:

- 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

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 methods of preparation, date of preparation, expiration date and preparer's name or initials. Preparation procedures are provided in the Method SOPs.

**21.4.2** All standards, reagents, and reference materials must be clearly labeled with a minimum of the following information:

- Expiration Date (include prep date for reagents)
- Standard ID (assigned by the 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 in the SDS documents available on the TestAmerica intranet site).

**21.4.3** In addition, the following information may be helpful:

- 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 does not provide sampling services. The laboratory's responsibility in the sample collection process lies in supplying the sampler with the necessary coolers, reagent water, sample containers, preservatives, sample labels, custody seals, COC forms, ice, and packing materials required to properly preserve, pack, and ship samples to the laboratory

### 22.2 Sampling Containers

The laboratory offers clean sampling containers for use by clients. These containers are obtained from reputable container manufacturers and meet EPA specifications as required. Any certificates of cleanliness that are provided by the supplier are maintained at the laboratory.

#### 22.2.1 Preservatives

Upon request, preservatives are provided to the client in pre-cleaned sampling containers. In some cases containers may be purchased pre-preserved from the container supplier. Whether prepared by the laboratory or bought pre-preserved, the grades of the preservatives are at a minimum:

- Hydrochloric Acid – Reagent ACS (Certified VOA Free) or equivalent
- Methanol – Purge and Trap grade
- Nitric Acid – Instra-Analyzed or equivalent
- Sodium Bisulfate – ACS Grade or equivalent
- Sodium Hydroxide – Instra-Analyzed or equivalent
- Sulfuric Acid – Instra-Analyzed or equivalent
- Sodium Thiosulfate – ACS Grade or equivalent

### 22.3 Definition of Holding Time

The date and time of sampling documented on the COC form establishes the day and time zero. As a general rule, when the maximum allowable holding time is expressed in "days" (e.g., 14 days, 28 days), the holding time is based on calendar day measured. Holding times expressed in "hours" (e.g., 6 hours, 24 hours, etc.) is measured from date and time zero. The first day of holding time ends twenty-four hours after sampling. Holding times for analysis include any necessary reanalysis. However, there are some programs that determine holding time compliance based on the date and specific time of analysis compared to the time of sampling regardless of how long the holding time is.

### 22.4 Sampling Containers, Preservation Requirements, Holding Times

The preservation and holding time criteria specified in the laboratory SOPs are derived from the source documents for the methods. 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. The laboratory SOP ST-PM-0002 contains a table listing preservation, container and holding time information.

### 22.5 Sample Aliquots / Subsampling

Taking a representative sub-sample from a container is necessary to ensure that the analytical results are representative of the sample collected in the field. The size of the sample container, the quantity of sample fitted within the container, and the homogeneity of the sample need consideration when sub-sampling for sample preparation. It is the laboratory's responsibility to take a representative subsample or aliquot of the sample provided for analysis.

Analysts should handle each sample as if it is potentially dangerous. At a minimum, safety glasses, gloves, and lab coats must be worn when preparing aliquots for analysis.

Guidelines on taking sample aliquots & sub-sampling are located in SOP ST-QA-0038, "Procedure for Compositing and Sub-sampling".

***NOTE:*** *Unless otherwise noted by individual preparation SOPs, the following statements apply to sample aliquots of volume (liquid) for testing analysis.*

- Density Requirement – If a sample is known or suspected (based upon client knowledge, project scope, or site history) to have a high density (>1.2 g/mL, e.g. a brine or waste) or a low density (<0.98 g/mL, e.g. mixed solvent), the sample density will be measured and the volume determined arithmetically (sample mass divided by the density equals the volume).

- Volume Determination – Aliquot volume is calculated by gravimetric determination assuming a sample density of 1. Samples that are not aqueous, or suspected of having a density greater than 1.2, will have aliquots taken for density analysis to correct volume for density

## 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.

### 23.1 Chain of Custody (COC)

The COC form is the written documented history of any sample and is initiated 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. 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 with the other login paperwork.

### 23.1.2 Legal / Evidentiary Chain-of-Custody

If samples are identified for legal/evidentiary purposes on the COC, login will complete the custody seal, retain the shipping record with the COC, and initiate an internal COC for laboratory use by analysts and a sample disposal record.

## 23.2 Sample Receipt

Samples are received at the laboratory by designated sample receiving personnel and a unique laboratory project identification number is assigned. Each sample container shall be assigned a unique sample identification number that is cross-referenced to the client identification number such that traceability of test samples is unambiguous and documented. Each sample container is affixed with a durable sample identification label. Sample acceptance, receipt, tracking and storage procedures are described in SOP ST-PM-0002, "Sample Receipt and Chain of Custody".

### 23.2.1 Laboratory Receipt

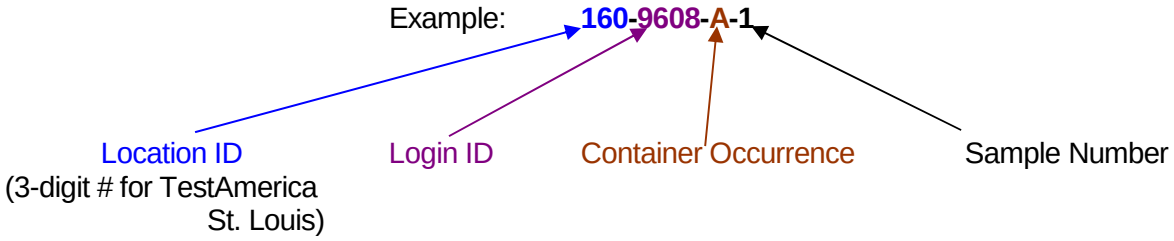
When samples arrive at the laboratory, sample receiving personnel inspect the coolers and samples. Coolers received from a known or potential radiologically contaminated site are frisked prior to opening. The integrity of each sample must be determined by comparing sample labels or tags with the COC and by visual checks of the container for possible damage. Any non-conformance, irregularity, or compromised sample receipt must be documented on a "Condition Upon Receipt" form (CUR, Figure 23-3) 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 four pieces of information:




The above example indicates TestAmerica St. Louis (location 160), Login ID 9608 (unique to a particular job/client), container "A" of sample number 1.

If the primary container goes through a prep step that creates a "new" container, then the new container is considered secondary and gets another ID. For example, when a 1-liter amber bottle is sent through a Liquid/Liquid Extraction and extraction vial is created from the prep step. The vial would be a secondary container and would be labeled as follows:

160-9608-A-1-**A**
Secondary Container Occurrence - the Secondary ID has five components

The IDs are 'bar-coded' on the LIMS generated laboratory sample label attached to each container.

These steps allow the samples to be tracked through the laboratory in every step from receipt to disposal.

### 23.3 Sample Acceptance Policy

The laboratory has a written sample acceptance policy (Figure 23-2) that clearly outlines the circumstances under which samples shall be accepted or rejected. These include:

- a COC filled out completely;
- samples must be properly labeled;
- proper sample containers with adequate volume for the analysis (Sampling Guide) and necessary QC;

- samples must be preserved according to the requirements of the requested analytical method (Sampling Guide);
- sample holding times must be adhered to (Sampling Guide);
- the Project Manager will be notified if any sample is received in damaged condition.

Data from samples which do not meet these criteria are flagged and the nature of the variation from policy is defined and noted in the Case Narrative.

**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** For samples received from a potentially radioactive site, an aliquot is removed from the container to perform a “rad screen.”

**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 ST-PM-0002.

### 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. In addition, samples to be analyzed for volatile organic parameters are stored in separate refrigerators designated for volatile organic parameters only. Samples having high levels of radiochemical contamination are labeled as such. 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 are analyzed every two weeks.

Analysts and technicians retrieve the sample container allocated to their analysis from the designated refrigerator and place them on carts, analyze the sample, and return the remaining sample or empty container to the refrigerator from which it originally came. All unused portions of samples, including empty sample containers, are returned to the secure sample control area. 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 a dry

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 analyses to be performed on the archived sample and assists clients in dealing with legal matters or regulatory issues**.**

Access to the laboratory is controlled such that sample storage need not be locked at all times unless a project specifically demands it. Samples are accessible to laboratory personnel only. Visitors to the laboratory are prohibited from entering the refrigerator and laboratory areas unless accompanied by an employee of TestAmerica.

### 23.5Hazardous Samples and Foreign Soils

To minimize exposure to personnel and to avoid potential accidents, hazardous and foreign soil samples are stored in an isolated area designated for hazardous waste only. The sample itself is clearly "HAZARDOUS" or "FOREIGN SOIL". 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, the sample is labeled as such. Potentially radioactive samples are "screened" prior to release to the laboratory. The RAD category is entered into the LIMS and alerts the analyst to the radiation level associated with the sample. All hazardous samples are either returned to the client or disposed of appropriately through a hazardous waste disposal firm that lab-packs all hazardous samples and removes them from the laboratory. Foreign soil samples are sent out for incineration by a USDA-approved waste disposal facility (*see SOPs ST-HS-0006,"Quarantine Soils Procedure", and the Radiation Protection SOPs for more details*).

### 23.6 Sample Shipping

In the event that the laboratory needs to ship samples, the samples are placed in a cooler with enough ice to ensure the samples remain just above freezing and at or below 6.0°C during transit. The samples are carefully surrounded by packing material to avoid breakage (yet maintain appropriate temperature). 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.

**Note:** If a client does not request trip blank analysis on the COC or other paperwork, the laboratory will not analyze the trip blanks that were supplied. However, in the interest of good client service, the laboratory will advise the client at the time of sample receipt that it was noted that they did not request analysis of the trip blank; and that the laboratory is providing the notification to verify that they are not inadvertently omitting a key part of regulatory compliance testing.

### 23.7Sample 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: ST-HS-0004, "Hazardous Waste Management Plan"). 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. Pertinent information includes the date of disposal, nature of disposal (such as sample depletion, hazardous waste facility disposal, and return to client), names of individuals who conducted the arrangements and physically completed the task. The laboratory will remove or deface sample labels prior to disposal unless this is accomplished through the disposal method (e.g., samples are incinerated). A Waste Disposal Record should be completed.

**Figure 23-1. Example: Chain of Custody (COC)**

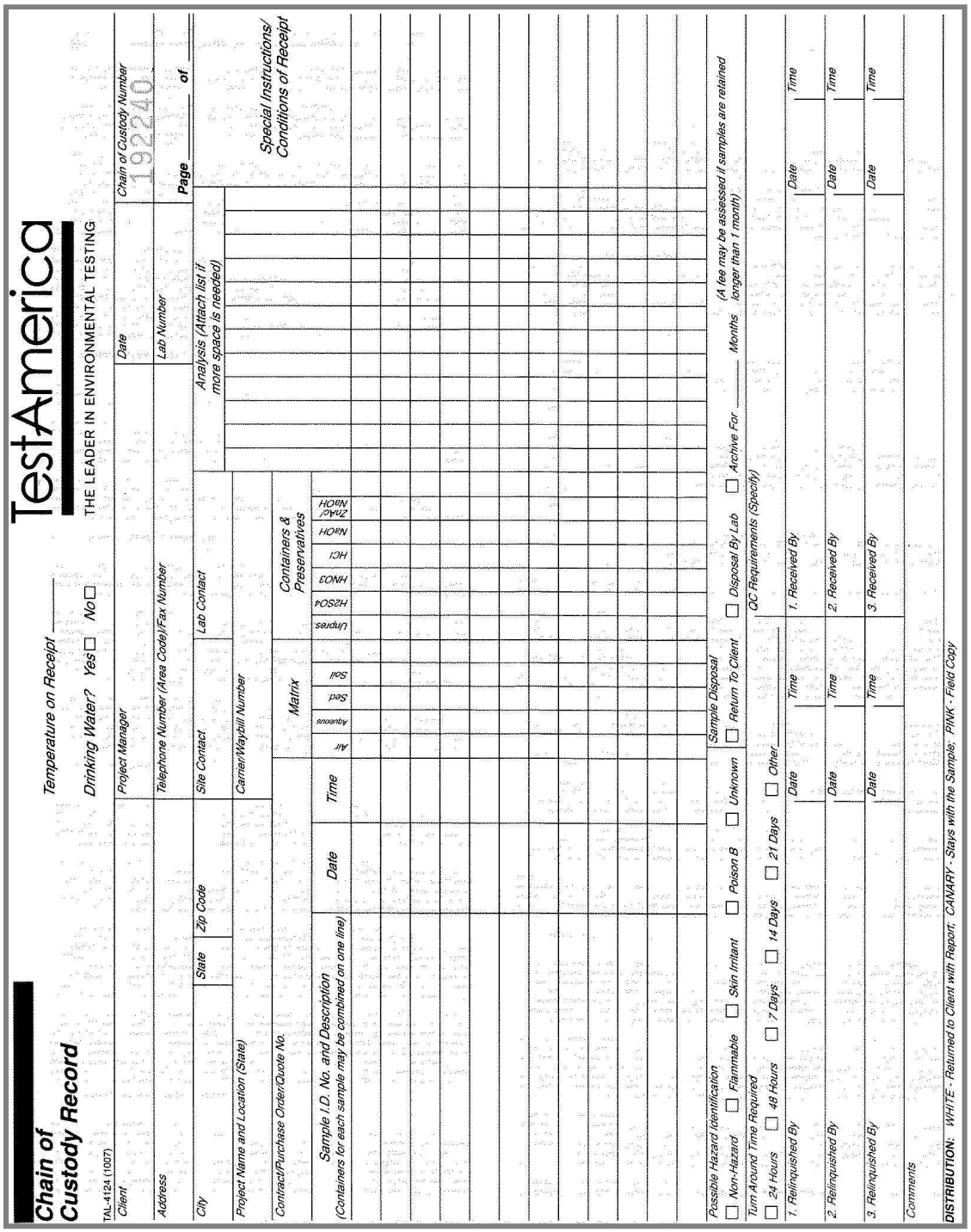


**Figure 23-2. Example: Sample Acceptance Policy**

## TestAmerica St. Louis
## Sample Acceptance Policy

NELAC specifies requirements under which any NELAC accredited laboratory will accept samples. STL St. Louis will review your sample shipment against those requirements listed below, and will communicate any discrepancies to you. Your project manager will assist you in the appropriate resolution of any issues related to sample receipt. Please contact your project manager with any questions.

When completing the chain of custody form, sign your name in the "relinquished by" box.

NELAC requirements are as follows:

- Proper, full and complete documentation, which includes sample identification, the location, date and time of collection, the collector's name, the preservation type, the sample matrix type, the requested testing method, and any special remarks concerning the samples shall be provided.
- Each sample shall be labeled with unique, durable and indelible identification.
- The samples shall be collected in the appropriate sample containers.
- The samples shall arrive at the laboratory within the specified holding time for the analyses requested.
- Sufficient sample volume must be available to perform the requested analyses.
- The laboratory will notify the client upon sample receipt if the samples exhibit obvious signs of damage, contamination or inadequate preservation.

## DoD QSM SAMPLE ACCETANCE POLICY:

NELAC specifies requirements under which any NELAC accredited laboratory will accept samples. TestAmerica St. Louis will review your sample shipment against those requirements listed below, and will communicate any discrepancies to you. Your project manager will assist you in the appropriate resolution of any issues related to sample receipt. Please contact your project manager with any questions.

When completing the chain of custody form, sign your name in the "relinquished by" box.

NELAC requirements are as follows:

-Proper, full and complete documentation, which includes sample identification, the location, date and time of collection, the collector's name, the preservation type, the sample matrix type, the requested testing method, and any special remarks concerning the samples shall be provided.
-Each sample shall be labeled with unique, durable and indelible identification.
-The samples shall be collected in the appropriate sample containers.
-The samples shall arrive at the laboratory within the specified holding time for the analyses requested.
-Sufficient sample volume must be available to perform the requested analyses.

The laboratory will notify the client upon sample receipt if the samples exhibit obvious signs of damage, contamination or inadequate preservation. Samples shall be considered “compromised” if the following conditions are observed upon sample receipt:

- Cooler and/or samples are received outside of temperature specification.
- Samples are received broken or leaking.
- Samples are received beyond holding time.
- Samples are received without appropriate preservative.
- Samples are received in inappropriate containers.
- COC does not match samples received.
- COC is not properly completed or not received.
- Breakage of any Custody Seal.
- Apparent tampering with cooler and/or samples.
- Headspace in volatiles samples.
- Seepage of extraneous water or materials into samples.
- Inadequate sample volume.
- Illegible, impermanent, or non-unique sample labelling.

When “compromised” samples are received, it must be documented on a Condition Upon Receipt Form (CUR) for the project records and the client must be contacted for instructions. If the client decides to proceed with analysis, the project report shall clearly indicate any of the above conditions and the resolution.

If the conditions listed on the Acceptance Policy are not satisfactory and when lacking direction from the client to the contrary, the sample will be rejected.

For DoD/DOE QSM project work, sample containers must be certified to meet the “less than” ½ the RL criteria for the analytes of concern. Analytes for which this certification can not be obtained will be noted in the Case Narrative. Upon DoD/DOE project approval, the laboratory will analyze method blanks prepared in the containers of concern, qualify and narrate the sample analytes which do not meet the criteria, or take other appropriate action as determined by the DoD/DOE project site.

**Figure 23-3. Example: Condition Upon Receipt (CUR) Form**

TestAmerica
THE LEADER IN ENVIRONMENTAL TESTING

CUR Form #: _____ Lot #(s): _____ _____

**CONDITION UPON RECEIPT FORM**

Client: _____

Quote No: _____

COC/RFA No: _____

Initiated By: _____ Date: _____ Time: _____

**Shipping Information**

Shipper: FedEx UPS DHL Courier Client Other: _____ Multiple Packages: Y N

| Shipping # (s):* | | | | Sample Temperature (s):** | | | |
|---|---|---|---|---|---|---|---|
| 1. | | 6. | | 1. | | 6. | |
| 2. | | 7. | | 2. | | 7. | |
| 3. | | 8. | | 3. | | 8. | |
| 4. | | 9. | | 4. | | 9. | |
| 5. | | 10. | | 5. | | 10. | |

*Numbered shipping lines correspond to Numbered Sample Temp lines

**Sample must be received at 4°C ± 2°C- If not, note contents below. Temperature variance does NOT affect the following: Metals-Liquid; Rad tests- Liquid or Solids; Perchlorate

**Condition** (Circle "Y" for yes, "N" for no and "N/A" for not applicable):

| | | | | | |
|---|---|---|---|---|---|
| 1. | Y N | Are there custody seals present on the cooler? | 8. | Y N | Are there custody seals present on bottles? |
| 2. | Y N N/A | Do custody seals on cooler appear to be tampered with? | 9. | Y N N/A | Do custody seals on bottles appear to be tampered with? |
| 3. | Y N | Were contents of cooler frisked after opening, but before unpacking? | 10. | Y N N/A | Was sample received with proper pH[1]? (If not, make note below) |
| 4. | Y N | Sample received with Chain of Custody? | 11. | Y N N/A | Containers for C-14, H-3 & I-129/131 marked with "Do Not Preserve" label? |
| 5. | Y N N/A | Does the Chain of Custody match sample ID's on the container(s)? | 12. | Y N | Sample received in proper containers? |
| 6. | Y N | Was sample received broken? | 13. | Y N N/A | Headspace in VOA or TOX liquid samples? (If Yes, note sample ID's below) |
| 7. | Y N | Is sample volume sufficient for analysis? | 14. | Y N N/A | Was Internal COC/Workshare received? |

[1] For DOE-AL (Pantex, LANL, Sandia) sites, pH of ALL containers received must be verified, EXCEPT VOA, TOX, Oil & Grease and soils

Notes: _____

Corrective Action:

☐ Client Contact Name: _____ Informed by: _____

☐ Sample(s) processed "as is"

☐ Sample(s) on hold until: _____ If released, notify: _____

Project Management Review: _____ Date: _____

THIS FORM MUST BE COMPLETED AT THE TIME THE ITEMS ARE BEING CHECKED IN. IF ANY ITEM IS COMPLETED BY SOMEONE OTHER THAN THE INITIATOR, THEN THAT PERSON IS REQUIRED TO APPLY THEIR INITIAL AND THE DATE NEXT TO THAT ITEM.

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# 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 measurements (e.g. Blanks, Laboratory Control Samples (LCS), Matrix Spikes (MS), duplicates (DUP), surrogates, Internal Standards (IS), tracers and carriers). These quality control checks are performed as required by the method or regulations to assess precision and accuracy. Quality control samples are to be treated in the ***exact*** same manner as the associated field samples being tested. In addition to the routine process quality control samples, Proficiency Testing (PT) Samples (concentrations unknown to laboratory) are analyzed to help ensure laboratory performance. PT samples must be evaluated the same as regular environmental samples. The laboratory shall employ the same quality control, sequence of analytical steps, and replicates as used when analyzing routine samples.

## 24.2 Controls

Sample preparation or pre-treatment is commonly required before analysis. Typical preparation steps include homogenization, grinding, solvent extraction, sonication, acid digestion, distillation, reflux, evaporation, drying and ashing. During these pre-treatment steps, samples are arranged into discreet manageable groups referred to as preparation (prep) batches. Prep batches provide a means to control variability in sample treatment. Control samples are added to each prep batch to monitor method performance and are processed through the entire analytical procedure with investigative/field samples.

## 24.3 Negative Controls

**Table 24-1. Example – Negative Controls**

| Control Type | Details |
|---|---|
| Method Blank (MB) | are used to assess preparation and analysis for possible contamination during the preparation and processing steps. |
| | The specific frequency of use for method blanks during the analytical sequence is defined in the specific standard operating procedure for each analysis. Generally it is 1 for each batch of samples; not to exceed 20 environmental samples. |
| | The method blank is prepared from a clean matrix similar to that of the associated samples that is free from target analytes (e.g., Reagent water, Ottawa sand, glass beads, etc.) and is processed along with and under the same conditions as the associated samples.<br><br>The method blank goes through all of the steps of the process (including as necessary: filtration, clean-ups, etc.). |
| | 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. |

**Table 24-1. Example – Negative Controls**

| Control Type | Details |
|---|---|
| Trip Blank [1] | 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. |
| Field Blanks [1] | are sometimes used for specific projects by the field samplers. A field blank prepared in the field by filling a clean container with pure reagent water and appropriate preservative, if any, for the specific sampling activity being undertaken. (EPA OSWER) |
| Equipment Blanks [1] | are also sometimes created in the field for specific projects. An equipment blank is a sample of analyte-free media which has been used to rinse common sampling equipment to check effectiveness of decontamination procedures. (NELAC) |
| Holding Blanks | also referred to as refrigerator or freezer blanks, are used to monitor the sample storage units for volatile organic compounds during the storage of VOA samples in the laboratory |

[1] When known, these field QC samples should not be selected for matrix QC as it does not provide information on the behavior of the target compounds in the field samples. Usually, the client sample ID will provide information to identify the field blanks with labels such as "FB", "EB", or "TB."

Evaluation criteria and corrective action for these controls are defined in the specific standard operating procedure for each analysis.

## 24.4 Positive Controls

Control samples (e.g., QC indicators) are analyzed with each batch of samples to evaluate data based upon (1) Method Performance (Laboratory Control Sample (LCS) or Blank Spike (BS)), which entails both the preparation and measurement steps; and (2) Matrix Effects (Matrix Spike (MS) or Sample Duplicate (MD, DUP), which evaluates field sampling accuracy, precision, representativeness, interferences, and the effect of the matrix on the method performed. Each regulatory program and each method within those programs specify the control samples that are prepared and/or analyzed with a specific batch

Note that frequency of control samples vary with specific regulatory, methodology and project specific criteria. Complete details on method control samples are as listed in each analytical SOP.

### 24.4.1 Method Performance Control - Laboratory Control Sample (LCS)

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.

The LCS is prepared from a clean matrix similar to that of the associated samples that is free from target analytes (for example: Reagent water, Ottawa sand, glass beads, etc.) and is processed along with and under the same conditions as the associated samples. The LCS is spiked with verified known amounts of analytes or is made of a material containing known and verified amounts of analytes, taken through all preparation and analysis steps along with the field samples. Where there is no preparation taken for an analysis (such as in aqueous

volatiles), or when all samples and standards undergo the same preparation and analysis process (such as Phosphorus), a calibration verification standard is reported as the LCS. In some instances where there is no practical clean solid matrix available, aqueous LCS's may be processed for solid matrices; final results may be calculated as mg/kg or ug/kg, assuming 100% solids and a weight equivalent to the aliquot used for the corresponding field samples, to facilitate comparison with the field samples.

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.).

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.

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 as they cover the range of all of the aroclors. Specific Aroclors may be used by request on a project specific basis.

## 24.5 Sample Matrix Controls

**Table 24-2. Sample Matrix Control**

| Control Type | Details | |
|---|---|---|
| Matrix Spikes (MS) | Use | Used to assess the effect sample matrix of the spiked sample has on the precision and accuracy of the results generated by the method used; |
| | Typical Frequency [1] | 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 [1] | 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[2] | 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 [1] | 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 [1] | 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. |
| Tracers and Carriers | Use | Chemically mimic and do not interfere with the target analytes through radiochemical separations. Isotopic tracers are typically radioactive materials while carriers are typically non-radioactive |
| | Typical Frequency [1] | Added to each client sample, method blank, LCS and matrix QC sample, as required by the specific method. |
| | Description | Added to samples to determine the overall chemical yield of the analytical preparation steps. Each sample is spiked separately with the same material and individual sample yields are determined. The tracer/carrier is added to the sample at the very beginning of the preparation steps. For solid samples the tracer/carrier is added after grinding, but before muffling or dissolution. |

[1] See the specific analytical SOP for type and frequency of sample matrix control samples.

[2] LCSD's are normally not performed except when regulatory agencies or client specifications require them. The recoveries for the spiked duplicate samples must meet the same laboratory established recovery limits as the accuracy QC samples. If an LCSD is analyzed both the LCS and LCSD must meet the same recovery criteria and be included in the final report. The precision measurement is reported as "Relative Percent Difference" (RPD). Poor precision between duplicates (except LCS/LCSD) may indicate non-homogeneous matrix or sampling.

## 24.6 Acceptance Criteria (Control Limits)

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 a semi-annual basis unless the method requires more frequent updating. Control limits are established per method (as opposed to per instrument) regardless of the number of instruments utilized.

Laboratory generated % Recovery acceptance (control) limits are generally established by taking $\pm$ 3 Standard Deviations (99% confidence level) from the average recovery of a minimum of 20-30 data points (more points are preferred).

- 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%.

- The maximum acceptable RPD limit will be 35% for waters and 40% for soils. The minimum RPD limit is 10%.

- If either the high or low end of the control limit changes by $\leq$ 5% from previous, the control chart is visually inspected and, using professional judgment, they may be left unchanged if there is no affect on laboratory ability to meet the existing limits.

**24.6.1** 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. The QA department can generate a Quality Control Limit summary that contains tables that summarize the precision and accuracy acceptability limits for the analyses performed at TestAmerica St. Louis. The information is stored in the LIMS and includes an effective date and is updated each time new limits are generated. Unless otherwise noted, these limits are laboratory generated. The limits are approved in the LIMS system after review by the QA department. The LIMS maintains an archive of all limits used in the laboratory. Historical limits can be found in the LIMS program . See laboratory SOP ST-QA-0014, "Evaluation of Analytical Accuracy and Precision through the Use of Control Charts".

**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 NELAC and DoD/DOE work, there are an allowable number of Marginal Exceedances (ME):

| | |
|---|---|
| <11 analytes | 0 marginal exceedances are allowed. |
| 11 – 30 Analytes | 1 marginal exceedance is allowed |
| 31-50 Analytes | 2 marginal exceedances are allowed |
| 51-70 Analytes | 3 marginal exceedances are allowed |
| 71-90 Analytes | 4 marginal exceedances are allowed |
| > 90 Analytes | 5 marginal exceedances are allowed |

- Marginal exceedances are recovery exceedances between 3 SD and 4 SD from the mean recovery limit (NELAC).

- 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.

**24.6.5** If radiochemical tracer or carrier recovery is outside limits the sample is re-analyzed to confirm matrix interference. If recoveries confirm, or there was obvious interference, results are reported from the original run and a note is included with the case narrative. If the re-analysis meets the recovery criteria, the second run is reported (or both are reported if requested by the client). When samples are non-detect for the target analytes and the carrier/tracer recovery indicates a high bias in the analysis, the samples are not re-run unless required by the client.

### 24.7 Additional Procedures to Assure Quality Control

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).

A discussion regarding MDLs, Limit of Detection (LOD) and Limit of Quantitation (LOQ) can be found in Section 19.

- Use of formulae to reduce data is discussed in the method SOPs and in Section 20.
- Selection of appropriate reagents and standards is included in Section 9 and 21.
- A discussion on selectivity of the test is included in Section 5.
- Constant and consistent test conditions are discussed in Section 18.
- 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, reviewed, and signed by the appropriate project manager. At a minimum, the standard laboratory report shall contain the following information:

**25.2.1** A report title (e.g. Analytical Report for Samples) with a "sample results" column header.

**25.2.2** Each report cover page printed on company letterhead, which includes the laboratory name, address and telephone number.

**25.2.3** A unique identification of the report (e.g. job number or SDG number) and on each page an identification in order to ensure the page is recognized as part of the report and a clear identification of the end.

**Note:** Page numbers of report are represented as page # of ##. Where the first number is the page number and the second is the total number of pages.

**25.2.4** A copy of the chain of custody (COC)

- Any COCs involved with Subcontracting are included.
- Any additional addenda to the report must be treated in a similar fashion so it is a recognizable part of the report and cannot accidentally get separated from the report (e.g., Sampling information).

**25.2.5** The name and address of client and a project name/number, if applicable.

**25.2.6** Client project manager or other contact

**25.2.7** Description and unambiguous identification of the tested sample(s) including the client identification code.

**25.2.8** Date of receipt of sample, date and time of collection, and date(s) of test preparation and performance, and time of preparation or analysis if the required holding time for either activity is less than or equal to 72 hours.

**25.2.9** Date reported or date of revision, if applicable.

**25.2.10** Method of analysis including method code (EPA, Standard Methods, etc).

**25.2.11** Practical quantitation limits or reporting limit.

**25.2.12** Method detection limits (if requested)

**25.2.13** Definition of Data qualifiers and reporting acronyms (e.g. ND).

**25.2.14** Sample results.

**25.2.15** QC data consisting of method blank, surrogate, LCS, and MS/MSD recoveries and control limits.

**25.2.16** Condition of samples at receipt including temperature. This may be accomplished in a narrative or by attaching sample login sheets (Refer to Sec. 25.2.4 regarding additional addenda).

**25.2.17** A statement to the effect that the results relate only to the items tested and the sample as received by the laboratory.

**25.2.18** A statement that the report shall not be reproduced except in full, without prior express written approval by the laboratory.

**25.2.19** A signature and title of the person(s) accepting responsibility for the content of the report and date of issue; authorized signatories are qualified Project Managers appointed by the Manager of Project Managers.

**25.2.20** When NELAC 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.

**25.2.21** 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.

**25.2.22** When soil samples are analyzed, a specific identification as to whether soils are reported on a “wet weight” or “dry weight” basis.

**25.2.23** Appropriate laboratory certification number for the state of origin of the sample, if applicable.

**25.2.24** If only part of the report is provided to the client (client requests some results before all of it is complete), it must be clearly indicated on the report (e.g., preliminary data). A complete report must be sent once all of the work has been completed.

**25.2.25** 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.

**25.2.26** A clear statement notifying the client that non-accredited tests were performed and directing the client to the laboratory’s accreditation certificates of approval shall be provided when non-accredited tests are included in the report.

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<u>Reporting Level or Report Type</u>**

The laboratory offers four levels of quality control reporting. Each level, in addition to its own specific requirements, contains all the information provided in the preceding level. The packages provide the following information in addition to the information described above:

- Level I is a report with all of the elements outlined in Section 25.2 above, excluding 25.2.15 (QC data).
- Level II is a Level I report plus summary information, including results for the method blank reported to the laboratory MDL, percent recovery for laboratory control samples and matrix spike samples, and the RPD values for all MSD and sample duplicate analyses.
- Level III contains all the information supplied in Level II, but presented on the CLP-like summary forms, and relevant calibration information. A Level II report is not included, unless specifically requested. No raw data is provided.
- Level IV is the same as Level III with the addition of all raw supporting data.

In addition to the various levels of QC packaging, the laboratory also provides reports in diskette deliverable form and as an electronic (pdf) file. Initial reports may be provided to clients by facsimile. 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 St. Louis offers a variety of EDD formats including Environmental Restoration Information Management System (ERPIMS), New Agency Standard (NAS), Format A, Excel, Dbase, GISKEY, and Text Files.

EDD specifications are submitted to the IT department by the PM for review and undergo the contract review process. 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 NELAC 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 No. 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 to meet all requirements of this document including a cover letter.

## 25.7 Format of Reports

The format of reports is designed to accommodate each type of environmental test carried out and to minimize the possibility of misunderstanding or misuse.

## 25.8 Amendments to Test Reports

Corrections, additions, or deletions to reports are only made when justification arises through supplemental documentation. Justification is documented using the laboratory's corrective action system (refer to Section 12).

The revised report is retained on the Archive data server, as is the original report. The revised report is stored in the Archive data server under the job number/SDG number followed by "rev".

When the report is re-issued, a notation of "Revised "is placed on the cover/signature page of the report *and at the top of the narrative page* with a brief explanation of reason for the re-issue.

## 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 work order 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.

## SECTION 26. REVISION HISTORY

### 26.1 CHANGES TO REVISION 0

**26.1.1** Updated to conform to new corporate Template. Information that was specific to the company at large and less specific to the individual laboratory was removed from the template and is now found in the Corporate Quality Management Plan (CQMP).

**26.1.2** The Quality Policy Statement was updated to include compliance with NELAC standards.

**26.1.3** Section 10 (Services to Client) was merged with Section 7 (renamed)

**26.1.4** Section 10 was left intentionally blank.

**26.1.5** Section 16 (Audits) was given new text.

**26.1.6** Section 17 (Management Reviews) revised QA report section, some tables were removed

**26.1.7** Section 21 (Calibrations) removed information that can be found in method SOPs

**26.1.8** Radiochemistry calculations in Appendix 6 were updated

**26.1.9** Tables, figures and appendices were updated and re-numbered

### 26.2 CHANGES TO REVISION 1(06/02/09)

**26.2.1** Added reference to ASME NQA-1-2000 to Section 3.1

**26.2.2** Updated Ethics Agreement in Appendix 1

**26.2.3** Updated radiochemistry calculations in Appendix 6.

### 26.3 CHANGES TO REVISION 2 (08/31/09)

**26.3.1** Added reference to DoD QSM 4.1 to Section 3.1

**26.3.2** Updated QA Manager job description in Section 4.2.3

**26.3.3** Updated laboratory organizational chart

**26.3.4** Added Quality Program objectives to Section 5.1; clarified staff responsibilities regarding QA documents

**26.3.5** Added QAM review cycle to Table 16-1

**26.3.6** Added freezer temperature criteria to Section 21.3.4

**26.3.7** Updated Calibration information in Table 21-3

**26.3.8** Added current Florida NELAC cert to Appendix 3

**26.3.9** Signatures moved from Title Page to Cover per DoD Requirements

### 26.4 CHANGES TO REVISION 3 (08/31/10)

**26.4.1** Section 2: list of Cross-walk references to the ISO 17025 requirements added

**26.4.2** Section 4.2: QA Manager responsibilities updated

**26.4.3** Section 4: Organizational Charts updated in figure 4-1

**26.4.4** Section 5.1: Addition to quality Policy Statement regarding continuous improvement

**26.4.5** Section 7: Figure 7-1 removed

**26.4.6** Section 13: Table 13-3 “General Corrective Actions” added

**26.4.7** Section 13.3.3: Root cause analysis added

**26.4.8** Sections 3.1 & 20.4: Source methods references updated

**26.4.9** Section 18.3: Evidence of successful training added

**26.4.10** Section 20.15.5: text on manual integrations and Mint Miner© expanded

**26.4.11** Section 21: Table 21-1 “instrument List”, updated

**26.4.12** Section 21.3.5: requirement for non-volumetric labware added
**26.4.13** Section 21.4: calibration standards section expanded
**26.4.14** Section 24.2.2: Unique sample ID section added
**26.4.15** Section 24.3: Sample Acceptance Policy moved to appear in Table of Contents
**26.4.16** Section 24.6: added note on Trip blanks
**26.4.17** Section 26.2.18: added narrative requirement reproduction of laboratory reports
**26.4.18** Information in Appendices 1,2,3,5 & 7 updated
**26.4.19** Added "End of Document" statement
**26.4.20** General grammatical edits and corrections

26.5 **CHANGES TO REVISION 4**

**26.5.1** 10/08/10: Added Section 20.4.2.4 to address DOCs for tests without analyte spikes
**26.5.2** 8/31/11: Removed the 'effective date' by section and applied it to the entire document. Continuous document pagination implemented.
**26.5.3** 2009 TNI Standard references added to the Table of Contents only – citations removed from the section titles within the document. Updated all references from the 2003 NELAC Standards to the 2009 TNI standard
**26.5.4** Use of the title 'Technical Manager' from the TNI Standard is defined and implemented.
**26.5.5** Section 10 (previously left empty) removed. Other section numbers adjusted accordingly.
**26.5.6** Section 4: Additional Quality Assurance and Technical Manager (a.k.a., Supervisors) responsibilities assigned based on the TNI Standard
**26.5.7** Section 8: Clarification of subcontracting procedures
**26.5.8** Table 12-1: Updated for additional corrective action procedures
**26.5.9** Section 15: Updates reflect current internal audit process as defined in CA-Q-S-004. Table 15-1 updated.
**26.5.10** Section 19: Verification of MDLs/RLs updated to TNI Standard
**26.5.11** Section 25: added statement regarding the listing of non-accredited methods in the lab report
**26.5.12** Appendix 2: updated laboratory floor plan
**26.5.13** Appendix 4: added/removed glossary terms/acronyms
**26.5.14** Appendix 5: Certification table updated
**26.5.15** Appendix 6: updated and clarified calculations
**26.5.16** Appendix 7: updated SOP list

**26.6 CHANGES TO REVISION 5**

**26.6.1** Grammatical and format corrections made throughout entire document
**26.6.2** Updated signature page
**26.6.3** REFERENCED CORPORATE SOPs AND POLICIES updated
**26.6.4** Section 4.3: Deputies updated
**26.6.5** Figure 4-1 Corporate and Laboratory Organization Charts updated
**26.6.6** Section 5.5: Criteria for Quality Indicators updated
**26.6.7** Changed TNI to NELAC where applicable
**26.6.8** Section 9.3.3: Specifications: updated compressed gasses paragraph
**26.6.9** Replaced Clouseau with LIMS where applicable

**26.6.10** Section 11.2: Responsibilities and Authorities removed COO
**26.6.11** Section 12: Removed Clouseau screen shots
**26.6.12** Section 14: Replaced reference to standards log program with LIMS
**26.6.13** Section 15: updated reference to Internal Auditing SOP to CA-Q-S-003
**26.6.14** Section 15: Added Audit Planning/Reporting section
**26.6.15** Sections 19.15.2 & 19.15.3: updated
**26.6.16** Section 20.2: Added "tagged-out" requirements
**26.6.17** Table 20-1, 20-2, 20-4 updated
**26.6.18** Section 22.5: Addition of aqueous sample aliquot density requirement and volume determination
**26.6.19** Section 23.2.1.1: Replaced QuantIMS with TALS unique sample identification.
**26.6.20** Section 23.3: Updated to indicate that variation from policy to be noted in case narrative
**26.6.21** Section 24.6.1: updated to reference LIMS instead of QC Browser
**26.6.22** Appendix 3: updated NELAC certification
**26.6.23** Appendix 4: added new glossary terms and acronyms
**26.6.24** Appendix 5: updated St. Louis certifications
**26.6.25** Appendix 6: added organic calculation “On column concentrations”
**26.6.26** Appendix 7: updated laboratory SOP listing

## 26.7 CHANGES TO REVISION 6

**26.7.1** Section 3.1, updated references
**26.7.2** Section 4.1, changed Chief Operating Officer to Chief Executive Officer
**26.7.3** Section 4.2, updated QA Manager, Technical Manager and Technical Director Responsibilities
**26.7.4** Section 4.3, updated responsibilities table of key personnel
**26.7.5** Figure 4-1, updated Corporate and Lab Org Chart
**26.7.6** Table 14-1, removed 7 year requirement and replaced it with reference to HR Manual
**26.7.7** Section 19.13.4, revised explanation of the meaning of the lab's uncertainty statement to more closely conform to A2LA and NIST language
**26.7.8** Table 20-4, updated to reflect practice
**26.7.9** Section 24.1, statement added to clarify and emphasize treatment of QC samples and PT samples
**26.7.10** Appendix 3: updated NELAC certification
**26.7.11** Appendix 5: updated St. Louis certifications
**26.7.12** Appendix 6: updated calculations
**26.7.13** Appendix 7: updated SOP listing

## 26.8 CHANGES TO REVISION 7 (02/02/2015)

**26.8.1** Section 4.3, updated Key Personnel Deputy table
**26.8.2** Figure 4-1, updated organizational charts
**26.8.3** Section 17.3, added reference to see SOP ST-QA-0044 Training
**26.8.4** Table 20-3, updated Example: Periodic Calibration
**26.8.5** Appendix 5, update lab certifications, accreditations, validations

## 26.9 CHANGES TO REVISION 8 (05/23/2016)

**26.9.1** Updated signatures page

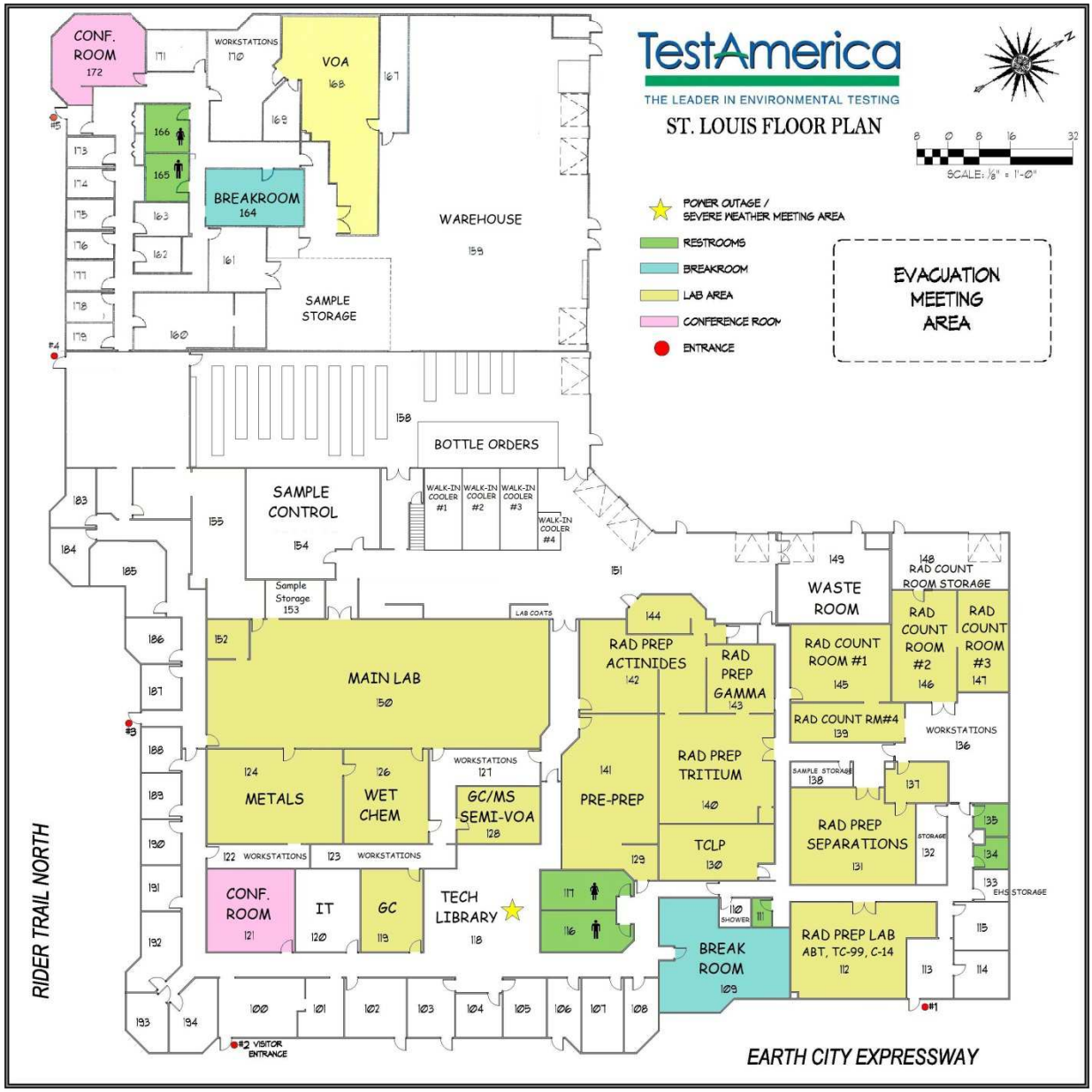
**26.9.2** Removed appendices: Ethics and confidentiality agreements; NELAC/TNI certified test
**26.9.3** Updated Corporate SOPs and Polices table as well as references throughout
**26.9.4** Added reference in section 3 to DOE Order 414.1D
**26.9.5** Updated corporate titles throughout
**26.9.6** Updated deputies, section 4.3
**26.9.7** Updated Org charts
**26.9.8** Updated section 5.5 with name of the app used
**26.9.9** Section 8.2.3, changed responsibilities from QAM to CSO
**26.9.10** Section 8.3 updated
**26.9.11** Section 9.3 & 9.3.2 updated
**26.9.12** Section 9.5 updated
**26.9.13** Section 11.2 & 11.3 updated
**26.9.14** Section 12.3.4 added information about iCAT
**26.9.15** Section 13, added list of opportunities for improvement
**26.9.16** Table 15-1, added SOP Method Compliance
**26.9.17** Section 15.1.3 updated
**26.9.18** Section 16.3 updated
**26.9.19** Section 20.3.3 added guidance of when a single point or range is required
**26.9.20** Section 25.2.19 changed from LD to PM as the one that appoints
**26.9.21** Section 25.3 added detail about Level I

## 26.10 CHANGES TO REVISION 9 (May 2017)

**26.10.1** Section 2 TOC Updated Corporate SOP Table, List of Documents
**26.10.2** Section 3 updated list of Compliance Regulations
**26.10.3** Section 4 Updated Table 4.3 – list of Deputies; Organization Chart
**26.10.4** Section 8 New test for Sections 8.2 & 8.3 (Sub-Contracting
**26.10.5** Section 9
26.10.5.1 removed outdated electronic order forms
26.10.5.2 Add that chemistry standards can also be re-verified (referenced SOP ST-QA-0002
26.10.5.3 Corrected DI check: resistivity daily, conductivity monthly
**26.10.6** Section 12 Added information on iCAT system
**26.10.7** Section 14 Updated Tables 14-1 & 14-2
**26.10.8** Section 15 Corrected SOP ID, from CA-Q-S-003 to CW-Q-S-003
**26.10.9** Section 16 Added items to Management Review
**26.10.10** Section 18 Corrected Appendix number for Floor Plan (changed from 2 to 1)
**26.10.11** Section 19
26.10.11.1 Updated MDL information (19.7)
26.10.11.2 Corrected Appendix number for SOPs (changed from 6 to 5)
**26.10.12** Section 20
26.10.12.1 Updated Instrument List and Maintenance Schedules
26.10.12.2 Removed turbidity meters
26.10.12.3 Changed “all standards are traceable to “when available,” standards are traceable
26.10.12.4 Clarified: *If an internal standard calibration is being used then bracketing calibration verification standards are not required <u>for some methods</u>, only daily verifications are needed except for DoD/DOE work..*
26.10.12.5 Added that bracketing QC may be analyzed out of 12 hour shift

**26.10.13** Section 21
26.10.13.1 Removed “comment box” from information “typically” recorded for standards. Comment box is not typically used.
26.10.13.2 Corrected MSDS to SDS
**26.10.14** Section 23 changed name for Figure 23-3, from “cooler receipt” to “condition upon receipt”
**26.10.15** Section 25 Updated 25.7 Format of Reports
**26.10.16** Appendix 1 Updated Floor Plan
**26.10.17** Appendix 2 Removed STU
**26.10.18** Appendix 3 Updated Certification information
**26.10.19** Appendix 5 Updated Laboratory SOP information
**26.10.20** Added DOE to all references of the QAM throughout the document
**26.10.21** Added hyperlinks though out document

**Appendix 1. Laboratory Floor Plan**



## Appendix 2. Glossary/Acronyms

**Glossary:**

**Acceptance Criteria:** Specified limits placed on characteristics of an item, process, or service defined in requirement documents. (ASQC)

**Accreditation:** The process by which an agency or organization evaluates and recognizes a laboratory as meeting certain predetermined qualifications or standards, thereby accrediting the laboratory.

**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)

**Activity, of radionuclides:** The expected number of spontaneous nuclear decays (transformations) in unit time from a specified energy state (excluding prompt decays from a lower nuclear level) for a given amount of a radionuclide. It's standard unit (SI) is the Becquerel (Bq), where one Bq equals one decay per second. Activity has often been expressed in curies (Ci), where 3.7 X 1010 Bq equals 1 Ci, exactly. (ANSI)

**Aliquot:** A discrete, measured, representative portion of a sample taken for analysis. (QSM)

**Analysis:** A combination of sample preparation and instrument determination. (QSM)

**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.

**Analyte:** The specific chemicals or components for which a sample is analyzed; it may be a group of chemicals that belong to the same chemical family and are analyzed together. (QSM)

**Analytical Uncertainty:** A subset of Measurement Uncertainty that includes all laboratory activities performed as part of the analysis. (NELAC)

**Assessment:** The evaluation process used to measure or establish the performance, effectiveness, and conformance of an organization and/or its systems to defined criteria (to the standards and requirements of laboratory accreditation). (NELAC)

**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. (NELAC)

**Background:** Ambient signal response recorded by measurement instruments that are independent of radioactivity contributed by the radionuclides being measured in the sample. (ANSI

**Batch:** 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) and/or those samples not requiring preparation, 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. (NELAC)

**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). (NELAC)

**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. (NELAC)

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 Curve:** The mathematical relationship between the known values, such as concentrations, of a series of calibration standards and their instrument response. (NELAC)

**Calibration Standard (Source):** A substance or reference material used to calibrate an instrument (QAMS)

**Carrier**: Carriers are stable counterparts of the radioactive isotope(s) to be measured. When used, carriers are added to all samples in an analytical batch so that each sample has a specific measurable QC parameter (yield). The carrier yield is used in the data calculation to correct for all sources of analytical losses. The term carrier can also be used for a non-radioactive compound added to assist in the isolation of the target analyte(s).

**Certified Reference Material (CRM):** A reference material

**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. (NELAC)

**Check source:** a radioactive source, not necessarily traceable to a national standards body such as NIST in the USA that is used to confirm the continuing satisfactory operation of an instrument. (ASTM)

**Clouseau**: TestAmerica custom software developed to document, track and trend non-conformances throughout the laboratory. The software interfaces with the laboratory information management system, QuantIMS and the report narrative generating software, KATO, to provide the laboratory with a corrective action system.

**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. NELAC 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 Second Column Confirmation; Alternate wavelength; Derivatization; Mass spectral interpretation; Alternative detectors or Additional Cleanup procedures. (NELAC)

**Conformance:** An affirmative indication or judgment that a product or service has met the requirements of the relevant specifications, contract, or regulation; also the state of meeting the requirements. (ANSI/ASQC E4-1994)

**Control Chart:** A graphical representation of data taken from a repetitive measurement or process. Control charts may be developed for various characteristics, (e.g., mean, standard deviation, range, etc.) of the data.

"A control chart has two basic uses: (1) as a tool to judge if a process was in control, and (2) as an aid in achieving and maintaining statistical control. For applications related to radiation detection instrumentation or radiochemical processes, the mean (center line) value of a historical characteristic (e.g., mean detector response), subsequent data values and control limits placed symmetrically above and below the center line are displayed on a control chart." (MARLAP)

**Count rate:** The rate at which detector pulses are being registered in a selected voltage interval. The unit is reciprocal seconds (i.e., $s^{-1}$). Generally the count rate is uncorrected for detector efficiency. The count rate divided by the detector efficiency for a specific particle and energy will yield the source activity.

**Count time:** The time interval for the counting of a sample or source by a radiation detector. Depending upon the context used, this can be either the "clock" time (the entire period required to count the sample), or "live" time (the period during which the detector is actually counting). Live time is always less than or equal to clock time. (MARLAP)

**Continuing Calibration Verification:** The verification of the initial calibration. Required prior to sample analysis and at periodic intervals. Continuing calibration verification applies to both external standard and internal standard calibration techniques, as well as to linear and nonlinear calibration models. (QSM)

**Correction:** Actions necessary to correct or repair analysis specific non-conformances (e.g. 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) A root cause analysis may not be necessary in all cases. (QSM)

**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. (NELAC)

**Decision Level (DL):** In the context of analyte detection, the minimum measured value (e.g., of the instrument signal or the analyte concentration) required to give confidence that a positive (nonzero) amount of analyte is present in the material analyzed. The DL is sometimes called the critical level (Lc) or critical value (MARLAP). It is the quantity of analyte at or above which an *a posteriori* decision is made that a positive quantity of the analyte is present. Confidence levels may be dictated by the project. For this document, the probability of a Type I error (probability of erroneously reporting a detectable nuclide in an appropriate blank or sample) is assumed to be set at 0.05.

**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. (NELAC)

**Detection Limit (DL):** The smallest analyte concentration that can be demonstrated to be different from zero or a blank concentration with 99% confidence. At the DL, the false positive rate (Type I error) is 1%. A DL may be used as the lowest concentration for reliably reporting a detection of a specific analyte in a specific matrix with a specific method with 99% confidence. (QSM)

**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 sub-samples 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)

**Energy Calibration:** The correlation of the multi-channel analyzer (MCA) channel number to decay photon energy, obtained from the location of peaks from known radioactive standards.

**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.

**False Negative:** A result that fails to identify (detect) an analyte or reporting an analyte to be present at or below a level of interest when the analyte is actually above the level of interest. (QSM)

**False Positive:** A result that erroneously identifies (detects) an analyte or reporting an analyte to be present above a level of interest when the analyte is actually present at or below the level of interest. (QSM)

**Field Blank:** Blank prepared in the field by filing a clean container with pure de-ionized water and appropriate preservative, if any, for the specific sampling activity being undertaken (EPA OSWER)

**Field of Accreditation:** Those matrix, technology/method, and analyte combinations for which the accreditation body offers accreditation.

**Holding Times:** The maximum time that samples may be held prior to analyses and still be considered valid or not compromised. (40 CFR Part 136)

**Initial Calibration Verification (ICV):** Verifies the initial calibration with a standard obtained or prepared from a source independent of the source of the initial calibration standards to avoid potential bias of the initial calibration. (QSM)

**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. (NELAC)

**Internal Standard Calibration:** Calibrations for methods that utilize internal standards to compensate for changes in instrument conditions.

**Instrument Blank:** A clean sample (e.g., distilled water) processed through the instrumental steps of the measurement process; used to determine instrument contamination. (EPA-QAD)

**Instrument 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 ± 100%. The IDL represents a range where qualitative detection occurs on a specific instrument. Quantitative results are not produced in this range.

**Laboratory Control Sample (however named, such as laboratory fortified blank, spiked blank, or QC check sample):** A sample matrix, free from the analytes of interest, spiked with verified known amounts of analytes or a material containing known and verified amounts of analytes, taken through all preparation and analysis steps 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 Information Management Systems (LIMS):** The entirety of an electronic data system (including hardware and software) that collects, analyzes, stores, and archives electronic records and documents. (QSM)

**Least Squares Regression (1$^{st}$ 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. (NELAC)

*QSM Clarification: The smallest concentration of a substance that must be present in a sample in order to be detected at the DL with 99% confidence. At the LOD, the false negative rate (Type II*

*error) is 1%. A LOD may be used as the lowest concentration for reliably reporting a non-detect of a specific analyte in a specific matrix with a specific method at 99% confidence.*

**LOD Verification [a.k.a., MDL Verification]:** A processed QC sample in the matrix of interest, spiked with the analyte at no more than 3X the LOD for single analyte tests and 4X the LOD for multiple analyte tests and processed through the entire analytical procedure.

**Limit(s) of Quantitation (LOQ) [a.k.a., Reporting Limit]:** The minimum levels, concentrations, or quantities of a target variable (e.g., target analyte) that can be reported with a specified degree of confidence. (NELAC)

*QSM Clarification: The smallest concentration that produces a quantitative result with known and recorded precision and bias. For DoD/DOE projects, the LOQ shall be set at or above the concentration of the lowest initial calibration standard and within the calibration range.*

**(QS) 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 or Saline/Estuarine. 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 & Emissions:* Whole gas or vapor samples including those contained in flexible or rigid wall containers and the extracted concentrated analytes of interest from a gas or vapor that are collected with a 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 replicate matrix spike prepared and analyzed to obtain a measure of the precision of the recovery for each analyte.

**Measurement Uncertainty:** An estimate of the error in a measurement often stated as a range of values that contain the true value, within a certain confidence level. The uncertainty generally includes many components which may be evaluated from experimental standard deviations based on repeated observations or by standard deviations evaluated from assumed probability distributions based on

experience or other information. For DoD/DOE, a laboratory's Analytical Uncertainty (such as use of LCS control limits) can be reported as the minimum uncertainty. (QSM)

**Method Blank:** A sample of a matrix similar to the batch of associated samples (when available) that is free from the analytes of interest and is processed simultaneously with and under the same conditions as samples through all steps of the analytical procedures, and in which no target analytes or interferences are present at concentrations that impact the analytical results for sample analyses.

**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)

**Minimum Detectable Activity or Concentration (MDA/MDC)**: The MDA is the smallest amount of an analyte in a sample that will be detected with a probability β of non-detection (Type II error), while accepting a probability α of erroneously deciding that a positive (non-zero) quantity of analyte is present in an appropriate blank sample (Type I error). Confidence levels may be dictated by the project. For the purposes of this module and the equations below, the α and β probabilities are assumed to be 0.05. MARLAP utilizes the Minimum Detectable Concentration (MDC) term instead of MDA

**Negative Control:** Measures taken to ensure that a test, its components, or the environment do not cause undesired effects, or produce incorrect test results.

**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.

**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.

**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. (NELAC)

**Proficiency Testing:** A means of evaluating a laboratory's performance under controlled conditions relative to a given set of criteria through analysis of unknown samples provided by an external source. (NELAC)

**Proficiency Testing Program:** The aggregate of providing rigorously controlled and standardized environmental samples to a laboratory for analysis, reporting of results, statistical evaluation of the results and the collective demographics and results summary of all participating laboratories. (NELAC)

**Proficiency Test Sample (PT):** A sample, the composition of which is unknown to the laboratory and is provided to test whether the laboratory can produce analytical results within specified acceptance criteria. (NELAC)

**Operator Aid:** A technical posting, other than formal procedures, rules, instructions (such as poster, operating manual, or notepad) that assists workers in routine tasks and are not required to be posted or displayed by any organization or procedure. All operator aids must be controlled by the facility.

**Qualitative Analysis:** Analysis designed to identify the components of a substance or mixture. (QSM)

**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. (NELAC)

**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. (NELAC)

**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. (NELAC)

**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. (NELAC)

**Quantitative Analysis:** analysis designed to determine the amounts or proportions of the components of a substance. (QSM)

**RadCapture**: Software used to process and report radiochemical data.

**Radioactive:** exhibiting radioactivity or containing radionuclides. (MARLAP)

**Radioactive decay:** Process by which a spontaneous change in nuclear state takes place. This process is accompanied by the emission of energy and subatomic particles.

**Radioactivity:** spontaneous emission of radiation, either directly from unstable atomic nuclei or as a consequence of a nuclear reaction.

**Radionuclide:** a nuclide that is radioactive (capable of undergoing radioactive decay). (MARLAP)

**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. (NELAC)

**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 homogeneous and well established to be used for the calibration of an apparatus, the assessment of a measurement method, or for assigning values to materials. (NELAC)

**Reference Standard:** Standard used for the calibration of working measurement standards in a given organization or a given location. (NELAC)

**Reporting Limit:** A customer-specified lowest concentration value that meets project requirements for quantitative data with known precision and bias for a specific analyte in a specific matrix. (QSM)

**Requested Limit**: The target MDA/MDC or critical value desired by the client.

**Sampling:** Activity related to obtaining a representative sample of the object of conformity assessment, according to a procedure.

**Second Order Polynomial Curve (Quadratic):** The $2^{nd}$ order curves are a mathematical calculation of a slightly curved 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 $2^{nd}$ order regression will generate a coefficient of determination (COD or $r^2$) that is a measure of the "goodness of fit" of the quadratic curvature the data. A value of 1.00 indicates a perfect fit. In order to be used for quantitative purposes, $r^2$ must be greater than or equal to 0.99.

**Selectivity:** 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. (NELAC)

**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. (NELAC)

**Standard Deviation:** the square root of a variance of a random variable. The variance is a measure of the variation of the observations within a measurement set. The standard deviation is often estimated using a set of measurements of the random variable. The standard deviation has the same units as the measured quantity and therefore, is particularly convenient when describing the variability of the measured quantity. (ANSI)

**Standard Operating Procedure (SOP):** 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. (NELAC)

**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)

**Systematic error:** An error component that produces a fixed bias in the underlying expected value of a determination, from measurement to measurement. (ANSI)

**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

**Technology:** A specific arrangement of analytical instruments, detection systems, and/or preparation techniques.

**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. (NELAC)

**Tracer**: Tracers are radioactive and/or massless. Where used, they are added to all samples in an analytical batch so that each sample has a specific measurable QC parameter (yield). Tracers are counted and the yield is used in data calculations to correct for and all sources of analytical loss.

**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.

**Unethical actions:** Deliberate falsification of analytical or quality control results, where failed method or contractual requirements are made to appear acceptable. (QSM)

### ***Acronyms*:**

| | |
|---|---|
| %R | Percent Recovery |
| ANSI | American National Standards Institute |
| App | Application |
| ASTM | American Society for Testing and Materials |
| Bq | becquerel |
| CAR | Corrective Action Report |
| CCV | Continuing Calibration Verification |
| CF | Calibration Factor |
| CFR | Code of Federal Regulations |
| Ci | Curie |
| CLP | Contract Laboratory Program |
| CoA | Certificate of Analysis |
| COC | Chain of Custody |
| cpm | Counts per minute |

| | |
|---|---|
| cps | Counts per second |
| CRM | Certified reference material |
| CSU | Combined standard uncertainty |
| CWA | Clean Water Act |
| DEQ | Department of Environmental Quality |
| DER | Duplicate Error Ratio |
| DOC | Demonstration of Capability |
| DoD | Department of Defense |
| DOE | Department of Energy |
| DOECAP | DOE Consolidated Audit Program |
| DOT | Department of Transportation |
| dpm | Disintegrations per minute |
| DQO | Data Quality Objectives |
| DUP | Duplicate |
| EDD | Electronic data deliverable |
| EHS | Environment, Health and Safety |
| EPA | Environmental Protection Agency |
| FWHM | Full width half maximum |
| GC | Gas Chromatography |
| GC/MS | Gas Chromatography/Mass Spectrometry |
| GFPC | Gas-flow Proportional Counter |
| HPGe | High-purity germanium |
| 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 |
| iDOC | Initial Demonstration of Capability |
| IH | Industrial Hygiene |
| IS | Internal Standard |
| ISO | International Organization of Standardization |
| keV | Kilo electron volts |
| LAN | Local area network |
| LCL | Lower control limits |
| LCS | Laboratory Control Sample |
| LCSD | Laboratory Control Sample Duplicate |
| LIMS | Laboratory Information Management System |
| LLD | Lower Level of Detection |
| LOD | Limit of Detection |
| LLQ | Lower Level of Quantitation |
| LOQ | Limit of Quantitation (PQL) |
| LSC | Liquid scintillation counter |
| MAPEP | Mixed Analyte Performance Evaluation Program |
| MARLAP | Multi-Agency Radiological Laboratory Analytical Protocol |
| MCL | Maximum contaminant limit |
| MDA/MDC | Minimum Detectable Activity/Concentration |
| MDL | Method Detection Limit |
| MDLCK | MDL Check Standard |
| MDLV | MDL Verification Check Standard |
| ME | Marginal exceedance |
| MeV | Mega electron volts |
| MQC | Minimum quantifiable concentration |
| MQO | Measurement quality objective |
| MRL | Method Reporting Limit Check Standard |

| | |
|---|---|
| MS | Matrix Spike |
| MSD | Matrix Spike Duplicate |
| NCM | Non-conformance memo |
| NELAC | National Environmental Laboratory Accreditation Conference |
| NELAP | National Environmental Laboratory Accreditation Program |
| NIST | National Institute of Standards and Technology |
| NVLAP | National Voluntary Laboratory Accreditation Program |
| pCi | picocurie |
| PE | Performance Evaluation |
| PT | Performance Testing |
| TNI | The NELAC Institute |
| QAM | Quality Assurance Manual |
| QA/QC | Quality Assurance / Quality Control |
| QAMS | Quality Assurance Management Systems |
| QAPP | Quality Assurance Project Plan |
| RCRA | Resource Conservation and Recovery Act |
| RDL | Required detection limit |
| RF | Response Factor |
| ROI | Region of interest |
| RPD | Relative Percent Difference |
| RPP | Radiation Protection Plan |
| RSD | Relative Standard Deviation |
| RSO | Radiation Safety Officer |
| SAP | Sample and analysis plan |
| SD | Standard Deviation |
| SDS | Safety Data Sheets |
| SMO | Sample Management Office |
| SOP | Standard Operating Procedure |
| SOW | Statement of work |
| SQC | Statistical quality control |
| SRM | Standard reference material |
| TAT | Turn-Around-Time |
| TCLP | Toxicity characteristic leaching procedure |
| TLD | Thermoluminescent dosimeter |
| TPU | Total propagated uncertainty |
| TSS | Total suspended solids |
| µohms | Resistivity unit of measure |
| WET | Whole effluent toxicity |
| WMP | Waste Management Plan |
| WP | Water pollution |
| VOA | Volatiles |
| VOC | Volatile Organic Compound |

**Appendix 3: Laboratory Certifications, Accreditations, Validations**

TestAmerica
THE LEADER IN ENVIRONMENTAL TESTING

## TestAmerica Certifications

| Laboratory | Program | Authority | Identification | Expiration Date |
|---|---|---|---|---|
| TestAmerica St. Louis | DoD ELAP | L-A-B | L2305 | 04/06/2019 |
| TestAmerica St. Louis | Federal | US Fish & Wildlife | LE058448-0 | 10/31/2017 |
| TestAmerica St. Louis | Federal | USDA | P330-17-0028 | 02/02/2020 |
| TestAmerica St. Louis | NELAP | Florida | E87689 | 06/30/2017 |
| TestAmerica St. Louis | NELAP | Illinois | 200023 | 11/30/2017 |
| TestAmerica St. Louis | NELAP | Kansas | E-10236 | 10/31/2017 |
| TestAmerica St. Louis | NELAP | Louisiana | 04080 | 06/30/2017 |
| TestAmerica St. Louis | NELAP | Louisiana (DW) | LA170011 | 12/31/2017 |
| TestAmerica St. Louis | NELAP | New Jersey | MO002 | 06/30/2017 |
| TestAmerica St. Louis | NELAP | New York | 11616 | 03/31/2017 |
| TestAmerica St. Louis | NELAP | Pennsylvania | 68-00540 | 02/28/2018 |
| TestAmerica St. Louis | NELAP | Texas | T104704193-16-10 | 07/31/2017 |
| TestAmerica St. Louis | NELAP | Utah | MO000542016-8 | 07/31/2017 |
| TestAmerica St. Louis | NELAP | Virginia | 460230 | 06/14/2017 |
| TestAmerica St. Louis | NRC | NRC | 24-24817-01 | 12/31/2022 |
| TestAmerica St. Louis | State Program | Alaska | MO00054 | 06/30/2017 |
| TestAmerica St. Louis | State Program | California | 2886 | 03/31/2018 |
| TestAmerica St. Louis | State Program | Connecticut | PH-0241 | 03/31/2017 |
| TestAmerica St. Louis | State Program | Iowa | 373 | 02/01/2018 |
| TestAmerica St. Louis | State Program | Kentucky (DW) | 90125 | 12/31/2017 |
| TestAmerica St. Louis | State Program | Maryland | 310 | 09/30/2017 |
| TestAmerica St. Louis | State Program | Missouri | 780 | 06/30/2017 |
| TestAmerica St. Louis | State Program | Nevada | MO000542017-1 | 07/31/2017 |
| TestAmerica St. Louis | State Program | North Dakota | R207 | 06/30/2017 |
| TestAmerica St. Louis | State Program | Oklahoma | 9997 | 08/31/2017 |
| TestAmerica St. Louis | State Program | South Carolina | 85002001 | 06/30/2017 |
| TestAmerica St. Louis | State Program | Washington | C592 | 08/30/2017 |
| TestAmerica St. Louis | State Program | West Virginia DEP | 381 | 08/31/2017 |

TestAmerica **St. Louis** maintains accreditations, certifications, and approvals 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:

*The certificates and parameter lists (which may differ) are available, upon request, from a laboratory representative. For each organization or 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: Calculations**

Common Calculations

- Percent Recoveries *(ICV, CCV, LCS, Surrogates) are calculated according to the equation:*

$$\%R = 100\left(\frac{Found}{True}\right)$$

  - o Tracers and Carriers

$$\text{Recovery}(\%) = \frac{measured}{added - native}\ x\,100$$

Where:

Measured is the amount of tracer/carrier measured
Added is the amount of tracer/carrier added (spiked) into the sample
Native is the amount of tracer/carrier analyte native to the sample

- Matrix Spike Recoveries *are calculated according to the following equation:*

$$\%R = 100\left(\frac{SSR - SR}{SA}\right)$$

Where:

SSR = Spike Sample Result
SR = Sample Result
SA = Spike Added

- The relative percent difference (RPD*) of matrix spike/matrix spike duplicates is calculated according to the following equation:*

$$RPD = 100\left[\frac{|MSD - MS|}{\left(\frac{MSD + MS}{2}\right)}\right]$$

Where:

MS = determined spiked sample concentration
MSD = determined matrix spike duplicate concentration

- Due to the nature of radioactive decay (random process) and the fact that Radiochemistry results are reported down to (and below) the MDC, dual criterion are used for replicate precision. When significant activity (well above the MDC) is present for a nuclide in the sample, the best representation of replicate precision is the RPD (Relative Percent Difference), which is calculated as follows:

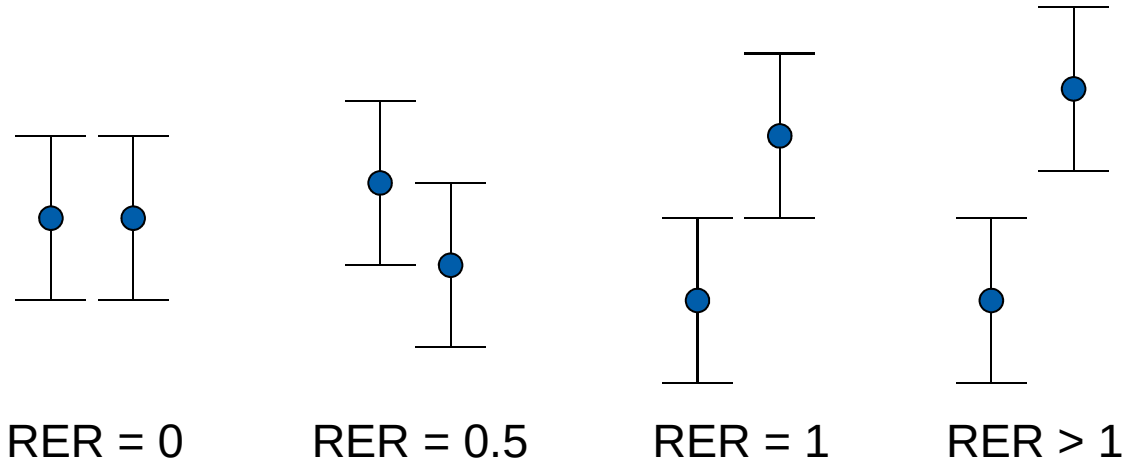
$$RPD = \frac{|Sample - Duplicate|}{\left(\frac{Sample + Duplicate}{2}\right)}$$

- Typically, the RPD is expected to be within a certain range (e.g. ±40%), dependent upon matrix and sample type. However, as the sample activity approaches the MDC, the RPD tends to "blow up" out of proportion due to the statistical error involved. Thus, we also look at the RER (Relative Error Ratio):

$$RER = \frac{|Sample - Duplicate|}{(Sample\ 2\sigma\ Unc. + Duplicate\ 2\sigma\ Unc.)}$$

The RER is most meaningful near or below the MDC, and is expected to be ≤ 1. As the sample activity increases, the RER tends to "blow up" out of proportion, and the RPD is more representative of replicate reproducibility.

Looking at the RER pictorially, when the 2σ error bars touch or overlap, the RER ≤ 1.




Thus, when evaluating replicate precision for Radiochemistry results, a dual criteria is applied. Either RPD ≤ control limit (e.g. ±40%) or RER ≤ 1.

- The percent difference (%D) is calculated as follows:

$$\%Difference = \frac{|R_1 - R_2|}{R_1} \times 100$$

Where:
$R_1$ = First result
$R_2$ = Second result

- Standard Deviation (SD) is calculated as follows:

$$SD = \sqrt{\sum_{i=1}^{N} \frac{(X_i - X)^2}{N-1}}$$

Where:

$X_i$ = Value of X as i through N
$N$ = Number of points
$\bar{X}$ = Average value of $X_i$

ADDITIONAL Calculations for Metals

- The final concentration for a digested aqueous sample is calculated as follows:

$$mg/L = \frac{C \times V1 \times D}{V2}$$

Where:
C = Concentration (mg/L) from instrument readout
D = Instrument dilution factor
V1 = Final volume in liters after sample preparation
V2 = Initial volume of sample digested in liters

- *The final concentration determined in digested solid samples when reported on a dry weight basis is calculated as follows:*

$$mg/Kg, dry\ weight = \frac{C \times V \times D}{W \times S}$$

Where:
C = Concentration (mg/L) from instrument readout
D = Instrument dilution factor
V = Final volume in liters after sample preparation
W = Weight in Kg of wet sample digested
S = Percent solids/100

Note: A Percent Solids determination must be performed on a separate aliquot when dry weight concentrations are to be reported. If the results are to be reported on wet weight basis the "S" factor should be omitted from the above equation.

Additional Calculations for Organics

- *The calibration factor for an external calibration standard is calculated as follows:*

$$Calibration\ Factor\ (CF) = \frac{Area\ or\ Height\ of\ Peak}{Mass\ Injected\ (ng)}$$

- *Relative Standard Deviation (%RSD), applicable to initial calibration, is calculated as follows:*

$$\%RSD = \frac{SD}{CF_{avg}} \times 100$$

Where:

*$CF_{avg}$ = The average of the initial CFs for a compound*

*SD = The standard deviation (using n-1) of the initial calibration CFs for a compound*

- *Aqueous sample concentration using external standard calibration is calculated as follows:*

$$Concentration\,(mg/L) = \frac{(A_x \times V_t \times D_f)}{(CF \times V_i \times V_s)}$$

Where:

$A_x$ = Response for the analyte in the sample
$V_i$ = Volume of extract injected, µL
$D_f$ = Dilution factor
$V_t$ = Volume of total extract, µL
$V_s$ = Volume of sample extracted or purged, mL
$CF$ = Calibration factor, area or height/ng

- *Non-aqueous sample concentration using external standard calibration is calculated as follows:*

$$Concentration\,(mg/kg) = \frac{(A_x \times V_t \times D_f)}{(CF \times V_i \times W \times D)}$$

Where:

$A_x$ = Response for the analyte in the sample
$V_i$ = Volume of extract injected, µL
$D_f$ = Dilution factor
$V_t$ = Volume of total extract, µL
$CF$ = Calibration factor, area or height/ng
$W$ = Weight of sample extracted or purged, g

$$D = \frac{100 - \%Moisture}{100}$$ (D = 1 if wet weight is required)

- *On column concentration*

*On Column Concentration (µg/mL):*

$$[OC] = \frac{A_x}{CF}$$

Where:

$[OC]$ = *On Column Concentration [typically expressed in µg/mL (ppm)]*

Then substitute/derive

$$[C] = [OC]\left(\frac{V_t * D}{V_i * V_s}\right)$$

When *on column concentration* $[OC]$ is equal to the *CAL-AMT (calibration amount)* of the low level standard needed to support the *reporting limit (µg/L)* and we solve the equation for *concentration (µg/L)*

Then

$$[C] \equiv RL \equiv [OC]\left(\frac{V_t * D}{V_i * V_s}\right)$$

Where:

*RL = Reporting Limit*

*Additional Calculations for GC/MS SVOA*

- Concentration calculation using average response factor:

$$C_{ex} = \frac{R_x C_{is}}{R_{is}\overline{RF}}$$

- Concentration calculation using linear fit:

$$C_{ex} = A + B\frac{(R_x C_{is})}{R_{is}}$$

Where:

$C_{ex}$ = Concentration in extract, µg/ml
$R_x$ = Response for analyte
$R_{is}$ = Response for internal standard
$C_{is}$ = Concentration of internal standard
$A$ = Intercept
$B$ = Slope

- Concentration calculation using quadratic fit:

$$C_{ex} = A + B\left(\frac{R_x C_{is}}{R_{is}}\right) + C\left(\frac{R_x C_{is}}{R_{is}}\right)$$

Where:

$C$ = Curvature

- Aqueous sample concentration is calculated as follows:

$$Concentration, ug/L = \frac{C_{ex} V_t}{V_o}$$

Where:

$V_t$ = Volume of total extract, µL, taking into account dilutions
$V_o$ = Volume of water extracted (ml)

- Sediment/soil, sludge and waste concentration is calculated as follows:

$$Concentration, ug/kg = \frac{C_{ex} V_t}{W_s D}$$

Where:

$W_s$ = Weight of sample extracted or diluted in grams
$D$ = (100 - % moisture in sample)/100, for a dry weight basis
or 1 for a wet weight basis

*Additional Calculations for GC/MS VOA*

- Calculation *(x)* for water and water-miscible waste:

$$x = \frac{(A_x)(I_s)(D_f)}{(A_{is})(V_o)}$$

Where:

$A_x$ = Area of characteristic ion for the compound being measured
$A_{is}$ = Area of the characteristic ion for the internal standard
$I_s$ = Amount of internal standard added in ng
$V_o$ = Volume of water purged, mL

$$D_f = Dilution\ Factor = \frac{Total\ volume\ purged\ (mL)}{Volume\ of\ original\ sample\ used\ (mL)}$$

- Calculation *(x)* for medium level soils:

$$x = \frac{(A_x)(I_s)(V_t)(1000)(D_f)}{(A_{is})(V_a)(W_s)(D)}$$

Where:

$A_x$, $I_s$, $D_f$, $A_{is}$ are the same as for water
$V_t$ = Volume of total extract, mL (typically 25 mL)

$V_a$ = Volume of extract added for purging, µL
$W_s$ = Weight of sample extracted, g

$$D = \frac{100 - \%moisture}{100}$$

- Calculation *(x)* for low level soils:

$$x = \frac{(A_x)(I_s)}{(A_{is})(W_s)(D)}$$

Where:

$A_x$, $I_s$, $A_{is}$ are the same as for water
$D$ is the same as for medium level soils
$W_s$ = Weight of sample added to the purge vessel, g

The Percent Difference is calculated as follows:

$$\% \text{ Difference} = \frac{\text{(CF(v) or RF(v)) - (Avg. CF or RF)}}{\text{(Avg. CF or RF)}} \times 100$$

Where:

CF(v) or RF(v) = CF or RF from verification standard
Avg. CF or RF = Average CF or RF from Initial Calibration.

The Percent Drift is calculated as follows:

$$\% \text{ Drift} = \frac{\text{Result - True Value}}{\text{True Value}} \times 100$$

The Percent Recovery is calculated as follows:

$$\% \text{ Recovery} = \frac{\text{Result}}{\text{True Value}} \times 100$$

**Gamma Activity Concentration**

The activity concentration of a sample will be calculated using the following equation.

$$ACT_S = \frac{Net\_Counts}{2.22 * E * t_S * Ab * V_A * D_C * D_S}$$

where:

| | | |
|---|---|---|
| $ACT_S$ | = | the activity in pCi/(units of the volume) |
| Net Counts=the net area of a peak | | |
| 2.22 | = | the correction factor to pCi |
| E | = | the efficiency – corrected for transmission |
| $t_S$ | = | the count time in minutes |

| | | |
|---|---|---|
| Ab | = | the gamma abundance factor |
| $V_A$ | = | the sample aliquot volume |
| $D_C$ | = | the decay correction during the analysis |
| $D_S$ | = | the decay correction from collection date to start of analysis |

Gamma Uncertainty of Concentration (at 2σ confidence level)

The Total Propagated Uncertainty (TPU) will be calculated using the following equation.

The software calculates the 2σ TPU term by incorporating the stochastic counting uncertainty and by examining the nuclide library for the error in the nuclide half-life and abundance for their respective contributions. The software routine also includes the standard certificate file and the calibration standard uncertainties. Finally, a 1% factor is added in quadrature due to the uncertainty in the preparation of the sample. This is attributed to the maximum allowable variability of the balances.

$$TPU_S = 1.96 * ACT_S * \sqrt{\left(\frac{\Delta P}{P}\right)^2 + \left(\frac{\Delta Ab}{Ab}\right)^2 + \left(\frac{\Delta \varepsilon}{\varepsilon}\right)^2 + \left(\frac{\Delta V}{V}\right)^2 + \left(\frac{sys}{100}\right)^2 + (\Delta Decay)^2}$$

Where:

$$\Delta\ Decay = \left[\frac{\Delta\ T_{1/2}}{T_{1/2}}\right] * \left[\frac{\lambda\ E_r}{1 - e^{-\lambda E_r}} - \lambda\ (T_S + E_r) - 1\right]$$

Where:

| | | |
|---|---|---|
| $TPU_S$ | = | the 2σ uncertainty of the activity of the sample |
| $ACT_S$ | = | the activity in pCi/(units of volume) |
| 1.96 | = | the statistical multiplication factor for 95% confidence level |
| ΔP | = | the uncertainty in the peak area |
| ΔAb | = | the uncertainty in gamma abundance |
| Δε | = | the uncertainty in the efficiency ε |
| ΔV | = | the uncertainty in the volume |
| sys | = | the systematic error estimate (in %)* |
| $\Delta T_{1/2}$ | = | the uncertainty in the half-life |
| $T_{1/2}$ | = | the half-life of the nuclide of interest |
| λ | = | the decay constant |
| $E_r$ | = | the elapsed real time during count |
| $T_s$ | = | the sample collection time |

Gamma MDC

The minimum detectable concentration will be calculated using the following equation.

$$MDC = \frac{4.65 * \sqrt{R_B * t_S} + 2.71}{2.22 * E * t_S * Ab * V_A * D_C * D_S}$$

Where:

| | | |
|---|---|---|
| MDC | = | Minimum Detectable Activity of the sample |
| $R_B$ | = | Count rate of detector background (in cpm) |
| $t_S$ | = | Count time for analysis |
| E | = | Detector efficiency |
| Ab | = | Abundance of the gamma emission |
| $V_A$ | = | sample aliquot volume |
| $D_C$ | = | Decay during sample analysis |
| $D_S$ | = | Decay from collection to start of analysis |

**Alpha Tracer Yield Recovery**

Tracer Yield Recovery

$$Y = \frac{(C_T - C_B)}{E * A_T * t_S}$$

Where:

| | | |
|---|---|---|
| Y | = | Chemical Yield |
| $C_T$ | = | Tracer Counts |
| $C_B$ | = | Tracer ROI background counts |
| $A_T$ | = | Tracer dpm |
| $t_s$ | = | Count time for analysis |
| E | = | Detector efficiency |

**Ra-226 Ingrowth factor:**

$$I = 1 + 3\left(1 - e^{-\lambda t}\right)$$

Where:

λ = ln(2)/Rn-222 Half-life (in days)
Rn-222 Half-life = 3.824 days
t = Time between BaPrecipitationTime and CountMidPoint (in days)

Note that for validation of data from TALS Level IV reports, BaPrecipitationTime = IngDecDate2 from the Ra-226 prep Batch Worksheet. CountMidPoint is the date Analyzed from the Analysis Detail Report plus one half of the count duration (Ts).

**Ra-228 Ingrowth/Decay factors:**

$$I = \left( \frac{1 - e^{-\lambda t_2}}{\lambda t_2} \right) \left(1 - e^{-\lambda t_3}\right) \left(e^{-\lambda t_1}\right)$$

Where:

$\lambda$ = ln(2)/Ac-228 Half-life (in days)
Ac-228 Half-life = 0.2563 days
$t_1$ = Time between YttriumPrecipitationTime and StartOfCount
$t_2$ = SampleCountDuration
$t_3$ = Time between YttriumIngrowthStartTime and YttriumPrecipitionTime

Note that for validation of data from TALS Level IV reports, YttriumPrecipitationTime = IngDecDate1 and YttriumPrecipitationTime = IngDecDate2 from the Ra-228 prep Batch Worksheet. StartofCount is the date Analyzed from the Analysis Detail Report.

**Total Strontium Ingrowth factor:**

$$I = 1 + \left(1 - e^{-\lambda t}\right)$$

Where:

$\lambda$ = ln(2)/Y-90 Half-life (in days)
Y-90 Half-life = 2.67 days
t = Time between StrontiumPrecipitationTime and CountMidPoint (in days)

Note that for validation of data from TALS Level IV reports, StrontiumPrecipitationTime = IngDecDate1 form the Total Strontium prep Batch Worksheet. CountMidPoint is the date Analyzed from the Analysis Detail Report plus one half of the count duration (Ts).

**Sr-90 Ingrowth/Decay factors:**

$$I = \left(1 - e^{-\lambda t_1}\right) \left(e^{-\lambda t_2}\right)$$

Where:

$\lambda$ = ln(2)/Y-90 Half-life (in days)
Y-90 Half-life = 2.67 days
$t_1$ = Time between StrontiumPrecipitationTime and YttriumPrecipitionTime
$t_2$ = Time between YttriumPrecipitationTime and CountMidPoint

Note that for validation of data from TALS Level IV reports, StrontiumPrecipitationTime = IngDecDate1 and YttriumPrecipitationTime = IngDecDate2 from the Sr-90 prep Batch Worksheet. StartofCount is the date Analyzed from the Analysis Detail Report.

**Additional Information for Radiochemistry Calculations:**

**Zero Count Uncertainty**

Certain analyses with intrinsic low background may lead to instances where both the background and the sample count results may be zero (e.g. alpha spec, Ni-59). In such circumstances, the counting uncertainty (CU) and total propagated uncertainty (TPU) will evaluate to zero. To provide a non-zero estimate of the counting uncertainty (and thus a non-zero TPU) in such an occasion, a value of one (1) will be substituted for the sample counts in the counting uncertainty and critical level equations.

**Crosstalk Calculation**

Alpha into Beta Crosstalk

$$\alpha >> \beta \ crosstalk = \frac{CPM_{XT}}{CPM_{\alpha} + CPM_{XT}} = y$$

$$yCPM_{\alpha} + yCPM_{XT} = CPM_{XT}$$

$$CPM_{XT} = \frac{y}{(1-y)} \ CPM_{\alpha} \quad where \ CPM_{\alpha} \ is \ net \ alpha \ CPM$$

Where:

| | | |
|---|---|---|
| CPM | = | counts per minute (S=Sample, B=Background, XT=crosstalk, α=alpha) |
| T | = | count duration in minutes (S=Sample, B=Background) |
| E | = | Efficiency |
| V | = | aliquot volume |
| UF | = | uncertainty factor (e.g. 0.05) |
| Act | = | activity |

**RadCapture Version 5.1.63**

Calculation equations for all methods were updated to create consistency. All methods now use the form:

$$Activity = \frac{\left(\frac{Cs}{Ts} - \frac{Cxt}{Ts} - \frac{Cb}{Tb}\right)}{D * E * I * V * R * A} * DF * UCF$$

$$\text{UncCnt}\ (1\sigma) = \frac{\sqrt{\frac{Cs}{Ts^2} + \frac{Cxt}{Ts^2} + \frac{Cb}{Tb^2} + Chi^2}}{D * E * I * V * R * A} * DF * UCF$$

$$\text{UncTot}\ (1\sigma) = \sqrt{\text{UncCnt}^{\,2} + (TPUFact * Activity)^2}$$

$$MDC = \left( \frac{3.29\sqrt{\frac{Cb}{Tb * Ts} + \frac{Cxt}{Ts^2} + \frac{Cb}{Tb^2} + Chi^2}}{D * E * I * V * R * A} + \frac{3}{D * E * I * V * Ts * R * A} \right) * DF * UCF$$

$$DLC = \left( \frac{1.645\sqrt{\frac{Cb}{Tb * Ts} + \frac{Cxt}{Ts^2} + \frac{Cb}{Tb^2} + Chi^2}}{D * E * I * V * R * A} \right) * DF * UCF$$

Where:

| | | |
|---|---|---|
| Cs | = | Sample Counts |
| Cb | = | Background Counts |
| Cxt | = | Crosstalk Counts (currently only gross beta) |
| Ts | = | Sample Count Duraton |
| Tb | = | Background Count Duration |
| D | = | Decay |
| E | = | Efficiency |
| I | = | Ingrowth |
| V | = | Aliquot Volume |
| R | = | Recovery |
| A | = | Abundance (Branching Ratio) |
| DF | = | Dilution Factor |
| UCF | = | Units Conversion Factor |
| Chi | = | non-Poisson variance |

For the count uncertainty, if both Cs and Cb = 0, then 1 is forced into Cs.
For the DLC, if Cb =0, then 1 is forced into Cb.

Gross Alpha/Beta is the only method which currently employs a crosstalk factor (and only for alpha into beta crosstalk). However, a crosstalk factor is included for all methods to create consistency. For all methods except Gross Alpha/Beta, Cxt is set to zero in the code.

Similarly, the non-Poisson variance (Chi) has only been employed for a specific client, and only for LSC methods. It is included for all methods to create consistency in the calculation equations. A table is set up in the database to list the Chi factor for each analyte. This factor may be updated on a periodic basis to reflect current operating conditions. This is controlled by an “active” date assigned in the table. The Chi factor is currently set to only be applied for

specific projects (client-based). When not directed to the Chi Table, the calculation uses zero (currently the default for all).

When both the crosstalk and Chi factors are zero, all equations are essentially equivalent to previous versions. The new DLC equation has a marked distinction modification in that it essentially represents a “non-paired” situation to take into account variation in count durations of the background and sample. When the sample and background count durations are the same, the DLC result of the new “non-paired” equation equals the result of the previous equation. Thus, for this verification only the DLC is calculated manually when the sample and background count durations are different. In addition, the factor in the second portion of the MDC equation has been changed to “3” (updated from “2.71” to reflect current generally accepted industry practice).

**Equations for Isotopes by Mass and Activity ICP-MS (Uranium by Mass)**

***Activity Calculation:***

$$A_c = M_c x\ S$$

*Where*:

$A_c$ = Activity concentration of Nuclide (e.g. pCi/g or pCi/L)

$M_c$ = Mass concentration of nuclide (e.g. ug/g or ug/L)

S = Specific Activity of the Nuclide

The specific activity of a nuclide is a constant based upon the halflife.

***Total Uranium, by Mass:***

$$M_{Total} = M_{U-233} + M_{U-234} + M_{U-235} + M_{U-236} + M_{U-}$$

*Where*:

M = Mass for each isotope from ICP - MS results

***Total Uranium, by Activity:***

$$A_{Total} = A_{U-233} + A_{U-234} + A_{U-235} + A_{U-236} + A_{U-238}$$

*Where*:

A = Activity for each isotope using conversion above from ICP - MS results

***Percent U-235 (by mass):***

$$\text{Percent U} - 235 = \left( \frac{M_{U-235}}{\left(M_{U-233} + M_{U-234} + M_{U-235} + M_{U-236} + M_{U-238}\right)} \right) \times 100$$

*Where*:

M = Mass for each isotope

*Specific Activity values utilized in the calculations above were obtained from NuclideNavigator Version 3.4 and are based upon the PCNUDAT data file from the National Nuclear Data Center (NNDC) at Brookhaven National Laboratory (BNL).*

| Nuclide | Specific Activity (pCi/ug) |
|---|---|
| Technetium | 17120 |
| Uranium 233 | 9636 |
| Uranium 234 | 6222 |
| Uranium 235 | 2.161 |
| Uranium 236 | 64.67 |
| Uranium 238 | 0.3361 |

***Uranium, by Mass:***

$$M = \frac{(A \times C) \times (G / L)}{N}$$

*Where*:

$A = Activity\ in\ pCi/L\ for\ liquid,\ pCi/g\ for\ soil$

$C = conversion\ factor\ from\ pCi\ to\ Bq = 0.037$

$G = gram\ formula\ weight$

$L = Lamda = 0.693\ /\ halflife\ in\ seconds$

$N = Avegadro's\ Number = 6.02252E + 23$

***Total Uranium, by Mass:***

$$M_{Total} = M_{U-234} + M_{U-235} + M_{U-238}$$

*Where*:

$M = Mass\ for\ each\ isotope\ from\ above\ equation$

***Percent U-235:***

$$Percent\ U-235 = \left( \frac{M_{U-235}}{\left(M_{U-234} + M_{U-235} + M_{U-238}\right)} \right) \times 100$$

*Where*:

$M = Mass\ for\ each\ isotope$

## Appendix 5 Laboratory SOP Listing

| SOP Number | SOP Title |
|---|---|
| ST-GC-0005 | Extractable Total Petroleum Hydrocarbons |
| ST-GC-0014 | Aromatic Volatiles and Volatile Petroleum Hydrocarbon |
| ST-GC-0015 | PCB GC Analysis |
| ST-GC-0016 | Pesticide GC Analysis |
| ST-GC-0017 | Herbicide GC Analysis |
| ST-GC-0018 | Analysis of Water Miscible non-Halogenated Organic Compounds by GC/FID |
| ST-HS-0001 | Waste Minimization Plan |
| ST-HS-0002 | Facility Addendum to Corporate Safety Manual |
| ST-HS-0003 | St. Louis Facility Contingency Plan |
| ST-HS-0004 | Hazardous Waste Management Plan |
| ST-HS-0005 | Laboratory Security Systems |
| ST-HS-0006 | Quarantine Soils Procedure |
| ST-HS-0007 | Fume Hood Calibration |
| ST-IP-0001 | Reactive Cyanide & Sulfide |
| ST-IP-0002 | Acid Digestion of soil |
| ST-IP-0004 | Labware Prep for Inorganic & Trace Metal Analysis |
| ST-IP-0013 | Acid Digestion of Aqueous Samples & Extracts |
| ST-IP-0014 | Alkaline digestion of Cr+6 |
| ST-IP-0015 | Filtration Procedure for Dissolved Metals Analysis |
| ST-IP-0019 | Sulfide Distillation |
| ST-IP-0020 | Distribution Coefficients of Inorganic Species by the Batch Method |
| ST-IS-0001 | Software Change Management |
| ST-IS-0002 | Software Testing, Validation & Verification |
| ST-IS-0003 | Information Systems |
| ST-LC-0002 | Analysis of Nitroaromatic & Nitroamine Explosives |
| ST-MS-0001 | GC/MS Analysis based on 8270C and 625 |
| ST-MS-0002 | Volatile Organics by GCMS |
| ST-MT-0001 | Metals by ICP/MS |
| ST-MT-0003 | Metals by ICP-AES |
| ST-MT-0005 | Mercury in Aqueous Samples by CVAA |
| ST-MT-0007 | Mercury in Solid Samples by CVAA |
| ST-OP-0001 | Labware Preparation for Organic Analysis |
| ST-OP-0002 | Extraction & Cleanup of Organic Compounds from Water |
| ST-OP-0007 | Extraction of Herbicides - Water & Soil |
| ST-OP-0008 | Extraction of Nitroaromatics |
| ST-OP-0009 | TCLP/SPLP and CWET Procedures |
| ST-PM-0001 | Project Setup and Quote |
| ST-PM-0002 | Sample Receipt & Chain of Custody |
| ST-PM-0003 | Bottle Kit Preparation |
| ST-QA-0002 | Standard and Reagent Preparation |
| ST-QA-0005 | Calibration & Verification Procedure for Thermometer |
| ST-QA-0014 | Evaluation of Accuracy and Precision via Control C |
| ST-QA-0016 | IDL/MDL Determination |
| ST-QA-0021 | Internal Surveillance |

| SOP Number | SOP Title |
|---|---|
| ST-QA-0023 | Document Control |
| ST-QA-0024 | Preventative Maintenance |
| ST-QA-0028 | Water System Maintenance & Monitoring |
| ST-QA-0031 | VOA Holding Blank Analysis |
| ST-QA-0035 | Preparation and Management of SOPs |
| ST-QA-0036 | Non-Conformance Memo Process |
| ST-QA-0037 | Procurement of Quality Related Items |
| ST-QA-0038 | Procedure for Compositing and Subsampling |
| ST-QA-0040 | Manual Integration Procedure |
| ST-QA-0041 | Lead Auditor |
| ST-QA-0042 | 10CFR 21 Defects and Non-Compliances |
| ST-QA-0044 | Training |
| ST-QA-0045 | Internal Chain-of-Custody (ICOC) TALS |
| ST-QA-0046 | Data Review, Verification and Report Completeness Review (formerly ST-PM-0004) |
| ST-QAM | Quality Assurance Manual |
| ST-RC-0002 | Planchet Prep for Radiochemistry & Radiological Sc |
| ST-RC-0003 | Drying & Grinding of Soil & Solid Samples |
| ST-RC-0004 | Prep of Soil, Sludge, Filter, Biota & )/G Samples |
| ST-RC-0010 | Screening Samples for Presence of Radioactive Mate |
| ST-RC-0014 | Bulk Drying and Grinding of Soil and Solid Samples |
| ST-RC-0015 | Total Activity Screening Procedure by LSC |
| ST-RC-0020 | Determination of Gross Alpha/Beta Activity |
| ST-RC-0021 | Gross Alpha Radiation in Water - Coprecipitation |
| ST-RC-0025 | Preparation of Samples for Gamma Spectroscopy |
| ST-RC-0030 | Determination of Tritium in Water, Fluids, Soil & |
| ST-RC-0036 | Chlorine-36 |
| ST-RC-0040 | Total Alpha Emitting Isotopes of Radium |
| ST-RC-0041 | Radium-226 & Radium-228 by Chemical Separation |
| ST-RC-0042 | Iodine-129 in Water |
| ST-RC-0050 | Preparation of Strontium 89 & 90 |
| ST-RC-0055 | Determination of Fe55, Ni59 & Ni63 by LSC |
| ST-RC-0057 | Carbon -14/Inert Gas |
| ST-RC-0058 | Soil Prep for Sr-89, Sr-90 & Total Sr using Extraction Chromatography |
| ST-RC-0100 | Actinide Co-precipitation |
| ST-RC-0125 | Determination of TC99 using Eichrom TEVA Resin |
| ST-RC-0210 | Determination of Po210 by Alpha Spectrometry |
| ST-RC-0211 | Determination of Pb210 by LSC |
| ST-RC-0232 | Isotopic Th/Np in Various Matrices by Eichrom TEVA |
| ST-RC-0238 | Isotopic U by Eichrom UTEVA Resin for Various Matrices |
| ST-RC-0240 | Isotopic Am/Cu/Pu/Th/U in Various Matrices Eichrom |
| ST-RC-0242 | Isotopic Th/Pu/U in Various Matrices by Eichrom Se |
| ST-RC-0245 | Determination of Pu241 by LSC |
| ST-RC-0247 | Promethium247 & Samarium151 Lanthide Resin Separation |
| ST-RC-0301 | Radium Isotopes by Alpha Spectrometry |
| ST-RC-0302 | Gross alpha/beta and Low Energy Beta by LSC |
| ST-RC-5006 | Decontamination of Lab Glassware, Labware & Equip. |

| SOP Number | SOP Title |
|---|---|
| ST-RD-0102 | Gamma Vision Analysis |
| ST-RD-0210 | Alpha spectroscopy |
| ST-RD-0302 | Liquid Scintillation Counter Analysis |
| ST-RD-0403 | Low Background Gas Flow Proportional Counting System |
| ST-RP-0001 | Radiation Protection Program |
| ST-RP-0005 | ALARA Program |
| ST-RP-0010 | Internal Exposure Control |
| ST-RP-0020 | External Exposure Control |
| ST-RP-0030 | Radiological Contamination |
| ST-RP-0031 | Radiation Work Permits |
| ST-RP-0032 | Instrumentation and surveillance |
| ST-RP-0033 | Radiological Areas and Posting |
| ST-RP-0034 | Engineered Controls |
| ST-RP-0042 | Handling of Sealed Sources |
| ST-RP-0050 | Purchase, Receipt, Handling and ID of Radioactive |
| ST-RP-0051 | Packaging/Transportation of Radioactive Material |
| ST-RP-0100 | Radiation Protection Records |
| ST-RP-0110 | Radiation Protection Training |
| ST-RP-0120 | Emergency Response & notification |
| ST-RP-0140 | Quality Assurance in Radiological Protection |
| ST-WC-0002 | Cyanide Analysis by Technicon TRAACS 800 Auto analyzer. |
| ST-WC-0004 | Chemical Oxygen Demand |
| ST-WC-0005 | Percent Solids Determination |
| ST-WC-0006 | Total Organic Halides in Water (TOX) |
| ST-WC-0011 | Analysis of pH in Water & Soil |
| ST-WC-0012 | Analysis of Sulfide in Water |
| ST-WC-0013 | Phosphorus, all Forms |
| ST-WC-0014 | Analysis of Ammonia as N in Water & Soil |
| ST-WC-0016 | Total Organic Carbon |
| ST-WC-0017 | Phenolics, Total Recoverable |
| ST-WC-0019 | Alkalinity in Water & Soil |
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# Quality Assurance Manual Cover Page

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# Title Page:

## Quality Assurance Manual
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## SECTION 3. INTRODUCTION, SCOPE AND APPLICABILITY

### 3.1 Introduction and Compliance References

TestAmerica Denver'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, 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, CA-Q-M-002) and the various accreditation and certification programs listed in Appendix 3. The CQMP provides a summary of TestAmerica's quality and data integrity system. It contains requirements and general guidelines under which all TestAmerica facilities shall conduct their operations.

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

- ANSI/ASQC, E4-1994, "Specifications and Guidelines for Quality Management Systems for Environmental Data Collection and Environmental Technology Programs" (American National Standard, January 5, 1995, or most recent version)
- "EPA Requirements for Quality Management Programs" (QA/R-2) (EPA/240/B-01/002, May 31, 2006).
- 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; Final Update V, August 2015.*
- *U.S. Department of Defense (DoD), Quality Systems Manual (QSM) for Environmental Laboratories, Version 4.2, October 2010.*
- *U.S. Department of Defense (DoD)/Department of Energy (DOE) Consolidated Quality Systems Manual (QSM) for Environmental Laboratories, Version 5.0, July 2013.*
- *U.S. Department of Defense (DoD)/Department of Energy (DOE) Consolidated Quality Systems Manual (QSM) for Environmental Laboratories, Version 5.1, 2017*
- Federal Register, 40 CFR Parts 136, 141, 172, 173, 178, 179 and 261.
- *Manual for the Certification of Laboratories Analyzing Drinking Water (EPA 815-R-05-004, January 2005)*

- APHA, *Standard Methods for* the Examination of Water and Wastewater, 18th Edition, 19th, 20th, 21st, 22nd, and on-line Editions.
- *U.S. Department of Energy* Order 414.1B, *Quality Assurance*, Approved April 29, 2004.
- *U.S. Department of Energy* Order 414.1C, *Quality Assurance*, June 17, 2005.
- *U.S. Department of Energy Order 414.1D, Quality Assurance, Aril, 25, 2011.*
- Nuclear Regulatory Commission (NRC) Quality Assurance Requirements.
- Toxic Substances Control Act (TSCA).

## 3.2 Terms and Definitions

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

Refer to Appendix 2 for the Glossary/Acronyms.

## 3.3 Scope / Fields of Testing

The laboratory analyzes a broad range of environmental and industrial samples every month. Sample matrices vary among drinking water, effluent water, groundwater, hazardous waste, sludge and soils. The Quality Assurance Program contains specific procedures and methods to test samples of differing matrices for chemical, 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 the LIMS. 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 and the Quality Assurance (QA) Manager. In some cases, QAPPs and DQOs may specify less stringent requirements. The Laboratory Director and the QA Manager must determine if it is in the lab's best interest to follow the less stringent requirements.

### 3.4 Management of the Manual

#### 3.4.1 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 itself is reviewed annually by senior laboratory management to assure that it reflects current practices and meets the requirements of the laboratory's clients and regulators as well as the CQMP. Occasionally, the manual may need changes in order to meet new or changing regulations and operations. The QA Manager will review the changes in the normal course of business and incorporate changes into revised sections of the document. All updates will be reviewed by the senior laboratory management staff. The laboratory updates and approves such changes according to its Document Control procedures (refer to SOPs DV-QA-001P and DV-QA-0010).

## SECTION 4. MANAGEMENT REQUIREMENTS

### 4.1 Overview

TestAmerica Denver 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 and Chief Executive Officer (CEO), Chief Operations Officer (COO), Vice President of Quality, etc.). The laboratory operational and support staff work under the direction of the Laboratory Director. The organizational structure for both Corporate and TestAmerica Denver 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. This manual is specific to the operations of TestAmerica's Denver laboratory.

#### 4.2.2 President and Chief Executive Officer (CEO)

The President and CEO is a member of the Board of Directors and is ultimately responsible for the quality and performance of all TestAmerica facilities. The President and CEO establishes the overall quality standard and data integrity program for the Analytical Business, providing the necessary leadership and resources to assure that the standard and integrity program are met.

### 4.2.3 Chief Operations Officer (COO)

The COO reports directly to the President and CEO of TestAmerica. The COO is responsible for the operations of TestAmerica's subsidiary companies and the company's strategic growth.

### 4.2.4 Senior Vice President (SVP) of Operations & Client Service

The SVP of Operations and Client Services leads the Client Service Organization (CSO); and oversees the operations of all TestAmerica laboratories, the Corporate Technical Services group and the Sales Opportunity Optimization efforts. The SVP provides direction to the VPs of Operations, Client Service Directors, Manager of Project Managers, Director of Technical Services and a Director of Sales. The SVP of Operations and Client Services reports directly to the President and CEO of TestAmerica.

### 4.2.5 Vice President of Operations

Each VP of Operations (VPO) reports directly to the SVP of Operations and Client Services. Each VPO is responsible for the overall administrative and operational management of their respective laboratories. The VPO's responsibilities include allocation of personnel and resources, long-term planning, goal setting, and achieving the financial, business, and quality objectives of TestAmerica. The VPO's ensure timely compliance with Corporate Management directives, policies, and management systems reviews. The VPO's are also responsible for restricting any laboratory from performing analyses that cannot be consistently and successfully performed to meet the standards set forth in this manual.

### 4.2.6 Vice President of Quality and Environmental Health and Safety (VP-QA/EHS)

The Vice President (VP) of QA/EHS reports directly to the President and CEO. With the aid of the Executive Committee, Laboratory Directors, Quality Directors, Safety Manager, EH&S Coordinators and QA Managers, the VP-QA/EHS has the responsibility for the establishment, general overview and corporate maintenance of the Quality Assurance and EH&S Programs within TestAmerica. Additional responsibilities include:

- Review of QA/QC and EH&S aspects of Corporate SOPs & Policies, national projects and expansions or changes in services.
- Work with various organizations outside of TestAmerica to further the development of quality and EHS standards and represent TestAmerica at various trade meetings.
- Prepare of a monthly report that includes quality metrics across the analytical laboratories and a summary of any quality related initiatives and issues.
- Prepare of a monthly report that includes EH&S metrics across the analytical laboratories and a summary of any EH&S related initiatives and issues.
- With the assistance of the Corporate Senior Management Teams and the EHS Director~~s~~, develop and implement the TestAmerica Environmental, Health and Safety Program.

### 4.2.7 Quality Assessment Director

The Quality Assessment Director reports to the VP-QA/EHS. The Quality Assessment Director has QA oversight of laboratories; responsible for the internal audit system, schedule and

procedure; monitors laboratory internal audit findings; identifies common laboratory weaknesses; and monitors corrective action closures. Together with the Quality Compliance Director, the Quality Systems Director, and the VP-QA/EHS, the Quality Assessment Director has the responsibility for the establishment, general overview and maintenance of the Analytical Quality Assurance Program within TestAmerica.

### 4.2.8 Quality Compliance Director

The Quality Compliance Director reports to the VP-QA/EHS. The Quality Compliance Director has QA oversight of laboratories; monitors and communicates DoD / DoE requirements; develops corporate tools for ensuring and improving compliance; develops corporate assessment tools; identifies common laboratory weaknesses; and monitors corrective action closures. Together with the Quality Assessment Director, Quality Systems Director and the VP-QA/EHS, the Quality Compliance Director has the responsibility for the establishment, general overview and maintenance of the Analytical Quality Assurance Program within TestAmerica.

### 4.2.9 Quality Systems Director

The Quality Systems Director reports to the VP-QA/EHS. The Quality Systems Director has QA oversight of laboratories; develops quality policies, procedures and management tools; monitors and communicates regulatory and certification requirements; identifies common laboratory weaknesses; and monitors corrective action closures. Together with the Quality Assessment Director, Quality Compliance Director and the VP-QA/EHS, the Quality Systems Director has the responsibility for the establishment, general overview and maintenance of the Analytical Quality Assurance Program within TestAmerica.

### 4.2.10 Quality Information Manager

The Quality Information Manager is responsible for managing all company official documents (e.g., Policies, Procedures, Work Instructions), the company's accreditation database, intranet websites, external laboratory subcontracting, regulatory limits for clients on the company's TotalAccess website; internal and external client support for various company groups (e.g., Client Services, EH&S, Legal, IT, Sales) for both quality and operational functions. The Quality Information Manager reports to the VP-QA/EHS; and works alongside the Quality Assessment, Quality Compliance and Quality System Directors and EHS Managers to support both the Analytical Quality Assurance and EHS Programs within TestAmerica.

### 4.2.11 Technical Services Director

The Technical Services Director is responsible for establishing, implementing and communicating TestAmerica's Analytical Business's Technical Policies, SOPs, and Manuals. Other responsibilities include conducting technical assessments as required, acting as a technical resource in national contracts review, coordinating new technologies, establishing best practices, advising staff on technology advances, innovations, and applications.

### 4.2.12 Ethics and Compliance Officers (ECOs)

TestAmerica has designated two senior members of the Corporate staff to fulfill the role of Ethics and Compliance Officer (ECO) – Corporate Counsel and VP of Human Resources and the VP-QA/EHS. Each ECO acts as a back-up to the other ECO and both are involved when

data investigations occur. Each ECO has a direct line of communication to the entire senior Corporate and lab management staff.

The ECOs ensure that the organization distributes the data integrity and ethical practices policies to all employees and ensures annual trainings and orientation of new hires to the ethics program and its policies. The ECO is responsible for establishing a mechanism to foster employee reporting of incidents of illegal, unethical, or improper practices in a safe and confidential environment.

The ECOs monitor and audit procedures to determine compliance with policies and to make recommendations for policy enhancements to the President and CEO, VPOs, Laboratory Director or other appropriate individuals within the laboratory. The ECO will assist the laboratory QA Manager in the coordination of internal auditing of ethical policy related activities and processes within the laboratory, in conjunction with the laboratories regular internal auditing function.

The ECOs will also participate in investigations of alleged violations of policies and work with the appropriate internal departments to investigate misconduct, remedy the situation, and prevent recurrence of any such activity.

### 4.2.13 Chief Information Officer (CIO)

The CIO is responsible for establishing, implementing and communicating TestAmerica's Information Technology (IT) Policies, SOPs and Manuals. Other responsibilities include coordinating new technologies, development of electronic communication tools such as TestAmerica's intranet and internet sites, ensuring data security and documentation of software, ensuring compliance with the NELAC standard, and assistance in establishing, updating, and maintaining Laboratory Information Management Systems (LIMS) at the various TestAmerica facilities.

### 4.2.14 Environmental Health and Safety Managers (Corporate)

The EHS Managers report directly to the VP-QA/EHS. The EHS Managers are responsible for the development and implementation of the TestAmerica Environmental, Health and Safety program. Responsibilities include:

- Consolidation and tracking all safety and health-related information and reports for the company, and managing compliance activities for TestAmerica locations.
- Coordination/preparation of the corporate Environmental, Health and Safety Manual Template that is used by each laboratory to prepare its own laboratory-specific Safety Manual/ CHP.
- Preparation of information and training materials for laboratory EHS Coordinators.
- Assistance in the internal and external coordination of employee exposure and medical monitoring programs to insure compliance with applicable safety and health regulations.
- Serving as Department of Transportation (DOT) focal point and providing technical assistance to location management.
- Serving as Hazardous Waste Management main contact and providing technical assistance to location management.

### 4.2.15 Laboratory Director

TestAmerica Denver's Laboratory Director is responsible for the overall quality, safety, financial, technical, human resource and service performance of the whole laboratory and reports to his/her respective VPO. The Laboratory Director provides the resources necessary to implement and maintain an effective and comprehensive Quality Assurance and Data Integrity Program.

Specific responsibilities include, but are not limited to:

- Providing one or more technical managers for the appropriate fields of testing. If the Technical Manager is absent for a period of time exceeding 15 consecutive calendar days, the Laboratory Director must designate another full time staff member meeting the qualifications of the Technical Manager to temporarily perform this function. If the absence exceeds 65 consecutive calendar days, the primary accrediting authority must be notified in writing.

- Ensuring that all analysts and supervisors have the appropriate education and training to properly carry out the duties assigned to them and ensures that this training has been documented.

- Ensuring that personnel are free from any commercial, financial and other undue pressures which might adversely affect the quality of their work.

- Ensuring TestAmerica's human resource policies are adhered to and maintained.

- Ensuring that sufficient numbers of qualified personnel are employed to supervise and perform the work of the laboratory.

- Ensuring 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.

- Reviewing and approving all SOPs prior to their implementation and ensures all approved SOPs are implemented and adhered to.

- Pursuing and maintaining appropriate laboratory certification and contract approvals.

- Supporting ISO 17025 requirements.

- Supporting DoD ELAP requirements.

- Ensuring client specific reporting and quality control requirements are met.

- Directing the management team, consisting of the QA Manager, the Inorganic Operations Manager, the Organic Operations Manager, the EH&S Coordinator and the Office Manager as direct reports.

### 4.2.16 Quality Assurance (QA) Manager

The QA Manager has responsibility and authority to ensure the continuous implementation of the quality system.

The QA Manager reports directly to the Laboratory Director and to his/her Corporate Quality Director. This person 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. This person has documented training and/or experience in QA/QC procedures and the laboratory's Quality System. The QA Manager directs the activities of the QA staff to accomplish specific responsibilities, which include, but are not limited to:

- Serving as the focal point for QA/QC in the laboratory.
- Having functions independent from laboratory operations for which he/she has quality assurance oversight.
- Maintaining and updating the QAM.
- Monitoring and evaluating laboratory certifications; scheduling proficiency testing samples.
- Monitoring and communicating regulatory changes that may affect the laboratory to management.
- Training and advising the laboratory staff on quality assurance/quality control procedures that are pertinent to their daily activities.
- Having documented training and/or experience in QA/QC procedures and the laboratory's Quality System.
- Having a general knowledge of the analytical test methods for which data audit/review is performed (and/or having the means of getting this information when needed).
- Arranging for or conducting internal audits on quality systems and the technical operation.
- Maintaining records of all ethics-related training, including the type and proof of attendance.
- Maintaining, improving, and evaluating the corrective action database and the corrective and preventive action systems.
- Notifying laboratory management of deficiencies in the quality system and ensuring corrective action is taken. Procedures that do not meet the standards set forth in the QAM or laboratory SOPs shall be investigated following procedures outlined in Section 12 and if deemed necessary may be temporarily suspended during the investigation.
- Objectively monitoring standards of performance in quality control and quality assurance without outside (e.g., managerial) influence.
- Coordinating document control of SOPs, MDLs, control limits, and miscellaneous forms and information.

- Reviewing a percentage of all final data reports for internal consistency. Reviewing Chain of Custody (COC), correspondence with the analytical request, batch QC status, completeness of any corrective action statements, manual calculations, format, holding time, sensibility and completeness of the project file contents.

- Reviewing external audit reports and data validation requests.

- Following-up with audits to ensure client QAPP requirements are met.

- Establishing reporting schedule and preparation of various quality reports for the Laboratory Director, clients and/or Corporate QA.

- Developing suggestions and recommendations to improve quality systems.

- Researching current state and federal requirements and guidelines.

- Directing the QA team to enable communication and to distribute duties and responsibilities.

- Ensuring communication with laboratory staff and monitoring standards of performance to ensure that systems are in place to produce the level of quality as defined in this document.

- Notifying laboratory management of deficiencies in the quality system and ensuring corrective action is taken. Procedures that do not meet the standards set forth in the QAM or laboratory SOPs are temporarily suspended following the procedures outlined in Section 12.

- Evaluating the thoroughness and effectiveness of training.

- Assuring compliance with ISO 17025.

- Assuring compliance with the DoD/DOE QSM.

#### 4.2.17 Quality Assurance Specialist

The Quality Assurance Specialist performs several roles. The QA Specialist reports to the facility QA Manager. The QA Specialist is responsible for QA documentation and involvement in the following activities:

- Assisting the QA Manager in performing the annual internal laboratory audits, compiling the evaluation, and coordinating the development of an action plan to address any deficiency identified.

- Facilitating external audits, coordinating with the QA Manager and Laboratory Staff to address any deficiencies noted at the time of the audit and subsequently presented in the final audit report.

- Assisting the QA Manager in the preparation of new SOPs and in the maintenance of existing SOPs, coordinating annual reviews and updates.

- Managing the performance testing (PT) studies, coordinating follow up studies for failed analytes and working with the QA Manager and Laboratory Staff to complete needed corrective action reports.

- Reviewing and maintaining personnel training records.
- Documenting control maintenance.
- Assisting the Quality Manager and Project Management Group in the review of program plans for consistency with organizational and contractual requirements. Summarize and convey to appropriate personnel anomalies or inconsistencies observed in the review process.
- Managing certifications and accreditations.
- Monitoring for compliance with the following QA Metrics: Temperature Monitoring of refrigeration units; thermometer calibrations; balance calibrations; Eppendorf/pipette calibrations; and proper standard/reagent storage.
- Periodically checking the proper use and review of instrument logs.
- Initiating the Mint-miner data file review process for organic instrumentation.
- Initiating the annual Instrument review.
- Assisting in the technical review of data packages which require QA review.
- Assisting the QA Manager in meeting the responsibilities of the QA Department as described in laboratory policies and SOPs.

#### 4.2.18 Quality Assurance Assistant

The Quality Assurance Assistant performs several roles. The QA Assistant reports to the facility QA Manager. The QA Assistant is responsible for QA documentation and involvement in the following activities:

- Assisting the QA Manager in performing the annual internal laboratory audits, compiling the evaluation, and coordinating the development of an action plan to address any deficiency identified.
- Serving as a project manager for proficiency testing samples and other QC samples. Processes and reports QC samples as routine samples to appropriate agencies.
- Assisting the QA Manager in maintaining the laboratory's reference data to keep it current and accurate.
- Preparing certification applications for states as directed by QA Manager.
- Reviewing and maintaining personnel training records.
- Performing document control maintenance.
- Assisting departments in generating MDL spreadsheets and calculations, reviewing MDL studies submitted to QA.
- Assisting in control limit generation.

- Ensuring maintenance of records archives.

- Maintaining historical indices for all technical records including SOPs, QC records, laboratory data, etc.

- Assisting the QA Manager in meeting the responsibilities of the QA Department as described in laboratory policies and SOPs.

### 4.2.19 Operations Manager (referred to in this document as Technical Manager)

The Inorganic and Organic Operations Managers (Technical Managers) report directly to the Laboratory Director. They are accountable for all analyses and analysts under their experienced supervision and for compliance with the ISO 17025 Standard. 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:

- Exercising day-to-day supervision of laboratory operations for the appropriate field of accreditation and reporting of results. Coordinating, writing, and 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. They insure that the SOPs are properly managed and adhered to at the bench. They develop standard costing of SOPs to include supplies, labor, overhead, and capacity (design versus demonstrated versus first-run yield) utilization.

- Reviewing and approving, with input from the QA Manager, proposals from marketing, in accordance with an established procedure for the review of requests and contracts. This procedure addresses the adequate definition of methods to be used for analysis and any limitations, the laboratory's capability and resources, the client's expectations. Differences are resolved before the contract is signed and work begins. A system documenting any significant changes is maintained, as well as pertinent discussions with the client regarding their requirements or the results of the analyses during the performance of the contract. All work subcontracted by the laboratory must be approved by the client. Any deviations from the contract must be disclosed to the client. Once the work has begun, any amendments to the contract must be discussed with the client and so documented.

- Monitoring the validity of the analyses performed and data generated in the laboratory. This activity begins with reviewing and supporting all new business contracts, insuring data quality, analyzing internal and external non-conformances to identify root cause issues and implementing the resulting corrective and preventive actions, facilitating the data review process (training, development, and accountability at the bench), and providing technical and troubleshooting expertise on routine and unusual or complex problems.

- Providing 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.

- Enhancing efficiency and improving quality through technical advances and improved LIMS utilization. Capital forecasting and instrument life cycle planning for second generation methods and instruments as well as asset inventory management.

- Coordinating sample management from “cradle to grave,” insuring that no time is lost in locating samples.
- Evaluating the level of internal/external non-conformances for all departments.
- Continuously evaluating turnaround time and addressing any problems that may hinder meeting the required and committed turnaround time from the various departments.
- Developing and improving the training of all analysts in cooperation with the Laboratory Director and QA Manager and in compliance with regulatory requirements.
- Working with the Department Managers/Supervisors to ensure that scheduled instrument maintenance is completed.
- Ensuring efficient utilization of supplies
- Constantly monitoring and modifying the processing of samples through the departments.
- Fully supporting the quality system.
- Ensuring Department Managers/Supervisors schedule all QA/QC-related requirements for compliance, e.g., MDLs, control chart review, etc.
- Directing department personnel to communicate quality, technical, personnel, and instrumental issues for a consistent team approach.
- Coordinating audit responses with the QA Manager.
- Assuring compliance with ISO 17025.
- Assuring compliance with the DoD/DOE QSM.

### 4.2.20 Radiation Safety Officer

The Radiation Safety Officer (RSO) is responsible for implementing TestAmerica Denver’s radiation safety program. The RSO reports directly to the Laboratory Director. The RSO’s duties consist of:

- Managing the personnel radiation dosimetry program
- Maintaining the Radioactive Materials License and radionuclide inventory
- Monitoring laboratory operations for compliance with the Radiation Safety Manual
- Training, documenting, and evaluating the TestAmerica Denver personnel for handling radioactive material
- Creating, releasing, and decontaminating Radiological Control Areas (RCAs)
- Monitoring and tracking radioactive materials

- Conducting the radioactive material waste disposal program in accordance with State and Federal regulations
- Maintaining all records related to the radiation safety program

### 4.2.21 Employee Health and Safety Coordinator

The Employee Health and Safety Coordinator (EH&S Coordinator) is responsible for administering the EH&S program that provides a safe, healthy working environment for all employees and the environment. The EH&S Coordinator reports directly to the Laboratory Director and the corporate Environmental Health and Safety Director. He/She monitors all areas for unsafe conditions, acts, and potential hazards. Specific responsibilities include, but are not limited to:

- Staying current with the hazardous waste regulations
- Continuing training on hazardous waste issues
- Reviewing and updating annually the Hazardous Waste Contingency Plan in the Environmental Health and Safety Manual
- Auditing the staff with regard to compliance with the Hazardous Waste Contingency Plan
- Contacting the hazardous waste subcontractors for review of procedures and opportunities for minimization of waste
- Conducting ongoing, necessary safety training and conducting new employee safety orientation
- Assisting in development and maintenance of the Chemical Hygiene/Safety Manual
- Administering dispersal of all Safety Data Sheet (SDS) information
- Performing regular chemical hygiene and housekeeping instruction
- Giving instruction on proper labeling and practice
- Serving as chairman of the laboratory safety committee
- Providing and training personnel on protective equipment
- Overseeing the inspection and maintenance of general safety equipment – fire extinguishers, safety showers, eyewash fountains, etc. and ensure prompt repairs as needed
- Supervising and scheduling fire drills and emergency evacuation drills
- Determining initial and subsequent exposure monitoring, if necessary, to determine potential employee exposure to chemicals used in the laboratory
- Conducting exposure monitoring assessments, as needed

- Determining when a complaint of possible over-exposure is “reasonable” and should be referred for medical consultation
- Assisting in the internal and external coordination of the medical consultation/monitoring program conducted by TestAmerica’s medical consultants
- Conducting weekly inspections of satellite accumulation areas and all hazardous waste storage areas
- Coordinating the proper storage, packing and disposal of laboratory wastes according to Department of Transportation (DOT) and Resource Conservation and Recovery Act (RCRA) regulations
- Maintaining waste disposal records
- Coordinating spill response activities including documentation for waste storage areas

### 4.2.22 Waste Disposal Technician

The Waste Disposal Technician is responsible for proper disposal of spent chemicals, process waste, and unused laboratory samples used in the laboratory according to corporate, federal, state, and local guidelines. The Waste Disposal Technician reports to the Hazardous Waste Specialist and EH&S Coordinator. The duties consist of:

- Packaging hazardous waste for transport per DOT, RCRA and TSCA guidelines
- Identifying waste streams and maintaining satellite accumulation areas
- Packaging expired chemicals for shipment or disposal
- Tracking volume of waste generated for reporting to corporate and EPA
- Preparing and tracking implementation of the Waste Minimization Plan
- Emptying satellite containers into bulk containers and returns to the laboratory for reuse

### 4.2.23 Department Manager/Supervisor

Department Managers/Supervisors report to the Operations Manager. At TestAmerica Denver these individuals may have the title of Department Manager I or II or Supervisor I or II. The title and level designation are based on the level of experience. He/she is accountable for all analyses and analysts under their experienced supervision and act as the Technical Managers in their assigned area in compliance with TNI requirements and for compliance with the ISO 17025 Standard. 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:

- Exercising day-to-day supervision of laboratory operations for the appropriate field of accreditation and reporting of results.
- Ensuring that analysts in their department adhere to applicable SOPs and the QA Manual. They perform frequent SOP and QA Manual review to determine if analysts are in

compliance and if new, modified, and optimized measures are feasible and should be added to these documents.

- Coordinating, writing, and reviewing documentation of all test methods, i.e., SOPs, with regard to quality, integrity, regulatory requirements and optimum and efficient production techniques, and subsequent analyst training and interpretation of the SOPs for implementation and unusual project samples.

- Reviewing and approving, with input from the QA Manager, Quality Assurance Project Plans (QAPPs). This procedure addresses the adequate definition of methods to be used for analysis and any limitations, the laboratory's capability and resources and 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. Any deviations from the contract must be disclosed to the client. Once the work has begun, any amendments to the contract must be discussed with the client and so documented.

- Monitoring the validity of the analyses performed and data generated in the laboratory. This activity begins with reviewing and supporting all new business contracts, insuring data quality, analyzing internal and external non-conformances to identify root cause issues and implementing the resulting corrective and preventive actions, facilitating the data review process (training, development, and accountability at the bench), and providing technical and troubleshooting expertise on routine and unusual or complex problems.

- Providing 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.

- Enhancing efficiency and improving quality through technical advances, improved LIMS utilization, capital forecasting and instrument life cycle planning for second generation methods and instruments as well as asset inventory management.

- Coordinating sample management from "cradle to grave," insuring that no time is lost in locating samples.

- Scheduling all QA/QC-related requirements for compliance, e.g., MDLs, etc.

- Directing department personnel to communicate quality, technical, personnel, and instrumental issues for a consistent team approach.

- Coordinating audit responses with the QA Manager.

- Complying with ISO 17025, The NELAC Institute (TNI) Standard, DOD ELAP and the various QC programs implemented at the Denver laboratory.

- Participating in the selection, training (familiarization with SOP, QC, Safety and computer systems), developing performance objectives and standards of performance, appraising (measurement of objectives), scheduling, counseling, disciplining, and motivating analysts and documenting these activities in accordance with systems developed by the QA and Personnel Departments.

- Evaluating staffing sufficiency and overtime needs.
- Encouraging the development of analysts to become cross-trained in various methods and/or operate multiple instruments efficiently while performing maintenance and documentation, self-supervise, and function as a department team.
- Providing guidance to analysts in resolving problems encountered daily during sample prep/analysis in conjunction with the Operations Manager, and/or QA Manager. Each is responsible for 100% of the data review and documentation, non-conformance and corrective actions, the timely and accurate completion of performance evaluation samples and MDLs, for his/her department.
- Ensuring all logbooks are maintained, current, reviewed, and properly labeled or archived.
- Reporting all non-conformance conditions to the QA Manager, Operations Manager, and/or Laboratory Director.
- Ensuring that preventive maintenance is performed on instrumentation as detailed in the QA Manual or SOPs. He/She has responsibility for developing and implementing a system for preventive maintenance, troubleshooting, and repairing or arranging for repair of instruments.
- Maintaining adequate and valid inventory of reagents, standards, spare parts, and other relevant resources required to perform daily analysis.
- Achieving optimum turnaround time on analyses and compliance with holding times.
- Conducting efficiency and cost control evaluations on an ongoing basis to determine optimization of labor, supplies, overtime, first-run yield, capacity (designed vs. demonstrated), second- and third-generation production techniques/instruments, and long-term needs for budgetary planning.
- Developing and implementing calibration programs.
- Providing written responses to external and internal audit issues.

### 4.2.24 Laboratory Analysts

Laboratory analysts are responsible for conducting analysis and performing all tasks assigned to them by the group leader or supervisor. The Analyst position at TestAmerica Denver is divided into levels. These levels range from Analyst I to Analyst V. The level designation is based on experience, expertise, and responsibilities. The responsibilities of the analysts are listed below:

- Performing 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.
- Documenting standard and sample preparation, instrument calibration and maintenance, data calculations, sample matrix effects, and any observed non-conformance on worklists, benchsheets, lab notebooks and/or the Non-Conformance Memo module in the LIMS.

- Reporting all non-conformance situations, instrument problems, matrix problems and QC failures, which might affect the reliability of the data, to their supervisor, the Technical Manager, and/or the QA Manager or member of QA staff.

- Performing 100% review of the data generated prior to entering and submitting for secondary level review.

- Suggesting method improvements to their supervisor, the Technical Manager, and the QA Manager. These improvements, if approved, will be incorporated. Providing 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.

- Working cohesively as a team in their department to achieve the goals of accurate results, optimum turnaround time, cost effectiveness, cleanliness, complete documentation, and personal knowledge of environmental analysis.

### 4.2.25 Laboratory Technician

Laboratory Technicians are responsible for the preparation of samples and performing all tasks assigned to them by the group leader or supervisor. The Laboratory Technician position at TestAmerica Denver is divided into three levels. These levels are Laboratory Technician I, Laboratory Technician II, and Laboratory Technician III. The level designation is based on experience, expertise, and responsibilities. The responsibilities of the Laboratory Technician are listed below:

- Retrieving samples from Sample Control for analysis

- Performing sample preparation 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.

- Documenting standard and sample preparation, sample matrix effects, and any observed non-conformance on worklists, benchsheets, lab notebooks and/or the Non-Conformance Database.

- Reporting all non-conformance situations, sample preparation problems, matrix problems and QC failures, which might affect the reliability of the data, to their supervisor, the Technical Manager, and/or the QA Manager or member of QA staff.

- Working cohesively as a team member in their department to achieve the goals of accurate results, optimum turnaround time, cost effectiveness, cleanliness, complete documentation, and personal knowledge of environmental analysis.

### 4.2.26 Laboratory Assistant

The Laboratory Assistant position is an entry-level position to learn basic laboratory technician skills. The Laboratory Assistant reports to their group leader or supervisor. The Laboratory Assistant's duties include the following:

- Assisting the Laboratory Technicians in preparation of samples for analysis

- Preparing routine forms and reports

- Collecting and preparing materials and supplies for the laboratory
- Assisting technicians in conducting routine analysis.

### 4.2.27 Sample Receiving Supervisor

The Sample Receiving Supervisor reports to the Operations Manager. The responsibilities are outlined below:

- Managing department resources in cooperation.
- Serving as liaison between the Sample Receiving, the Customer Service Organization (CSO) and operations.
- Directing the logging of incoming samples into the LIMS
- Ensuring the verification of data entry from login
- Providing daily assessments of sample receipts
- Monitoring the preparation and shipment of bottle kits to clients
- Overseeing the receipt, log in, and storage of samples
- Scheduling couriers for sample pickup from customer sites

### 4.2.28 Sample Control Technician

The Sample Control Technician reports to the Sample Receiving Supervisor. The Sample Control Technician position at TestAmerica Denver is divided into levels. These levels range from Sample Control Technician I to Sample Control Technician IV. The level designation is based on experience and responsibilities of the Technician. The Sample Control Technician responsibilities include the following:

- Receiving and unloading samples or consignments in accordance with DOT regulations
- Verifying samples against the Chain of Custody (COC)
- Logging samples into the LIMS to assign a lot number for tracking purposes and distribute the paperwork to the Project Managers and Department Managers/Supervisors
- Labeling samples with lot number assigned and deliver the samples to the appropriate labs for analysis daily
- Monitoring freezer and cooler temperatures daily to confirm that the readings are within SOP guidelines
- Shipping all subcontracted samples to designated lab in accordance with DOT regulations as needed

### 4.2.29 Shipping/Maintenance Technician

The Shipping/Maintenance Technician reports to the Sample Receiving Supervisor. The Shipping/Maintenance Technician duties include the following:

- Maintaining the inventory control system
- Receiving and distributing incoming supplies
- Preparing and shipping bottle sampling kits to clients or on-site crews
- Maintaining bottle and cooler inventory
- Packing in-house samples for shipment to other laboratories

### 4.2.30 Courier

The Courier reports to the Sample Receiving Supervisor. The Courier's duties include the following:

- Picking up and delivering samples and reports to clients and the laboratory
- Receiving and signing the chain of custody for samples
- Preparing and shipping bottle sampling kits to clients or on-site crews
- Performing preventative maintenance on company vehicles

### 4.2.31 Manager of Client Relations Managers

The Manager of Client Relations Managers mentors a regional team of Client Relations Managers (CRMs), coordinating workload and ensuring that bids and proposals are provided on schedule. The Manager of Client Relations Managers reports to the Client Services Director. The responsibilities of this position include:

- Managing the CRM team to ensure that bids are provided to clients in a timely manner
- Providing oversight to CRMs regarding pricing decisions
- Providing guidance to the CRMs for compilation of large bids and proposals such as strategy and content
- Providing guidance for professional development of the CRMs

### 4.2.32 Client Relations Manager

The Client Relations Manager (CRM) is accountable for new client setup, account maintenance, document review, quotes and proposals, and project kick-off. The CRM role fosters and develops client relationships in support of the CSO mission. The duties of this position include:

- Verifying that that lab certification meets project requirements for new quotes

- Verifying that lab compound lists and limits meet project requirements for new quotes
- Confirming that EDD format is available for new quotes
- Engaging workshare labs, service centers and non-TA locations as needed for new quotes
- Providing supporting documentation to the client as needed
- Setting up new clients initiating project QAPP review with operations, QA and subcontract labs as needed
- Initiating technical support from operations as needed
- Providing quotes
- Communicating with clients to ensure client requirements are being met and complaints are communicated to the appropriate personnel within TestAmerica for resolution

#### 4.2.33 Manager of Project Management

The Manager of Project Management reports to the Regional Client Services 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 duties of this position are outlined below:

- Managing technical training and growth of the Project Management team
- Serving as technical liaison for the Project Management team
- Providing human resource management of the Project Management team
- Overseeing response to client inquiries concerning sample status
- Assisting clients regarding the resolution of problems concerning COC
- Ensuring that client specifications are met by communicating project and quality assurance requirements to the laboratory
- Notifying the supervisors of incoming projects and sample delivery schedules
- Being accountable to clients for communicating sample progress in daily status meeting with agreed-upon due dates
- Discussing with clients any project-related problems, resolving service issues, and coordinating technical details with the laboratory staff
- Monitoring the status of all data package projects in-house to ensure timely and accurate delivery of reports
- Informing clients of data package-related problems and resolve service issues

### 4.2.34 Project Manager

The Project Managers report to the Manager of Project Management (MPM) and serve as liaisons between the laboratory's technical departments and the laboratory's clients. At TestAmerica Denver there are two levels of Project Managers (I or II). The level designation is based on experience, expertise, and responsibilities. The Project Manager's responsibilities include:

- Ensuring client specifications are met by communicating project and quality assurance requirements to the laboratory
- Notifying laboratory personnel of incoming projects and sample delivery schedules
- Monitoring the status of all projects in-house to ensure timely delivery of reports
- Informing clients of project-related problems, resolving service issues and coordinating technical issues with the laboratory staff
- Assisting clients regarding the resolution of problems concerning COC
- Coordinating client requests for sample containers and ensuring clients receive the proper sampling supplies
- Scheduling sample pick-ups from client offices or project sites and notifying the laboratory staff of incoming samples
- Coordinating subcontract work
- Responding to client inquiries concerning sample status
- Assisting clients with resolution of problems concerning Chains-of-Custody

### 4.2.35 Manager of Project Management Assistants

The Manager of Project Management Assistants (PMAs) reports to the Manager of Project Management. The Manager of PMAs responsibilities include:

- Supporting Project Management staff to meet the mission of the Client Service Organization
- Supervising the Project Management Assistants
- Managing technical training and growth of the Project Management Assistants team

### 4.2.36 Project Management Assistant

The Project Management Assistant reports to the Manager of Project Management Assistants and designated Project Managers. The Project Management Assistant assists the Project Manager in servicing the client's needs and communicating those needs to the laboratory. The Project Management Assistant's responsibilities include:

- Collating data reports, expanded deliverables, and electronic data deliverables (EDD's) for

delivery to clients.

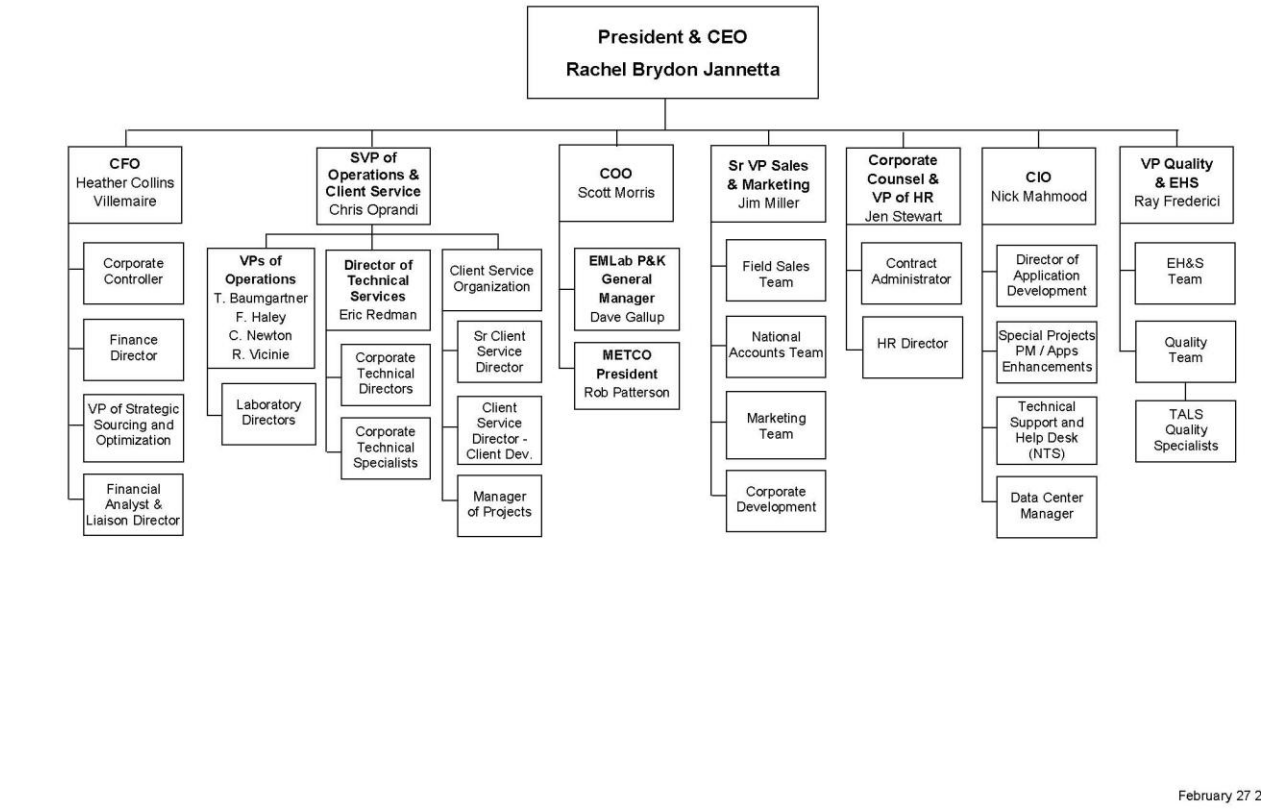
- Writing case narratives accompanying data packages to communicate anomalies to clients
- Proof reading and filing data reports received from the laboratory
- Assisting Project Managers in changing compound lists, TAT, and setting up tables in Word or Excel
- Monitoring report due dates for timely delivery
- Invoicing completed data packages
- Generating credit or debit invoices to ensure proper payment
- Copying and paginating reports

### 4.3 Deputies

The following table defines who assumes the responsibilities of key personnel in their absence. See WI-DV-0071 for the current list of personnel in these roles.

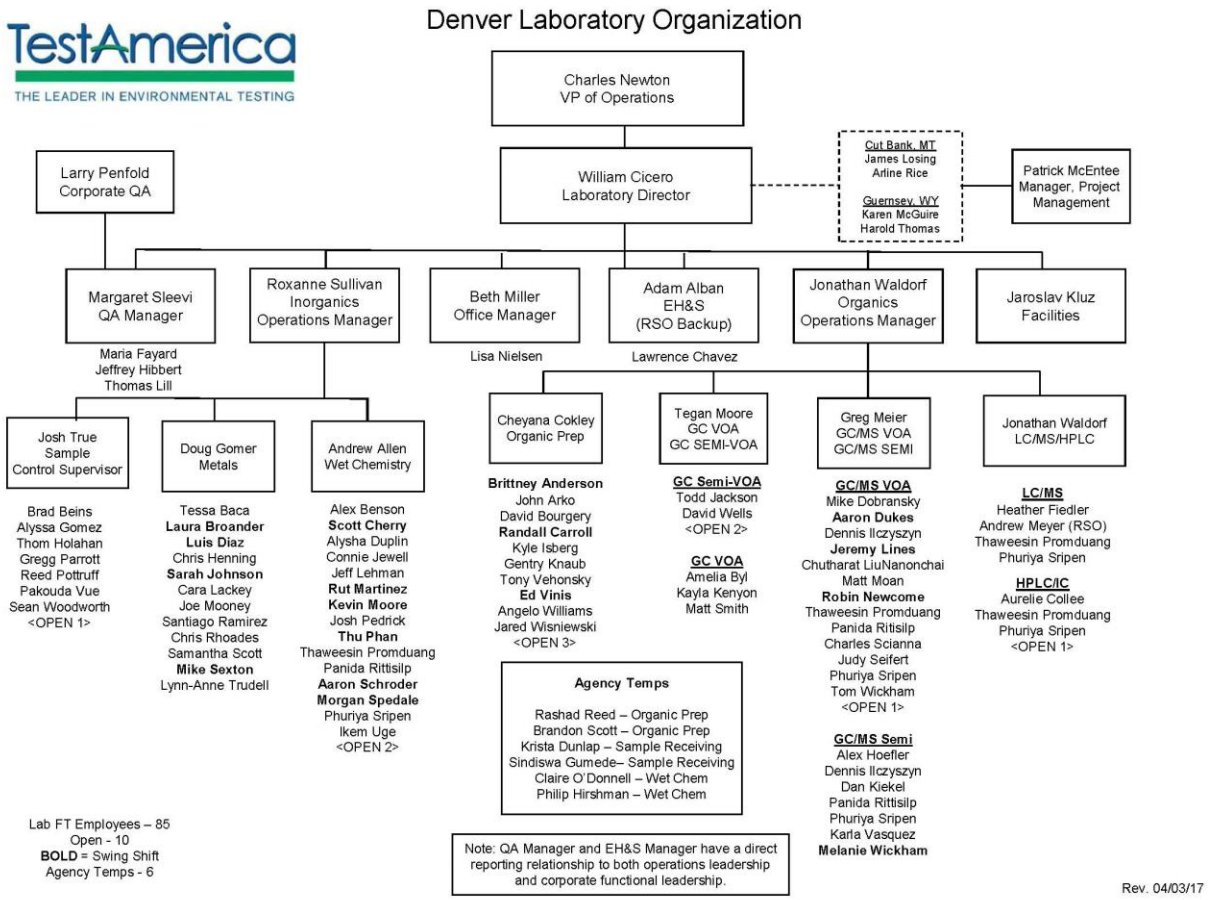
| Key Personnel | Deputy |
|---|---|
| Laboratory Director | Administrative Duties: Office Manager<br>Technical and Operations Duties: Operations Managers |
| Quality Assurance Manager | Quality Assurance Specialist |
| Inorganic Operations Manager | Organic Operations Manager |
| Organic Operations Manager | Inorganic Operations Manager |
| EHS Coordinator | Backup EHS Coordinator |
| Radiation Safety Officer | Backup Radiation Safety Officer |
| CSO Manager of Project Management | CSO Manager of Project Management Assistants |

**Figure 4-1. Corporate and Laboratory Organization Charts**



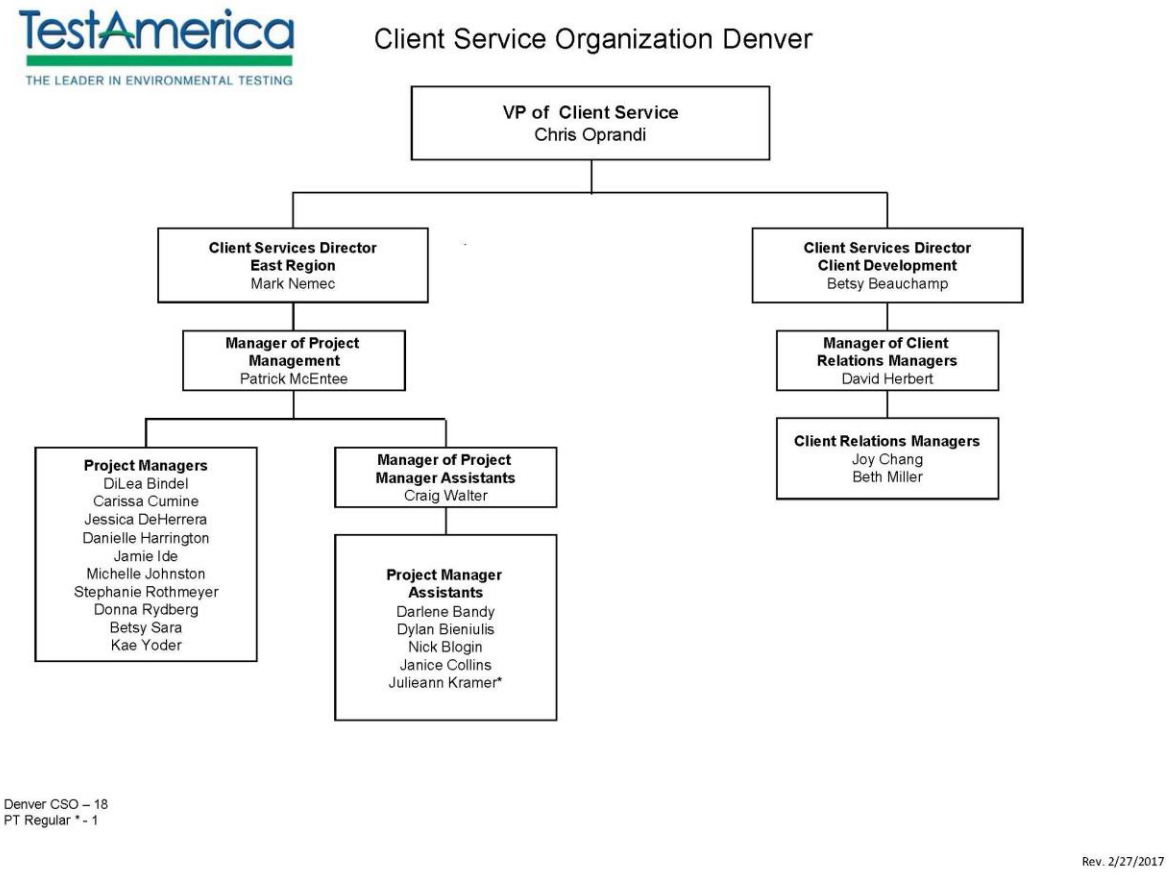

The current version of this organization chart is available on Oasis.

**Figure 4-1. Corporate and Laboratory Organization Charts (cont.)**




The current version of this organization chart is available on the public drive:
G:\HR\Read\Org Charts.

**Figure 4-1. Corporate and Laboratory Organization Charts (cont.)**




The current version of this organization chart is available on the public drive:
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## 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 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 CW-L-S-002).
- Procedures and guidance for recalling data if necessary (Corporate SOP CW-Q-S-005).
- Effective external and internal monitoring system that includes procedures for internal audits (Section 15).

These elements assure that the laboratory is able to:

- Produce results, which are accurate and include QA/QC information that meet 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 this industry.
- Operate the 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 this 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 Policy Memoranda – Quality or Administrative policies issued by the QA Manager and/or Laboratory Director to address requirements not otherwise detailed in SOPs or Work Instructions.

#### 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 Policy Memoranda
- Laboratory Quality Assurance Manual (QAM)
- Laboratory SOPs and Policies
- Other (Work Instructions (WI), memos, forms, flow charts, checklists, 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).

### 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 maintains a *Quality Control Limit Summary* in the LIMS referred to as a *"Method Limit Group"* that summarizes the precision and accuracy acceptability limits for each performed analysis. This summary includes an effective date, is updated each time new limits are generated and are managed by the laboratory's QA department. Unless otherwise noted, limits within these tables are laboratory generated. Some acceptability limits are derived from US

EPA methods when they are required or other programs such as DoD/DOE QSM. Where US EPA method limits are not required or program limits are not available, the laboratory has developed limits from evaluation of data from similar matrices. Criteria for development of control limits are contained in SOP DV-QA-003P, *Quality Control Program*.

### 5.6 Statistical Quality Control

Statistically-derived precision and accuracy limits are required by selected methods (such as SW-846). The laboratory routinely utilizes statistically-derived limits to evaluate method performance and determine when corrective action is appropriate. The analysts are instructed to use the current limits in the Laboratory Information Management System (LIMS). Limits are entered into the Method Limit Groups according to the effective date. Historical limits can be obtained using the "Historical" feature in the LIMS. If a method defines the QC limits, the method limits are used.

If a method requires the generation of historical limits, the laboratory 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. These are stored in the project in the LIMS and are used only for samples assigned to that project.

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

As the QC limits are calculated in the Control Chart Module in the LIMS, QC charts are generated showing warning and control limits for the purpose of evaluating trends. Refer to SOP DV-QA-003P for a description of the control chart process and evaluation of trends. The Department Managers/Supervisors evaluate these to determine if adjustments need to be made or for corrective actions to methods and submits requests for limits updates to the QA Manager. The QA manager assesses the limits to determine if they will be updated or requests corrective action to the method procedure. All findings are documented and kept on file.

### 5.7 Quality System Metrics

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

## 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 CW-Q-S-001, *Corporate Document Control and Archiving*. The laboratory's internal document control procedure is defined in SOP DV-QA-0010, *Document Control*.

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

The laboratory maintains control of records for raw analytical data and supporting records such as audit reports and responses, logbooks, standard logs, training files, MDL studies, Proficiency Testing (PT) studies, certifications and related correspondence, and corrective action reports. 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. In order to develop a new document, a department manager/supervisor submits an electronic draft to the QA Department for suggestions and approval before use. Upon approval, QA personnel add the identifying version information to the document and retain 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 every year and revised as appropriate. Changes to documents occur when a procedural change warrants.

## 6.3 Procedures for Document Control Policy

For changes to the QA Manual, and SOPs refer to SOP DV-QA-001P, *Preparation and Management of Standard Operating Procedures (SOPs) and Other Controlled Documents* and

SOP DV-QA-0010, *Document Control.* Uncontrolled copies must not be used within the laboratory. Previous revisions and back-up data are stored by the QA department. Electronic copies are stored on the Public server in the QA folder (R:\QA\Read\SOPS\ESOPS\ALL) for the current revision.

Forms, worksheets, work instructions and information are organized by department and document type in the QA office. Electronic versions are kept on the network. The procedure for the care of these documents is in SOP DV-QA-001P and SOP DV-QA-0010.

### 6.4 Obsolete Documents

All invalid or obsolete documents are removed, or otherwise prevented from unintended use. The laboratory has specific procedures as described above to accomplish this. In general, obsolete documents are collected from employees according to distribution lists and are marked obsolete on the cover or destroyed. At least one copy of the obsolete document is archived according to SOP DV-QA-0005, *Document Archiving Procedure*.

## 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 its 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 (Percent 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 client's data quality and reporting requirements and that the lab has the capacity to meet the client's 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 Client Relations Manager or Proposal Team, 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 Program.

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)

- Contract Administrator
- VP of Operations
- Laboratory Client Relations Manager
- Laboratory Project Manager
- Laboratory and/or Corporate Technical Managers / Directors
- Laboratory and/or Corporate Information Technology Managers/Directors
- Account Executives

- Laboratory and/or Corporate Quality
- Laboratory and/or Corporate Environmental Health and Safety Managers/Directors
- The Laboratory Director reviews the formal laboratory quote and makes final acceptance for their facility.

The Sales Director, Contract Administrator, Account Executive or Proposal Coordinator 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.

### 7.3 Documentation

Appropriate records are maintained for every contract or work request. All stages of the contract review process are documented and include records of any significant changes. The Contracts Department maintains copies of all signed contracts. TestAmerica Denver's Customer Service Organization maintains copies of all signed contracts for reference locally on the network. The contract will be distributed to and maintained by the appropriate sales/marketing personnel and the Account Executive. A copy of the contract and formal quote will be filed with the laboratory 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.

#### 7.3.1 Project-Specific Quality Planning

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

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

Prior to work on a new project, the dissemination of project information and/or project opening meetings may occur to discuss schedules and unique aspects of the project. Items to be discussed may include the project technical profile, turnaround times, holding times, methods, analyte lists, reporting limits, deliverables, sample hazards, or other special requirements. The PM introduces new projects to the laboratory staff through project kick-off meetings or to the supervisory staff during production meetings. These meetings provide direction to the laboratory staff in order to maximize production and client satisfaction, while maintaining quality. In addition, project notes may be associated with each sample batch as a reminder upon sample receipt and analytical processing. Unique or large programs generally have a Quality Assurance Summary prepared by the PM. This summary is posted on the public Outlook folders for anyone in the lab to access. The Quality Assurance Summary documents all requirements that are non-standard that cannot effectively be done in TALS method comments.

During the project, any change that may occur within an active project is agreed upon between the client/regulatory agency and the PM/laboratory. These changes (e.g., use of a non-standard method or modification of a method) and approvals must be documented prior to implementation. Documentation pertains to any document, e.g., letter, e-mail, variance, contract addendum, which has been signed by both parties.

Such changes are also communicated to the laboratory during production meetings and updated in the Quality Assurance Summary, when applicable. Changes are also updated in the project notes and are introduced to the managers at these meetings or via email. The laboratory staff is introduced to the modified requirements via the PM. the Technical Manager or the individual laboratory supervisor. 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". This topic is discussed in Section 7.

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

- Providing reasonable access for clients or their representatives to the relevant areas of the laboratory for the witnessing of tests performed for the client.
- Assisting client-specified third party data validators as specified in the client's contract.
- Providing 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.

The Technical Manager, department managers / supervisors and/or the QA Manager are available to discuss any technical questions or concerns that the client may have.

## 7.6 Reporting

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

**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 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 clients because project scope, changes in laboratory capabilities, capacity or unforeseen circumstances, it must be assured that the subcontractors or work sharing laboratories understand the requirements and will meet the same commitments the laboratory has made to the client. Refer to TestAmerica's Corporate SOPs on Subcontracting (CW-L-S-004) 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 TNI/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 accredited work where required.

Project Managers (PMs), Client Relations Managers (CRM), or Account Executives (AE) for the Export Lab (TestAmerica laboratory that transfers samples to another laboratory) are responsible for obtaining client approval prior to subcontracting any samples. The laboratory will advise the client of a subcontract arrangement in writing and when possible approval from the client shall be retained in the project folder. Standard TestAmerica Terms and Conditions include the flexibility to subcontract samples within the TestAmerica laboratories. Therefore, additional advance notification to clients for intra-laboratory subcontracting is not necessary unless specifically required by a client contract.

**Note:** In addition to the client, some regulating agencies (e.g., USDA) or contracts (e.g., DoD or DOE projects) require notification prior to placing such work**.**

### 8.2 Qualifying and Monitoring Subcontractors

Whenever a PM (or Account Executive (AE) or Client Relationship Manager, etc.) 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:

- Subcontractors specified by the client - In these circumstances, the client assumes

responsibility for the quality of the data generated from the use of a subcontractor.

- Subcontractors reviewed by TestAmerica – Firms which have been reviewed by the company and are known to meet standards for accreditations (e.g., State, TNI and DoD/DOE); technical specifications; legal and financial information.

A listing of vendors is available on the TestAmerica intranet site.

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). For DoD/DOE clients, the approval must be in writing. 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).

**8.2.1** 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 Client Relations Manager (CRM) or Laboratory Director. The CRM or Laboratory Director requests that the PM begin the process of approving the subcontract laboratory as outlined in Corporate SOP No. CW-L-S-004, *Subcontracting*.

Once the appropriate accreditation and legal information is received by the laboratory, it is evaluated for acceptability (where applicable) and forwarded to the Corporate Quality Information Manager (QIM) for review. After the Corporate QIM reviews the documents for completeness, the information is forwarded to the Finance Department for formal signature and contracting with the laboratory. The approved vendor will be added to the approved subcontractor list on the intranet site and the finance group is concurrently notified for JD Edwards.

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 subcontractors on our approved list can only be recommended to the extent that we would use them.

## 8.3 Oversight and Reporting

**8.3.1** The status and performance of qualified subcontractors will be monitored by the Corporate Quality department. Any problems identified will be brought to the attention of TestAmerica's Corporate Finance, Legal and 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. CSO personnel 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 CSO Personnel, Laboratory Directors, QA Managers and Sales Personnel.

Prior to initially sending samples to the subcontracted laboratory, the PM confirms their certification status to determine if it is current and scope-inclusive. The information is documented within the project records.

**8.3.2** For continued use of a subcontractor, verification of certification is placed upon the subcontractor for the defined project. Samples are subcontracted under Chain of Custody with the program defined as 'Accreditation Required' and the following statement for verification upon sample receipt:

**Note:** Since laboratory accreditations are subject to change, TestAmerica Laboratories, Inc. places the ownership of method, analyte & accreditation compliance upon our subcontract laboratories. This sample shipment is forwarded under Chain of Custody. If the laboratory does not currently maintain accreditation in the State of Origin listed above for analytes/tests/matrix being analyzed, the samples must be shipped back to the TestAmerica laboratory or other instructions will be provided. Any changes to accreditation status should be brought to TestAmerica Laboratories, Inc. attention immediately. If all requested accreditations are current to date, return the signed Chain of Custody attesting to said compliance to TestAmerica Laboratories, Inc.

For TestAmerica laboratories, certifications can be viewed on the company's TotalAccess Database.

**8.3.3** All subcontracted samples must be accompanied by a TestAmerica Chain of Custody (COC). A copy of the original COC sent by the client must be available in TALS for 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 accredited work must be identified in the subcontractor's report as appropriate. If TNI accreditation is not required, the report does not need to include this information.

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

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

### 8.4 Contingency Planning

The full qualification of a subcontractor may be waived to meet emergency needs; however, this decision and 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 COC.

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 time.

The use of any emergency subcontractor will require the PM to complete a JDE New Vendor Add Form in order to process payment to the vendor and add them to TALS. This form requires the user to define the subcontractor's category/s of testing and the reason for testing.

## 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 *Capital Expenditure, Controlled Purchase Requests and Fixed Asset Capitalization*, SOP CW-F-S-007.

Contracts will be signed in accordance with TestAmerica's *Company-Wide Authorization Matrix*, Policy 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 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 and Supplies

Purchasing guidelines for equipment, consumables, 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 Acid and Solvent

Lot Testing and Approval, SOP CA-Q-S-001 and CA-Q-S-001 DV-1, *Procedure for Testing Acetonitrile and Solvents from CYCLE-TAINERS®.* Approval information for the solvents and acids tested under SOP CA-Q-S-001 is stored on the TestAmerica SharePoint, under Solvent Approvals. A master list of all tested materials, as well as the certificates of analysis for the materials, is stored in the same location.

### 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 analyst completes the order template when requesting reagents, standards, or supplies or the analyst may check the item out of the on-site consignment system that contains items approved for laboratory use.

The analyst must provide the master item number (from the master item list that has been approved by the Technical Manager), item description, package size, catalogue page number, and the quantity needed. If an item being ordered is not the exact item requested, approval must be obtained from the Technical Manager prior to placing the order. The purchasing manager or designee places the order.

### 9.3.2 Receiving

It is the responsibility of the purchasing manager or designee to receive the shipment. It is the responsibility of the analyst who ordered the materials to document the date materials were received. Once the ordered reagents or materials are received, the analyst compares the information on the label or packaging to the original order to ensure that the purchase meets the quality level specified. This is documented through the addition of the received date and initials on the packing sheet. These are scanned and saved in the department.

The purchasing manager verifies the lot numbers of received solvents and acids against the pre-approval lists. If a received material is listed as unapproved, or is not listed, it is sequestered and returned to the vendor. Alternatively, the laboratory may test the material for the intended use, and if it is acceptable, document the approval on the approval list. Records of any testing performed locally are maintained on the shared “public” folder on the computer network.

Materials may not be released for use in the laboratory until they have been inspected, verified as suitable for use, and the inspection/verification has been documented as described in WI-DV-0098, *Acceptance of Materials Used for Testing.*

Safety Data Sheets (SDSs) are available online through the Company’s intranet website. Anyone may review these for relevant information on the safe handling and emergency precautions of on-site chemicals.

### 9.3.3 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. 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 manufacturer's or SOPs expiration date unless 'verified' as described 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 attached to the new entry for the standard in the TALS reagent module as described in SOP DV-QA-0015, *Verification and Storage of Chemical Standards and Reagents.*

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.

Water used in the preparation of standards or reagents must have a specific conductivity of less than 1- µmho/cm (or specific resistivity of greater than 1.0 megohm-cm) at 25$^{o}$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 QA Manager (or designee) must be notified immediately in order to notify all departments, decide on cessation (based on intended use) of activities, and make arrangements for correction. See SOP DV-QA-0026, *DI Water Monitoring*.

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 maintained in electronic files. These records include date of receipt, lot number (when applicable), and expiration date (when applicable). Incorporation of the item into the record indicates that the analyst has compared the new certificate with the previous one for the same purpose and that no difference is noted, unless approved and so documented by the QA Manager or designee. For all standards recorded in the Reagent Module in the LIMS, the certificate of analysis must be attached to the record for the source material. See SOP DV-QA-0015, *Verification and Storage of Chemical Standards and Reagents.*

### 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 and Safety Manual (Corp. Doc. 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 Manager and/or the Laboratory Director. If they agree with the request, the procedures outlined in TestAmerica's Corporate Policy CA-T-P-001, *Qualified Products List*, are followed. A decision is made as to which piece of equipment can best satisfy the requirements. The appropriate written requests are completed and purchasing places the order.

Upon receipt of a new or used piece of equipment, 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. The equipment's capability is assessed to determine if it is adequate or not for the specific application. For instruments, a calibration curve is generated, followed by MDLs, Demonstration of Capabilities (DOCs), and other relevant criteria (refer to Section 19). For software, its operation must be deemed reliable and evidence of instrument verification must be retained by the IT Department or QA Department. Software certificates supplied by the vendors are filed with the LIMS Administrator or Data Center Manager, as appropriate. The manufacturer's operation manual is retained at the bench or available through the laboratory's computer network.

## 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 Department Managers/Supervisors. The service providers that perform the services are approved by the Technical Manager and/or Laboratory Director.

Analytical balances are serviced and calibrated annually in accordance with SOP DV-QA-0014, *Selecting and Using Balances*. The calibration and maintenance services are performed on-

site, and the balances are returned to use immediately following successful calibration. When the calibration certificates are received (usually within two weeks of the service), they are reviewed, and documentation of the review is filed with the certificates. If the calibration was unsuccessful, the balance is immediately removed from service and segregated pending either further maintenance or disposal.

Calibration services for support equipment such as thermometers, weight sets, autopipettors, etc., are obtained from vendors with current and valid ISO 17025 accreditation for calibration of the specific piece of equipment. Prior to utilizing the vendor's services, the vendor's accreditation status is verified. Once the equipment has been calibrated, the calibration certificates are reviewed by the QA department, and documentation of the review is filed with the calibration certificates.

### 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 *Procurement and Contracts Policy* (Policy 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 Director of Technical Services 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 process to be of significant business and strategic value. Listening to and documenting client concerns captures 'client knowledge' that enables the laboratory's 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 the laboratory's business services (e.g., communications, responsiveness, data, reports, invoicing and other functions) expressed by any party, whether received verbally or in written form. Client inquiries, complaints or noted discrepancies are documented, communicated to management, and addressed promptly and thoroughly.

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

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

The process of complaint resolution and documentation utilizes the procedures outlined in Section 12 (Corrective Actions) and is documented following SOP DV-QA-013P, *Customer Complaints*.

### 10.2 External Complaints

An employee that receives a complaint initiates the complaint resolution process by first documenting the complaint according to SOP DV-QA-013P, *Customer Complaints*.

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 Quality 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 supervisor for resolution. The supervisor may elect to discuss it with the Technical Manager or have a representative contact the client to decide on a logical course of action. Once an approach is agreed upon, the analyst documents it using the laboratories corrective action system described in Section 12. This information can then be supplied to the client in the form of a footnote or a case narrative with the report. Refer to SOP DV-QA-0031, *Nonconformance and Corrective Action System* for the procedure to handle such situations.

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 Manager or Department Supervisor 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. The laboratory must also disclose if an analyte is not listed on the appropriate accreditation or certification documents for a given state when that status is available. 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

Under certain circumstances, the Laboratory Director, the Technical 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 using a Nonconformance Memo (NCM). This information may also be documented in the batch record, logbooks and/or data review checklists as appropriate. Any impacted data must be referenced in a case narrative and/or flagged with an appropriate data qualifier.

Any misrepresentation or possible misrepresentation of analytical data discovered by any laboratory staff member must be reported to facility Senior Management within 24-hours. The Senior Management staff is comprised of the Laboratory Director, the QA Manager, and the Technical Manager. The reporting of issues involving alleged violations of the company's Data Integrity or Manual Integration procedures must be conveyed to an ECO, (e.g., VP-QA/EHS) 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, and the Quality Directors have the authority and responsibility to halt work, withhold final reports, or suspend an analysis for due cause as well as authorize the resumption of work.

## 11.3 Evaluation of Significance and Actions Taken

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

Corporate SOP entitled *Data Recalls* (CW-Q-S-005) is the procedure to be followed when it is discovered that erroneous or biased data may have been reported to clients or regulatory agencies.

Corporate SOP entitled *Internal Investigation* (CW-L-S-002) is the procedure to be followed for investigation and correction of situations involved alleged incidents of misconduct or violation of the company's ethics policy.

Laboratory level decisions are documented and approved using the laboratory's standard nonconformance/corrective action reporting described in SOP DV-QA-0031 *Non-Conformance and Corrective Action System*, and SOP DV-QA-0034, *Root Cause Analysis, Corrective Actions*

*and Preventive Action Plans,* in lieu of the data recall determination form contained in TestAmerica's Corporate SOP CW-Q-S-005.

### 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 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 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 VPO 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.). 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 Manager, QA 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.

Communications to all external parties (e.g., clients) who need to be notified will be made as quickly as possible. Reports will be revised and reissued as part of the corrective action as

identified by the investigation and corrective action plan. The procedures to be used for investigation and notification are described in SOP CW-L-S-002, *Internal Investigation,* SOP CW-Q-S-005, *Data Recalls,* and SOP DV-QA-019P*, Results and Report Revisions,* as applicable.

## 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. Corrective actions are documented using Non-Conformance Memos (NCM) and 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 Non-Conformance Memo (NCM)** is used to document the following types of corrective actions:

- Deviations from an established procedure or SOP
- QC outside of limits (non-matrix related)
- Isolated reporting / calculation errors
- Client complaints
- Discrepancies in materials / goods received vs. manufacturer packing slips.

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

- Questionable trends that are found in the review of NCMs.
- Issues found while reviewing NCMs that warrant further investigation.
- Internal and external audit findings.

- Failed or unacceptable PT results.
- Corrective actions that cross multiple departments in the laboratory.
- Systematic reporting / calculation errors
- Client complaints
- Data recall investigations
- Identified poor process or method performance trends
- Excessive revised reports
- Health and Safety violations identified in audits

This 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. An NCM or Corrective Action Report (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 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 NCM or CAR is used for this documentation.

#### 12.3.3 Root Cause Analysis

Root Cause Analysis is a class of problem solving (investigative) methods aimed at identifying the basic or causal factor(s) that underlie variation in performance or the occurrence of a significant failure. The root cause may be buried under seemingly innocuous events, many steps preceding the perceived failure. At first glance, the immediate response is typically

directed at a symptom and not the cause. Typically, root cause analysis would be best with three or more incidents to triangulate a weakness. Corporate SOP CA-Q-S-009, *Root Cause Analysis* describes the procedure.

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 the 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 five 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 Technical Manager and QA Manager are responsible to ensure that the corrective action taken was effective.
- Ineffective actions are documented and re-evaluated until acceptable resolution is achieved. Technical Managers are accountable to the Laboratory Director to ensure final acceptable resolution is achieved and documented appropriately.
- Each CAR is entered into a database for tracking purposes and at least monthly a summary of all corrective actions is printed out for review to aid in ensuring that the corrective actions have taken effect.
- TestAmerica laboratories began using the Incident/Corrective Action Tracker (iCAT) database developed by the company in 2015. An incident is an event triggering the need for one or more corrective actions as distinct from a corrective action, a potential deficiency stemming from an incident that requires investigation and possibly fixing. The database is independent of TALS, available to all local and corporate managers, and capable of notifying and tracking multiple corrective actions per event, dates, and personnel. iCAT allows associated document upload, categorization (such as, external/internal audit, client service concerns, data quality issues, proficiency testing, etc.), and trend analysis. The Denver Laboratory also uses its previous audit database to provide custom reports for tracking and trending.
- The QA Manager reviews NCMs and CARs for trends. Highlights are included in the QA monthly report (refer to Section 16). If a significant trend develops that adversely affects quality, an audit of the area is performed and corrective action implemented.
- Any out-of-control situations that are not addressed acceptably at the laboratory level may be reported to the Corporate Quality Director by the QA Manager, indicating the nature of the 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 an NCM or CAR.

Table 12-1 includes examples of general technical corrective actions. For specific criteria and corrective actions, refer to the analytical methods or specific method SOPs. The laboratory may also 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 the QAM Sections 19 and 20. All corrective actions are reviewed monthly, at a minimum, by the QA Manager and highlights are included in the QA monthly report.

To the extent possible, samples shall be reported only if all quality control measures are acceptable. If the deficiency does not impair the usability of the results, data will be reported with an appropriate data qualifier and/or the deficiency will be noted in the case narrative. Where sample results may be impaired, the Project Manager is notified by an NCM and appropriate corrective action (e.g., reanalysis) is taken and documented**.**

## 12.5 Basic Corrections

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

This same process applies to adding additional information to a record. All additions made later than the initial documentation 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**

## *TestAmerica Corrective Action Plan*

*TAL Audit #* *Program:* *Requirements Document:*

*Purpose:* Not entered *Company Auditing:*

*Date Audited:* *Lead Auditor:*

*Date Report Received:* *Response Due Date:*

*TAL Issue Number* *Status:* *Title:*

*Reference Citation:* *Lab Process:* *Lab Section:*

*Client Issue #:* *Type of Issue:* *Method #:*

*Finding Description:*

*Cause Analysis:*

*Corrective Action Plan:*

*Lab Responsible Party:*

*Planned Completion Date:*

*Page 1 of 1*

**Table 12-1. Example – General Corrective Action Procedures**

| QC Activity *(Individual Responsible for Initiation/Assessment)* | Acceptance Criteria | Recommended Corrective Action |
|---|---|---|
| Initial Instrument Blank<br><br>*(Analyst, Data Reviewer)* | - Instrument response < ½ RL (or method specific criteria). | - Prepare another blank.<br>- If same response, determine cause of contamination: reagents, environment, instrument equipment failure, etc. |
| Initial Calibration Standards<br><br>*(Analyst, Data Reviewer)* | - Correlation coefficient > 0.99 or method or program requirement.<br>- % Recovery within acceptance range.<br>- See details in Method SOP. | - Reanalyze standards.<br>- If still unacceptable, remake standards and recalibrate instrument. |
| Independent Calibration Verification (Second Source)<br><br>*(Analyst, Data Reviewer)* | - % Recovery within control limits as defined in Method SOPs. | - Remake and reanalyze standard.<br>- If still unacceptable, then remake calibration standards or use new primary standards and recalibrate instrument. |
| Continuing Calibration Standards<br><br>*(Analyst, Data Reviewer)* | % Recovery within control limits as defined in Method SOPs. | - Reanalyze standard.<br>- If still unacceptable, then recalibrate and rerun affected samples. |
| Matrix Spike / Matrix Spike Duplicate (MS/MSD)<br><br>*(Analyst, Data Reviewer)* | - % Recovery within limits documented in the LIMS. | - 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.<br>- If the LCS is within acceptable limits the batch is acceptable.<br>- The results of the duplicates, matrix spikes and the LCS are reported with the data set.<br>- For matrix spike or duplicate results outside criteria the data for that sample shall be reported with qualifiers.<br><br>**NOTE:** The laboratory must eliminate lab error as the cause of the deviation. |
| Laboratory Control Sample (LCS)<br><br>*(Analyst, Data Reviewer)* | - % Recovery within limits documented in the LIMS. | - Batch must be re-prepared and re-analyzed. This includes any allowable marginal exceedance.<br>When not using marginal exceedances, the following exceptions apply:<br>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;<br>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 |

| QC Activity *(Individual Responsible for Initiation/Assessment)* | Acceptance Criteria | Recommended Corrective Action |
|---|---|---|
| | | regulatory limit/decision level with data qualifying codes.<br><br>**Note:** If there is insufficient sample or the holding time cannot be met, contact client and report with flags. |
| Surrogates<br><br>*(Analyst, Data Reviewer)* | - % Recovery within limits of method or within three standard deviations of the historical mean as documented in the LIMS. | - Individual sample must be repeated. Place comment in LIMS.<br>- Surrogate results outside criteria shall be reported with qualifiers. |
| Method Blank (MB)<br><br>*(Analyst, Data Reviewer)* | < ½ Reporting Limit | - Reanalyze blank.<br>- If still positive, determine source of contamination. If necessary, reprocess (i.e., digest or extract) entire sample batch. Report blank results.<br>- Qualify the result(s) if the concentration of a targeted analyte in the MB is at or above one-half the reporting limit AND is > 1/10 of the amount measured in the sample.<br>See SOP DV-QA-003P. |
| Proficiency Testing (PT) Samples<br><br>*(Department Manager(s) /Supervisor(s), QA Manager)* | - Criteria supplied by PT Supplier. | - Any failures or warnings must be investigated for cause. Failures may result in the need to repeat a PT sample to show the problem is corrected. |
| Internal / External Audits<br><br>*(QA Manager, Department Manager(s) /Supervisor(s)/ Technical Manager/, Laboratory Director)* | - Defined in Quality System documentation such as SOPs, QAM, etc. | - Non-conformances must be investigated through CAR system and necessary corrections must be made. |
| Reporting / Calculation Errors<br><br>(Depends on issue – possible individuals include: *Analysts, Data Reviewers, Project Managers, Technical Manager, Department Manager(s) /Supervisor(s),QA Manager, Corporate QA, Corporate Management)* | - SOP CW-Q-S-005, *Data Recalls* | - Corrective action is determined by type of error. Follow the procedures in SOP CW-L-S-002 and SOP DV-QA-019P. |
| Client Complaints<br><br>*(Project Managers, Lab Director/Manager, Sales* | -[Issue specific] | - Corrective action is determined by the type of complaint. For example, a complaint regarding an incorrect address on a report will result in the |

| QC Activity *(Individual Responsible for Initiation/Assessment)* | Acceptance Criteria | Recommended Corrective Action |
|---|---|---|
| *and Marketing)* | | report being corrected and then follow-up must be performed on the reasons the address was incorrect (e.g., database needs to be updated). See SOP DV-QA-013P. |
| QA Monthly Report<br><br>(*QA Manager, Lab Director/Technical 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<br><br>(*EHS Coordinator, Lab Director/Technical Manager, Department Manager(s) /Supervisor(s))* | - Environmental Health and Safety (EHS) Manual. | - Non-conformance is investigated and corrected through EHS procedures. |

**Note:**

1. Except as noted below for certain compounds, the method blank should be below one-half the reporting limit. Concentrations up to five times the reporting limit will be allowed for the ubiquitous laboratory and reagent contaminants: methylene chloride, acetone, 2-butanone, phthalates, sodium, zinc, and iron, **provided** they appear in similar levels in the reagent blank and samples. This allowance presumes that the reporting limit is significantly below any regulatory limit to which the data are to be compared and that blank subtraction will not occur.

## 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, the laboratory continually strives to improve customer service and client satisfaction through continuous improvements to laboratory systems.

Opportunities for improvement may be discovered through any of the following:

- review of the monthly QA Metrics Report,
- trending NCMs,

- review of control charts and QC results,
- trending proficiency testing (PT) results,
- performance of management system reviews,
- trending client complaints,
- review of processing operations, or
- staff observations.

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. The metrics report is reviewed monthly by laboratory management, Corporate QA and TestAmerica's Executive Committee. These metrics are used to evaluate the management and quality system performance on an ongoing basis and provide a tool for identifying areas for improvement.

Items identified as continuous improvement opportunities to the management system may be issued as goals from the annual management systems review, recommendations from internal audits, white papers, Lesson Learned, Technical Services audit report, Technical Best Practices, or as Corporate or management initiatives.

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 and non-conformances provides a valuable mechanism for identifying preventive action opportunities.

**13.1.1** The following elements are part of a preventive action/process improvement system:

- Identification of an opportunity for preventive action or process improvement.
- Identification of process for the preventive action or improvement.
- Definition of the measurements to assess the effectiveness of the process once undertaken.
- Execution of the preventive action or improvement.
- Evaluation of the plan using the defined measurements.
- Verification of the effectiveness of the preventive action or improvement.
- Documentation of any permanent changes to the Quality System as a result of the Preventive Action or Process Improvement to close out the process. Documentation of Preventive Action/Process improvement is incorporated into the monthly QA reports, corrective action process and management review.

**13.1.2** Any Preventive Actions/Process Improvement 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.

### 13.2 Management of Change

The Management of Change process is designed to manage significant events and changes that occur within the laboratory. Through these procedures, the potential risks inherent with a new event or change are identified and evaluated. The risks are minimized or eliminated through pre-planning and the development of preventive measures. The types of changes covered under this system include: Facility Changes, Major Accreditation Changes, Addition or Deletion to Laboratory's Capabilities or Instrumentation, Key Personnel Changes, Laboratory Information Management System (LIMS) changes. This process is discussed in further detail in SOP DV-QA-028P, *Management of Change*.

## 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. Exceptions for programs with longer retention requirements are discussed in Section 14.1.2.

### 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. More detailed information on retention of specific records is provided in CW-L-P-001, *Records Retention Policy* and CW-L-WI-001, *TestAmerica Records Retention/Storage Schedule*. Quality records are maintained by the QA department in a database or electronic files that list the contents of archived data, 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 Department Manager/Supervisor or their designee.

**Table 14-1. Record Index[1]**

| | Record Types [1]: | Retention Time: |
|---|---|---|
| **Technical Records** | - Raw Data<br>- Logbooks[2]<br>- Standards<br>- Certificates<br>- Analytical Records<br>- MDLs/IDLs/DOCs<br>- Lab Reports | 5 Years from analytical report issue* |
| **Official Documents** | - Quality Assurance Manual (QAM)<br>- Work Instructions<br>- Policies<br>- SOPs<br>- Policy Memoranda<br>- Manuals<br>- Published Methods | Indefinitely |

**Table 14-1. Record Index[1]**

| | Record Types [1]: | Retention Time: |
|---|---|---|
| **QA Records** | - Certifications<br>- Method and Software Validation / Verification Data | Indefinitely |
| **QA Records** | - Internal and External Audits/Responses<br>- Corrective/Preventive Actions<br>- Management Reviews<br>- Data Investigation | 5 Years from archival*<br><br>**Data Investigation:** 5 years or the life of the affected raw data storage whichever is greater (beyond 5 years if ongoing project or pending investigation) |
| **Project Records** | - Sample Receipt and COC Documents<br>- Contracts and Amendments<br>- Correspondence<br>- QAPP<br>- SAP<br>- Telephone Logbooks<br>- Lab Reports | 5 Years from analytical report issue* |
| **Administrative Records** | - Financial and Business Operations | Refer to CW-L-WI-001 |
| | - EH&S Manual, Permits | Indefinitely |
| | - Disposal Records | Indefinitely |
| | - Employee Handbook | Indefinitely |
| | - Personnel files, Employee Signature and Initials, Administrative Training Records (e.g., Ethics) | Refer to HR Manual |
| | - Administrative Policies | Indefinitely |
| | - Technical Training Records | 7 years |
| | - Legal Records | Indefinitely |
| | - HR Records | Refer to CW-L-WI-001 |
| | - IT Records | Refer to CW-L-WI-001 |
| | - Corporate Governance Records | Refer to CW-L-WI-001 |
| | - Sales & Marketing | 5 years |
| | - Real Estate | Indefinitely |

[1] Record Types encompass hardcopy and electronic records.
[2] Examples of Logbook types: Maintenance, Instrument Run, Preparation (standard and samples), Standard and Reagent Receipt, Archiving, Balance Calibration, Temperature (hardcopy or electronic records).
* Exceptions listed in Table 14-2.

**14.1.1** All records are stored and retained in such a way that they are secure and readily retrievable at the laboratory facility or the Iron Mountain data storage 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**.** Records archived off-site are stored in a secure location where a record is maintained of any entry into the storage facility. Whether on-site or off-site storage is used, logs are maintained in each storage box to note removal and return of records. Records are

maintained for a minimum of five years unless otherwise specified by a client or regulatory requirement.

For raw data and project records, record retention shall be calculated from the date the project report is issued. For other records, such as Controlled Documents, QA, or Administrative Records, the retention time is calculated from the date the record is formally retired. Records related to the programs listed in Table 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**

| Program | [1]Retention Requirement |
|---|---|
| Drinking Water – All States | 10 years (lab reports and raw data)<br>10 years - Radiochemistry (project records) |
| Drinking Water Lead and Copper Rule | 12 years (project records) |
| Commonwealth of MA – All environmental data 310 CMR 42.14 | 10 years |
| FIFRA – 40 CFR Part 160 | Retain for life of research or marketing permit for pesticides regulated by EPA |
| Housing and Urban Development (HUD) Environmental Lead Testing | 10 years |
| Alaska | 10 years |
| Louisiana – All | 10 years |
| Michigan Department of Environmental Quality – all environmental data | 10 years |
| Navy Facilities Engineering Service Center (NFESC) | 10 years |
| Ohio VAP | 10 years and State contacted prior to disposal |
| TSCA - 40 CFR Part 792 | 10 years after publication of final test rule or negotiated test agreement |
| OSHA | 30 years |

[1]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 are 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. In addition, refer to SOP DV-QA-025P, *Electronic Data Backup*.

**14.1.4** The record keeping system allows for historical reconstruction of all laboratory activities that produced the analytical data, as well as rapid recovery of historical data. (Records stored off site should be accessible within 2 days of a request for such records). The history of the sample from when the laboratory took possession of the samples must be readily understood through the documentation. This shall include inter-laboratory transfers of samples and/or extracts.

- The records include the identity of personnel involved in sampling, sample receipt, preparation, or testing. All analytical work contains the initials (at least) of the personnel involved. The laboratory's copy of the COC is maintained electronically with the project records. (Only the state of California requires the original COC be maintained.) 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 the project record in the LIMS. All other documents are scanned and attached to the project record in the LIMS.
- 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 per SOP DV-QA-0005, *Document Archiving Procedure).* Instrument data are 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 re-constructing an analytical sequence. Where an analysis is performed without an instrument, bound logbooks or electronic bench sheets are used to record and file data. Standard and reagent information is 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 are lost. The data files and storage media must be tested to verify the laboratory's ability to retrieve the information prior to the destruction of the hard copy that was scanned.
- Also refer to Section 19.14.1 'Computer and Electronic Data Related Requirements'**.**

## 14.2 Technical and Analytical Records

**14.2.1** The laboratory retains records of original observations, derived data and sufficient information to establish an audit trail, calibration records, staff records and a copy of each analytical report issued, for a minimum of five years unless otherwise specified by a client or regulatory requirement. The records for each analysis shall contain sufficient information to enable the analysis to be repeated under conditions as close as possible to the original. The records shall include the identity of laboratory personnel responsible for the sampling, performance of each analysis and reviewing 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, on a benchsheet or in the batch information in the LIMS.
- instrumentation identification and instrument operating conditions/parameters. Operating conditions/parameters are typically described 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**.**

**14.2.4** All logbooks used during receipt, preparation, storage, analysis, and reporting of samples or monitoring of support equipment shall undergo a documented supervisory or peer review on a bi-monthly basis.

## 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 instrument logbooks, standards logbooks, and records for data reduction, validation, storage and reporting. Logbooks are issued on a per instrument basis, and are numbered sequentially. All data are recorded sequentially within a series of sequential logbooks or directly in the LIMS

or on batch-specific bench sheets. Scanned copies of bench sheets are filed sequentially on the laboratory network or attached specifically to the batch in the LIMS. Standards are maintained in the LIMS – no logbooks are used to record that data. Records are considered archived when noted as such in the records management system (a.k.a., document control).

### 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 *Internal Auditing*, SOP CW-Q-S-003. 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<br>QA Technical Audits | Joint responsibility:<br>a) QA Manager or designee<br>b) Technical Manager or Designee<br>(Refer to CW-Q-S-003) | QA Technical Audits Frequency:<br>50% of methods annually |
| SOP Method Compliance | Joint responsibility:<br>a) QA Manager or designee<br>b) Technical Manager or Designee<br>(Refer to CW-Q-S-003) | SOP Compliance Review Frequency:<br>100% of SOPs 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 and sustainability. The audit is divided into sections for each operating or support area of the lab, and the audit is comprehensive for a given area. The 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 assess data authenticity and analyst integrity. These 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., Chrom AuditMiner) 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 Manager or qualified designee at least annually. 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 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: Nonpotable Water (WP), Soil, and Underground Storage Tank (UST).

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 the 2009 TNI standards.

### 15.3 Audit Findings

Audit findings are documented using the corrective action process and database. The laboratory’s corrective action responses for both types of audits may include action plans that could not be completed within a predefined timeframe. In these instances, a completion date must be set and agreed to by operations management and the QA Manager.

Developing and implementing corrective actions to findings is the responsibility of the Department Manager/Supervisor 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 laboratory’s 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, their Quality Director as well as the VPO. 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, VPO or Corporate QA may request that additional information be added to the report.

On a monthly basis, Corporate QA compiles information from all the monthly laboratory reports. The Corporate Quality Directors prepare a report that includes a compilation of all metrics and notable information and concerns regarding the QA programs within the laboratories. The report also includes a listing of new regulations that may potentially impact the laboratories. This report is presented to the Senior Management Team and VPs of Operations.

## 16.2 Annual Management Review

The senior lab management team (Laboratory Director, Technical Manager, and QA Manager) conducts a review annually of its quality systems and LIMS to ensure its continuing suitability and effectiveness in meeting client and regulatory requirements and to introduce any necessary changes or improvements. It will also provide a platform for defining goals, objectives and action items that feed into the laboratory planning system. Corporate Operations and Corporate QA personnel are 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 cannot be solved by the lab and report them to Corporate IT.

This management systems review (Corporate SOP CW-Q-S-004 and Work Instruction CW-Q-WI-003) 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.
- Compliance to the Ethics Policy and Data Integrity Plan. Including any evidence/incidents of inappropriate actions or vulnerabilities related to data integrity.
- Review radiation safety practices:
    - Radiation health and safety
    - Radioactive hazardous waste management
    - Radioactive materials management

A report is generated by the QA Manager and management. The report is distributed to the appropriate VPO 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 Internal Investigation SOP shall be followed (SOP 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 President and CEO, COO, VP of Client Services, VPs of Operations and Quality Directors receive a monthly report from the VP-QA/EHS summarizing any current data integrity or data recall investigations. The VPs of Operations 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, or BS) in an applied science with some chemistry in the curriculum. Exceptions can be made based upon the individual's experience and ability to learn. Selection of qualified candidates for laboratory employment begins with documentation of minimum education, training, and experience prerequisites needed to perform the prescribed task. Minimum education and training requirements for TestAmerica employees are outlined in job descriptions and are generally summarized for analytical staff in the table below.

The laboratory maintains job descriptions for all personnel who manage, perform or verify work affecting the quality of the environmental testing the laboratory performs. Job Descriptions are located on the TestAmerica intranet site's Human Resources web-page (Also see Section 4 for position descriptions/responsibilities).

Experience and specialized training are occasionally accepted in lieu of a college degree (basic lab skills such as using a balance, colony counting, aseptic or quantitation techniques, etc., are also considered).

As a general rule for analytical staff:

| Specialty | Education | Experience |
|---|---|---|
| Extractions, Digestions, some electrode methods (pH, DO, Redox, etc.), or Titrimetric and Gravimetric Analyses | H.S. Diploma | On the job training (OJT) |
| GFAA, CVAA, 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 |
| Spectral Interpretation | A college degree in an applied science or 2 years of college chemistry | and 2 years relevant experience<br>or<br>5 years of prior analytical experience |
| Department Managers/Supervisors – **General** | Bachelor's Degree in an applied science or engineering with 24 semester hours in chemistry<br><br>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 |

| Specialty | Education | Experience |
|---|---|---|
| Department Managers/Supervisors – **Wet Chem** only (no advanced instrumentation) | Associates degree in an applied science or engineering or 2 years of college with 16 semester hours in chemistry | and 2 years relevant experience |

When an analyst does not meet these requirements, they can perform a task under the direct supervision of a qualified analyst, peer reviewer or Department Manager/Supervisor, 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. Below are examples of various areas of required employee training:

| Required Training | Time Frame | Employee Type |
|---|---|---|
| Environmental Health and 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 |
| Radiation Safety | 30 days of hire | All |
| Ethics – Comprehensive Refresher | Annually | All |
| Initial Demonstration of Capability (DOC) | Prior to unsupervised method performance | Technical |

The laboratory maintains records of relevant authorization/competence, education, professional qualifications, training, skills and experience of technical personnel (including contracted personnel) as well as the date that approval/authorization was given. These records are kept on file at the laboratory. Also refer to "Demonstration of Capability" in Section 19.

The training of technical staff is kept up to date by:

- Documentation in each employee's 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).

- Evidence of annual ethics training and an Ethics Agreement signed by each staff member (renewed each year).
- A Confidentiality Agreement signed by each staff member signed at the time of employment.
- Documentation and attestation forms on employment status and records; benefit programs; timekeeping/payroll; and employee conduct (e.g., ethics violations), maintained by Human Resources in the employee's secured personnel file.

Evidence of successful training could include such items as:

- Adequate documentation of training within operational areas, including one-on-one technical training for individual technologies, and particularly for people cross-trained.
- Analysts' knowledge to refer to QA Manual for quality issues.
- Analysts following SOPs, i.e., practice matches SOPs.
- Analysts regularly communicate to supervisors and QA if SOPs need revision, rather than waiting for auditors to find problems.

Further details of the laboratory's training program are described in SOPs DV-QA-0024, *Training* and DV-QA-0037, *New Employee and On-going 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 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 54,000 $ft^2$ 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, organic sample analysis, inorganic 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 such as:

- Volatile organic chemical handling areas, including sample preparation and waste disposal, and volatile organic chemical analysis areas.

Access to and use of all areas affecting the quality of analytical testing is defined and controlled by secure access to the laboratory building as described below in the Building Security section.

Adequate measures are taken to ensure good housekeeping in the laboratory and to ensure that any contamination does not adversely affect data quality. These measures include regular cleaning to control dirt and dust within the laboratory. Work areas are available to ensure an unencumbered work area. Work areas include:

- Access and entryways to the laboratory.
- Sample receipt areas.
- Sample storage areas.
- Chemical and waste storage areas.
- Data handling and storage areas.
- Sample processing areas.
- Sample analysis areas.

### 18.4 Floor Plan

A floor plan can be found in Appendix 1.

### 18.5 Building Security

Building keys and alarm codes are distributed to employees as necessary.

Visitors to the laboratory sign in and out in a visitor's logbook. A visitor is defined as any person who visits the laboratory who is not an employee of the laboratory. In addition to signing into

the laboratory, the Environmental, Health and Safety Manual contains requirements for visitors and vendors. There are specific safety forms that must be reviewed and signed. Visitors (with the exception of company employees) are escorted by laboratory personnel at all times. Contractors may work in the building without an escort at all times as long as his/her location 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 clients' requirements and that are within the scope of the laboratory's capabilities. These include sampling, handling, transport, storage and preparation of samples, and, where appropriate, an estimation of the measurement of uncertainty as well as statistical techniques for analysis of environmental data.

Instructions are available in the laboratory for the operation of equipment as well as for the handling and preparation of samples. All instructions, Standard Operating Procedures (SOPs), reference methods and manuals relevant to the working of the laboratory are readily available to all staff. Deviations from published methods are documented (with justification) in the laboratory's approved SOPs. SOPs are submitted to clients for review at their request. Significant deviations from published methods require client approval and regulatory approval where applicable.

### 19.2 Standard Operating Procedures (SOPS)

The laboratory maintains SOPs that accurately reflect all phases of the laboratory such as assessing data integrity, corrective actions, handling customer complaints as well as all analytical methods and sampling procedures. The method SOPs are derived from the most recently promulgated/approved, published methods and are specifically adapted to the laboratory facility. Modifications or clarifications to published methods are clearly noted in the SOPs. All SOPs are controlled in the laboratory.

- All SOPs contain a revision number, effective date, and appropriate approval signatures. Controlled copies are available to all staff.
- Procedures for writing an SOP are incorporated by reference to TestAmerica's Corporate SOP entitled *Writing a Standard Operating Procedure*, CW-Q-S-002 and are detailed in the laboratory's SOP DV-QA-001P.
- SOPs are reviewed at a minimum of every year, 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

Routine analytical services are performed using standard EPA-approved methodology. In some cases, modification of standard approved methods may be necessary to provide accurate analyses of particularly complex matrices. When the use of specific methods for sample analysis is mandated through project or regulatory requirements, only those methods shall be used.

When clients do not specify the method to be used or methods are not required, the methods used will be clearly validated and documented in an SOP and available to clients and/or the end user of the data.

The analytical methods used by the laboratory are those currently accepted and approved by the U. S. EPA and the state or territory from which the samples were collected. Reference methods include:

- *Guidelines Establishing Test Procedures for the Analysis of Pollutants Under the Clean Water Act; Analysis and Sampling Procedures; 40CFR Part 136 as amended by Method Update Rule; May 18, 2012*
- *Methods for Chemical Analysis of Water and Wastes, EPA 600 (4-79-020), 1983.*
- *Methods for the Determination of Inorganic Substances in Environmental Samples, EPA-600/R-93/100, August 1993.*
- *Methods for the Determination of Metals in Environmental Samples, EPA/600/4-91/010, June 1991. Supplement I: EPA-600/R-94/111, May 1994.*
- *Methods for the Determination of Organic Compounds in Drinking Water, EPA-600/4-88-039, December 1988, Revised, July 1991, Supplement I, EPA-600-4-90-020, July 1990, Supplement II, EPA-600/R-92-129, August 1992. Supplement III EPA/600/R-95/131 - August 1995 (EPA 500 Series) (EPA 500 Series methods)*
- *Technical Notes on Drinking Water Methods, EPA-600/R94-173, October 1994*
- *Standard Methods for the Examination of Water and Wastewater, 18$^{th}$/19$^{th}$ /20$^{th}$/21$^{st}$/22$^{nd}$/ on-line edition; Eaton, A.D. Clesceri, L.S. Greenberg, A.E. Eds; American Water Works Association, Water Pollution Control Federation, American Public Health Association: Washington, D.C.*
- *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; Final Update V, August 2015.*

- *Annual Book of ASTM Standards, American Society for Testing and Materials (ASTM), Philadelphia, PA.*
- *National Status and Trends Program, National Oceanographic and Atmospheric Administration, Volume I-IV, 1985-1994.*
- *Manual for the Certification of Laboratories Analyzing Drinking Water (EPA 815-R-05-004, January 2005)*
- *Code of Federal Regulations (CFR) 40, Parts 136, 141, 172, 173, 178, 179 and 261*
- *RSK SOP-175 Revision 0, R.S. Kerr Environmental Research Laboratory; August 11, 1994.*
- *Alaska Method AK101, "For the Determination of Gasoline Range Organics", Version 04/08/02.*
- *Alaska Method AK102, "For the Determination of Diesel Range Organics", Version 04/08/02.*
- *Alaska Method AK103, "For the Determination of Residual Range Organics", Version 04/08/02.*
- *Kansas Method for the Determination of Low-range Hydrocarbons (LRH), Kansas Department of Health and Environment, Office of Laboratory Services and Bureau of Environmental Remediation, Revision 1.0, Nov. 2015.*
- *Kansas Method for the Determination of Mid-Range Hydrocarbons (MRH) and High-Range Hydrocarbons (HRH), Revision 1.0, Kansas Department of Health and Environment, Nov 2015.*
- *NWTPH-Gx, "Volatile Petroleum Products Method for Soil and Water", Manchester Environmental Laboratory, Dept. of Ecology, State of Washington.*
- *NWTPH-HCID "Hydrocarbon Identification Method for Soil and Water," Manchester Environmental Laboratory, Dept. of Ecology, State of Washington.*
- *Methods 8020/8015 (modified) Gasoline Range Organics (GRO), Oklahoma Department of Environmental Quality Revision 4.0, 02/24/96.*
- *Methods 8000/8100 (modified), Diesel Range Organics (DRO), Oklahoma Department of Environmental Quality, October 22, 1997, Rev. 4.1.*

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 (DOC), is performed whenever there is a change in instrument type (e.g., new instrumentation), matrix, method or personnel (e.g., analyst hasn't performed the test within the last twelve months) as described in SOP DV-QA-0024, *Training*.

**Note:** The laboratory shall have a DOC for all analytes included in the methods that the laboratory performs, and proficiency DOCs for each analyst shall include all analytes that the laboratory routinely performs. Addition of non-routine analytes does not require new DOCs for all analysts if those analysts are already qualified for routine analytes tested using identical chemistry and instrument conditions.

The initial demonstration of capability must be thoroughly documented and approved by the Department Manager/Supervisor and QA Manager prior to independently analyzing client samples. All associated documentation must be retained in accordance with the laboratories 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 client request is documented and the lab informs the client of its procedure for working with unusual compounds. The final report must be footnoted: *Reporting Limit based on the low standard of the calibration curve* and the case narrative must include a statement that the results for this analyte are not for compliance purposes.

### 19.4.3 Initial Demonstration of Capability (IDOC) Procedures

**19.4.3.1** Refer to SOP DV-QA-0024, *Training*.

Note: Results of successive LCS analyses can be used to fulfill the DOC requirement.

A certification statement (refer to Figure 19-1) shall be used to document the completion of each initial demonstration of capability. A copy of the certification is archived in the analyst's training folder.

## 19.5 Laboratory Developed Methods and Non-Standard Methods

Any new method developed by the laboratory must be fully defined in an SOP and validated by qualified personnel with adequate resources to perform the method. Method specifications and the relation to client requirements must be clearly conveyed to the client if the method is a non-standard method (not a published or routinely accepted method). The client must also be in agreement to the use of the non-standard method.

## 19.6 Validation of Methods

Validation is the confirmation by examination and the provision of objective evidence that the particular requirements for a specific intended use are fulfilled.

All non-standard methods, laboratory designed/developed methods, standard methods used outside of their scope, and major modifications to published methods must be validated to confirm they are fit for their intended use. The validation will be as extensive as necessary to meet the needs of the given application. The results are documented with the validation procedure used and contain a statement as to the fitness for use.

### 19.6.1 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 are 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 are to be reported in this region, it must be done 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 may 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, describing the specific differences in the new method in a revision of the existing SOP 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 and periodic 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 can be differentiated from blanks. 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. Generally, the analyst prepares at least seven replicates of solution spiked at one to five times the estimated method detection limit (most often at the lowest standard in the calibration curve) into the applicable matrix with all the analytes of interest. Each of these aliquots is extracted (including any applicable clean-up procedures) and

analyzed in the same manner as the samples. Where possible, the seven replicates should be analyzed over 2-4 days to provide a more realistic MDL. To allow for some flexibility, this low level sample may be analyzed every batch or every week or some other frequency rather than doing the study all at once. In addition, a larger number of data points may be used if the appropriate t-value multiplier is used.

Refer to the Corporate SOP CA-Q-S-006, *Detection Limits* and the laboratory's SOP DV-QA-005P, *Determination of Method Detection Limits* for details on the laboratory's MDL process.

NOTE: The LOD referenced above is the Limit of Detection per the TNI definition, equivalent to the MDL. The LOD in the DoD/DOE QSM is the spike level at which the method detection limit is verified. The latter LOD is 2-4 times the MDL (i.e., 2-4x the TNI LOD).

## 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 often 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 seven replicate spike analyses, like the MDL but without sample preparation, or by the analysis of ten instrument blanks and calculating three times the absolute value of the standard deviation.

If the IDL is greater than the MDL, it may be used as the reported MDL.

## 19.9 Verification of Detection and Reporting Limits

Once the MDL is determined, it must be verified on each instrument used for the given method. TestAmerica defines the DoD/DOE QSM Detection Limit (DL) as being equal to the MDL. TestAmerica also defines the DoD/DOE QSM Limit of Detection (LOD) as being equal to the lowest concentration standard that successfully verifies the MDL, also referred to as the MDLV standard. MDL and MDLV standards are extracted/digested and analyzed through the entire analytical process. The MDL and MDLV determinations do 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 MDLV standard is not successful, then the laboratory will redevelop their MDL or perform and pass two consecutive MDLVs at a higher concentration and set the LOD at the higher concentration. Initial and quarterly verification is required for all methods listed in the laboratory's DoD ELAP Scope of Accreditation. For methods that are not listed in this Scope of Accreditation, annual verification is performed. If an analyte is not on the laboratory's DoD ELAP accreditation, the MDL must be verified annually. Refer to the laboratory SOP DV-QA-005P, *Determination of Method Detection Limits* for further details.

The laboratory quantitation limit is equivalent to the DoD/DOE Limit of Quantitation (LOQ), which is at a concentration equal to or greater than the lowest non-zero calibration standard. The DoD/DOE QSM requires the laboratory to perform an initial characterization of the bias and precision at the LOQ and quarterly LOQ verifications thereafter. If the quarterly verification results are not consistent with three-standard deviation confidence limits established initially, then the bias and precision will be reevaluated and clients contacted for any on-going projects. For DoD/DOE projects, TestAmerica makes a distinction between the Reporting Limit (RL) and the LOQ unless the client has requested other reporting formats. The RL is a level at or above

the LOQ that is used for specific project reporting purposes, as agreed to between the laboratory and the client. The RL cannot be lower than the LOQ concentration, but may be higher. If an analyte is not on the laboratory's DoD ELAP accreditation the LOQ must be verified at least annually unless an annual MDL verification is performed.

## 19.10 Retention Time Windows

Most organic analyses and some inorganic analyses use chromatography techniques for qualitative and quantitative determinations. For every chromatography analysis each analyte will have a specific time of elution from the column to the detector. This is known as the analyte's retention time. The variance in the expected time of elution is defined as the retention time window. As the key to analyte identification in chromatography, retention time windows must be established on every column for every analyte used for that method. These records are kept with the files associated with an instrument for later quantitation of the analytes. Complete details are available in the laboratory 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": 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 uncertainties at approximately the 99% confidence level with a coverage factor of k = 3. As an example, for a reported result of 1.0 mg/L with an LCS recovery range of 50 to 150%, the estimated uncertainty in the result would be 1.0 $\pm$ 0.5 mg/L.

**19.12.5** In the case where a well-recognized test method specifies limits to the values of major sources of uncertainty of measurement (e.g., 524.2, 525, etc.) and specifies the form of presentation of calculated results, no further discussion of uncertainty is required.

## 19.13 Sample Reanalysis Guidelines

Because there is a certain level of uncertainty with any analytical measurement, a sample 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.

**Note:** Client specific Contractual Terms and 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. If no additional data are available (e.g., historical data, matrix interference, non-routine sample matrix, etc.) it may be necessary to 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/Supervisor, Technical 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 the laboratory's computer security procedures and policies are shown below. The laboratory is currently running the TestAmerica Laboratory Information Management System (TALS) which is a custom in-house developed LIMS system that has been highly customized to meet the needs of the laboratory. It is referred to as LIMS for the remainder of this section. The LIMS utilizes Sequel Server which is an industry standard relational database platform. It is referred to as Database for the remainder of this section.

**19.14.1.1 Maintain the Database Integrity:** Assurance that data are 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. More detail is provided in SOP DV-QA-0010, *Document Control.*
- 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. More detail is provided in SOP DV-QA-025P, *Electronic Data Backup.*

**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. See Policies CW-I-P-007, *Computer Systems Password Policy* and CW-I-P-001, *Internet Access and Use Policy*.

### 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 are reduced by the analyst and then verified by the Data Reviewer prior to updating the data in LIMS. This review is documented on the data review checklist. These checklists are saved electronically.

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 SOP DV-QA-011P, *Acceptable Manual Integration Practices.*

Analytical results are reduced to appropriate concentration units specified by the analytical method, taking into account factors such as dilution, sample weight or volume, etc. Blank correction will be applied only when required by the method or per manufacturer's indication; otherwise, it should not be performed. Calculations are independently verified by appropriate laboratory staff. Calculations and data reduction steps for various methods are summarized in the respective analytical SOPs or program requirements.

**19.14.2.1** All raw data must be retained in the worklist folder, computer file (if appropriate), and/or runlog. All criteria pertinent to the method must be recorded. The documentation is recorded at the time observations or calculations are made and must be signed or initialed/dated (month/day/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 (µg/L) for liquids and milligrams per kilogram (mg/kg) or micrograms per kilogram (µg/kg) for solids. For values greater than 10,000 mg/L, results can be reported in percent, i.e., 10,000 mg/L = 1**%.** Units are defined in each laboratory 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 two significant figures on the final report. Refer to SOP DV-QA-004P, *Rounding and Significant Figures* for details regarding the number of significant figures to report for each step in the process.

**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 verifies that the data were uploaded correctly. All electronic data files are 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', i.e., observations and measurements are recorded as they are made, 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 paper logbooks used in the laboratory.
- Unused portions of pages must be "Z"'d out, signed and dated.

- Worksheets and electronic forms are created with the approval of the 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 several SOPs (e.g., DV-QA-0003, *Sample Management and Chain of Custody,* DV-QA-0020, *Data Review*, and DV-QA-0022, *Data Package Assembly),* to ensure that reported data are free from calculation and transcription errors, and that QC parameters have been reviewed and evaluated before data are reported. The laboratory also has an SOP discussing Manual Integrations to ensure the authenticity of the data (DV-QA-011P, *Acceptable Manual Integration Practices*). The general review concepts are discussed below, more specific information can be found in the SOPs.

**19.14.4.1** Log-In Review - The data review process starts at the sample receipt stage. Sample control personnel review chain-of-custody forms and project instructions from the project management group. This is the basis of the sample information and analytical instructions entered into the LIMS. The log-in instructions are reviewed by the personnel entering the information, and a second level review is conducted by the project management staff.

**19.14.4.2** First Level Data Review - The next level of data review occurs with the analysts. As data are generated, analysts review their work to ensure that the results meet project and SOP requirements. First level reviews include inspection of all raw data (e.g., instrument output for continuous analyzers, chromatograms, spectra, and manual integrations), evaluation of calibration/calibration verification data in the day's analytical run, evaluation of QC data, and reliability of sample results. The analyst transfers data into LIMS, data qualifiers are verified or added as needed. All first level reviews are documented.

**19.14.4.3** Second Level Data Review – All analytical data are subject to review by a second qualified analyst or supervisor. Second level reviews include inspection of all raw data (e.g., instrument output, chromatograms, and spectra) including 100% of data associated with any changes made by the primary analyst, such as manual integrations or reassignment of peaks to different analytes, or elimination of false negative analytes. The second review also includes evaluation of initial calibration/calibration verification data in the day's analytical run, evaluation of QC data, reliability of sample results, qualifiers and NCM narratives. Manual calculations are checked in second level review. All second level reviews are documented.

Issues that deem further review include the following:

- QC data are outside the specified control limits for accuracy and precision
- Reviewed sample data do not match with reported results
- Unusual reporting 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

**19.14.4.4** 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, Technical Manager, Department Manager/Supervisor, or Quality Director for further investigation. Corrective action is initiated whenever necessary.

**19.14.4.5** The results are then entered or directly transferred into the computer database and a hard copy (or .pdf) is printed for the client.

**19.14.4.6** 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 the COC is followed, cover letters/ narratives are present, flags are appropriate, and project specific requirements are met. The Project Manager may also evaluate the validity of results for different test methods given expected chemical relationships.

**19.14.4.7** 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.

**19.14.4.8** 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 internal SOP DV-QA-011P, *Acceptable Manual Integration Practices*.

**19.14.5.1** The analyst must adjust the 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 integration is required. Analysts are encouraged to ask for assistance from a senior analyst or manager when in doubt.

**19.14.5.2** Analysts shall not increase or decrease peak areas for the sole purpose of achieving acceptable QC recoveries that would have otherwise been unacceptable. The intentional recording or reporting of incorrect information (or the intentional omission of correct information) is against company principles and policy and is grounds for immediate termination.

**19.14.5.3** Client samples, performance evaluation samples, and quality control samples are all treated equally when determining whether or not a peak area or baseline should be manually adjusted.

**19.14.5.4** All manual integrations receive a second level review. Manual integrations must be indicated on an expanded scale “after” chromatograms such that the integration performed can be easily evaluated during data review. Expanded scale “before” chromatograms are also required for all manual integrations on QC parameters (calibrations, calibration verifications, laboratory control samples, internal standards, surrogates, etc.).

**Figure 19-1. Example - Demonstration of Capability Documentation**

TestAmerica
THE LEADER IN ENVIRONMENTAL TESTING

| | |
|---|---|
| IDOC | ☐ |
| DOC | ☐ |

| | |
|---|---|
| **Analyst Name** | |
| **Date** | |
| **SOP Number** | |
| **Method** | |
| **Analysis** | |
| **Matrix** | |

We the undersigned, CERTIFY that:

1. The analyst identified above, using the cited test method with the specifications in the cited SOP, which is in use at this facility for the analysis of samples under the TestAmerica Quality Assurance Manual, has met the Initial or Ongoing Demonstration of Capability.
2. The test method was performed by the analyst identified on this certification following the TestAmerica SOP and source method.
3. A copy of the laboratory-specific SOP and source method is available for all personnel on-site. These documents have been reviewed by the analyst as part of this Demonstration of Capability.
4. The data associated with the initial/ongoing demonstration of capability are true, accurate, complete and self-explanatory (*). These data are attached to this certification statement.
5. All raw data (including a copy of this certification form) necessary to reconstruct and validate these analyses have been retained at the facility, and that the associated information is well organized and available for review by authorized inspectors.

Comments/Observations:

Analyst's Name (Print) Signature & Date

Supervisor (Print) Signature & Date

Margaret S. Sleevi
QA Manager's Name Signature & Date

* *True: Consistent with supporting data.*
*Accurate: Based on good laboratory practices consistent with sound scientific principles/practices.*
*Complete: Includes the results of all supporting performance testing.*
*Self-explanatory: Data properly labeled and stored so that the results are traceable and require no additional explanation.*

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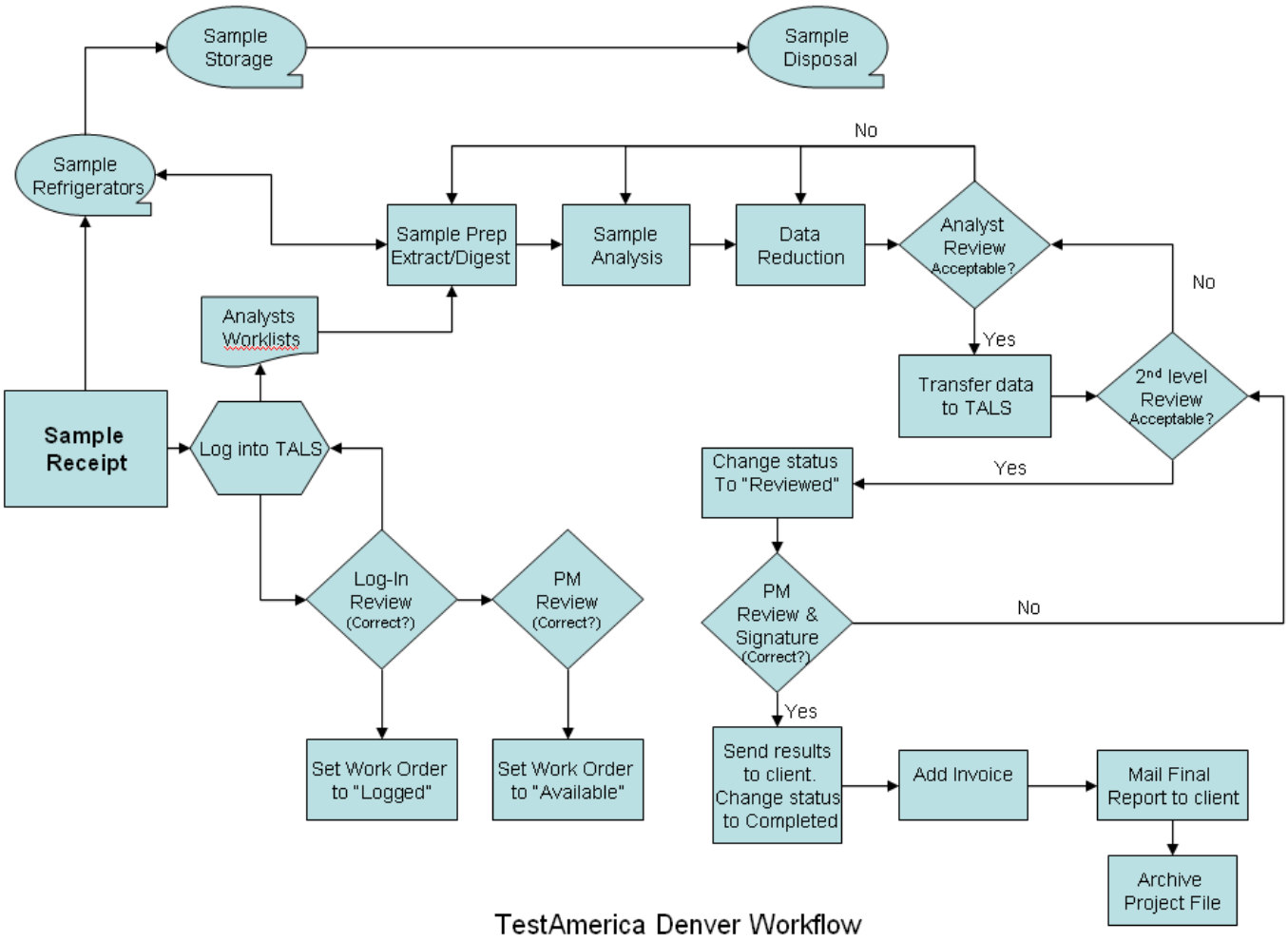
Rev 4 – 4/16/2014

**Figure 19-2. Example: Work Flow**



# SECTION 20. EQUIPMENT and CALIBRATIONS

## 20.1 Overview

Instrumentation is purchased on the basis of accuracy, dependability, efficiency and sensitivity. The laboratory is furnished with all items of sampling, preparation, analytical testing and measurement equipment necessary to correctly perform the tests for which the laboratory has capabilities. Each piece of equipment is capable of achieving the required accuracy and complies with specifications relevant to the method being performed. Before being placed into use, the equipment (including sampling equipment) is calibrated and checked to establish that it meets its intended specification. The calibration routines for analytical instruments establish the range of quantitation. Calibration procedures are specified in laboratory SOPs. A list of laboratory instrumentation and support equipment is presented in Table 20-1.

Equipment is only operated by authorized and trained personnel. Manufacturer's instructions for equipment use are readily accessible to all appropriate laboratory personnel.

## 20.2 Preventive Maintenance

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 Department Manager/Supervisor to ensure that instrument maintenance logs are kept for all equipment in his/her department. Preventative maintenance procedures are also outlined in analytical SOPs or instrument manuals. (Note: for some equipment, the log used to monitor performance is also the maintenance log.

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 taped in page must be signed across the page entered, the tape and the logbook so that it is clear that a page is missing if only half a signature is found in the logbook. Alternatively the maintenance event can be documented in the log and the location where the service receipts are stored referenced in this entry.

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.

At a minimum, if an instrument is sent out for service or transferred to/from another facility, it must be recalibrated and the laboratory MDL verified (using an MDLV) prior to return to lab operations. If equipment is transferred from another facility and the method and/ or analytes have not previously been performed the lab must complete a method validation for those analytes.

### 20.3 Support Equipment

This section applies to all devices that may not be the actual test instrument, but are necessary to support laboratory operations. These include but are not limited to: balances, ovens, refrigerators, freezers, incubators, water baths, temperature measuring devices, 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 of this information is recorded in logs, and the recalibration/recertification certificates are kept on file. Refer to SOP DV-QA-0014, *Selecting and Using Balances.*

### 20.3.2 pH, Conductivity, and Turbidity Meters

The pH meters used in the laboratory are accurate to ± 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 calibrated before each use with a known standard to demonstrate the meters do not exceed an error of 1% or one µmhos/cm. The cell constant must be verified annually.

Turbidity meters are calibrated at least monthly and the calibration is verified before each use against the three calibration standards.

All of this information is documented in logs, on bench sheets or in the batch record. Consult pH, Conductivity, and Turbidity SOPs for further information.

### 20.3.3 Thermometers

All thermometers are calibrated on an annual basis with a NIST-traceable thermometer. IR thermometers, digital probes and thermocouples are calibrated quarterly.

- If the temperature measuring device is used over a range of 10°C or less, then a single point verification within the range of use is acceptable;
- If the temperature measuring device is used over a range of greater than 10°C, then the verification must bracket the range of use.

IR Thermometers should be calibrated over the full range of use, including ambient, iced (4°C) and frozen (0°C to -5°C), per the Drinking Water Manual.

The mercury NIST thermometer is recalibrated every five years and the digital NIST thermometer is recalibrated annually (unless thermometer has been exposed to temperature extremes or apparent separation of internal liquid) by an approved outside service and the provided certificate of traceability is kept on file. The NIST thermometer(s) have increments of 1 degree, and have ranges applicable to method and certification requirements. The NIST traceable thermometer is used for no other purpose than to calibrate other thermometers.

Internal calibration records are documented in electronic spreadsheets. Certificates documenting calibration by outside vendors is maintained in files in the QA office. Monitoring method-specific temperatures, including incubators, heating blocks, water baths, and ovens, is documented in method-specific logbooks or batch records. More information on this subject can be found in SOP DV-QA-0001, *Thermometer Calibration Procedure.*

### 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 7 days a week.

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 > 0ºC and ≤ 6 ºC.

Specific temperature settings/ranges for other refrigerators, ovens, waterbaths, and incubators can be found in method specific SOPs.

All of this information is documented in Daily Temperature Logs, log tag (datalogger) downloads method-specific logbooks, or batch records. See SOP DV-QA-0012, *Monitoring Refrigerator Temperature and Power Failure Contingency Plan* or method specific SOPs.

### 20.3.5 Autopipettors, Dilutors, and Syringes

Mechanical volumetric dispensing devices (except Class A Glassware and Glass microliter syringes) are given unique identification numbers and the delivery volumes are verified gravimetrically, daily (if used).

For those dispensers that are not used for analytical measurements, a label is applied to the device stating that it is not calibrated. Any device not regularly verified cannot be used for any quantitative measurements.

Micro-syringes are purchased from Hamilton Company. Each syringe is traceable to NIST. The laboratory keeps on file an "Accuracy and Precision Statement of Conformance" from Hamilton attesting established accuracy.

Refer to SOP DV-QA-0008, *Volumetric Verification.*

## 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**.** Isotope dilution methods are calibrated initially and as needed. There is no minimum requirement for recalibration for isotope dilution methods.

### 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 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 exceptions to these rules are ICP and ICPMS methods which define the working range with periodic linear dynamic range studies, rather than through the range of concentrations of daily calibration 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). For unique situations, such as Disodium Iminodiacetate (IDA) 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. 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. The process of calibration verification applies to both external standard and internal standard calibration techniques, as well as to linear and non-linear calibration models. Initial calibration verification is with a standard source secondary (second source standard) to the calibration standards, but continuing calibration verifications may use the same source standards as the calibration curve.

**Note:** The process of calibration verification referred to 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.

**Note:** If an internal standard calibration is being used (e.g., GC or GCMS) then bracketing calibration verification standards are not required, only daily verifications are needed. The results from these verification standards must meet the calibration verification criteria and the retention time criteria (if applicable).

Generally, the initial calibrations must be verified at the beginning of each 12-hour analytical shift during which samples are analyzed. (Some methods may specify more or less frequent verifications). The 12-hour analytical shift begins with the injection of the calibration verification standard (or the MS tuning standard in MS methods). The shift ends after the completion of the analysis of the last sample, QC, or standard that can be injected within 12-hours of the beginning of the shift.

A continuing instrument calibration verification (CCV) must be repeated at the beginning and, for methods that have quantitation by external calibration models, at the end of each analytical batch. Some methods have more frequent CCV requirements see specific SOPs. Most Inorganic methods require the CCV to be analyzed after ever ten 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 and 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 and reported based upon discussion and approval of the client:

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 two 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 Percent Difference from the average CF or RF of the initial calibration or based on Percent Drift or Percent 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.

- When the acceptance criteria for the calibration verification are exceeded high, i.e., high bias, and there are associated samples that are non-detects, then those non-detects may be reported. Otherwise, the samples affected by the unacceptable calibration verification shall be reanalyzed after a new calibration curve has been established, evaluated and accepted.

- When the acceptance criteria for the calibration verification are exceeded low, i.e., low bias, those sample results may be reported if they exceed a maximum regulatory limit/decision level. Otherwise, the samples affected by the unacceptable verification shall be reanalyzed after a new calibration curve has been established, evaluated and accepted. Alternatively, a reporting limit standard may be analyzed to demonstrate that the laboratory can still support non-detects at their reporting limit.

### 20.5 Tentatively Identified Compounds (TICs) – GC/MS Analysis

For samples containing components not associated with the calibration standards, a library search may be made for the purpose of tentative identification. The necessity to perform this type of identification will be determined by the purpose of the analyses being conducted. Data system library search routines should not use normalization routines that would misrepresent the library or unknown spectra when compared to each other.

**Note:** If the TIC compound is not part of the client target analyte list but is calibrated by the laboratory and is both qualitatively and/or quantitatively identifiable, it should not be reported as a TIC. If the laboratory does not routinely analyze for this compound and may not have verified MDLs, the compound is reported as a “targeted TIC” as it is reported compared to a known standard and can be quantitatively (if verification is in control) and qualitatively measured. The result should be qualified if this is the case.

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 spectrometer, 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 and Support Equipment List**

| Instrument Type | Number |
|---|---|
| GC Semivolatiles | 14 |
| GC Volatiles | 4 |
| GC/MS Semivolatiles | 9 |
| GCMS Volatiles | 15 |
| HPLC | 4 |
| HPLC/MS/MS | 5 |
| IC/MS/MS | 2 |
| ICP | 2 |
| ICP/MS | 2 |
| HPLC/ICP/MS | 1 |
| Mercury Analyzer | 2 |
| Graphite Furnace | 1 |
| Ion Chromatograph | 7 |
| TOC Analyzer | 3 |
| TOX Analyzer | 2 |
| Autoanalyzer | 5 |
| Autotitrator | 2 |
| pH Meter | 4 |
| Conductivity Meter | 1 |
| Dissolved Oxygen Meter | 1 |
| Turbidimeter | 1 |
| Flashpoint | 1 |
| Spectrophotometer | 2 |
| Balances | 26 |
| Refrigerators & Freezers | 54 |
| Ovens | 13 |

NOTE: A complete listing of all analytical and support equipment is available from the laboratory. The QA Department maintains a Master List of Equipment.

**Table 20-2. Example: Schedule of Routine Maintenance**

| Instrument | Procedure | Frequency |
|---|---|---|
| Cetac Mercury Analyzers | • Change Lamp<br>• Clean cell and GLS as needed<br>• Check pump tubing and pump flow<br>• Check Waste Container<br>• Fill reductant bottle with 10% Stannous Chloride and check acid reagent | As needed<br>As needed<br>Daily<br>Daily<br>Daily |
| GFAA | • Check fluid level in rinse and waste containers<br>• Check condition of autosampler tubing<br>• Check the condition of graphite tube and replace as needed<br>• Check coolant level in chiller and replace as needed<br>• Check condition of Contact Rings and replace as needed<br>• Clean or replace air filters | Daily<br>Daily<br>Daily<br>Daily<br>Daily<br>As needed |
| ICP | • Check pump tubing<br>• Check fluid level in waste container<br>• Clean or replace air filters<br>• Check torch for residue<br>• Check nebulizer flow<br>• Clean nebulizer and drain chamber<br>• Fill rinse solution/ IS solution<br>• Replace capillary tubing/sipper probe<br>• Change internal cooling fluid | Daily<br>Daily<br>As needed<br>Daily<br>Daily<br>Daily<br>Daily<br>As needed<br>Quarterly |
| ICP MS | • Change pump tubing<br>• Check level of tuning solution<br>• Check waste container<br>• Load printer with paper<br>• Check air filters<br>• Replace coolant on chiller<br>• Clean or change nebulizer<br>• Clean or replace torch<br>• Replace sample tubing<br>• Change oil in vacuum pumps<br>• Remove and clean cones | Daily<br>Daily<br>Daily<br>Daily<br>As needed<br>As needed<br>As needed<br>As needed<br>As needed<br>As needed<br>As needed |
| UV-Vis Spectrophotometer | • Clean ambient flow cell<br>• Precision check/alignment of flow cell<br>• Wavelength verification check | As required<br>As required<br>Semi-annually |
| Colorimetric Analyzer | • Clean detector<br>• Clean filters<br>• Check tubing<br>• Clean sample probe shaft<br>• Clean pump, diluter, and XYZ sampler.<br>• Lubricate pump roller | Daily<br>Daily<br>Daily<br>Daily<br>Monthly<br>Semi-annually |

| Instrument | Procedure | Frequency |
|---|---|---|
| Ion Chromatograph | • Check plumbing for leaks | Daily |
| | • Check gases | Daily |
| | • Check pump pressure | Daily |
| | • Check eluent level | Daily |
| | • Check conductivity meter | Daily |
| | • De-gas pump head when flow is erratic | As needed |
| | • Change analytical columns and bed supports guard | As needed<br>As needed |
| | • Check and replace any damaged/discolored tubing | As needed<br>As needed |
| Total Organic Halide Analyzer | • Check electrodes/polish if needed | Daily |
| | • Replace dehydrating fluid /electrolyte fluid | Daily |
| | • Clean quartz boat | Daily |
| | • Perform cell performance check | Daily |
| | • At the end of each day of use, wash out the absorption module, empty the electrolyte and fill chamber with DI water, empty dehydrator tube | Daily |
| | • Clean or replace pyrolysis tube | As needed |
| | • Clean titration cell | As needed |
| | • Replace reference electrode fluid | As needed |
| | • Change quartz wool | As needed |
| | • Replace o-rings and seals | As needed |
| Hewlett Packard GC/MS | • Check Septa and clean injection port | Daily |
| | • Check carrier gas supply | Daily |
| | • Check tune parameters | Daily |
| | • Check oil levels in mechanical pumps and the diffusion pump if the vacuum is insufficient | As needed |
| | • Replace electron multiplier | As needed |
| | • Clean Source | As needed |
| | • Replace filaments | As needed |
| | • Change rough pump oil and exhaust filters | Annually |
| | • Relubricate the turbomolecular pump-bearing wick | Annually |
| Gas Chromatograph | • Check carrier gas supply | Daily |
| | • Check temperatures of inlet, detectors, verify temperature program | Daily |
| | • Check septa clean injection port or replace injection port liner and cut column if needed | As needed |
| | • Reactivate carrier gas drying agents | As needed |
| | • Replace or repair flow controllers if constant flow cannot be maintained | As needed |
| Electron Capture Detector (ECD) | • Detector wipe test (Ni-63) | Semi-annually |
| | • Detector cleaning | As needed |
| Flame Ionization Detector (FID) | • Detector cleaning | As needed |
| Nitrogen Phosphorus Detector (NPD) | • Replace bead | As needed |
| | • Replace ceramic rings | As needed |
| Photoionization Detector (PID) | • Change O-rings | As needed |
| | • Clean lamp window | As needed |

| Instrument | Procedure | Frequency |
|---|---|---|
| HPLC | • Check level of eluent vessels | Daily |
| | • Change pump seals | As needed |
| | • Change the column frit | As needed |
| | • Change fuses in power supply | As needed |
| | • Filter all samples | Daily |
| | • Change autosampler rotor or oil autosampler slides | As needed |
| | • Change or backflush columns | As needed |
| | • Replace needle | As needed |
| | • Replace needle seat assembly | As needed |
| | • Replace Active Inlet Valve (AIV) cartridge | As needed |
| | • Replace lamps | As needed |
| APCI/ESI LC/MS/MS | • Check solvent reservoirs | Daily |
| | • Verify that pump is primed and operating pulse free | Daily |
| | • Verify temperatures for capillary heater/vaporizer heater | Daily |
| | • Verify pressure of manifold/fore-pump | Daily |
| | • Verify that corona and multiplier are functional | Daily |
| | • Clean Lenses | As needed |
| | • Clean skimmer | As needed |
| | • Replace column | As needed |
| | • Oil autosampler | As needed |
| | • Change autosampler filters | As needed |
| | • Replace sample inlet tube | As needed |
| | • Replace fused silica tubing at ESI interface | As needed |
| | • Replace rough pump oil | Annually |
| | • Replace turbo pump oil | Annually |
| | • Vacuum system components including fans and fan covers | Annually |
| Balances | • Class “S” traceable weight check | Daily, when used |
| | • Clean pan and check if level | Daily |
| | • Field service | At least Annually |
| Sonicator | • Inspect probe for etching/pitting | Daily |
| | • Tune sonicator assembly | Weekly |
| | • Disassemble and clean probe tips | As needed |
| Conductivity Meter | • Standardize with KCL | Daily |
| | • Conductivity cell cleaning | As needed |
| | • Check probes and cables | As needed |
| Flash Point Tester | • Check stirrer | Daily |
| | • Check tubing | Daily |
| | • Check gas supply | Daily |
| | • Check thermometer against NIST thermometer | Quarterly by QA |
| Digestion Block | • Check with NIST thermometer | Annually |
| Turbidimeter | • Check light bulb | Daily, when used |
| | • Inspect cells | Monthly |
| | • Clean housing | Monthly |

| Instrument | Procedure | Frequency |
|---|---|---|
| Deionized/Distilled Water | • Conductivity check<br>• System cleaning<br>• Replace cartridge and large mixed bed resins | Daily<br>As needed<br>As needed |
| Drying Ovens | • Temperature monitoring<br>• Temperature adjustments | Daily<br>As required |
| Refrigerators/ Freezers | • Temperature monitoring<br>• Temperature adjustment<br>• Defrosting/cleaning | Daily<br>As required<br>As required |
| pH/Specific Ion Meter | • Calibration/check slope<br>• Clean electrode | Daily<br>As required |
| Dissolved Oxygen Meter | • Calibration/barometric pressure check<br>• Inspect probe for scratches or cracks.<br>• Change membrane | Daily<br>Daily<br>As required |
| BOD Incubator | • Temperature monitoring | Daily |
| Water baths | • Temperature monitoring<br>• Water replaced | Daily<br>Monthly or as 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, daily accuracy and precision 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), or another accreditation organization that is a signatory to a MRA (Mutual Recognition Arrangement) of one or more of the following cooperations – ILAC (International Laboratory Accreditation Cooperation) or APLAC (Asia–Pacific Laboratory Accreditation Cooperation). A calibration certificate and scope of accreditation is kept on file at the laboratory.

The calibration laboratory's policy for achieving measurement traceability is defined and includes the subsequent elements of uncertainty.

The uncertainty calculations of the calibration laboratory are supported by uncertainty budgets and are represented by expanded uncertainties typically using a coverage factor of k=2 to approximate the 95% confidence level. This explanation accompanies the measurement result and the associated uncertainty.

The tolerance uncertainty ratio (TUR) is calculated using the expanded uncertainty of the measurement, not the collective uncertainty of the measurement standards. A statement to this effect accompanies the TUR along with the coverage factor and confidence level.

The calibration report or certificate submitted to TestAmerica Denver contains, in a well-designed format, a traceability statement, the conditions under which the calibrations were made in the context of any potential influence, a compliance statement with an identified metrological specification and the pertinent clauses, a clearly identified record of the quantities and functional test results before and after re-calibration, and no recommendation on the calibration interval. Opinions and interpretations of results are presented along with the basis upon which they were made and identified as such. The report may be submitted by facsimile

or other electronic means as long as the requirements of the International Standard are achieved. If significant amendments are made to a calibration certificate, a supplemental certificate for the serial-number-specified piece of equipment is so identified. When a new certificate is offered, it uniquely identifies and references the one it replaces. All calibration reports are filed in the QA Office.

The calibration laboratory supports in-house calibration systems: documented procedures for in-house calibrations, evidence by a report, certificate, or sticker, for an appropriate amount of time; training records of calibration personnel; certificates from accreditation services demonstrating traceability to national or international standards of measurement; procedures for evaluating measurement uncertainty; timely and documented recalibration of reference standards. When subcontracting to a calibration laboratory, TestAmerica Denver does not use a firm who subcontracts the work.

An external certified service engineer services laboratory balances on an annual basis. This service is documented on each balance with a signed and dated certification sticker. Balance calibrations are checked each day of use. All mercury thermometers are calibrated annually against a traceable reference thermometer. Temperature readings of ovens, refrigerators, and incubators are checked on each day of use.

### 21.3 Reference Standards / Materials

Reference standards/materials, where commercially available, are traceable to certified reference materials. Commercially prepared reference standards, to the extent available, are purchased from vendors that are accredited to ISO Guide 34 and ISO/IEC Guide 17025. All reference standards from commercial vendors shall be accompanied with a certificate that includes at least the following information:

- Manufacturer
- Analytes or parameters calibrated
- Identification or lot number
- Calibration method
- Concentration with associated uncertainties
- Purity

If a standard cannot be purchased from a vendor that supplies a Certificate of Analysis, the purity of the standard is documented by analysis. The receipt of all reference standards must be documented. Reference standards are labeled with a unique Standard Identification Number and expiration date. All documentation received with the reference standard is retained as a QC record and references the Standard Identification Number.

All reference, primary and working standards/materials, whether commercially purchased or laboratory prepared, must be checked regularly to ensure that the variability of the standard or material from the ‘true’ value does not exceed method requirements. The accuracy of calibration standards is checked by comparison with a standard from a second source. In cases where a second standard manufacturer is not available, a vendor certified different lot is acceptable for use as a second source. For unique situations, such as Disodium Iminodiacetate (IDA) analysis where no other source or lot is available, a standard made by a different analyst would be considered a second source. The appropriate Quality Control (QC) criteria for specific

standards are defined in laboratory SOPs. In most cases, the analysis of an Initial Calibration Verification (ICV) or LCS (where there is no sample preparation) is used as the second source confirmation. These checks are generally performed as an integral part of the analysis method (e.g., calibration checks, laboratory control samples).

All standards and materials must be stored and handled according to method or manufacturer's requirements in order to prevent contamination or deterioration. Refer to the Corporate Environmental Health and Safety Manual or laboratory SOPs. For safety requirements, please refer to method SOPs and the laboratory Environmental Health and Safety Manual.

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 procedures for re-verifying expired standards are documented in SOP DV-QA-0015, *Verification and Storage of Chemical Standards and Reagents*.

## 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 Certificate of Analysis or Purity must be retained, stored appropriately, and readily available for use and inspection. These records are maintained by the analytical groups and by QA on the laboratory network or TestAmerica Intranet Oasis. Certificates of analysis for standards are also attached to the standard record in the Reagent Module in the 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 documentation and labeling, please refer to SOP DV-QA-0015, *Verification and Storage of Chemical Standards and Reagents*.

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. Blended gas standard cylinders use a nominal concentration if the certified value is within +/-15%, otherwise the certified values are used for the canister concentration.

**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 (source or intermediate)
- Standard type (spike, surrogate, other)
- Parent standard ID (if applicable)
- Parent Standard Analyte Concentration (if applicable)
- Parent Standard Amount used (if applicable)
- Component Analytes
- Final concentration of each analyte
- Comment box (text field)

Records are maintained electronically for standard and reference material preparation. These records show the traceability to purchased stocks or neat compounds. These records also include method of preparation, date of preparation, expiration date and preparer's name or initials. Preparation procedures are provided in the Method SOPs.

**21.4.2** All standards, reagents, and reference materials must be clearly labeled with a minimum of the following information:

- Expiration Date (include prep date for reagents)
- Standard ID from LIMS
- Special Health/Safety warnings if applicable

Records must also be maintained of the date of receipt and date opened 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 in the LIMS and TestAmerica Intranet Oasis.

**21.4.3** In addition, the following information may be helpful:

- Date opened (Required by DOE in QSM 5.1)
- 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 preparation/analytical batch records.

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 does not provide sampling services. The laboratory's responsibility in the sample collection process lies in supplying the sampler with the necessary coolers, reagent water, sample containers, preservatives, sample labels, custody seals, COC forms, and packing materials required to properly preserve, pack, and ship samples to the laboratory

### 22.2 Sampling Containers

The laboratory offers clean sampling containers for use by clients. These containers are obtained from reputable container manufacturers and meet EPA specifications as required. Certificates of cleanliness for the bottles and preservatives are provided by the supplier and are maintained at the laboratory or are available to the laboratory on-line.

#### 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:

- Hydrochloric Acid – Reagent ACS (Certified VOA Free) or equivalent
- Methanol – Purge and Trap grade
- Nitric Acid – Instra-Analyzed or equivalent
- Sodium Bisulfate – ACS Grade or equivalent
- Sodium Hydroxide – Instra-Analyzed or equivalent
- Sulfuric Acid – Instra-Analyzed or equivalent
- Sodium Thiosulfate – ACS Grade or equivalent

### 22.3 Definition of Holding Time

The date and time of sampling documented on the COC form establishes the day and time zero. As a general rule, when the maximum allowable holding time is expressed in "days" (e.g., 14 days, 28 days), the holding time is based on calendar day measured without regard to time zero. Holding times expressed in "hours" (e.g., 6 hours, 24 hours, etc.) are measured from date and time zero, the time sampled. Holding times for analysis include any necessary reanalysis.

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 the length of the

holding time. This must be documented as part of the project records prior to acceptance of samples.

### 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. 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 subsample or aliquot of the sample provided for analysis.

Analysts should handle each sample as if it is potentially dangerous. At a minimum, safety glasses, gloves, and lab coats must be worn when preparing aliquots for analysis.

Guidelines on taking sample aliquots and subsampling are located in SOP DV-QA-0023, *Subsampling.*

## 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.

### 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 collector's 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 independent couriers is maintained as part of the job record.

### 23.1.2 Legal / Evidentiary Chain-of-Custody

If samples are identified for legal/evidentiary purposes on the COC, Login will complete the custody seal, retain the shipping record with the COC, and initiate an internal COC for laboratory use by analysts and a sample disposal record.

## 23.2 Sample Receipt

Samples are received at the laboratory by designated sample receiving personnel and a unique laboratory project identification number is assigned. Each sample container shall be assigned a unique sample identification number that is cross-referenced to the client identification number such that traceability of test samples is unambiguous and documented. Each sample container is affixed with a durable sample identification label. Sample acceptance, receipt, tracking and

storage procedures are summarized in the following sections. Refer to SOP DV-QA-0003, *Sample Management and Chain of Custody* for detailed information on receipt of samples.

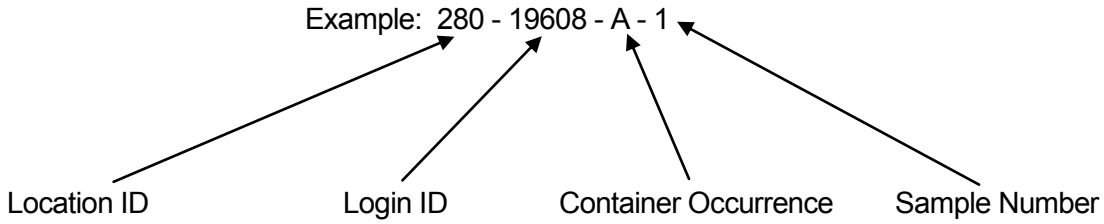
### 23.2.1 Laboratory Receipt

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 Condition Upon Receipt Anomaly Form (CUR) 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 four components):




The above example states that the laboratory is TestAmerica Denver (Location 280). Login ID is 19608 (unique to a particular client/job occurrence). The container code indicates it is the first container ("A") of Sample #1.

If the primary container goes through a prep step that creates a "new" container, then the new container is considered secondary and gets another ID. An example of this being a client sample in a 1-Liter amber bottle is sent through a Liquid/Liquid Extraction and an extraction vial is created from this step. The vial would be a SECONDARY container. The secondary ID has 5 components.

Example: 280 - 19608 - A - 1 - **A** ← **Secondary Container Occurrence**

Example: 280-19608-A-1-A, would indicate the PRIMARY container listed above went through a step that created the first occurrence of a SECONDARY container.

With this system, a client sample can literally be tracked throughout the laboratory in every step from receipt to disposal.

## 23.3 Sample Acceptance Policy

The laboratory has a written sample acceptance policy (Figure 23-2) that clearly outlines the circumstances under which samples shall be accepted or rejected. These include:

- a COC filled out completely;
- samples must be properly labeled;
- proper sample containers with adequate volume for the analysis (Sampling Guide) and necessary QC;
- samples must be preserved according to the requirements of the requested analytical method (Sampling Guide);
- sample holding times must be adhered to (Sampling Guide);
- all samples submitted for water/solid Volatile Organic analyses must have a Trip Blank submitted at the same time;
- the project manager will be notified if any sample is received in damaged condition.

Data from samples which do not meet these criteria are flagged and the nature of the variation from policy is defined.

**23.3.1** After inspecting the samples, the sample receiving personnel sign and date the COC form, make any necessary notes of the samples' conditions and store them in appropriate refrigerators or storage locations.

**23.3.2** Any deviations from these checks 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.

> Note: North Carolina requires that they be notified when samples are processed that do not meet sample acceptance criteria.

The samples are logged into the LIMS according to DV-QA-0003, *Sample Management and Chain of Custody.* Deviations from the sample acceptance criteria are noted on the Condition Upon Receipt Form (CUR) by sample receiving staff. These deviations are resolved with the client by the PM or PMA.

## 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, except metals sample containers for only ICP or ICPMS analysis which may be stored unrefrigerated. In

addition, samples to be analyzed for volatile organic parameters are stored in separate refrigerators designated for volatile organic parameters only. Samples are never to be stored with reagents, standards or materials that may create contamination.

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(s) allocated to their analysis from the designated refrigerator, place them on carts, document the transfer of containers in LIMS, analyze the sample, and return the remaining sample or empty container to the refrigerator from which it originally came, documenting the return in LIMS. Empty containers are stored in the sample archive area until disposal. This transfer is documented in LIMS. All unused samples are kept in the refrigerators until the project is invoiced. At this time, the samples will be retained for an additional thirty days, typically in the sample archive area. Special arrangements may be made to store samples for longer periods of time. This extended holding period allows additional analyses to be performed on the archived sample and assists clients in dealing with legal matters or regulatory issues. Upon disposal, the drum number used for disposal is logged into LIMS.

Access to the laboratory is controlled such that sample storage need not be locked at all times unless a project specifically demands it. Samples are accessible to laboratory personnel only. Visitors to the laboratory are prohibited from entering the refrigerator and laboratory areas unless accompanied by an employee of TestAmerica.

## 23.5 Hazardous Samples and Foreign Soils

Any sample that is received from a foreign country or from a USDA quarantine area within the United States must be sent with a copy of the laboratory's soil import permit and each cooler must have affixed a soil import permit label (Form 550) with the accompanying soil import permit number. See SOP DV-QA-0019, *Quarantine Soils Procedure.*

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, this is documented in a nonconformance memo. Analysts will notify the entire laboratory of any sample determined to be hazardous during handling or analysis by sending an email. All hazardous samples are either returned to the client or disposed of appropriately through a hazardous waste disposal firm that lab-packs all hazardous samples and removes them from the laboratory. Foreign soil samples are sent out for incineration by a USDA-approved waste disposal facility.

## 23.6 Sample Shipping

In the event that the laboratory needs to ship samples, the samples are placed in a cooler with enough ice to ensure the samples remain just above freezing and at or below 6°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. Details of the procedure for shipping samples to another location are described in SOP DV-QA-0036, *Sub-out Work Sample Management and Chain of Custody.* Samples are generally shipped overnight express or hand-delivered by a TestAmerica courier to maintain sample integrity. All personnel involved with shipping and receiving samples must be trained to maintain the proper chain-of-custody

documentation and to keep the samples intact and on ice. The Environmental, Health and Safety Manual contains additional shipping requirements.

**Note:** If a client does not request trip blank analysis on the COC or other paperwork, the laboratory will not analyze the trip blanks that were supplied. However, in the interest of good client service, the laboratory will advise the client at the time of sample receipt that it was noted that they did not request analysis of the trip blank; and that the laboratory is providing the notification to verify that they are not inadvertently omitting a key part of regulatory compliance testing.

### 23.7 Sample Disposal

Samples should be retained for a minimum of 30 days after the project report is sent, however, provisions may be made for earlier disposal of samples once the holding time is exceeded. Some samples are required to be held for longer periods based on regulatory or client requirements (e.g., 60 days after project report is sent). The laboratory must follow the longer sample retention requirements where required by regulation or client agreement. Several possibilities for sample disposal exist: the sample may be consumed completely during analysis, the sample may be returned to the customer or location of sampling for disposal, or the sample may be disposed of in accordance with the laboratory's waste disposal procedures (SOP: DV-HS-0005, *Excess Sample Material 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. Pertinent information includes the date of disposal, nature of disposal (such as sample depletion, hazardous waste facility disposal, return to client), names of individuals who conducted the arrangements and physically completed the task. The laboratory will remove or deface sample labels prior to disposal unless this is accomplished through the disposal method (e.g., samples are incinerated). A Hazardous Waste Manifest will be prepared to document the disposal of each drum. Additional detail is in SOP DV-HS-0004, *Hazardous Waste Manifesting.*

**Figure 23-1. Example: Chain of Custody (COC)**

>>> Select a Laboratory <<<
#N/A
#N/A
#N/A
#N/A

## Chain of Custody Record

TestAmerica
THE LEADER IN ENVIRONMENTAL TESTING

**TestAmerica Laboratories, Inc.**

**Regulatory Program:** ☐ DW ☐ NPDES ☐ RCRA ☐ Other:

| **Client Contact** | **Project Manager:** | **Site Contact:** | **Date:** | COC No: |
|---|---|---|---|---|
| Your Company Name here | **Tel/Fax:** | **Lab Contact:** | **Carrier:** | _____ of _____ COCs |
| Address | **Analysis Turnaround Time** | | | Sampler: |
| City/State/Zip | ☐ CALENDAR DAYS ☐ WORKING DAYS | | | **For Lab Use Only:** |
| (xxx) xxx-xxxx Phone | TAT if different from Below _____ | | | Walk-in Client: |
| (xxx) xxx-xxxx FAX | ☐ 2 weeks | | | Lab Sampling: |
| Project Name: | ☐ 1 week | | | |
| Site: | ☐ 2 days | | | Job / SDG No.: |
| P O # | ☐ 1 day | | | |

| Sample Identification | Sample Date | Sample Time | Sample Type (C=Comp, G=Grab) | Matrix | # of Cont. | Filtered Sample (Y / N) | Perform MS / MSD (Y / N) | | | | | | | | | | | | | | | Sample Specific Notes: |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
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**Preservation Used: 1= Ice, 2= HCl; 3= $H_2SO_4$; 4=$HNO_3$; 5=NaOH; 6= Other _____**

**Possible Hazard Identification:**
Are any samples from a listed EPA Hazardous Waste? Please List any EPA Waste Codes for the sample in the Comments Section if the lab is to dispose of the sample.

☐ Non-Hazard ☐ Flammable ☐ Skin Irritant ☐ Poison B ☐ Unknown

**Sample Disposal ( A fee may be assessed if samples are retained longer than 1 month)**

☐ Return to Client ☐ Disposal by Lab ☐ Archive for _____ Months

**Special Instructions/QC Requirements & Comments:**

Custody Seals Intact: ☐ Yes ☐ No | Custody Seal No.: | Cooler Temp. (°C): Obs'd: _____ Corr'd: _____ Therm ID No.: _____

| Relinquished by: | Company: | Date/Time: | Received by: | Company: | Date/Time: |
|---|---|---|---|---|---|
| Relinquished by: | Company: | Date/Time: | Received by: | Company: | Date/Time: |
| Relinquished by: | Company: | Date/Time: | Received in Laboratory by: | Company: | Date/Time: |

**Form No. CA-C-WI-002, Rev. 4.4, dated 07/15/2014**

**Figure 23-2. Example: Sample Acceptance Policy**

All incoming work will be evaluated against the criteria listed below. Where applicable, data from any samples that do not meet the criteria listed below will be noted on the laboratory report defining the nature and substance of the variation. In addition the client will be notified either by telephone, fax or e-mail as soon as possible after the receipt of the samples.

Per State and/or Federal Regulation, the client is responsible to ensure that samples are shipped in accordance with DOT/IATA requirements, and that radioactive materials may only be delivered to licensed facilities. Any samples containing (or suspected to contain) Source, Byproduct, or Special Nuclear Material as defined by 10 CFR should be delivered directly to facilities licensed to handle such radioactive material. Natural material or ores containing naturally occurring radionuclides may be delivered to any TestAmerica facility or courier as long as the activity concentration of the material does not exceed 270 pCi/g alpha or 2700 pCi/g beta (49 CFR Part 173).

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 collector's name*
   - *The matrix description*
   - *The container description*
   - *The total number of each type of container*
   - *Preservatives used*
   - *Analysis requested*
   - *Requested turnaround time (TAT)*
   - *Any special instructions*
   - *Purchase Order number or billing information (e.g., quote number) if available*
   - *The date and time that each person received or relinquished the sample(s), including their signed name.*
   - *The date and time of receipt must be recorded between the last person to relinquish the samples and the person who receives the samples in the lab, and they must be exactly the same.*
   - Information must be legible
2) Samples must be properly labeled.
   - Use durable labels (labels provided by TestAmerica are preferred)
   - Include a unique identification number
   - Include sampling date and time and sampler ID
   - Include preservative used.
   - Use indelible ink
   - Information must be legible
3) Proper sample containers with adequate volume for the analysis and necessary QC are required for each analysis requested. See Lab Sampling Guide.
4) Samples must be preserved according to the requirements of the requested analytical method (See Sampling Guide**.**
5) Most analytical methods require chilling samples to $4^{o}$ C (other than water samples for metals analysis). For these methods, the criteria are met if the samples are chilled to below $6^{o}$ C and above freezing ($0^{o}$C). For methods with other temperature criteria (e.g., some bacteriological methods require $\leq$ 10 $^{o}$C), the samples must arrive within $\pm$ $2^{o}$ C of the required temperature or within the method specified range.

5i.) Samples that are delivered to the laboratory on the same day they are collected may not meet the requirements of Section 5. In these cases, the samples shall be considered acceptable if the samples were received on ice.

5ii.) If sample analysis is begun within fifteen (15) minutes of collection, thermal preservation is not required.

5iii.) Thermal preservation is not required in the field if the laboratory receives and refrigerates the sample within fifteen (15) minutes of collection.

- Chemical preservation (pH) will be verified prior to analysis and documented, either in sample control or by the analyst. The project manager will be notified immediately if there is a discrepancy. If analyses will still be performed, all affected results will be flagged to indicate improper preservation.
- For Volatile Organic analyses in drinking water (Methods 502.2 or 524.2) residual chlorine must be neutralized prior to preservation. If there is prior knowledge that the samples are not chlorinated, state it on the COC and use the VOA vials pre-preserved with HCl. The following are other options for a sampler and laboratory where the presence of chlorine is not known:
  - 1. Test for residual chlorine in the field prior to sampling.
    - If no chlorine is present, the samples are to be preserved using HCl as usual.
    - If chlorine is present, add either ascorbic acid or sodium thiosulfate prior to adding HCl.
  - 2. Use VOA vials pre-preserved with sodium thiosulfate or ascorbic acid and add HCl after filling the VOA vial with the sample.
- **FOR WATER SAMPLES TESTED FOR CYANIDE (by Standard Methods or EPA 335)**
  - In the Field: Samples are to be tested for Sulfide using lead acetate paper prior to the addition of Sodium Hydroxide (NaOH). If sulfide is present, the sample must be treated with Cadmium Chloride and filtered prior to the addition of NaOH.
    - If the sulfide test and treatment is not performed in the field, the lab will test the samples for sulfide using lead acetate paper at the time of receipt and if sulfide is present in the sample, the client will be notified and given the option of retaking the sample and treating in the field per the method requirements or the laboratory can analyze the samples as delivered and qualify the results in the final report.
  - It is the responsibility of the client to notify the laboratory if thiosulfate, sulfite, or thiocyanate are known or suspected to be present in the sample. This notification may be on the chain of custody. The samples may need to be subcontracted to a laboratory that performs a UV digestion. If the lab does not perform the UV digestion on samples that contain these compounds, the results must be qualified in the final report.
  - The laboratory must test the sample for oxidizing agents (e.g., chlorine) prior to analysis and treat according to the methods prior to distillation. (Ascorbic acid or sodium arsenite are the preferred choice.)

6) Sample Holding Times

- TestAmerica will make every effort to analyze samples within the regulatory holding time. Samples must be received in the laboratory with enough time to perform the sample analysis. Except for short holding time samples (< 48 hr HT) sample must be received with at least 48 hr (working days) remaining on the holding time for the laboratory to ensure analysis.
- Analyses that are designated as “field” analyses (Odor, pH, Dissolved Oxygen, Disinfectant Residual; a.k.a. Residual Chlorine, and Redox Potential) should be analyzed ASAP by the field sampler prior to delivering to the lab (within 15 minutes). However, if the analyses are to be performed in the laboratory, TestAmerica will make every effort to analyze the samples within 24 hours from receipt of the samples in the testing laboratory. Samples for “field” analyses received

after 4:00 pm on Friday or on the weekend will be analyzed no later than the next business day after receipt (Monday unless a holiday). Samples will remain refrigerated and sealed until the time of analysis. The actual times of all "field" sample analyses are noted on the "Short Hold Time Detail Report" in the final report. Samples analyzed in the laboratory will be qualified on the final report with an 'H' to indicate holding time exceedance.

7) All samples submitted for Volatile Organic analyses must have a Trip Blank submitted at the same time. TestAmerica will supply a blank with the bottle order.

8) The project manager will be notified if any sample is received in damaged condition. TestAmerica will request that a sample be resubmitted for analysis.

9) Recommendations for packing samples for shipment:

   - Pack samples in Ice rather than "Blue" ice packs.
   - Soil samples should be placed in plastic zip-lock bags. The containers often have dirt around the top and do not seal very well and are prone to intrusion from the water from melted ice.
   - Water samples would be best if wrapped with bubble-wrap or paper (newspaper, or paper towels work) and then placed in plastic zip-lock bags.
   - Fill extra cooler space with bubble wrap.

## Figure 23-3. Example: Cooler Receipt Form

*TestAmerica Denver* **Sample Receiving Checklist**
**DV-QA-0003**

Login #:_____ Date/Time Received:_____

Company Name & Sampling Site: _____

Time Zone: • EDT/EST • CDT/CST • MDT/MST • PDT/PST • OTHER State:

Document any problems or discrepancies and the actions taken to resolve them on a Condition Upon Receipt Anomaly Report (CUR)

Temp_____ IR#_____ Temp_____ IR#_____ Temp_____ IR#_____ Temp_____ IR#_____

CF_____Initials_____ CF_____Initials_____ CF_____Initials_____ CF_____Initials_____

Date_____ Date_____ Date_____ Date_____

| N/A | Yes | No | | ***Initials*** _____ |
|---|---|---|---|---|
| ☐ | ☐ | ☐ | 1. | Is radioactivity at or below background? BKG CPM:_____ CPM Reading: _____ |
| ☐ | ☐ | ☐ | 2a. | Is a custody seal present on the cooler? |
| ☐ | ☐ | ☐ | 2b. | If yes, is the cooler's custody seal intact? |
| ☐ | ☐ | ☐ | 2c. | Do cooler or samples appear to not have been compromised or tampered with? |
| ☐ | ☐ | ☐ | 3a. | Were samples received on ice? |
| ☐ | ☐ | ☐ | 3b. | Is cooler temperature acceptable? |
| ☐ | ☐ | ☐ | 3c. | Has temperature been recorded? |
| ☐ | ☐ | ☐ | 4. | Is COC present; filled out in ink and legible; and filled out with all pertinent information? |
| ☐ | ☐ | ☐ | 5. | Is the Field Sampler's name present on the COC? |
| ☐ | ☐ | ☐ | 6a. | Are there no discrepancies between the **sample IDs** and/or **collection date and time** on the containers and the COC? |
| ☐ | ☐ | ☐ | 6b. | Are there no discrepancies between the container types and those listed on the COC? |
| ☐ | ☐ | ☐ | 7. | Are samples received within Holding Time? |
| ☐ | ☐ | ☐ | 8. | Do sample containers have legible labels? |
| ☐ | ☐ | ☐ | 9. | Are all sample containers intact (not broken or leaking)? |
| ☐ | ☐ | ☐ | 10a. | Are appropriate sample containers used? |
| ☐ | ☐ | ☐ | 10b. | Are sample bottles completely filled? (Perchlorate bottles ≥ 1/3 head space) |
| ☐ | ☐ | ☐ | 10c. | Is sufficient vol. for all requested analyses, incl. any requested MS/MSDs provided? |
| ☐ | ☐ | ☐ | 11. | No splitting or compositing of samples required? |
| ☐ | ☐ | ☐ | 12. | Do all VOA sample vials have no headspace or bubbles >6 mm (1/4") in diameter? |
| ☐ | ☐ | ☐ | 13. | Were VOA vials labeled as preserved? ☐ HCl ☐ 0-6°C ☐ Sodium Thiosulfate ☐ Ascorbic Acid ☐ Other |
| ☐ | ☐ | ☐ | 14. | Are all samples single phase? (i.e., no multiphasic samples are present.) |

**Login Checks:** ***Initials*** _____

| | | | | |
|---|---|---|---|---|
| ☐ | ☐ | ☐ | 15. | Was a Priority Form completed for any short holds or quick TATs? |
| ☐ | ☐ | ☐ | 16. | Were any tests logged for subcontract? |
| ☐ | ☐ | ☐ | 17. | Were special archiving instructions and login instructions indicated in the Project Notes? |

Note Archive Requirements: _____

| | | | | |
|---|---|---|---|---|
| ☐ | ☐ | ☐ | 18. | Were multiple Series logged for this job? |

**Labeling and Storage Checks:** ***Initials*** _____

pH Checks Required? ☐ Yes ☐ No Residual chlorine check required: ☐ Yes ☐ No Quarantined: ☐ Yes ☐ No

| | | | | |
|---|---|---|---|---|
| ☐ | ☐ | ☐ | 19. | Was Sample Preservation verified and found to be correct? (excluding VOA, Oil & Grease, and TOC volumes) |
| ☐ | ☐ | ☐ | 20. | Was Residual Chlorine checked and noted on the CUR if present? |
| ☐ | ☐ | ☐ | 21. | If subcontract work was requested, was volume placed on sub shelf? |
| ☐ | ☐ | ☐ | 22. | Were Terracore/Encores delivered to VOA lab? |
| ☐ | ☐ | ☐ | 23. | Did the sample ID on TA label match the client's sample ID on container? |
| ☐ | ☐ | ☐ | 24. | Were stickers for special archiving instructions affixed to each box? |

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# SECTION 24. ASSURING THE QUALITY OF TEST RESULTS

## 24.1 Overview

In order to assure clients of the validity of their data, the laboratory continuously evaluates the quality of the analytical process. The analytical process is controlled not only by instrument calibration as discussed in Section 20, but also by routine process quality control measurements (e.g., Blanks, Laboratory Control Samples (LCS), Matrix Spikes (MS), duplicates (DUP), surrogates, Internal Standards (IS)). These quality control checks are performed as required by the method or regulations to assess precision and accuracy. Quality control samples are to be treated in the exact same manner as the associated field samples being tested. In addition to the routine process quality control samples, Proficiency Testing (PT) Samples (concentrations unknown to laboratory) are analyzed to help ensure laboratory performance.

## 24.2 Controls

Sample preparation or pre-treatment is commonly required before analysis. Typical preparation steps include homogenization, grinding, solvent extraction, sonication, acid digestion, distillation, reflux, evaporation, drying and ashing. During these pre-treatment steps, samples are arranged into discreet manageable groups referred to as preparation (prep) batches. Prep batches provide a means to control variability in sample treatment. Control samples are added to each prep batch to monitor method performance and are processed through the entire analytical procedure with investigative/field samples.

## 24.3 Negative Controls

**Table 24-1. Example – Negative Controls**

| Control Type | Details |
|---|---|
| Method Blank (MB) | Used to assess preparation and analysis for possible contamination during the preparation and processing steps. |
| | The specific frequency of use for method blanks during the analytical sequence is defined in the specific standard operating procedure for each analysis. Generally it is one for each batch of samples; not to exceed 20 environmental samples. |
| | The method blank is prepared from a clean matrix similar to that of the associated samples that is free from target analytes (e.g., Reagent water, Ottawa sand, glass beads, etc.) and is processed along with and under the same conditions as the associated samples.<br><br>The method blank goes through all of the steps of the process (including, as necessary: filtration, clean-ups, etc.). |
| | Reanalyze or qualify associated sample results when the concentration of a targeted analyte in the blank is at or above one-half the reporting limit as established by the method or by regulation, AND is greater than 1/10 of the amount measured in the sample. For some wet chemistry methods, the allowable blank may contain up to the reporting limit, as defined in the method SOP. |
| Calibration Blanks | 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 | Blank reagents or reagent water that may be processed during an analytical sequence in order to assess contamination in the analytical system. In general, instrument blanks are used to differentiate between contamination caused by the analytical system and that caused by the sample handling or sample prep process. Instrument blanks may also be inserted throughout the analytical sequence to minimize the effect of carryover from samples with high analyte content. |

**Table 24-1. Example – Negative Controls**

| Control Type | Details |
|---|---|
| Trip Blank [1] | 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 free of any volatile compounds. Appropriate preservatives are also added to the container. The trip blank is sent with the bottle order and is intended to reflect the environment that the containers are subjected to throughout shipping and handling and help identify possible sources if contamination is found. The field sampler returns the trip blank in the cooler with the field samples. |
| Field Blanks [1] | Sometimes used for specific projects by the field samplers. A field blank prepared in the field by filling a clean container with pure reagent water and appropriate preservative, if any, for the specific sampling activity being undertaken. (EPA OSWER) |
| Equipment Blanks [1] | 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) |
| 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. |

[1] When known, these field QC samples should not be selected for matrix QC as it does not provide information on the behavior of the target compounds in the field samples. Usually, the client sample ID will provide information to identify the field blanks with labels such as "FB", "EB", or "TB."

Evaluation criteria and corrective action for these controls are defined in the specific standard operating procedure for each analysis.

## 24.4 Positive Controls

Control samples (e.g., QC indicators) are analyzed with each batch of samples to evaluate data based upon (1) Method Performance (Laboratory Control Sample (LCS) or Blank Spike (BS)), which entails both the preparation and measurement steps; and (2) Matrix Effects (Matrix Spike (MS) (Matrix spikes are not applicable to air) or Sample Duplicate (MD, DUP), which evaluates field sampling accuracy, precision, representativeness, interferences, and the effect of the matrix on the method performed. Each regulatory program and each method within those programs specify the control samples that are prepared and/or analyzed with a specific batch.

Note that frequency of control samples vary with specific regulatory, methodology and project specific criteria. Complete details on method control samples are as listed in each analytical SOP.

### 24.4.1 Method Performance Control - Laboratory Control Sample (LCS)

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.

The LCS is prepared from a clean matrix similar to that of the associated samples that is free from target analytes (for example: reagent water, Ottawa sand, glass beads, etc.) and is processed along with and under the same conditions as the associated samples. The LCS is spiked with verified known amounts of analytes or is made of a material containing known and verified amounts of analytes, taken through all preparation and analysis steps along with the field samples. Where there is no preparation taken for an analysis (such as in aqueous volatiles), or when all samples and standards undergo the same preparation and analysis process (such as Phosphorus), a calibration verification standard is reported as the LCS. In some instances where there is no practical clean solid matrix available, aqueous LCS's may be

processed for solid matrices; final results may be calculated as mg/kg or µg/kg, assuming 100% solids and a weight equivalent to the aliquot used for the corresponding field samples, to facilitate comparison with the field samples.

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.).

The specific frequency of use for LCS during the analytical sequence is defined in the specific standard operating procedure for each analysis. It is generally one for each batch of samples; not to exceed 20 environmental samples.

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. 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 as they cover the range of all of the aroclors. Specific aroclors may be used by request on a project specific basis.

## 24.5 Sample Matrix Controls

**Table 24-2. Sample Matrix Control**

| Control Type | Details | |
|---|---|---|
| Matrix Spikes (MS) | Use | 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 [1] | 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 the same set of compounds in both the Laboratory Control Sample and Matrix Spike. Refer to the method SOP for complete details. |
| | Description | A sample fortified with a known amount of the test analyte(s). |
| Surrogate | Use | Measures method performance to sample matrix (organics only). |

**Table 24-2. Sample Matrix Control**

| Control Type | Details | |
|---|---|---|
| | Typical Frequency [1] | 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 | 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[2] | Use | As 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 [1] | 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 | 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 [1] | 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. |

[1] See the specific analytical SOP for type and frequency of sample matrix control samples.
[2] LCSD's are normally not performed except when regulatory agencies or client specifications require them. The recoveries for the spiked duplicate samples must meet the same laboratory established recovery limits as the accuracy QC samples. If an LCSD is analyzed both the LCS and LCSD must meet the same recovery criteria and be included in the final report. The precision measurement is reported as "Relative Percent Difference" (RPD). Poor precision between duplicates (except LCS/LCSD) may indicate non-homogeneous matrix or sampling.

## 24.6 Acceptance Criteria (Control Limits)

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 or program requires more frequent updating. 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 ± 3 standard deviations (99% confidence level) from the average recovery of a minimum of 20-30 data points (more points are preferred).

- 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 minimum RPD limit is 10%.
- If either the high or low end of the control limit changes by $\leq$ 5% from previous, the control chart is visually inspected and, using professional judgment, they may be left unchanged if there is no affect on laboratory ability to meet the existing limits.

**24.6.1** 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 DV-QA-003P, *Quality Control Program* for a detailed description of the control charting procedure.

**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.
- The analytical results are above the relevant regulatory limit and the LCS is below the lower control limit.

For TNI and DoD/DOE work, there are an allowable number of Marginal Exceedances (ME):

| | |
|---|---|
| <11 analytes | 0 marginal exceedances are allowed. |
| 11 – 30 Analytes | 1 marginal exceedance is allowed |
| 31-50 Analytes | 2 marginal exceedances are allowed |
| 51-70 Analytes | 3 marginal exceedances are allowed |
| 71-90 Analytes | 4 marginal exceedances are allowed |
| > 90 Analytes | 5 marginal exceedances are allowed |

- 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. For any 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. For any project that does not allow marginal exceedances this requirement must be communicated to the laboratory.

**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

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).

A discussion regarding MDLs, Limit of Detection (LOD) and Limit of Quantitation (LOQ) can be found in Section 19.

- Use of formulae to reduce data is discussed in the method SOPs and in Section 20.
- Selection of appropriate reagents and standards is included in Section 9 and 21.
- A discussion on selectivity of the test is included in Section 5.
- Constant and consistent test conditions are discussed in Section 18.
- The laboratories sample acceptance policy is included in Section 23.

## SECTION 25. REPORTING RESULTS

### 25.2 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.

### 25.3 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, containing the laboratory name on the cover page, is reviewed, and signed by the appropriate project manager (or designee). At a minimum, the standard laboratory report shall contain the following information:

**25.3.1** A report title (e.g., Analytical Report For Samples) with a "sample results" column header.

**25.3.2** Each report cover page includes the laboratory name, address and telephone number.

**25.3.3** A unique identification of the report (e.g., job number) and on each page an identification in order to ensure the page is recognized as part of the report and a clear identification of the end.

**Note:** Page numbers of the report are represented as page # of ##. Where the first number is the page number and the second is the total number of pages.

**25.3.4** A copy of the chain of custody (COC) and any COCs involved with Subcontracting are included.

**25.3.5** The name and address of client and a project name/number, if applicable.

**25.3.6** Client project manager or other contact

**25.3.7** Description and unambiguous identification of the tested sample(s) including the client identification code.

**25.3.8** 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.

**25.3.9** Date reported or date of revision, if applicable.

**25.3.10** Method of analysis including method code (EPA, Standard Methods, etc.).

**25.3.11** Reporting limit.

**25.3.12** Method detection limits (if requested)

**25.3.13** Definition of Data qualifiers and reporting acronyms (e.g., ND).

**25.3.14** Sample results.

**25.3.15** QC data consisting of method blank, surrogate, LCS, and MS/MSD recoveries and control limits.

**25.3.16** Condition of samples at receipt including temperature. This may be accomplished in a narrative or by attaching sample login sheets.

**25.3.17** A statement expressing the validity of the results, that the source methodology was followed and all results were reviewed for error.

**25.3.18** A statement to the effect that the results relate only to the items tested and the sample as received by the laboratory.

**25.3.19** A statement that the report shall not be reproduced except in full, without prior express written approval by the laboratory.

**25.3.20** A signature and title of the person(s) accepting responsibility for the content of the report and date of issue. Authorized signatories are qualified Project Managers appointed by the Manager of Project Managers.

**25.3.21** 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.

**25.3.22** Where applicable, a narrative to the report that explains the issue(s) and corrective action(s) taken in the event that a specific accreditation or certification requirement was not met.

**25.3.23** When soil samples are analyzed, a specific identification as to whether soils are reported on a “wet weight” or “dry weight” basis.

**25.3.24** Appropriate laboratory certification number for the state of origin of the sample, if applicable.

**25.3.25** If only part of the report is provided to the client (client requests some results before all of it is complete), it must be clearly indicated on the report (e.g., partial report or preliminary report). A complete report must be sent once all of the work has been completed.

**25.3.26** Any non-TestAmerica subcontracted analysis results are provided as a separate report on the official letterhead of the subcontractor. All TestAmerica subcontracting is clearly identified on the report as to which laboratory performed a specific analysis.

**25.3.27** A Certification Summary Report, where required, will document that, unless otherwise noted, all analytes tested and reported by the laboratory were covered by the noted certifications.

**Note:** Refer to Corporate SOP CA-I-P-002, *Electronic Reporting and Signature Policy*, for details on internally applying electronic signatures of approval.

## 25.4 Reporting Level or Report Type

The laboratory offers four levels of quality control reporting. Each level, in addition to its own specific requirements, contains all the information provided in the preceding level. The packages provide the following information in addition to the information described above:

- Level I is a report with the elements described in Section 25.2 above, excluding 25.2.15 (QC Data)

- Level II is a Level I report plus summary information, including results for the method blank, percent recovery for laboratory control samples and matrix spike samples, and the RPD values for all MSD and sample duplicate analyses.
- Level III contains all the information supplied in Level II, but presented on the CLP-like summary forms, and relevant calibration information. A Level II report is not included, unless specifically requested. No raw data are provided.
- Level IV is the same as Level III with the addition of all raw supporting data.

In addition to the various levels of QC packaging, the laboratory also provides reports in diskette deliverable form. Initial (preliminary) reports may be provided to clients by facsimile. Procedures used to ensure client confidentiality are outlined in Section 25.6.

### 25.4.1 Electronic Data Deliverables (EDDs)

EDDs are routinely offered as part of TestAmerica's services. The Denver laboratory offers a variety of EDD formats including Environmental Restoration Information Management System (ERPIMS), New Agency Standard (NAS), Format A, Excel, Dbase, GISKEY, SEDD2A, and Text Files.

EDD specifications are submitted to the IT department by the PM for review and undergo the contract review process. Once the facility has committed to providing data in a specific electronic format, the coding of the format may need to be performed. This coding is documented and validated. The validation of the code is retained by the IT staff coding the EDD.

EDDs shall be subject to a review to ensure their accuracy and completeness. If EDD generation is automated, review may be reduced to periodic screening if the laboratory can demonstrate that it can routinely generate that EDD without errors. Any revisions to the EDD format must be reviewed until it is demonstrated that it can routinely be generated without errors. If the EDD can be reproduced accurately and if all subsequent EDDs can be produced error-free, each EDD does not necessarily require a review.

## 25.5 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.

## 25.6 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 CW-L-S-004).

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.

## 25.7 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.

**25.7.1** Report deliverable formats are discussed with each new client. If a client requests that reports be faxed or e-mailed, the reports are to meet all requirements of this document and include a cover letter.

## 25.8 Format of Reports

The format of reports is designed to accommodate each type of environmental test carried out and to minimize the possibility of misunderstanding or misuse.

## 25.9 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 job number followed by "Rev#". The revised report will have the word "revised" or "amended" next to the date rather than the word "reported".

When the report is re-issued, a notation of "Revision #" is placed on the cover/signature page of the report. The revision history, revision number and date, is listed in the narrative with a brief explanation of reason for the re-issue. *For Example*: *Revision 1: June 19, 2014 This revision was necessary to change the 8270 SVOC analyte bis(2-Chloroisopropyl)ether to 2,2'-oxybis(1-chloropropane) per client request. No changes to the data results were required. The Level IV report has been revised to reflect this change.*

## 25.10 Policies on Client Requests for Amendments

### 25.10.1 Policy on Data Omissions or Reporting Limit Increases

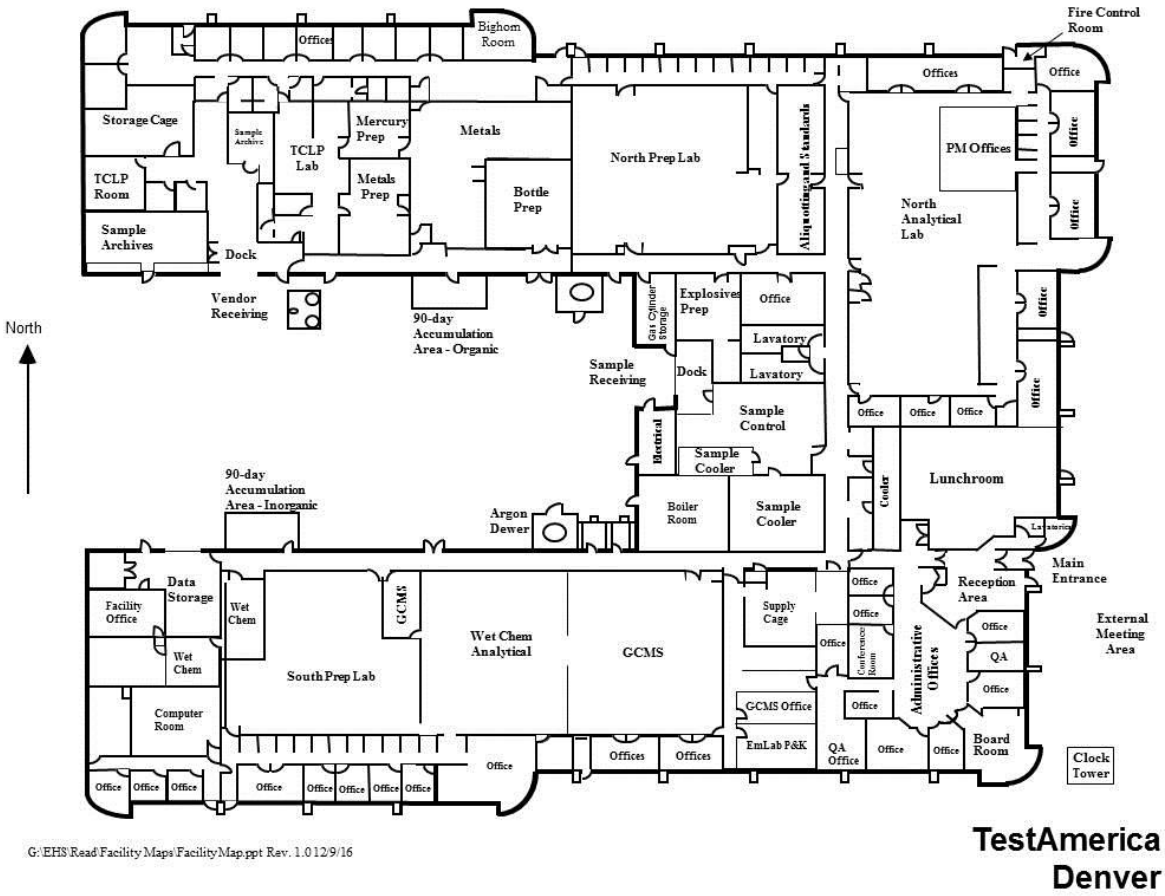
Fundamentally, laboratory policy is simply to not omit previously reported results (including data qualifiers) or to not raise reporting limits and report sample results as ND. This policy has few exceptions. Exceptions are:

- 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 TestAmerica.

### 25.10.2 Multiple Reports

TestAmerica does not issue multiple reports for the same work order where there is different information on each report (this does not refer to copies of the same report) unless required to meet regulatory needs and approved by QA.

## Appendix 1. Laboratory Floor Plan



## Appendix 2. Glossary/Acronyms (EL-V1M2 Sec. 3.1)

**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.

**Analytical Uncertainty:** A subset of Measurement Uncertainty that includes all laboratory activities performed as part of the analysis. (TNI)

**Anomaly:** A condition or event, other than a deficiency, that may affect the quality of the data, whether in the laboratory's control or not.

**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 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 Curve:** The mathematical relationship between the known values, such as concentrations, of a series of calibration standards and their instrument response. (TNI)

**Calibration Standard:** A substance or reference material used to calibrate an instrument (QAMS)

**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 (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 safeguard identified CBI and to maintain all information identified as such in full confidentiality.

**Confirmation:** Verification of the identity of a component through the use of an approach with a different scientific principle from the original method. These may include, but are not limited to Second Column Confirmation; Alternate wavelength; Derivatization; Mass spectral interpretation; Alternative detectors or Additional Cleanup procedures. (TNI)

**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)

**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, whether in the laboratory's control or not. (ASQC)

**Demonstration of Capability:** A procedure to establish the ability of the analyst to generate analytical results of acceptable accuracy and precision. (TNI)

**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 filing a clean container with pure de-ionized water and appropriate preservative, if any, for the specific sampling activity being undertaken (EPA OSWER)

**Field of Accreditation:** Those matrix, technology/method, and analyte combinations for which the accreditation body offers accreditation.

**Holding Times:** The maximum time that samples may be held prior to analyses and still be considered valid or not compromised. (40 CFR Part 136)

**Internal Standard:** A known amount of standard added to a test portion of a sample 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 ± 100%. The IDL represents a range where qualitative detection occurs on a specific instrument. Quantitative results are not produced in this range.

**Laboratory Control Sample (however named, such as laboratory fortified blank, spiked blank, or QC check sample):** A sample matrix, free from the analytes of interest, spiked with verified known amounts of analytes or a material containing known and verified amounts of analytes, taken through all preparation and analysis steps 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.

**Least Squares Regression (1st Order Curve):** The least squares regression is a mathematical calculation of a straight line over two axes. The y axis represents the instrument response (or Response ratio) of a standard or sample and the x axis represents the concentration. The regression calculation will generate a correlation coefficient (r) that is a measure of the "goodness of fit" of the regression line to the data. A value of 1.00 indicates a perfect fit. 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)

**LOD Verification [a.k.a., MDL Verification]:** A processed QC sample in the matrix of interest, spiked with the analyte at no more than 3X the LOD for single analyte tests and 4X the LOD for multiple analyte tests and processed through the entire analytical procedure.

**Limit(s) of Quantitation (LOQ) [a.k.a., Reporting Limit]:** The minimum levels, concentrations, or quantities of a target variable (e.g., target analyte) that can be reported with a specified degree of confidence. (TNI)

**(QS) 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 or Saline/Estuarine. 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 and Emissions:* Whole gas or vapor samples including those contained in flexible or rigid wall containers and the extracted concentrated analytes of interest from a gas or vapor that are collected with a sorbent tube, impinger solution, filter, or other device. (TNI)

**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 replicate matrix spike prepared and analyzed to obtain a measure of the precision of the recovery for each analyte.

**Method Blank:** A sample of a matrix similar to the batch of associated samples (when available) that is free from the analytes of interest and is processed simultaneously with and under the same conditions as

samples through all steps of the analytical procedures, and in which no target analytes or interferences are present at concentrations that impact the analytical results for sample analyses.

**Method Detection Limit:** The minimum concentration of a substance (an analyte) that can be measured and reported with 99% confidence that the analyte concentration is greater than zero and is determined from analysis of a sample in a given matrix containing the analyte. (40 CFR Part 136, Appendix B)

**Negative Control:** Measures taken to ensure that a test, its components, or the environment do not cause undesired effects, or produce incorrect test results.

**Non-conformance:** An indication, judgment, or state of not having met the requirements of the relevant specifications, contract, or regulation.

**Observation:** A record of phenomena that (1) may assist in evaluation of the sample data; (2) may be of importance to the project manager and/or the client, and yet not at the time of the observation have any known effect on quality.

**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.

**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.

**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. (TNI)

**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 laboratory and is provided to test whether the 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. (TNI)

**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)

**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 homogeneous and well established to be used for the calibration of an apparatus, the assessment of a measurement method, or for assigning values to materials. (TNI)

**Reference Standard:** Standard used for the calibration of working measurement standards in a given organization or a given location. (TNI)

**Sampling:** Activity related to obtaining a representative sample of the object of conformity assessment, according to a procedure.

**Second Order Polynomial Curve (Quadratic):** The $2^{nd}$ order curves are a mathematical calculation of a slightly curved line over two axis. The y axis represents the instrument response (or Response ratio) of a standard or sample and the x axis represents the concentration. The $2^{nd}$ order regression will generate a coefficient of determination (COD or $r^2$) that is a measure of the "goodness of fit" of the quadratic curvature the data. A value of 1.00 indicates a perfect fit. In order to be used for quantitative purposes, $r^2$ must be greater than or equal to 0.99.

**Selectivity:** 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. (TNI)

**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

**Technology:** A specific arrangement of analytical instruments, detection systems, and/or preparation techniques.

**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.

**Acronyms:**

CAR – Corrective Action Report
CCV – Continuing Calibration Verification
CF – Calibration Factor
CFR – Code of Federal Regulations
COC – Chain of Custody
DOC – Demonstration of Capability
DQO – Data Quality Objectives
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
LOD – Limit of Detection
LOQ – Limit of Quantitation
MDL – Method Detection Limit
MDLCK – MDL Check Standard
MDLV – MDL Verification Check Standard
MRL – Method Reporting Limit Check Standard
MS – Matrix Spike
MSD – Matrix Spike Duplicate
SDS - 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
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 Denver maintains accreditations, certifications, and approvals 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:

TestAmerica
THE LEADER IN ENVIRONMENTAL TESTING

### TestAmerica Certifications

| Laboratory | Program | Authority | Identification | Expiration Date |
|---|---|---|---|---|
| TestAmerica Denver | A2LA | Wyoming (UST) | 2907.01 | 10/31/2017 |
| TestAmerica Denver | DoD ELAP | A2LA | 2907.01 | 10/31/2017 |
| TestAmerica Denver | Federal | USDA | P330-16-00397 | 12/15/2019 |
| TestAmerica Denver | ISO/IEC 17025 | A2LA | 2907.01 | 10/31/2017 |
| TestAmerica Denver | NELAP | Florida | E87667 | 06/30/2017 |
| TestAmerica Denver | NELAP | Illinois | 200017 | 04/30/2018 |
| TestAmerica Denver | NELAP | Kansas | E-10166 | 04/30/2017 |
| TestAmerica Denver | NELAP | Louisiana | 02096 | 06/30/2017 |
| TestAmerica Denver | NELAP | Minnesota | 8-999-405 | 12/31/2017 |
| TestAmerica Denver | NELAP | New Hampshire | 205310 | 04/28/2017 |
| TestAmerica Denver | NELAP | New Jersey | CO004 | 06/30/2017 |
| TestAmerica Denver | NELAP | New York | 11964 | 04/01/2018 |
| TestAmerica Denver | NELAP | Oregon | 4025 | 01/08/2018 |
| TestAmerica Denver | NELAP | Pennsylvania | 68-00664 | 07/31/2017 |
| TestAmerica Denver | NELAP | Texas | T104704183-16-12 | 09/30/2017 |
| TestAmerica Denver | NELAP | Utah | CO00026 | 07/31/2017 |
| TestAmerica Denver | NELAP | Virginia | 460232 | 06/14/2017 |
| TestAmerica Denver | State Program | Alaska (UST) | UST-30 | 04/05/2018 |
| TestAmerica Denver | State Program | Arizona | AZ0713 | 12/20/2017 |
| TestAmerica Denver | State Program | Arkansas DEQ | 88-0687 | 06/01/2017 |
| TestAmerica Denver | State Program | California | 2513 | 01/08/2018 |
| TestAmerica Denver | State Program | Connecticut | PH-0686 | 09/30/2018 |
| TestAmerica Denver | State Program | Georgia | N/A | 01/08/2018 |
| TestAmerica Denver | State Program | Iowa | 370 | 12/01/2018 |
| TestAmerica Denver | State Program | Maine | CO0002 | 03/03/2019 |
| TestAmerica Denver | State Program | Nevada | CO0026 | 07/31/2017 |
| TestAmerica Denver | State Program | North Carolina (WW/SW) | 358 | 12/31/2017 |
| TestAmerica Denver | State Program | North Dakota | R-034 | 01/09/2018 |
| TestAmerica Denver | State Program | Oklahoma | 8614 | 08/31/2017 |
| TestAmerica Denver | State Program | South Carolina | 72002001 | 01/09/2017 * |
| TestAmerica Denver | State Program | Washington | C583 | 08/02/2017 |
| TestAmerica Denver | State Program | West Virginia DEP | 354 | 11/30/2017 |
| TestAmerica Denver | State Program | Wisconsin | 999615430 | 08/31/2017 |

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* Certification Valid - Laboratory is Pending Renewal with the Program Authority

**For more information, or to contact a local TestAmerica representative nearest you, please visit our website at www.testamericainc.com**



The certificates and accredited parameter lists are available for each State/Program at www.testamericainc.com under Analytical Services Search – Certifications.

# APPENDIX C
# LABORATORY STANDARD OPERATING PROCEDURES (LAB SOPs)

[provided on CD only]

# APPENDIX D
# ADR AND EQUIS EDITION ELECTRONIC DATA DELIVERABLE FILE SPECIFICATIONS

# ADR Electronic Data Deliverable (EDD) File Specifications

The ADR EDD consists of three separate, comma-delimited ASCII text files or Excel CSV files (two, if instrument calibration information is not required by the project). Each file corresponds to a table in the ADR application. These tables are identified as the Analytical Results Table (A1), Laboratory Instrument Table (A2), and Sample Analysis Table (A3). Each file follows the naming convention of using the Laboratory Reporting Batch ID (SDG Number or some other identifier for the EDD) followed by the table identifier (A1, A2, or A3), and then a ".txt" or ".csv" extension. For example, the EDD file names for a laboratory reporting batch identified as SDG001 that includes instrument calibration data would be as follows.

SDG001A1.txt or SDG001A1.csv
SDG001A2.txt or SDG001A2.csv (A2 file is optional)
SDG001A3.txt or SDG001A3.csv

## Analytical Results Table (A1 File)

The Analytical Results table contains analytical results and related information on an analyte level for field samples and associated laboratory quality control samples (excluding calibrations and tunes). Field QC blanks and laboratory method blanks must report a result record for each analyte reported within a method. The method target analyte list is matrix dependent and specified in the project library. Laboratory control samples (LCS and LCSD) and matrix spike samples (MS and MSD) must report a result record for every analyte specified as a spiked analyte in the project library. The project library is a reference table ADR uses for both EDD error checking and automated data review. The project library is populated with information from the project QAPP. Refer to the User Manual for detailed information on project libraries. Table 1 in this document lists all field names and their descriptions for the Analytical Results Table (A1).

## Laboratory Instrument Table (A2 File)

The Laboratory Instrument table contains results and related information on an analyte level for instrument initial calibration standards, initial calibration verification standards, continuing calibration standards, and GC/MS tunes. A record must exist for each target analyte reported in a method (specified in the project library), for every calibration type (the field named QCType) associated to samples reported in the EDD. Initial calibrations, initial calibration verifications, and associated samples are linked to each other using a unique Run Batch ID for every distinct initial calibration within a method. Continuing calibrations and associated samples are linked to each other using a unique Analysis Batch ID for every distinct continuing calibration within a method. GC/MS tunes are linked to initial and continuing calibrations (and hence samples) using the Run Batch and Analysis Batch IDs respectively. The Laboratory Instrument Table (A2) is optional. Depending on the level of validation required by the data user, the Laboratory Instrument table may not be requested in the deliverable. Table 2 in this document lists field names and descriptions for the Laboratory Instrument Table (A2).

## Sample Analysis Table (A3 File)

The Sample Analysis table contains information on a sample level for field samples and laboratory quality control analyses (excluding calibrations and tunes). A sample record exists for each sample/method/matrix/analysis type combination. Table 3 in this document lists field names and descriptions for the Sample Analysis Table (A3).

## EDD Field Properties

Tables 1, 2, and 3 in this document specify the EDD field properties for each file. These include the field name and sequence, field name description, data type and length for each field, and whether or not a particular field requires a standard field. Field elements in the EDD must be sequenced according to the order they appear in Tables 1, 2, and 3. For example, in the Analytical Result table (the A1 file), the field "ClientSampleID" will always be the first piece of information to start a new line of data (or database record), followed by the fields "LabAnalysisRefMethodID", "AnalysisType", and so on.

Table 4 in this document lists standard values for those fields that hold standard values. Required field constraints depend on the combination of sample, matrix, method, analyte type, and calibration or QC type information reported in a record. Tables 5 through 9 in this document indicate required fields for each EDD file (table) according to the method category, matrix, analyte type, sample, and QC or calibration type reported in a record.

When creating an EDD as a text file, use the ASCII character set in a file of lines terminated by a carriage return and line feed. No characters are allowed after the carriage return and line feed. Enclose each data set in double quotes (") and separate each field by a comma (comma delimited). Data fields with no information (null) may be represented by two consecutive commas. For example, in the Sample Analysis table, since the "Collected", "ShippingBatchID", and "Temperature" fields do not apply to laboratory generated QA/QC samples, the record for a Laboratory Control Sample by Method 8270C would be entered as follows. Note that the first two fields ("ProjectNumber" and "ProjectName") are omitted in this example.

…"LCSW100598",,"AQ","LCSW100598","LCS",,"8270C",… (and so on)

Do not pad fields with leading or trailing spaces if a field is populated with less than the maximum allowed number of characters. In the above example, although the "MatrixID" field can accommodate up to 10 characters, only 2 characters were entered in this field.

The EDD can be constructed within Excel and saved as .csv file for import into the application. Be sure to format all cells as text beforehand, otherwise Excel will reformat entered values in some cases.

# Table 1
# Field Descriptions for the Analytical Results Table (A1 file)

**Contains laboratory test results and related information for field and QC samples (excluding instrument calibrations) on an analyte level for environmental chemistry including radiochemistry**

| Field Name | Field Name Description | Field Type | Field Length | Standard Value List |
|---|---|---|---|---|
| ClientSampleID | Client or contractor's identifier for a field sample as reported on the chain-of-custody<br><br>If a sample is analyzed as a laboratory duplicate, matrix spike, or matrix spike duplicate, append suffixes DUP, MS and MSD respectively to the Client Sample ID with no intervening spaces or hyphens (i.e. MW01DUP, MW01MS, and MW01MSD). For Method Blanks, LCS, and LCSD enter the unique LaboratorySampleID into this field<br><br>Do not append suffixes to the ClientSampleID for dilutions, reanalyses, or re-extracts (the AnalysisType field is used for this distinction). For example, MW01DL and MW01RE are not allowed<br><br>Parent sample records must exist for each MS and MSD. If an MS/MSD is shared between two EDDs, records for the MS/MSD and its parent sample must exist in the Analytical Results table for both EDDs. | Text | 25 | NO |
| LabAnalysisRefMethodID | Laboratory reference method ID. The method ID may be an EPA Method number or a Lab Identifier for a method such as a SOP Number, however; method ID is specified by the project. The method ID must be entered into the standard list. | Text | 25 | YES (specified in project plan) |
| AnalysisType | Defines the analysis type (i.e., Dilution, Reanalysis, etc.). This field provides distinction for sample result records when multiple analyses are submitted for the same sample, method, and matrix; for example dilutions, re-analyses, and re-extracts. | Text | 10 | YES (See Table 4) |
| LabSampleID | Laboratory tracking number for field samples and lab generated QC samples such as method blank, LCS, and LCSD. There are no restrictions for the LabSampleID except for field length and that the LabSampleID must be distinct for a given field sample or lab QC sample and method.<br><br>Suffixes may be applied to the LabSampleID to designate dilutions, reanalysis, etc. | Text | 25 | NO |
| LabID | Identification of the laboratory performing the analyses. | Text | 7 | NO |
| ClientAnalyteID | CAS Number or unique client identifier for an analyte or isotope.<br><br>If a CAS Number is not available, use a unique identifier provided by the client or contractor. The ClientAnalyteID for a particular target analyte or isotope should be specified by the project and must exist in the standard value tables for Analytes.<br><br>For the LCS, LCSD, MS, and MSD, it is only necessary to report the compounds designated as spikes in the library (and surrogates for organic methods.)<br><br>For TICs from GC/MS analyses, enter the retention time in decimal minutes as the Client Analyte ID. | Text | 12 | YES (specified by project) |

# Table 1
# Field Descriptions for the Analytical Results Table (A1 file)

**Contains laboratory test results and related information for field and QC samples (excluding instrument calibrations) on an analyte level for environmental chemistry including radiochemistry**

| Field Name | Field Name Description | Field Type | Field Length | Standard Value List |
|---|---|---|---|---|
| AnalyteName | Chemical name for the analyte or isotope. The project specifies how an analyte or isotope is named. The analyte name must be associated to a ClientAnalyteID in the standard values table for Analytes (excluding compounds designated as TIC's). | Numeric | 60 | YES (specified by project) |
| Result | Result value for the analyte or isotope.<br><br>Entries must be numeric. For non-detects of target analytes or isotopes and spikes, do not enter "ND" or leave this field blank. If an analyte or spike was not detected, enter the reporting limit value corrected for dilution and percent moisture as applicable. Do not enter "0" | Text | 10 | NO |
| ResultUnits | The units defining how the values in the Result, DetectionLimit, and ReportingLimit fields are expressed. For radiochemistry this also includes how the value in the Error field is expressed. | Text | 10 | YES (specified by project in the library) |
| LabQualifiers | A string of single letter result qualifiers assigned by the lab based on client-defined rules and values.<br><br>The "U" Lab Qualifier must be entered for all non-detects. Other pertinent lab qualifiers may be entered with the "U" qualifier. Order is insignificant. Lab qualifiers other than those listed in the standard values table may be used. If so, these must be added to the standard value table in the application. | Text | 7 | YES (See Table 4) |
| DetectionLimit | For radiochemistry methods, the minimum detectable activity for the isotope being measured.<br><br>For all other methods: The minimum detection limit value for the analyte being measured.<br><br>**For DoD QSM enter the Limit of Detection (LOD)** | Numeric | 10 | NO |
| DetectionLimitType | Specifies the type of detection limit (i.e., MDA, MDL, IDL, etc.). | Text | 10 | YES (See Table 4) |
| RetentionTime or Error | For radiochemistry methods only, enter the 2 Sigma Counting Error. The units for error are entered in the ResultUnits field.<br><br>For GC/MS methods only, enter the time expressed in decimal minutes between injection and detection for GC/MS TICs only<br><br>For target analytes in all other methods, leave this field blank. Note: GC retention times are not evaluated at this time. | Text | 5 | NO |
| AnalyteType | Defines the type of result, such as tracer, surrogate, spike, or target compound. | Text | 7 | YES (See Table 4) |

# Table 1
# Field Descriptions for the Analytical Results Table (A1 file)

**Contains laboratory test results and related information for field and QC samples (excluding instrument calibrations) on an analyte level for environmental chemistry including radiochemistry**

| Field Name | Field Name Description | Field Type | Field Length | Standard Value List |
|---|---|---|---|---|
| PercentRecovery | For radiochemistry methods: The tracer yield, if applicable.<br><br>For all other analytical methods: The percent recovery value of a spiked compound or surrogate.<br><br>If the spike or surrogate was not recovered because of dilution, enter "DIL". If a spike or surrogate was not recovered because of matrix interference, enter "INT". If a spike or surrogate was not recovered because it was not added to the sample, enter "NS". | Numeric | 5 | NO |
| RelativePercentDifference | The relative percent difference (RPD) of two QC results, such as MS/MSD, LCS/LCSD, and Laboratory Duplicates. Report RPD in Laboratory Duplicate, LCSD, and MSD records only.<br><br>If the RPD is not calculable, enter "NC". | Numeric | 5 | NO |
| ReportingLimit | Reporting limit value for the measured analyte or isotope<br>Factor in the dilution factor and percent moisture correction, if applicable. The Reporting Limit for each analyte and matrix in a given method is specified in the project library or QAPP.<br><br>**For DoD QSM enter the Limit of Quantitation (LOQ)** | Numeric | 10 | NO |
| ReportingLimitType | Specifies the type of reporting limit (i.e., CRQL, PQL, SQL, RDL, etc). The Reporting Limit Type for each method and matrix is specified in the project library or QAPP. | Text | 10 | YES (specified by the project) |

# Table 1
# Field Descriptions for the Analytical Results Table (A1 file)

**Contains laboratory test results and related information for field and QC samples (excluding instrument calibrations) on an analyte level for environmental chemistry including radiochemistry**

| Field Name | Field Name Description | Field Type | Field Length | Standard Value List |
|---|---|---|---|---|
| | | | | |
| ReportableResult | This field indicates whether or not the laboratory chooses an individual analyte or isotope result as reportable. Enter "YES" if the result is reportable. Enter "NO" if the result is not reportable. This field applies to target analytes only.<br><br>If only one analysis is submitted for a particular sample and method, enter "YES" for all target compounds (where Analyte Type = TRG). For GC/MS methods enter yes for tentatively identified compounds ( where Analyte Type = TIC).<br><br>If two or more analyses are submitted for a particular sample and method (i.e. initial analysis, reanalysis and/or dilutions), enter "YES" from only <u>one</u> of the analyses for each target compound. For example: a sample was run a second time at dilution because benzene exceeded the calibration range in the initial, undiluted analysis. All target analytes are reported in each analysis. For the initial analysis, (Analysis Type = RES), enter "NO" for benzene and enter "YES" for all other compounds. For the diluted analysis (Analysis Type = DL), enter "YES" for benzene and enter "NO" for all other compounds.<br><br>For TICs (Analyte Type = TIC), if more than one analysis is submitted for a particular sample and method, choose only one of the analyses where Reportable Result = YES for <u>all</u> TICs. For example, a sample was run a second time because one or more target compounds exceeded the calibration range in the undiluted analysis. Choose a particular analysis and enter "YES" for all TICS. In the other analysis enter "NO" for all TICs.<br><br>Note that it is not necessary to report the full target analyte list for the initial result, dilution, re-analysis, or re-extraction. However, each target analyte must be reported YES once and once only in the case of multiple analyses for a given sample, method, and matrix. In the case of organics, all surrogates must be reported for all analyses submitted for a given sample, method, and, matrix. | Text | 3 | YES (See Table 4) |
| MDL_DoD | This field is not part of the standard ADR EDD format.<br><br>**For DoD QSM enter the MDL, otherwise leave blank. (ADR does not perform error checks on this field)** | Numeric | 10 | NO |

# Table 2
# Field Descriptions for the Laboratory Instrument Table (A2 file)

**Contains related to laboratory instrument calibration on an analyte level and GC/MS Tune information. This table is optional depending on project requirements. Do not report Table A2 for radiochemistry methods.**

| Field Name | Field Name Description | Field Type | Field Length | Standard Value List |
|---|---|---|---|---|
| InstrumentID | Laboratory instrument identification. | Text | 15 | NO |
| QCType | Type of instrument QC (i.e., Instrument_Performance_Check or type of calibration standard). | Text | 10 | YES (See Table 4) |
| Analyzed | Analysis date/time for BFB, DFTPP, initial calibration verification standards, calibration verification standards, and continuing calibration standards. For the initial calibration, enter date and time of the last standard analyzed. Also, see comments about initial calibrations in the Alternate_Lab_Analysis_ID field name description. | Date/ Time | * | NO |
| AlternateLab_AnalysisID | Common laboratory identification used for standards (i.e., VOA STD50, CCAL100, BFB50, etc). For initial calibration, enter ICAL. Information from the initial calibration is entered as one record for each analyte that summarizes the results of the initial calibration (i.e. %RSD, correlation coefficient, and avg RF). Records are not entered for each individual standard within the initial calibration. | Text | 12 | NO |
| LabAnalysisID | Unique identification of the raw data electronic file associated with the calibration standard or tune (i.e., 9812101MS.DV). Leave this field blank for the initial calibration. See comments about initial calibrations in the Alternate_Lab_Analysis_ID field description. This field is only applicable where an electronic instrument file is created as part of the analysis. | Text | 15 | NO |
| LabAnalysisRefMethodID | Laboratory reference method ID (i.e., 8260B, 8270C, 6010B, etc.). The method ID is specified by the project. The LabAnalysisRefMethodID must be in the standard value list for Method IDs. | Text | 25 | YES (specified by the project) |
| ClientAnalyteID | CAS number or unique client identifier for an analyte. If a CAS number is not available, use a unique identifier provided by the client. The unique identifier for a particular analyte should be specified by the project and must exist in the standard value list for ClientAnalyteID.<br><br>Records for each calibration must report the full target analyte list including surrogates as applicable. The target analyte list is specified for each method and matrix in the project | Text | 12 | YES (specified by the project) |
| AnalyteName | The chemical name for the analyte. The project specifies how an analyte is named. The AnalyteName must be associated to a ClientAnalyteID in the standard values. | Text | 60 | YES (specified by the project) |

# Table 2
# Field Descriptions for the Laboratory Instrument Table (A2 file)

**Contains related to laboratory instrument calibration on an analyte level and GC/MS Tune information. This table is optional depending on project requirements. Do not report Table A2 for radiochemistry methods.**

| Field Name | Field Name Description | Field Type | Field Length | Standard Value List |
|---|---|---|---|---|
| RunBatch | Unique identifier for a batch of analyses performed on one instrument under the control of one initial calibration and initial calibration verification. The Run Batch ID links both the initial calibration and initial calibration verification to subsequently analyzed and associated continuing calibrations, field samples, and QC analyses. For GC/MS methods, the Run_Batch ID also links a BFB or DFTPP tune and the initial calibration and initial calibration verification standards to associated samples and method QC analyses. A new and unique Run Batch ID must be used with every new initial calibration. | Text | 12 | NO |
| AnalysisBatch | Unique laboratory identifier for a batch of analyses performed on one instrument and under the control of a continuing calibration or continuing calibration verification. The Analysis Batch ID links the continuing calibration or calibration verification to subsequently analyzed and associated field sample and QC analyses. For GC/MS methods, the Analysis Batch ID also links the BFB or DFTPP tune. A new and unique Analysis Batch ID must be used with every new continuing calibration or continuing calibration verification.<br><br>For GC methods, only report opening standards, do not include closing standards (unless the closing standard functions as the opening standard for a subsequent set of analyses, in which case a new and unique Analysis Batch ID is assigned).<br><br>When dual or confirmation columns/detectors are used, enter results from the primary column/detector only (this is similar to CLP Pesticide reporting). | Text | 12 | NO |
| LabReportingBatch | Unique laboratory identifier for a batch of samples including associated calibrations and method QC, reported as a group by the lab (i.e., lab work order #, log-in #, or SDG). Links all instrument calibrations, samples, and method QC reported as a group or SDG. | Text | 12 | NO |
| PercentRelativeStandard Deviation | The standard deviation relative to the mean used to evaluate initial calibration linearity. Organic methods may use either %RSD or Correlation Coefficient.<br><br>If applicable, enter the %RSD. Leave this field blank if the Correlation Coefficient is used. | Numeric | 5 | NO |
| CorrelationCoefficient | The correlation coefficient resulting from linear regression of the initial calibration. For metals by ICAP, enter '1.0' if a two-point initial calibration was analyzed. Organic methods may use either %RSD or Correlation Coefficient.<br><br>If applicable, enter the Correlation Coefficient. Leave this field blank if the %RSD is used | Numeric | 5 | NO |
| RelativeResponseFactor | This field applies to GC/MS only.<br>For continuing calibration enter the relative response factor.<br><br>For initial calibration enter the average relative response factor. Refer to comments about initial calibration records in the field description for Alternate_Lab_Analysis_ID. | Numeric | 5 | NO |

# Table 2
# Field Descriptions for the Laboratory Instrument Table (A2 file)

**Contains related to laboratory instrument calibration on an analyte level and GC/MS Tune information. This table is optional depending on project requirements. Do not report Table A2 for radiochemistry methods.**

| Field Name | Field Name Description | Field Type | Field Length | Standard Value List |
|---|---|---|---|---|
| Percent_Difference (or Percent Recovery) | For organic methods, this field is the difference between 2 measured values expressed as a percentage.<br><br>If %RSD is reported, enter the % difference between the average response factor of the initial calibration (IC) and the response factor of the initial calibration verification (ICV) or continuing calibration (CCV).<br><br>If correlation coefficient is used, enter the % difference between the true value and the measured value.<br><br>The Percent_Difference is expressed as a negative or positive value. Do not express Percent_Difference as an absolute value. Use a negative value if the CCV or ICV response factor is less than the IC average response factor or, in the case of correlation coefficient, the CCV or ICV measured value is less than the true value. Use a positive value if the CCV or ICV response factor is greater than the IC average response factor, or in the case of correlation coefficient, the CCV or ICV measured value is greater than the true value.<br><br>For inorganic methods, this field is the recovery of an analyte expressed relative to the true amount (i.e., %R for a metal in the continuing calibration or initial calibration verification by Method 6010B). | Numeric | 5 | NO |
| PeakID01 | Identifies individual m/z ions for GC/MS tuning compounds. For BFB enter 50, for DFTPP enter 51. | Numeric | 10 | NO |
| PercentRatio01 | For BFB enter the relative percent abundance of m/z 50 measured relative to the raw abundance of m/z 95.<br><br>For DFTPP enter the relative percent abundance of m/z 51 measured relative to the raw abundance of m/z 198. | Numeric | 10 | NO |
| PeakID02 | Identifies individual m/z ions for GC/MS tuning compounds. For BFB enter 75, for DFTPP enter 68. | Numeric | 10 | NO |
| PercentRatio02 | For BFB enter the relative percent abundance of m/z 75 measured relative to the raw abundance of m/z 95.<br><br>For DFTPP enter the relative percent abundance of m/z 68 measured relative to the raw abundance of m/z 69. | Numeric | 10 | NO |
| PeakID03 | Identifies individual m/z ions for GC/MS tuning compounds. For BFB enter 95, for DFTPP enter 69. | Numeric | 10 | NO |
| PercentRatio03 | For BFB enter the ion abundance of m/z 95 as 100 percent.<br><br>For DFTPP enter the relative percent abundance of m/z 69 measured relative to the raw abundance of m/z 198. | Numeric | 10 | NO |
| PeakID04 | Identifies individual m/z ions for GC/MS tuning compounds. For BFB enter 96, for DFTPP enter 70. | Numeric | 10 | NO |

# Table 2
# Field Descriptions for the Laboratory Instrument Table (A2 file)

**Contains related to laboratory instrument calibration on an analyte level and GC/MS Tune information. This table is optional depending on project requirements. Do not report Table A2 for radiochemistry methods.**

| Field Name | Field Name Description | Field Type | Field Length | Standard Value List |
|---|---|---|---|---|
| PercentRatio04 | For BFB enter the relative percent abundance of m/z 96 measured relative to the raw abundance of m/z 95.<br><br>For DFTPP enter the relative percent abundance of m/z 70 measured relative to the raw abundance of m/z 69 | Numeric | 10 | NO |
| PeakID05 | Identifies individual m/z ions for GC/MS tuning compounds. For BFB enter 173, for DFTPP enter 127. | Numeric | 10 | NO |
| PercentRatio05 | For BFB enter the relative percent abundance of m/z 173 measured relative to the raw abundance of m/z 174.<br><br>For DFTPP enter the relative percent abundance of m/z 127 measured relative to the raw abundance of m/z 198 | Numeric | 10 | NO |
| PeakID06 | Identifies individual m/z ions for GC/MS tuning compounds. For BFB enter 174, for DFTPP enter 197. | Numeric | 10 | NO |
| PercentRatio06 | For BFB enter the relative percent abundance of m/z 174 measured relative to the raw abundance of m/z 95.<br><br>For DFTPP enter the relative percent abundance of m/z 197 measured relative to the raw abundance of m/z 198. | Numeric | 10 | NO |
| PeakID07 | Identifies individual m/z ions for GC/MS tuning compounds. For BFB enter 175, for DFTPP enter 198. | Numeric | 10 | NO |
| PercentRatio07 | For BFB enter the relative percent abundance of m/z 175 measured relative to the raw abundance of m/z 174.<br><br>For DFTPP enter the ion abundance of m/z 198 as 100 percent. | Numeric | 10 | NO |
| PeakID08 | Identifies individual m/z ions for GC/MS tuning compounds. For BFB enter 176, for DFTPP enter 199. | Numeric | 10 | NO |
| PercentRatio08 | For BFB enter the relative percent abundance of m/z 176 measured relative to the raw abundance of m/z 174.<br><br>For DFTPP enter the relative percent abundance of m/z 199 measured relative to the raw abundance of m/z 198. | Numeric | 10 | NO |
| PeakID09 | Identifies individual m/z ions for GC/MS tuning compounds. For BFB enter 177, for DFTPP enter 275. | Numeric | 10 | NO |
| PercentRatio09 | For BFB enter the relative percent abundance of m/z 177 measured relative to the raw abundance of m/z 176.<br><br>For DFTPP enter the relative percent abundance of m/z 275 measured relative to the raw abundance of m/z 198. | Numeric | 10 | NO |
| PeakID10 | Identifies individual m/z ions for GC/MS tuning compounds. For BFB leave blank, for DFTPP enter 365. | Numeric | 10 | NO |

# Table 2
# Field Descriptions for the Laboratory Instrument Table (A2 file)

**Contains related to laboratory instrument calibration on an analyte level and GC/MS Tune information. This table is optional depending on project requirements. Do not report Table A2 for radiochemistry methods.**

| Field Name | Field Name Description | Field Type | Field Length | Standard Value List |
|---|---|---|---|---|
| PercentRatio10 | For BFB leave blank.<br><br>For DFTPP enter the relative percent abundance of m/z 365 measured relative to the raw abundance of m/z 198. | Numeric | 10 | NO |
| PeakID11 | Identifies individual m/z ions for GC/MS tuning compounds. For BFB leave blank, for DFTPP enter 441. | Numeric | 10 | NO |
| PercentRatio11 | For BFB leave blank.<br><br>For DFTPP the percent abundance of m/z 441 measured relative to the raw abundance of m/z 443 | Numeric | 10 | NO |
| PeakID12 | Identifies individual m/z ions for GC/MS tuning compounds. For BFB leave blank, for DFTPP enter 442. | Numeric | 10 | NO |
| PercentRatio12 | For BFB leave blank.<br><br>For DFTPP enter the relative percent abundance of m/z 442 measured relative to the raw abundance of m/z 198. | Numeric | 10 | NO |
| PeakID13 | Identifies individual m/z ions for GC/MS tuning compounds. For BFB leave blank, for DFTPP enter 443. | Numeric | 10 | NO |
| PercentRatio13 | For BFB leave blank.<br><br>For DFTPP enter the relative percent abundance of m/z 443 measured relative to the raw abundance of m/z 442. | Numeric | 10 | NO |

* Date/time format is: MM/DD/YYYY hh:mm where MM = month, DD = day, YYYY = four digits of the year, hh = hour in 24 hour format, and mm = minutes.

# Table 3
# Field Description for the Sample Analysis (A3 file)

**This table contains information related to analyses of field samples and laboratory QC samples (excluding calibrations and tunes) on a sample level for environmental chemical analyses including radiochemistry**

| Field Name | Field Name Description | Field Type | Field Length | Standard Value List |
|---|---|---|---|---|
| ProjectNumber | Project number assigned by the client. | Text | 30 | YES (specified by project) |
| ProjectName | Project name assigned by the client. | Text | 90 | YES (specified by project) |
| ClientSampleID | Client or contractor's identifier for a field sample<br><br>If a sample is analyzed as a laboratory duplicate, matrix spike, or matrix spike duplicate, append suffixes DUP, MS and MSD respectively to the Client Sample ID with no intervening spaces or hyphens (i.e. MW01DUP, MW01MS, and MW01MSD). For Method Blanks, LCS, and LCSD enter the unique LaboratorySampleID into this field<br><br>Do not append suffixes to the ClientSampleID for dilutions, reanalyses, or re-extracts (the Analysis_Type field is used for this distinction). For example, MW01DL and MW01RE are not allowed<br><br>Parent sample records must exist for each MS and MSD. If an MS/MSD is shared between two EDDs, records for the MS/MSD and its parent sample must exist in the Sample Analysis table for both EDDs. | Text | 25 | NO |
| Collected | For radiochemistry methods the Date of sample collection. Refer to the date format for radiochemistry methods at the end of this table.<br><br>For all other methods the Date and Time of sample collection. Refer to the date/time format at the end of this table.<br><br>Leave this field blank for Method Blank, LCS, and LCSD | Date/ Time | 16* | NO |
| MatrixID | Sample matrix (i.e., AQ, SO, etc.) | Text | 10 | YES (See Table 4) |
| LabSampleID | Laboratory tracking number for field samples and lab generated QC samples such as method blank, LCS, and LCSD.<br><br>There are no restrictions for the LabSampleID except field length and that the LabSampleID must be unique for a given field sample or lab QC sample and method. | Text | 25 | NO |
| QCType | This record identifies the type of quality control sample QC (i.e., Duplicate, LCS, Method Blank, MS, or MSD). For regular samples, leave this field blank. | Text | 10 | YES (See Table 4) |
| ShippingBatchID | Unique identifier assigned to a cooler or shipping container used to transport client or field samples. Links all samples to a cooler or shipping container. No entry for method blanks, LCS, and LCSD. This field is optional. | Text | 25 | NO |
| Temperature | Temperature (in centigrade degrees) of the sample as received.<br><br>This field is not required for radiochemistry methods. | Numeric | 10 | NO |

# Table 3
# Field Description for the Sample Analysis (A3 file)

**This table contains information related to analyses of field samples and laboratory QC samples (excluding calibrations and tunes) on a sample level for environmental chemical analyses including radiochemistry**

| Field Name | Field Name Description | Field Type | Field Length | Standard Value List |
|---|---|---|---|---|
| LabAnalysisRefMethodID | Laboratory reference method ID. The method ID may be an EPA Method number or laboratory identifier for a method such as a SOP number, however; values used for Laboratory Method IDs are specified by the project and must be contained in the standard value list for method IDs. | Text | 25 | YES (Specified by the project) |
| PreparationType | Preparation Method Number (i.e., 3010A, 3510C, 3550C, 5030B, etc.)<br><br>For analytical procedures that do not have a specific preparation method number, use "Gen Prep". | Text | 25 | YES (See Table 4) |
| AnalysisType | Defines the type of analysis such as initial analysis, dilution, re-analysis, etc. This field provides distinction for sample records when multiple analyses are submitted for the same sample, method, and matrix, for example: dilutions, re-analyses, and re-extracts. | Text | 10 | YES (See Table 4) |
| Prepared | For radiochemistry leave this field blank.<br><br>For all other methods enter the date and time of sample preparation or extraction. Refer to the date/time format at the end of this table. | Date/ Time | 16* | NO |
| Analyzed | For radiochemistry methods the date of sample analysis. Refer to the date format for radiochemistry methods at the end of this table.<br><br>For all other methods the date and time of sample analysis. Refer to the date and time format at the end of this table. | Date/ Time | * | NO |
| LabID | Identification of the laboratory performing the analysis. | Text | 7 | NO |
| QCLevel | The level of laboratory QC associated with the analysis reported in the EDD. If only the Analytical Results Table (A1) and the Sample Analysis Table (A3) information are submitted for the sample, enter "COA". If the Laboratory Instrument Table (A2) information is also submitted for the sample, enter "COCAL" | Text | 6 | YES (See Table 4) |
| ResultBasis | Indicates whether results associated with this sample record are reported as wet or percent moisture corrected. This field is only required for soils and sediments. Enter "WET" if results are not corrected for percent moisture. Enter "DRY" if percent moisture correction is applied to results. | Text | 3 | YES (See Table 4) |
| TotalOrDissolved | This field indicates if the results related to this sample record are reported as a total or dissolved fraction. This field is only required for metal methods. For all other methods leave this field blank. | Text | 3 | YES (See Table 4) |
| Dilution | Dilution of the sample aliquot. Enter "1" for method blanks, LCS, and LCSD, or if the field samples was analyzed without dilution. | Numeric | 10 | NO |
| HandlingType | Indicates the type of leaching procedure, if applicable (i.e., SPLP, TCLP, WET).<br><br>Leave this field blank if the sample analysis was not performed on a leachate. | Text | 10 | YES (See Table 4) |

# Table 3
# Field Description for the Sample Analysis (A3 file)

**This table contains information related to analyses of field samples and laboratory QC samples (excluding calibrations and tunes) on a sample level for environmental chemical analyses including radiochemistry**

| Field Name | Field Name Description | Field Type | Field Length | Standard Value List |
|---|---|---|---|---|
| HandlingBatch | Unique laboratory identifier for a batch of samples prepared together in a leaching procedure (i.e., SPLP, TCLP, or WET preparation). The HandlingBatch links samples with leaching blanks.<br><br>Leave this field blank if the sample analysis was not performed on a leachate | Text | 12 | NO |
| LeachateDate | Date and time of leaching procedure (i.e., date for SPLP, TCLP, or WET preparation). Refer to the date and time format at the end of this table.<br><br>Leave this field blank if the sample analysis was not performed on a leachate | Date /Time | 16* | NO |
| Percent_Moisture | Percent of sample composed of water. Enter for soil and sediment samples only. | Numeric | 10 | NO |
| MethodBatch | Unique laboratory identifier for a batch of samples of similar matrices analyzed by one method and treated as a group for matrix spike, matrix spike duplicate, or laboratory duplicate association<br><br>The method batch links the matrix spike and/or matrix spike duplicate or laboratory duplicates to associated samples. Note, the MethodBatch association may coincide with the PreparationBatch association. The MethodBatch is specifically used to link the MS/MSD and/or DUP to associated samples. | Text | 12 | NO |
| PreparationBatch | Unique laboratory identifier for a batch of samples prepared together for analysis by one method and treated as a group for method blank, LCS and LCSD association.<br><br>The PreparationBatch links method blanks and laboratory control samples (blank spikes) to associated samples. Note, the PreparationBatch association may coincide with the MethodBatch association but the PreparationBatch specifically links the Method Blank and LCS to associated samples. | Text | 12 | NO |
| RunBatch | For radiochemistry methods leave this field blank.<br><br>For all other methods the RunBatch is the unique identifier for a batch of analyses performed on one instrument under the control of one initial calibration and initial calibration verification. The RunBatch links both the initial calibration and initial calibration verification to subsequently analyzed and associated continuing calibrations, field samples, and QC analyses. For GC/MS methods, the RunBatch also links a BFB or DFTPP tune. A distinct RunBatch must used with every new initial calibration within a method<br><br>The value entered in this field links a particular sample/method/analysis type record to a set of associated initial calibration and initial calibration verification records from Table A2.<br><br>This field is only required if the A2 table is included with the EDD. | Text | 12 | NO |

# Table 3
# Field Description for the Sample Analysis (A3 file)

**This table contains information related to analyses of field samples and laboratory QC samples (excluding calibrations and tunes) on a sample level for environmental chemical analyses including radiochemistry**

| Field Name | Field Name Description | Field Type | Field Length | Standard Value List |
|---|---|---|---|---|
| AnalysisBatch | For radiochemistry methods leave this field blank.<br><br>For all other methods the AnalysisBatch is the unique identifier for a batch of analyses performed on one instrument and under the control of a continuing calibration or continuing calibration verification. The AnalysisBatch links the continuing calibration or calibration verification to subsequently analyzed and associated field sample and QC analyses. For GC/MS methods, the AnalysisBatch also links the BFB or DFTPP tune. A distinct AnalysisBatch must be used with every new continuing calibration or continuing calibration verification within a method<br><br>The value entered in this field links a particular sample/method/analysis type record to a set of associated continuing calibration records in the Laboratory Instrument table.<br><br>This field is only required if the A2 table is included with the EDD. | Text | 12 | NO |
| LabReportingBatch | Unique laboratory identifier for the EDD. This is equivalent to the sample delivery group, lab work number, login ID, etc. The LabReportingBatch links all records in the EDD reported as one group. The value entered in this field must be the same in all records. | Text | 12 | NO |
| LabReceipt | Date and time the sample was received in the lab. A time value of 00:00 may be entered. Refer to the date/time format at the end of this table. | Date/ Time | 16* | |
| LabReported | Date and time hard copy reported delivered by the lab. A time value of 00:00 may be entered. Refer to the date/time format at the end of this table. | Date/ Time | 16* | |

* For radiochemistry methods format Date as MM/DD/YYYY (where MM = two digit month, DD = two digit day, and YYYY = four digit year)

For all other methods format Date and Time as MM/DD/YYYY hh:mm YYYY (where MM = two digit month, DD = two digit day, and YYYY = four digit year, hh = hour in 24 hour format, and mm = minutes)

# Table 4
# Standard Value List

| Field Name | Standard Value | Standard Value Description |
|---|---|---|
| Analysis_Type | DL | Dilution of the original sample |
| | DL2 | Second dilution of the original sample |
| | DL3 | Third dilution of the original sample |
| | DL4 | Fourth dilution of the original sample |
| | RE | Reanalysis/re-extraction of sample |
| | RE2 | Second reanalysis/re-extraction of sample |
| | RE3 | Third reanalysis/re-extraction of sample |
| | RE4 | Fourth reanalysis/re-extraction of the original sample |
| | RES | The initial or original sample. |
| | | |
| Analyte_Name | Refer to QAPP and Project Library | Analyte names are specified by the project and entered into the library for each method and matrix. Analyte Names used in project libraries must first exist in the standard value table. The same holds true for the ClientAnalyteID |
| | | |
| Analyte_Type | IS | Internal standard as defined per CLP usage |
| | SPK | Spiked analyte |
| | SURR | Surrogate as defined as per CLP usage |
| | TIC | Tentatively identified compound for GC/MS analysis |
| | TRG | Target compound |
| | | |
| Detection_Limit_Type [1] | CRDL | Contract required detection limit |
| | IDL | Instrument detection limit |
| | MDA | Minimum detectable activity |
| | MDL | Method detection limit |
| | | |
| Handling_Type [2] | WET | Wet leaching procedure |
| | SPLP | Synthetic Precipitation Leaching Procedure |
| | TCLP | Toxicity Characteristic Leaching Procedure |
| | | |
| Lab_Analysis_Ref_Method_ID | Refer to QAPP and Project Library | Method IDs are specified by the project and entered into the library. Methods used in project libraries must first exist in the standard value table |
| | | |
| Lab_Qualifiers [3] | * | INORG: Duplicate analysis was not within control limits |
| | * | ORG: Surrogate values outside of contract required QC limits |
| | + | INORG: Correlation coefficient for the method of standard additions (MSA) was less than 0.995 |
| | A | ORG: Tentatively identified compound (TIC) was a suspected aldol-condensation product |
| | B | INORG: Value less than contract required detection limit but greater than or equal to instrument detection limit |
| | B | ORG: Compound is found in the associated blank as well as in the sample |
| | C | ORG: Analyte presence confirmed by GC/MS |
| | D | Result from an analysis at a secondary dilution factor |
| | E | INORG: Reported value was estimated because of the presence of interference |
| | E | ORG: Concentrations exceed the calibration range of the instrument |
| | H | Analysis performed outside method or client-specified holding time requirement |
| | J | Estimated value |
| | M | INORG: Duplicate injection precision was not met |
| | N | INORG: Spiked sample recovery was not within control limits |
| | N | ORG: Presumptive evidence of a compound |
| | P | ORG: Difference between results from two GC columns unacceptable (>25% Difference) |
| | S | Reported value was determined by the method of standard additions (MSA) |
| | U | Compound was analyzed for but not detected. Analyte result was below the Reporting Limit. |
| | W | INORG: Post digestion spike was out of control limits |
| | X | Reserved for a lab-defined data qualifier |
| | Y | Reserved for a lab-defined data qualifier |
| | Z | Reserved for a lab-defined data qualifier |
| | | |
| Matrix_ID | AIR | Air |
| | AQ | Water |
| | ASH | Ash |

# Table 4
# Standard Value List

| Field Name | Standard Value | Standard Value Description |
|---|---|---|
| Matrix_ID (continued) | BIOTA | Biological matter |
| | FILTER | Filter |
| | LIQUID | Non-aqueous liquid |
| | OIL | Oil |
| | SED | Sediment |
| | SLUDGE | Sludge |
| | SO | Soil |
| | SOLID | Non-soil/sediment solid |
| | TISSUE | Tissue |
| | WASTE | Waste |
| | WIPE | Wipe |
| | | |
| Preparation_Type [4] | 3005A | Acid Digestion of Waters for Total Recoverable or Dissolved Metals by FLAA or ICP |
| | 3010A | Acid of Aqueous Samples and Extracts for Total Metals by FLAA or ICP |
| | 3015 | Microwave Assisted Acid Digestion of Aqueous Samples and Extracts |
| | 3020A | Acid Digestion of Aqueous Samples and Extracts for Total Metals by GFAA |
| | 3031 | Acid Digestion of Oils for Metals Analysis by AA or ICP |
| | 3050B | Acid Digestion of Sediments, Sludges, and Soils |
| | 3051 | Microwave Assisted Acid Digestion of Sediments, Sludges, Soils and Oils |
| | 3052 | Microwave Assisted Acid Digestion of Siliceous and Organically Based Matrices |
| | 3060A | Alkaline Digestion for Hexavalent Chromium |
| | 3510C | Separatory Funnel Liquid-Liquid Extraction |
| | 3520C | Continuous Liquid-Liquid Extraction |
| | 3535 | Solid Phase Extraction |
| | 3540C | Soxhlet Extraction |
| | 3541 | Automated Soxhlet Extraction |
| | 3545 | Pressurized Fluid Extraction |
| | 3550B | Ultrasonic Extraction |
| | 3560 | Supercritical Fluid Extraction of Total Recoverable Petroleum Hydrocarbons |
| | 5030B | Purge and Trap for Aqueous Samples |
| | 5035 | Closed-System Purge-and-Trap and Extraction for Volatile Organics in Soil and Waste Samples |
| | 7470A | Acid digestion of waters for Mercury analysis |
| | 7471A | Acid digestion of soils and solids for Mercury analysis |
| | Gen Prep | Generic preparation type when a preparation method ID does not exist (used mostly for general chemistry methods) |
| | | |
| QC_Level | COA | Certificate of Analysis (accuracy and precision, no calibration) |
| | COACAL | Certificate of Analysis (accuracy and precision including calibration) |
| | | |
| QC_Type | MB | Analytical control consisting of all reagents and standards that is carried through the entire procedure (Method Blank) |
| | CV | (Calibration Verification) Analytical standard run at a specified frequency to verify the calibration of the analytical system |
| | CCV | (Continuing Calibration Verification) Analytical standard run every 12 hours to verify the calibration of the GC/MS system |
| | DUP | A second aliquot of a sample that is treated the same as the original aliquot to determine the precision of the method |
| | IC | (Initial Calibration) Analysis of analytical standards for a series of different specified concentrations |
| | ICV | (Initial Calibration Verification) Analytical standard run at a specified frequency to verify the accuracy of the initial calibration of the analytical system |
| | IPC | (Instrument Performance Check) Analysis of DFTPP or BFB to evaluate the performance of the GC/MS system |
| | LCS | (Laboratory Control Sample) A control sample of known composition |
| | LCSD | (Laboratory Control Sample Duplicate) A duplicate control sample of known composition |
| | MS | (Matrix Spike) Aliquot of a matrix spiked with known quantities and subjected to the entire analytical procedure to measure recovery |
| | MSD | (Matrix Spike Duplicate) A second aliquot of the same matrix as the matrix spike that is spiked in order to determine the precision of the method |
| | | |
| Reporting_Limit_Type [1] | CRDL | Contract required detection limit |
| | CRQL | Contract required quantitation limit |

# Table 4
# Standard Value List

| Field Name | Standard Value | Standard Value Description |
|---|---|---|
| Reporting_Limit_Type (continued) | PQL | Practical quantitation limit |
| | SQL | Sample quantitation limit |
| | RDL | Reportable detection limit |
| | | |
| Result_Basis | DRY | Result was calculated on a dry weight basis |
| | WET | Result was calculated on a wet weight basis |
| | | |
| Result_Units [5] | ug/L | Micrograms per liter |
| | mg/L | Milligrams per liter |
| | ug/Kg | Micrograms per kilogram |
| | mg/Kg | Milligrams per kilogram |
| | pg/L | Picograms per liter |
| | ng/Kg | Nanograms per kilogram |
| | | |
| Total_Or_Dissolved | DIS | Dissolved |
| | TOT | Total |

1 **Additional Detection Limit Types and Reporting Limit Types may be used. These must be added to the application standard values.**
2 **Additional Handling Types (leachate procedures) may be used. These must be added to the application standard values**
3 **Additional Lab Qualifiers may be used, or listed Lab Qualifiers may be used in a different manner than described in this table. New lab qualifiers must be added to the application standard value tables. NOTE: The "U" Lab Qualifier must be used for all non-detects.**
4 **Additional Preparation Types may be used. These must be added to the application standard value tables.**
5 **Additional Result Units may be used. The project library specifies the reporting limit used for each method and matrix**

**Note: If new standard values are used then these standard values must be entered in the software standard values for both the lab and contractor. The application will automatically update the standard values tables if an importing library contains standard values (method, client analyte ID, and analyte name) that do not exist in the software importing the new library.**

# Table 5
# Required Fields in the Analytical Results Table for GC/MS, GC, and HPLC Methods

| | GC/MS Methods | | | GC and HPLC Methods | | |
|---|---|---|---|---|---|---|
| Field | Regular Sample* | MS/MSD | Method Blank, LCS/LCSD | Regular Sample* | MS/MSD | Method Blank, LCS/LCSD |
| Client_Sample_ID | X | X | X | X | X | X |
| Lab_Analysis_Ref_Method_ID | X | X | X | X | X | X |
| Analysis_Type | X | X | X | X | X | X |
| Lab_Sample_ID | X | X | X | X | X | X |
| Lab_ID | X | X | X | X | X | X |
| | | | | | | |
| Client_Analyte_ID | X | X | X | X | X | X |
| Analyte_Name | X | X | X | X | X | X |
| Result | X | X | X | X | X | X |
| Result_Units | X | X | X | X | X | X |
| Lab_Qualifiers | Q | Q | Q | Q | Q | Q |
| | | | | | | |
| Detection Limit | X | X | X | X | X | X |
| Detection_Limit_Type | X | X | X | X | X | X |
| Retention_Time | T | | T | | | |
| Analyte_Type | X | X | X | X | X | X |
| Percent_Recovery | S | R | R | S | R | R |
| | | | | | | |
| Relative_Percent_Difference | | D | D | | D | D |
| Reporting_Limit | X | X | X | X | X | X |
| Reporting_Limit_Type | X | X | X | X | X | X |
| Reportable_Result | X | X | X | X | X | X |

Key

- X Required Field
- D Required field for spiked compounds in the LCSD and MSD only
- Q Required field if laboratory has qualified result. The “U” qualifier MUST be entered if the result is non-detect.
- R Required field if Analyte_Type = “SPK” or “SURR”
- S Required field for surrogate compounds only
- T Required field for tentatively identified compounds by GC/MS only
- * Also includes Equipment Blanks, Field Blanks, and Trip Blanks

# Table 6
# Required Fields in the Analytical Results Table for ICAP, AA, and IC Methods

| Field | ICAP and AA Methods | | | IC and Wet Chemistry Methods | | |
|---|---|---|---|---|---|---|
| | Regular Sample* | Sample Duplicate, MS/MSD | Method Blank, LCS/LCSD | Regular Sample* | Sample Duplicate MS/MSD | Method Blank, LCS/LCSD |
| Client_Sample_ID | X | X | X | X | X | X |
| Lab_Analysis_Ref_Method_ID | X | X | X | X | X | X |
| Analysis_Type | X | X | X | X | X | X |
| Lab_Sample_ID | X | X | X | X | X | X |
| Lab_ID | X | X | X | X | X | X |
| | | | | | | |
| Client_Analyte_ID | X | X | X | X | X | X |
| Analyte_Name | X | X | X | X | X | X |
| Result | X | X | X | X | X | X |
| Result_Units | X | X | X | X | X | X |
| Lab_Qualifiers | Q | Q | Q | Q | Q | Q |
| | | | | | | |
| Detection Limit | X | X | X | X | X | X |
| Detection_Limit_Type | X | X | X | X | X | X |
| Retention_Time | | | | | | |
| Analyte_Type | X | X | X | X | X | X |
| Percent_Recovery | | S | S | | S | S |
| | | | | | | |
| Relative_Percent_Difference | | R | R | | R | R |
| Reporting_Limit | X | X | X | X | X | X |
| Reporting_Limit_Type | X | X | X | X | X | X |
| Reportable_Result | X | X | X | X | X | X |

Key

X Required field
Q Required field if laboratory has qualified result. The “U” qualifier MUST be entered if the result is non-detect
R Required field for spiked compounds in LCSD or MSD, or target compounds in the Sample Duplicate only
S Required field if Analyte_Type = “SPK”
* Also includes Trip Blanks, Equipment Blanks, and Field Blanks

# Table 7
# Required Fields in the Laboratory Instrument Table

| | GC/MS Tunes | | Initial Calibration | | | | Initial Calibration Verification | | | | Calibration Verification, Continuing Calibration |
|---|---|---|---|---|---|---|---|---|---|---|---|
| Field | VOA | SVOA | GC/MS | GC HPLC | ICP/AA | IC* | GC/MS | GC HPLC | ICP/AA | IC* | ALL METHODS |
| Instrument_ID | X | X | X | X | X | X | X | X | X | X | X |
| QC_Type | X | X | X | X | X | X | X | X | X | X | X |
| Analyzed | X | X | X | X | X | X | X | X | X | X | X |
| Alternate_Lab_Analysis_ID | X | X | X | X | X | X | X | X | X | X | X |
| Lab_Analysis_ID | X | X | | | | | X | X | X | X | X |
| Lab_Analysis_Ref_Method_ID | X | X | X | X | X | X | X | X | X | X | X |
| Client_Analyte_ID | X | X | X | X | X | X | X | X | X | X | X |
| Analyte_Name | X | X | X | X | X | X | X | X | X | X | X |
| Run_Batch | X | X | X | X | X | X | X | X | X | X | X |
| Analysis_Batch | C | C | | | | | | | | | X |
| Lab_Reporting_Batch | X | X | X | X | X | X | X | X | X | X | X |
| Percent_Relative_Standard_Deviation | | | X | X | | | | | | | |
| Correlation_Coefficient | | | B | B | X | X | | | | | |
| Relative_Response_Factor | | | X | | | | X | | | | M |
| Percent_Difference | | | | | | | X | X | X | X | X |
| Peak_ID_01 | X | X | | | | | | | | | |
| Percent_Ratio_01 | X | X | | | | | | | | | |
| Peak_ID_02 | X | X | | | | | | | | | |
| Percent_Ratio_02 | X | X | | | | | | | | | |
| Peak_ID_03 | X | X | | | | | | | | | |
| Percent_Ratio_03 | X | X | | | | | | | | | |
| Peak_ID_04 | X | X | | | | | | | | | |
| Percent_Ratio_04 | X | X | | | | | | | | | |
| Peak_ID_05 | X | X | | | | | | | | | |
| Percent_Ratio_05 | X | X | | | | | | | | | |
| Peak_ID_06 | X | X | | | | | | | | | |
| Percent_Ratio_06 | X | X | | | | | | | | | |
| Peak_ID_07 | X | X | | | | | | | | | |
| Percent_Ratio_07 | X | X | | | | | | | | | |
| Peak_ID_08 | X | X | | | | | | | | | |
| Percent_Ratio_08 | X | X | | | | | | | | | |
| Peak_ID_09 | X | X | | | | | | | | | |
| Percent_Ratio_09 | X | X | | | | | | | | | |
| Peak_ID_10 | | X | | | | | | | | | |
| Percent_Ratio_10 | | X | | | | | | | | | |
| Peak_ID_11 | | X | | | | | | | | | |
| Percent_Ratio_11 | | X | | | | | | | | | |
| Peak_ID_12 | | X | | | | | | | | | |
| Percent_Ratio_12 | | X | | | | | | | | | |
| Peak_ID_13 | | X | | | | | | | | | |
| Percent_Ratio_13 | | X | | | | | | | | | |

Key

X Required field (some fields are not applicable to some General (Wet) Chemistry tests)

B Required field if reporting best fit

C Required field if BFB or DFTPP associated with a continuing calibration only

M Required field for GC/MS continuing calibration only

*IC Includes Ion Chromatography and Classical or Wet Chemistry methods. Methods such as pH, Conductivity, and others do not use traditional calibration procedures: therefore some fields marked as a required field under the "IC" column do not apply for these methods.

# Table 8
# Required Fields in the Sample Analysis Table

| Field | GC, GC/MS, HPLC Methods | | ICAP and AA Methods | | IC and Wet Chemistry Methods | |
|---|---|---|---|---|---|---|
| | Method Blanks, LCS/LCSD | Regular Samples*, Sample Duplicate, MS/MSD | Method Blanks, LCS/LCSD | Regular Samples*, Sample Duplicate, MS/MSD | Method Blanks, LCS/LCSD | Regular Samples*, Sample Duplicate, MS/MSD |
| Client_Sample_ID | X | X | X | X | X | X |
| Collected | | X | | X | | X |
| Matrix_ID | X | X | X | X | X | X |
| Lab_Sample_ID | X | X | X | X | X | X |
| QC_Type | X | Q | X | Q | X | X |
| | | | | | | |
| Shipping_Batch_ID | | X | | X | | X |
| Temperature | | X | | | | X |
| Lab_Analysis_Ref_Method_ID | X | X | X | X | X | X |
| Preparation_Type | X | X | X | X | X | X |
| Analysis_Type | X | X | X | X | X | X |
| | | | | | | |
| Prepared | A | A | X | X | N | N |
| Analyzed | X | X | X | X | X | X |
| Lab_ID | X | X | X | X | X | X |
| QC_Level | X | X | X | X | X | X |
| Results_Basis | | S | | S | | S |
| | | | | | | |
| Total_Or_Dissolved | | | W | W | | |
| Dilution | X | X | X | X | X | X |
| Handling_Type | L | L | L | L | L | L |
| Handling_Batch | L | L | L | L | L | L |
| Leachate_Date | L | L | L | L | L | L |
| | | | | | | |
| Percent Moisture | | S | | S | | S |
| Method_Batch | X | X | X | X | X | X |
| Preparation_Batch | X | X | X | X | X | X |
| Run_Batch | C | C | C | C | C | C |
| Analysis_Batch | C | C | C | C | C | C |
| | | | | | | |
| Lab_Reporting_Batch | X | X | X | X | X | X |
| Lab_Receipt | | X | | X | | X |
| Lab_Reported | X | X | X | X | X | X |

Key

X Required field
A Required field for samples prepared by methanol extraction
C Required field if Instrument Calibration Table (A2) is included in EDD
L Required field if analysis performed on SPLP, TCLP, or WET extracts
N Required field only for samples that require preparation before analysis
Q Required field for Sample Duplicate, MS, and MSD only
S Required field if “Matrix_ID” = “SO” or “SED”
W Required field for aqueous samples only
* Includes Trip Blanks, Equipment Blanks, and Field Blanks

*DISCUSSION DRAFT*

# LABORATORY
# ELECTRONIC DATA DELIVERABLE
# FORMAT SPECIFICATION

## EQuIS Edition

February 2009

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# 1 GENERAL REQUIREMENTS

## 1.1 Transfer Media

There are 4 acceptable modes of data transfer to ENVIRON International Corporation (ENVIRON)'s Emeryville, CA office:

1. Laboratory maintained website with links to datafile downloads;
2. File share service, such as Box or Sharefile;
3. MS-Windows readable disk (CD or DVD);
4. Email to an email address arranged between ENVIRON and the lab.

Each Sample Delivery Group (SDG) (i.e., data pack) should be packaged separately for transfer.

## 1.2 Character Set

ENVIRON Corporation data files must be provided in the ASCII Character Set. Furthermore, all character information, except for *analyte* field values, must be provided in ***UPPER CASE***. The *analyte* field may be provided in mixed case.

## 1.3 Record Terminator

Within each data file, the individual records must be terminated by a carriage return (ASCII Character 013).

## 1.4 Field Delimiter

Per EarthSoft, the preferred field delimiter is the tab character (ASCII Character 009). Comma (","; ASCII Character 044) will also be accepted as a delimiter.

To further ensure the field delimitation, ENVIRON requires the inclusion of double quotes (", ASCII Character 034) on either side of text data field values (e.g., "1,2,3-ethane",34.4,"B",10.0). Double quotes must not be placed around numeric values.

## 1.5 White space

All extraneous white space characters (e.g., spaces, tabs, blanks) must be eradicated from the data file. All data fields must be trimmed (i.e., clipped) to remove leading and trailing white space.

## 1.6 Chain of Custody Correspondence

The information provided in the analytical sample results data records must strictly correspond to the information reported to the laboratory on the Chain of Custody. This information may not be altered, have information appended or prefixed to it. For example, if the sample identifier reported on the chain of custody is 1786H-MW01-950501, that is the

string which must be returned -- not 1786H-MW01-950501DL, not 1786H-MW01-950501RE. These types of additions are acceptable on the Lab Sample ID.

NOTE: This constraint does not apply for laboratory QC samples that are cloned from field samples (e.g., Matrix Spike, Lab Duplicates).

### 1.7 Air Samples

For air samples, both sets of results (by volume and by cubic meter) must be reported. Please append a VOL to the back of the method for the "by volume" (e.g., ppbv) results, so they are not considered duplicate records by the EQuIS checker.

For example:

| sys_sample_code | lab_anl_method_name | cas_rn | result_unit |
|---|---|---|---|
| SG-01-060908 | TO15 | 156-59-2 | ug/m3 |
| SG-01-060908 | TO15VOL | 156-59-2 | ppbv |
| SG-02-060908 | TO15 | 156-59-2 | ug/m3 |
| SG-02-060908 | TO15VOL | 156-59-2 | ppbv |

Granted, the ppbv results sent on the EDD can be simply converted to ug/m3; however, ENVIRON's reference information (e.g., molecular weight) may not match the inputs that the lab used in its calculations, therefore, not receiving both values results in discrepancies between the database and the hard copy report.

# 2 EQuIS Formats

## 2.1 Overview

The Emeryville Office of ENVIRON International Corporation has elected to implement EQuIS Chemistry (version 5) from EarthSoft, Inc. as its internal data repository standard. The 4-file format, including the refinements noted below, is the required format. The generic documentation for these specifications is available directly from EarthSoft at http://www.earthsoft.com/support/edd.asp and will not be repeated in this document.

Exceptions may be made to accept the EZ-EDD at the discretion of the ENVIRON project manager and the database administrator in specific cases (e.g., geotechnical analyses).

## 2.2 EQuIS 4-File Record Structures

### 2.2.1 Sample File

The sample file should contain the required information for all samples, regardless of their source (e.g., field, lab). Information that is not marked required should be provided in all cases where the information is available.

Shaded columns denote fields that are included in the default EQuIS sample loader file, but contain information that is generally not provided to the laboratory. For consistency with the import utility, these fields must remain in the EDD; however, population of these fields is not expected.

| Pos# | Field Name | Data Type | Required | Comments |
|---|---|---|---|---|
| 1 | sys_sample_code | Text(40) | Y | Unique sample identifier. Each sample must have a unique value, including spikes and duplicates. Laboratory QC samples must also have unique identifiers. As noted in Section 1.6 above, for field samples, this should match the value which appears on the chain of custody. |
| 2 | sample_name | Text(30) | Y | Standardized sample name across all permutations. It is not required to be unique (i.e., duplicates are OK). As noted in Section 1.6 above, for field samples, this should match the value which appears on the chain of custody. |

| Pos# | Field Name | Data Type | Required | Comments |
|---|---|---|---|---|
| 3 | sample_matrix_code | Text(10) | Y | Code which distinguishes between different type of sample matrix. For example, blank samples must be distinguished from ground water samples, etc. See Section 3.1 to this document for the set of valid values. |
| 4 | sample_type_code | Text(20) | Y | Code which distinguishes between different types of samples. For example, normal field samples must be distinguished from laboratory method blank samples, etc. See Section 3.2 to this document for the set of valid values. |
| 5 | sample_source | Text(10) | Y | This field identifies where the sample came from, either **FIELD** or **LAB**. |
| 6 | parent_sample_code | Text(40) | N | The value of "sys_sample_code" that uniquely identifies the sample that was the source of this sample. For example, the value of this field for a duplicate sample would identify the normal sample of which this sample is a duplicate.<br>Required in the laboratory EDD for all laboratory "clone" samples (e.g., spikes and duplicates). Field duplicates may be submitted blind to the laboratory, so this field is not required in the laboratory EDD for field "clones". Must be blank for samples which have no parent (e.g., normal field samples, LCS samples, method blanks, etc.). |
| 7 | sample_delivery_group | Text(10) | Y | The lab job identifier, consistent with the labeling on the final report. |
| 8 | sample_date | Date | Y | Date sample was collected (in MM/DD/YYYY format for EDD). |
| 9 | sample_time | Time | N | Time of sample collection in 24-hr (military) HH:MM format. |
| 10 | sys_loc_code | Text(20) | N | Sample collection location. |
| 11 | start_depth | Double | N | Beginning depth (top) of soil sample. |
| 12 | end_depth | Double | N | Ending depth (bottom) of soil sample. |
| 13 | depth_unit | Text(15) | N | Unit of measurement for the sample begin and end depths. |
| 14 | chain_of_custody | Text(15) | N | Chain of custody identifier. A single sample may be assigned to only one chain of custody. If the chains are not serialized, please use the collection date of the samples, formatted as YYYYMMDD. |

| Pos# | Field Name | Data Type | Required | Comments |
|---|---|---|---|---|
| 15 | sent_to_lab_date | Date | N | Date sample was sent to lab (in MM/DD/YYYY format for EDD). |
| 16 | sample_receipt_date | Date | N | Date that sample was received at laboratory (in MM/DD/YYYY format for EDD). |
| 17 | sampler | Text(30) | N | Name or initials of sampler. |
| 18 | sampling_company_code | Text(10) | N | Name or initials of sampling company (no controlled vocabulary). |
| 19 | sampling_reason | Text(30) | N | Optional reason for sampling. |
| 20 | sampling_technique | Text(40) | N | Sampling technique. |
| 21 | task_code | Text(10) | N | Code used to identify the task under which the field sample was retrieved. |
| 22 | collection_quarter | Text(5) | N | Quarter of the year sample was collected (e.g., "1Q96"). |
| 23 | composite_yn | Text(1) | N | Boolean field used to indicate whether a sample is a composite sample. |
| 24 | composite_desc | Text(255) | N | Description of composite sample. |
| 25 | sample_class | Text(10) | N | Navy sample class code. |
| 26 | custom_field_1 | Text(255) | N | Custom sample field. |
| 27 | custom_field_2 | Text(255) | N | Custom sample field. |
| 28 | custom_field_3 | Text(255) | N | Custom sample field. |
| 29 | comment | Text(255) | N | Sample comments as necessary (e.g., broken jar, cooler issues). |
| 30 | sample_receipt_time | Text(5) | N | Time of lab receipt sample in 24-hr (military) HH:MM format. |

### 2.2.2 Test File

The test file should contain the required information for all samples, regardless of their source (e.g., field, lab). Information that is not marked required should be provided in all cases where the information is available.

| Pos# | Field Name | Data Type | Required | Comments |
|---|---|---|---|---|
| 1 | sys_sample_code | Text(40) | Y | Unique sample identifier. Each sample must have a unique value, including spikes and duplicates. Laboratory QC samples must also have unique identifiers. As noted in Section 1.6 above, for field samples, this should match the value which appears on the chain of custody. |
| 2 | lab_anl_method_name | Text(35) | Y | Laboratory analytic method name or description. |
| 3 | analysis_date | Date | Y | Date of sample analysis in MM/DD/YYYY format. |
| 4 | analysis_time | Text(5) | Y | Time of sample analysis in 24-hr (military) HH:MM format. |
| 5 | total_or_dissolved | Text(1) | Y | Type of analysis. Valid values include:<br>"T"=Total analysis;<br>"D"=Dissolved or Filtered analysis;<br>"N"=constituents for which neither "total" nor "dissolved" is applicable.<br>**This differs from the default EQuIS specification, which constrains the use of T and D to metals analyses**. |
| 6 | column_number | Text(2) | N | Column identifier for dual column analyses. |
| 7 | test_type | Text(10) | Y | Type of test. Valid values include:<br>"INITIAL";<br>“DILUTION”;<br>"REEXTRACT";<br>"REANALYSIS".<br>Contact DBA if other values are needed. |
| 8 | lab_matrix_code | Text(10) | N | The matrix of the sample as analyzed may be different from the matrix of the sample as retrieved (e.g. leachates). |

| Pos# | Field Name | Data Type | Required | Comments |
|---|---|---|---|---|
| 9 | analysis_location | Text(2) | Y | Valid values include:<br>"FI" for field instrument or probe;<br>"FL" for mobile field laboratory analysis;<br>"LB" for fixed-based laboratory analysis.<br>Contact DBA if other values are needed. |
| 10 | basis | Text(10) | Y | Valid values include:<br>"WET" for wet-weight basis reporting;<br>"DRY" for dry-weight basis reporting;<br>"NA" where this distinction is not applicable.<br>Contact DBA if other values are needed. |
| 11 | container_id | Text(30) | N | Sample container identifier. |
| 12 | dilution_factor | Single | N | Effective test dilution factor. |
| 13 | prep_method | Text(35) | N | Laboratory sample preparation method name or description. |
| 14 | prep_date | Date | N | Date of sample preparation in MM/DD/YYYY. This field, in conjunction with extraction time, is used to determine whether holding times for field samples have been exceeded. |
| 15 | prep_time | Text(5) | N | Time of sample preparation in 24-hr (military) HH:MM format. This field, in conjunction with extraction date, is used to determine whether holding times for field samples have been exceeded. |
| 16 | leachate_method | Text(15) | N | Laboratory leachate generation method name or description. |
| 17 | leachate_date | Date | N | Date of leachate preparation in MM/DD/YYYY format. |
| 18 | leachate_time | Text(5) | N | Time of leachate preparation in 24-hr (military) HH:MM format. |
| 19 | lab_name_code | Text(10) | N | Unique identifier of the laboratory. Must be consistent across all projects. |
| 20 | qc_level | Text(10) | N | Laboratory QC level associated with the analysis. |
| 21 | lab_sample_id | Text(20) | Y | Unique sample ID internally assigned by the laboratory. |

| Pos# | Field Name | Data Type | Required | Comments |
|---|---|---|---|---|
| 22 | percent_moisture | Text(5) | N | Percent moisture of the sample portion used in this test; this value may vary from test to test for any sample. Numeric format is "NN.MM", i.e., 70.1% should be reported as "70.1" but not as .701. |
| 23 | subsample_amount | Text(14) | N | Amount of sample used for test. This is an optional field for the laboratory EDD unless otherwise specified by the EQuIS Chemistry project manager. |
| 24 | subsample_amount_ unit | Text(15) | N | Unit of measurement for subsample amount. |
| 25 | analyst_name | Text(30) | N | Name or initials of laboratory analyst. |
| 26 | instrument_id | Text(50) | N | Instrument identifier. |
| 27 | comment | Text(255) | N | Sample comments as necessary (e.g., broken jar, cooler issues). |
| 28 | preservative | Text(50) | N | Sample preservative used. |
| 29 | final_volume | Text(15) | N | The final amount of the sample after sample preparation. |
| 30 | final_volume_unit | Text(15) | N | The unit of measure that corresponds to the final_amount. |

### 2.2.3 Batch File

The batch file should contain the required information for all samples, regardless of their source (e.g., field, lab). Information that is not marked required should be provided in all cases where the information is available.

| Pos# | Field Name | Data Type | Required | Comments |
|---|---|---|---|---|
| 1 | sys_sample_code | Text(40) | Y | Unique sample identifier. Each sample must have a unique value, including spikes and duplicates. Laboratory QC samples must also have unique identifiers. As noted in Section 1.6 above, for field samples, this should match the value which appears on the chain of custody. |
| 2 | lab_anl_method_ name | Text(35) | Y | Laboratory analytic method name or description. |
| 3 | analysis_date | Date | Y | Date of sample analysis in MM/DD/YYYY format. |
| 4 | analysis_time | Text(5) | Y | Time of sample analysis in 24-hr (military) HH:MM format. |

| Pos# | Field Name | Data Type | Required | Comments |
|---|---|---|---|---|
| 5 | total_or_dissolved | Text(1) | Y | Type of analysis. Valid values include:<br>"T"=Total analysis;<br>"D"=Dissolved or Filtered analysis;<br>"N"=constituents for which neither "total" nor "dissolved" is applicable.<br>**This differs from the default EQuIS specification, which constrains the use of T and D to metals analyses**. |
| 6 | column_number | Text(2) | N | Column identifier for dual column analyses. |
| 7 | test_type | Text(10) | Y | Type of test. Valid values include:<br>"INITIAL";<br>“DILUTION”;<br>"REEXTRACT";<br>"REANALYSIS".<br>Contact DBA if other values are needed. |
| 8 | test_batch_type | Text(10) | Y | Lab batch type. Valid values include:<br>"PREP";<br>"ANALYSIS";<br>"LEACH" |
| 9 | test_batch_id | Text(20) | Y | Unique identifier for all lab batches. Must be unique within EQuIS Chemistry database. For example, the same identifier can not be used for a prep batch and an analysis batch. |

### 2.2.4 Result File

The result file should contain the required information for all samples, regardless of their source (e.g., field, lab). Information that is not marked required should be provided in all cases where the information is available.

| Pos# | Field Name | Data Type | Required | Comments |
|---|---|---|---|---|
| 1 | sys_sample_code | Text(40) | Y | Unique sample identifier. Each sample must have a unique value, including spikes and duplicates. Laboratory QC samples must also have unique identifiers. As noted in Section 1.6 above, for field samples, this should match the value which appears on the chain of custody. |
| 2 | lab_anl_method_name | Text(35) | Y | Laboratory analytic method name or description. |
| 3 | analysis_date | Date | Y | Date of sample analysis in MM/DD/YYYY format. |
| 4 | analysis_time | Text(5) | Y | Time of sample analysis in 24-hr (military) HH:MM format. |
| 5 | total_or_dissolved | Text(1) | Y | Type of analysis. Valid values include:<br>"T"=Total analysis;<br>"D"=Dissolved or Filtered analysis;<br>"N"=constituents for which neither "total" nor "dissolved" is applicable.<br>**This differs from the default EQuIS specification, which constrains the use of T and D to metals analyses**. |
| 6 | column_number | Text(2) | N | Column identifier for dual column analyses. |
| 7 | test_type | Text(10) | Y | Type of test. Valid values include:<br>"INITIAL";<br>“DILUTION”;<br>"REEXTRACT";<br>"REANALYSIS".<br>Contact DBA if other values are needed. |

| Pos# | Field Name | Data Type | Required | Comments |
|---|---|---|---|---|
| 8 | cas_rn<br>(CAS_Number) | Text(15) | Y | Unique analyte identifier. Use assigned CAS number when one is identified for an analyte.<br>Tentatively Identified Compounds (TICs) are not assigned a standard CAS number. The laboratory is required to assign a UNIQUE identifier for each TIC. The unique identifier must be placed in this field. Since retention time for TICs are unique per sample and sample analysis method, this information is the recommended value to use as the unique identifier. |
| 9 | chemical_name | Text(60) | Y | Chemical name as it appears in the lab pack. |
| 10 | result_value | Text(20) | N | Must only be a numeric value. It is stored as a string of characters so that significant digits can be retained. Must be identical with values presented in the hard copy.<br>It must be blank for non-detects. |
| 11 | result_error_delta | Text(20) | N | Error range applicable to the result value; typically used only for radiochemistry results. |
| 12 | result_type_code | Text(10) | Y | Type of result. Valid values include:<br>"TRG" for a target or regular result;<br>"TIC" for tentatively identified compounds;<br>"SUR" for surrogates;<br>"IS" for internal standards;<br>"SC" for spiked compounds. |
| 13 | reportable_result | Text(10) | Y | Valid values include:<br>"YES" for results which are reportable;<br>"NO" for other results.<br>For a given sample/method/analyte combination there should only be ONE result record with YES in the reportable_result field. |
| 14 | detect_flag | Text(2) | Y | Valid values include:<br>"Y" for detected analytes ;<br>"N" for non-detects. |
| 15 | lab_qualifiers | Text(7) | Y | Qualifier flags assigned by the laboratory in accordance with the CLP SOW documents (e.g., U=non-detect, not ND, not <). |

| Pos# | Field Name | Data Type | Required | Comments |
|---|---|---|---|---|
| 16 | organic_yn | Text(1) | Y | Valid values include:<br>"Y" for organic constituents;<br>"N" for inorganic constituents. |
| 17 | method_detection_limit | Text(20) | Y | Method Detection Limit (MDL). The MDL is the minimum amount of an analyte that can be routinely identified using a specific method. |
| 18 | reporting_detection_limit | Text(20) | Y | Practical Quantitation Limit (PQL). The PQL, defined in SW846 methods, is the lowest level that can be reliably achieved within specified limits of precision and accuracy during routine laboratory operating conditions. |
| 19 | quantitation_limit | Text(20) | Y | Sample quantitation limit (SQL). Per USEPA guidance, the SQL is the MDL adjusted to reflect sample-specific action such as dilution or use of a smaller sample aliquot for analysis due to matrix effects or the high concentration of some analytes. |
| 20 | result_unit | Text(15) | Y | Units of measurement for the result. |
| 21 | detection_limit_unit | Text(15) | N | Units of measurement for the detection limit(s). |
| 22 | TIC_retention_time | Text(8) | N | For tentatively identified compounds. May be used in the CAS number field to identify individual TICs as long as each retention time per sample per method of analysis is unique. |
| 23 | result_comment | Text(255) | N | Any comments related to the analysis. |
| 24 | qc_original_conc | Text(14) | N | The concentration of the analyte in the original (unspiked) sample. |
| 25 | qc_spike_added | Text(14) | N | The concentration of the analyte added to the original sample. |
| 26 | qc_spike_measured | Text(14) | N | The measured concentration of the analyte. Use zero for spiked compounds that were not detected in the sample. |
| 27 | qc_spike_recovery | Text(14) | N | The percent recovery calculated as specified by the laboratory QC program. Report as percentage value (e.g., report "120%" as "120", not 1.2). |
| 28 | qc_dup_original_conc | Text(14) | N | The concentration of the analyte in the original (unspiked) sample. |
| 29 | qc_dup_spike_added | Text(14) | N | The concentration of the analyte added to the original sample. |

| Pos# | Field Name | Data Type | Required | Comments |
|---|---|---|---|---|
| 30 | qc_dup_spike_measured | Text(14) | N | The measured concentration of the analyte in the duplicate. |
| 31 | qc_dup_spike_recovery | Text(14) | N | The duplicate percent recovery calculated as specified by the laboratory QC program. Report as percentage value (e.g., report "120%" as "120", not 1.2). |
| 32 | qc_rpd | Text(8) | N | The relative percent difference calculated as specified by the laboratory QC program. Report as percentage value (e.g., report "120%" as "120", not 1.2). |
| 33 | qc_spike_lcl | Text(8) | N | Lower control limit for spike recovery. Report as percentage value (e.g., report "120%" as "120", not 1.2). |
| 34 | qc_spike_ucl | Text(8) | N | Upper control limit for spike recovery. Report as percentage value (e.g., report "120%" as "120", not 1.2). |
| 35 | qc_rpd_cl | Text(8) | N | Relative percent difference control limit. Required for any duplicated sample. Report as percentage multiplied by 100 (e.g., report "120%" as "120"). |
| 36 | qc_spike_status | Text(10) | N | Used to indicate whether the spike recovery was within control limits. Use the "*" character to indicate failure, otherwise leave blank. Required for spikes, spike duplicates, surrogate compounds, LCS and any spiked sample. |
| 37 | qc_dup_spike_status | Text(10) | N | Used to indicate whether the duplicate spike recovery was within control limits. Use the "*" character to indicate failure, otherwise leave blank. |
| 38 | qc_rpd_status | Text(10) | N | Used to indicate whether the relative percent difference was within control limits. Use the "*" character to indicate failure, otherwise leave blank. Required for any duplicated sample. |

## 2.3 EQuIS EZ Result Import (aka EZEDD)

The EZEDD file should contain the required information for all samples, regardless of their source (e.g., field, lab). Information that is not marked required should be provided in all cases where the information is available.

| Pos# | Field Name | Data Type | Required | Comments |
|---|---|---|---|---|
| 1 | project_code | Text(20) | Y | Unique identifier assigned to a project site or delivery order. |
| 2 | sample_name | Text(30) | Y | Standardized sample name across all permutations. It is not required to be unique (i.e., duplicates are OK). As noted in Section 1.6 above, for field samples, this should match the value which appears on the chain of custody. |
| 3 | sys_sample_code | Text(40) | Y | Unique sample identifier. Each sample must have a unique value, including spikes and duplicates. Laboratory QC samples must also have unique identifiers. As noted in Section 1.6 above, for field samples, this should match the value which appears on the chain of custody. |
| 4 | sample_date | Date | N | Date sample was collected (in MM/DD/YYYY format for EDD). |
| 5 | sample_time | Text(5) | N | Time of sample collection in 24-hr (military) HH:MM format. |
| 6 | analysis_location | Text(2) | Y | Valid values include:<br>"FI" for field instrument or probe;<br>"FL" for mobile field laboratory analysis;<br>"LB" for fixed-based laboratory analysis.<br>Contact DBA if other values are needed. |
| 7 | lab_name_code | Text(20) | Y | Unique identifier of the laboratory. Must be consistent across all projects. |
| 8 | lab_sample_id | Text(20) | Y | Unique sample ID internally assigned by the laboratory. |

| Pos# | Field Name | Data Type | Required | Comments |
|---|---|---|---|---|
| 9 | sample_type_ code | Text(20) | Y | Code which distinguishes between different types of samples. For example, normal field samples must be distinguished from laboratory method blank samples, etc. See Section 3.2 to this document for the set of valid values. |
| 10 | lab_del_group | Text(20) | N | The lab job identifier, consistent with the labeling on the final report. Commonly referenced as Sample Delivery Group (SDG). |
| 11 | lab_batch_number | Text(20) | N | Sample preparation batch number assigned by the laboratory. |
| 12 | lab_anl_method_name | Text(35) | Y | Laboratory analytic method name or description. |
| 13 | cas_rn<br>(CAS_Number) | Text(15) | Y | Unique analyte identifier. Use assigned CAS number when one is identified for an analyte.<br>Tentatively Identified Compounds (TICs) are not assigned a standard CAS number. The laboratory is required to assign a UNIQUE identifier for each TIC. The unique identifier must be placed in this field. Since retention time for TICs are unique per sample and sample analysis method, this information is the recommended value to use as the unique identifier. |
| 14 | chemical_name | Text(60) | Y | Chemical name as it appears in the lab pack. |
| 15 | result_value | Text(20) | N | Must only be a numeric value. It is stored as a string of characters so that significant digits can be retained. Must be identical with values presented in the hard copy.<br>It must be blank for non-detects. |
| 16 | lab_qualifiers | Text(7 | N | Qualifier flags assigned by the laboratory in accordance with the CLP SOW documents (e.g., U=non-detect, not ND, not <). |
| 17 | result_unit | Text(15) | Y | Units of measurement for the result. |
| 18 | result_type_code | Text(10) | Y | Type of result. Valid values include:<br>"TRG" for a target or regular result;<br>"TIC" for tentatively identified compounds;<br>"SUR" for surrogates;<br>"IS" for internal standards;<br>"SC" for spiked compounds. |

| Pos# | Field Name | Data Type | Required | Comments |
|---|---|---|---|---|
| 19 | detect_flag | Text(2) | Y | Valid values include:<br>"Y" for detected analytes;<br>"N" for non-detects. |
| 20 | reporting_detection_limit | Text(20) | N | Practical Quantitation Limit (PQL). The PQL, defined in SW846 methods, is the lowest level that can be reliably achieved within specified limits of precision and accuracy during routine laboratory operating conditions. |
| 21 | dilution_factor | Single | N | Effective test dilution factor. |
| 22 | sample_matrix_code | Text(10) | Y | Code which distinguishes between different type of sample matrix. For example, blank samples must be distinguished from ground water samples, etc. See Section 3.1 to this document for the set of valid values. |
| 23 | total_or_dissolved | Text(1) | N | Type of analysis. Valid values include:<br>"T"=Total analysis;<br>"D"=Dissolved or Filtered analysis;<br>"N"=constituents for which neither "total" nor "dissolved" is applicable.<br>**This differs from the default EQuIS specification, which constrains the use of T and D to metals analyses**. |
| 24 | basis | Text(10) | Y | Valid values include:<br>"WET" for wet-weight basis reporting;<br>"DRY" for dry-weight basis reporting;<br>"NA" where this distinction is not applicable.<br>Contact DBA if other values are needed. |
| 25 | analysis_date | Date | N | Date of sample analysis in MM/DD/YYYY format. |
| 26 | analysis_time | Text(5) | N | Time of sample analysis in 24-hr (military) HH:MM format. |
| 27 | method_detection_limit | Text(20) | N | Method Detection Limit (MDL). The MDL is the minimum amount of an analyte that can be routinely identified using a specific method. |
| 28 | lab_prep_method_name | Text(35 | N | Description of sample prep or extraction method. |
| 29 | prep_date | Date | N | Date of sample preparation in MM/DD/YYYY. This field, in conjunction with extraction time, is used to determine whether holding times for field samples have been exceeded. |

| Pos# | Field Name | Data Type | Required | Comments |
|---|---|---|---|---|
| 30 | prep_time | Text(5) | N | Time of sample preparation in 24-hr (military) HH:MM format. This field, in conjunction with extraction date, is used to determine whether holding times for field samples have been exceeded. |
| 31 | test_batch_id | Text(20) | N | Unique identifier for all lab batches. Must be unique within EQuIS Chemistry database. For example, the same identifier can not be used for a prep batch and an analysis batch. |
| 32 | result_error | Text(20) | N | Applicable only when reporting radiological sample results. |
| 33 | TIC_retention_time | Text(8) | N | For tentatively identified compounds. May be used in the CAS number field to identify individual TICs as long as each retention time per sample per method of analysis is unique. |
| 34 | qc_level | Text(10) | N | Laboratory QC level associated with the analysis. |
| 35 | result_comment | Text(255) | N | Any comments related to the analysis. |
| 36 | parent_sample_code | Text(40) | N | The value of "sys_sample_code" that uniquely identifies the sample that was the source of this sample. For example, the value of this field for a duplicate sample would identify the normal sample of which this sample is a duplicate.<br><br>Required in the laboratory EDD for all laboratory "clone" samples (e.g., spikes and duplicates). Field duplicates may be submitted blind to the laboratory, so this field is not required in the laboratory EDD for field "clones". Must be blank for samples which have no parent (e.g., normal field samples, LCS samples, method blanks, etc.). |

# 3 Valid Values

These valid value lists may be amended on a project specific basis. A full set of valid values tables for use with EDP is available upon request.

## 3.1 Matrix Codes

| Matrix_code | Matrix_desc |
|---|---|
| AA | Ambient Air |
| GS | Soil Gas |
| LA | Aqueous Phase of a Multiple Phase Liquid or Solid Sample |
| LM | Multiple Phase Liquid Waste Sample |
| SC | Cement |
| SD | Drill Cuttings, Solid Matrix |
| SE | Sediment |
| SL | Sludge |
| SM | Water Filter (Solid Material used to filter Water) |
| SO | Soil |
| SQ | Soil/Solid Quality Control Matrix |
| SR | Water Filter Residue (Solid that gets filtered out of Water) |
| ST | Solid Waste |
| SW | Swab or Wipe |
| TA | Animal Tissue |
| TP | Plant Tissue |
| WA | Drill Cuttings, Aqueous Matrix |
| WC | Drilling Water (Used for Well Construction) |
| WD | Well Development Water |
| WG | Ground Water |
| WH | Equipment Wash Water, i.e., Water used for Washing |
| WL | Leachate |
| WO | Ocean Water |
| WP | Potable (i.e., Drinking) Water |
| WQ | Water Quality Control Matrix |
| WS | Surface Water |
| WV | Water From Vadose Zone |
| WW | Waste Water |

## 3.2 Sample Types

| Sample_type_code | Sample_type_desc | Sample Source |
|---|---|---|
| EB | Equipment Blank | Field |
| FD | Field Duplicate | Field |
| FS | Field Spike | Field |
| N | Normal Environmental Sample | Field |
| RB | Material Rinse Blank | Field |
| RD | Regulatory Duplicate | Field |
| TB | Trip Blank | Field |
| AB | Ambient Conditions Blank | Lab |
| BD | Blank Spike Duplicate | Lab |
| BS | Blank Spike | Lab |
| BSD | Blank Spike and Duplicate considered as one sample | Lab |
| LB | Lab Blank | Lab |
| LR | Lab Replicate | Lab |
| MB | Material Blank | Lab |
| MS | Lab Matrix Spike | Lab |
| MSD | Lab Matrix Spike Duplicate, pair considered as one sample | Lab |
| SD | Lab Matrix Spike Duplicate | Lab |

# 4 Appendix A

## *EarthSoft EDD Format Definition*
## *EQuIS Chemistry 4 File EDD*

**EQuIS Chemistry 4 File Import Format (EFWEDD)**

# EQuIS Chemistry 4 File EDD

Version 11e – 8/23/2004
Provided by EarthSoft, Inc.
Spreadsheet Templates: EFWEDD01.xls
Former Title: Analytical Results - Electronic Data Transfer Format

## Table of Contents



 

## EQuIS Chemistry 4 File Import Format (EFWEDD)

## Introduction

The purpose of this document is to describe the 4 file import templates available in EQuIS Chemistry. The Electronic Data Deliverable, or EDD, referred to is EFWEDD01.xls. This Microsoft Excel spreadsheet contains 5 tabs, each with a format for importing various data into different parts of the EQuIS Chemistry data structure. Each template has a corresponding import format available by the same name in the EQuIS Chemistry General Import module. It should be noted that, although there are two sample formats, EFW2FSample and EFW2LabSMP, only one should be used, depending on the type of data to be imported. It is also noted that the EDD is simply a data format. EarthSoft distributes the format as a Microsoft Excel document, but it could be created in Lotus or any other spreadsheet. Ultimately, the files that are actually imported into EQuIS Chemistry must be saved from the EDD as text (.txt) or comma-delimited (.csv) files, terminated with a carriage return.

In the following tables, fields with **Y** in the **Req** column are required but are not part of the key. Fields with **Y/K** in the **Req** column are part of the key and are used to determine the uniqueness of the row in the EDD file. The designation "FK (table_name)" in the Description column indicate that the field is a foreign key to the specified table; the data value in this field must exist in the table indicated. Column headers with the names of the fields may be included. A second header line with the column numbers may also be included. The header lines are not required.

Questions about this document or the Geo3EDD may be referred to the EarthSoft Help Desk at help@earthsoft.com.

## General Information

### File Format

All data from the laboratory must be stored in an ASCII file using the following standard format. Each data field must be either separated by tabs or enclosed in double quotes (") and separated by commas. Data fields with no information may be represented by two commas. Maximum length of text fields is indicated in the parentheses. If the information is less than the maximum length, do not pad the record with spaces.

Each record must be terminated with a carriage return/line feed (i.e., standard DOS text file). The file can be produced using any software with the capability to create ASCII files. Date is reported as MM/DD/YY (month/day/year) and time as HH:MM (hour:minute). Time uses a 24 hour clock, thus 3:30 p.m. will be reported as 15:30.

Four files are required: one each for samples, tests, results, and batches, although the user may choose to utilize the Field Sample import format (EFW2Fsample), for importing field sample data. The filename extensions are used to indicate the file type as follows:

*.SMP for sample rows
*.TST for test rows
*.RES for result rows
*.BCH for batch rows

The character portion of the filenames must be the same for each group of four files. Filename conventions may be defined however the laboratory and EQuIS Chemistry project manger determine. For example, the date, sample delivery group, or project name may be encoded in the filename if desired. Although we anticipate that all four files will be prepared and loaded into EQuIS Chemistry together in one group, this is not necessary. Each file can be loaded separately if desired.

## EQuIS Chemistry 4 File Import Format (EFWEDD)

### Data Integrity Rules

If a field is to be considered part of the primary key of a table, it is indicated below by the presence of "PK" in the *PK* column. The combination of values in each primary key must be unique within the file. Also, referential integrity must be enforced between tables. That is, the values of `sys_sample_code` present in the Result and Test tables must also be present in the Sample table. Logical relationships between the tables are shown in the entity relationship diagram, which is available from the EarthSoft Help Desk.

The key fields in the test table may appear complicated, so they are discussed further here. The EQuIS Chemistry user has the flexibility to choose uniqueness constraints on the analytic test event table (i.e. `dt_test`). By default, only two fields are defined as part of a unique key: `sys_sample_code` and `lab_anl_method_name`. This means that each combination of sample ID and lab method can be used to uniquely define a lab test event. For example, by default a given combination of sample ID and lab method may have only one analysis date or dilution factor. Other users might wish to store retests or re-dilutions as separate test events. One way to achieve this would be to include `analysis_date` as part of the unique key of `dt_test`. This would allow multiple occurrences of a given combination of sample ID and lab method, provided that analysis date is different for each retest. Other common situations are discussed below. The fields that may be included as part of a unique key on `dt_test` are indicated below by the presence of "PK?" in the *PK* column. If these fields are part of the uniqueness constraint needed by the EQuIS Chemistry user, then they must be required in the EDD. This is indicated by the symbol Y/K? in the *Required* column of the tables shown below.

A. Some EQuIS Chemistry users intend to import the full suite of test level information, including column_ number and analysis_time. Other users do not need these fields. If these two fields are not required by the EQuIS Chemistry user, than this field may be left null (i.e., empty).

B. Some metal analyses can be done on unfiltered samples (to obtain total concentrations) or can be done on filtered samples (to get dissolved concentrations). Some EQuIS Chemistry users may choose to distinguish between these types of tests by using different method names. However, other users need to use the same method name value for both of these tests, and therefore require another field to distinguish between these test types. If the total_or_dissolved field is not required to distinguish these types of tests, than this field may be left null (i.e., empty).

### Null Format

Many fields are optional, and the list of valid values may be defined in a project or lab specific manner, as determined by the laboratory and EQuIS Chemistry project manager. When a field is <u>not</u> listed as required, this means that a null or blank may be appropriate. However, the blank value must still be surrounded by commas. In other words, the number of fields is always the same, whether or not the fields include data is optional. Refer to the example below where the second of three fields shown below is considered optional,

"Data-one","Data-two","Data-three",…→OK
"Data-one","Data-three",…→Not OK
"Data-one",,"Data-three",…→OK

## EQuIS Chemistry 4 File Import Format (EFWEDD)

### Necessary Steps

Several decisions must be made by the lab and by the EQuIS Chemistry users before the EDDs are prepared. These decisions include the following:

1. Decide if analysis_date, test_type, column_number, total_or_dissolved, and analysis_time may be left blank (see above discussion). This decision must apply for the duration of the EQuIS Chemistry project. This decision must correspond to the unique index defined by the user for the project.

2. Decide whether a *controlled vocabulary* is needed for lab_anl_method_name and provide to lab if necessary (EQuIS Chemistry can manage lab_anl_method_name aliases internally, and the lab does not necessarily need to use controlled vocabulary). By controlled vocabulary, we mean an explicit list of valid values for a field. For example, a list of valid analytic method names might include "SW8240" but not "SW-8240" nor "EPA 8240".

3. Decide whether a controlled vocabulary is needed for prep_method and provide to lab if necessary (EQuIS Chemistry can manage prep_method aliases internally, and the lab does not necessarily need to use controlled vocabulary).

4. Select the controlled vocabulary for cas_rn (required by EQuIS Chemistry).

5. Decide whether the following “optional” fields will be required:

   **Sample level optional fields**
   comment
   sample_date
   sample_time
   sample_receipt_date
   sample_delivery_group
   standard_solution_source
   sample_receipt_time

   **Test level optional fields**
   lab_matrix_code
   analysis_location
   basis
   container_id
   dilution_factor
   prep_method
   prep_date
   prep_time
   leachate_method
   leachate_date
   leachate_time
   lab_name_code
   qc_level
   lab_sample_id
   percent_moisture
   subsample_amount
   subsample_amount_unit
   analyst_name
   instrument_id
   comment
   preservative

## EQuIS Chemistry 4 File Import Format (EFWEDD)

final_volume
final_volume_unit

**Result level optional fields**
result_error_delta
lab_qualifiers
organic_yn
method_detection_limit
reporting_detection_limit
quantitation_limit
detection_limit_unit
tic_retention_time
result_comment
qc_original_conc
qc_spike_added
qc_spike_measured
qc_spike_recovery
qc_dup_original_conc
qc_dup_spike_added
qc_dup_spike_measured
qc_dup_spike_recovery
qc_rpd
qc_spike_lcl
qc_spike_ucl
qc_rpd_cl
qc_spike_status
qc_dup_spike_status
qc_rpd_status

## EQuIS Chemistry 4 File Import Format (EFWEDD)

### Examples

*QC fields in a normal field sample (i.e., Sample_type_code = N, TB, etc.)*

The following table shows some of the fields in the result file for a normal field sample. Notice that all QC fields are blank.

| cas_rn | result value | qc original conc | qc spike added | qc spike measured | qc spike recovery | qc dup original conc | qc dup spike added | qc dup spike measured | qc dup spike recovery |
|---|---|---|---|---|---|---|---|---|---|
| 93-76-5 | 1.56 | | | | | | | | |
| 94-75-7 | 3.17 | | | | | | | | |
| 94-82-6 | 2.31 | | | | | | | | |

*QC fields in a normal field sample with surrogates (i.e., Sample_type_code = N, TB, etc.)*

The following table shows some of the fields in the result file for a normal field sample. Notice that QC fields are blank except on surrogate rows. Many users will need to complete only the recovery field data; the spike added and spike measured fields will not be needed in most situations.

| Cas_rn | result value | result unit | result type code | qc original conc | qc spike added | qc spike measured | qc spike recovery |
|---|---|---|---|---|---|---|---|
| 93-76-5 | 1.56 | mg/l | TRG | | | | |
| 94-75-7 | 3.17 | mg/l | TRG | | | | |
| PHEN2F | | mg/l | SUR | | 12.5 | 12.9 | 103 |

*QC fields in a matrix spike (i.e., Sample_type_code = MS)*

The following table shows some of the fields in the result file for a matrix spike sample. Notice that all "dup" QC fields are blank, and that the result_value field is not needed. Also, the qc_rpd field would be blank for these rows. Many users will need to complete only the calculated recovery field.

| Cas_rn | result value | qc original conc | qc spike added | qc spike measured | qc spike recovery | qc dup original conc | qc dup spike added | qc dup spike measured | qc dup spike recovery |
|---|---|---|---|---|---|---|---|---|---|
| 93-76-5 | | 1.56 | 4.18 | 5.36 | 90.9 | | | | |
| 94-75-7 | | 3.17 | 4.18 | 7.15 | 95.2 | | | | |
| 94-82-6 | | 2.31 | 4.22 | 5.66 | 79.3 | | | | |

*QC fields in a matrix spike duplicate (i.e., Sample_type_code = SD)*

The table on the following page shows some of the fields in the result file for a matrix spike duplicate sample. Notice that all "dup" QC fields are completed, and that the result_value field is not needed. Also, the qc_rpd field would be completed for these rows. Many users will need to complete only the calculated recovery field.

## EQuIS Chemistry 4 File Import Format (EFWEDD)

| cas_rn | result value | qc original conc | qc spike added | qc spike measured | qc spike recovery | qc dup original conc | qc dup spike added | qc dup spike measured | qc dup spike recovery |
|---|---|---|---|---|---|---|---|---|---|
| 93-76-5 | | | | | | 1.56 | 4.23 | 5.70 | 97.8 |
| 94-75-7 | | | | | | 3.17 | 4.23 | 7.62 | 105 |
| 94-82-6 | | | | | | 2.31 | 4.13 | 5.33 | 73.1 |

*QC fields in a matrix spike/matrix spike duplicate (i.e., Sample_type_code = MSD)*

The following table shows some of the fields in the result file for a matrix spike/matrix spike duplicate considered as single sample (they can be reported this way, or as two separate samples as shown above). Notice that all QC fields are completed, and that the result_value field is not needed. Also, the qc_rpd field would be completed for these rows. Many users will need to complete only the calculated recovery field.

| Cas_rn | result value | qc original conc | qc spike added | qc spike measured | qc spike recovery | qc dup original conc | qc dup spike added | qc dup spike measured | qc dup spike recovery |
|---|---|---|---|---|---|---|---|---|---|
| 93-76-5 | | 1.56 | 4.18 | 5.36 | 90.9 | 1.56 | 4.23 | 5.70 | 97.8 |
| 94-75-7 | | 3.17 | 4.18 | 7.15 | 95.2 | 3.17 | 4.23 | 7.62 | 105 |
| 94-82-6 | | 2.31 | 4.22 | 5.66 | 79.3 | 2.31 | 4.13 | 5.33 | 73.1 |

*QC fields in an LCS (i.e., laboratory control sample, blank spike, Sample_type_code = BS)*

The following table shows some of the fields in the result file for an LCS sample. The qc_rpd field would be blank for these rows. Many users will need to complete only the calculated recovery field. LCS duplicate samples (i.e., Sample_type_code = BD) and LCS/LCSD samples (i.e., Sample_type_code = BSD) follow the patterns similar to the SD and MSD samples described above.

| Cas_rn | result value | qc original conc | qc spike added | qc spike measured | qc spike recovery | qc dup original conc | qc dup spike added | qc dup spike measured | qc dup spike recovery |
|---|---|---|---|---|---|---|---|---|---|
| 93-76-5 | | | 5.00 | 5.26 | 105 | | | | |
| 94-75-7 | | | 1.00 | 1.02 | 102 | | | | |
| 94-82-6 | | | 12.5 | 12.9 | 103 | | | | |

*Retests*

The following table shows how to report retests in an example where a sample was retested at dilution. The end user would see the first two constituents (75-25-2, and 67-66-3) in the initial test, and constituent 95-95-4 in the diluted retest. The other results would be "turned off" by setting the reportable_result field to "No". Note that the user might not require this level of detail. In such cases, the rows flagged below as not reportable would not need to be included in the EDD.

| Test_type | cas_rn | result_value | reportable_result |
|---|---|---|---|
| initial | 75-25-2 | 1.2 | Yes |
| initial | 67-66-3 | 3.4 | Yes |
| initial | 95-95-4 | 100 | No |
| retest | 75-25-2 | 0 | No |
| retest | 67-66-3 | 0 | No |
| retest | 95-95-4 | 78.3 | Yes |

*Second Columns*

The following table shows how to report first and second column confirmation results. The end user would see the first and third constituents (75-25-2, and 95-95-4) as "primary" in the first column, and constituent 67-66-3 as "primary" in the second column. The other results would be "turned off" by setting the reportable_result field to "No". Note that the user might not require this level of detail. In such cases, the rows flagged below as not reportable would not need to be included in the EDD, and the test could be set to "NA".

| test_type | cas_rn | result_value | reportable_result |
|---|---|---|---|
| 1C | 75-25-2 | 1.2 | Yes |
| 1C | 67-66-3 | 3.4 | No |
| 1C | 95-95-4 | 5.6 | Yes |
| 2C | 75-25-2 | 1.3 | No |
| 2C | 67-66-3 | 3.7 | Yes |
| 2C | 95-95-4 | 5.4 | No |

## EQuIS Chemistry 4 File Import Format (EFWEDD)

### Field Sample Import Format

| Pos# | Field Name | DataType | PK | Required | Field Definition |
|---|---|---|---|---|---|
| 1 | sys_sample_code | Text(40) | PK | Y/K | Unique sample identifier. Each sample must have a unique value, including spikes and duplicates. Laboratory QC samples must also have unique identifiers. The laboratory and the Chem user have considerable flexibility in the methods they use to derive and assign unique sample identifiers, but uniqueness throughout the database is the only restriction enforced by Chem. |
| 2 | sample_name | Text(30) | | N | Additional sample identification information as necessary. Is not required to be unique (i.e., duplicates are OK). |
| 3 | sample_matrix_code | Text(10) | | Y | Code which distinguishes between different type of sample matrix. For example, soil samples must be distinguished from ground water samples, etc. IRPIMS-style sample matrix codes are understood by Chem, and other valid sample types can be added by the Chem user. The matrix of the sample as analyzed may be different from the matrix of the sample as retrieved (e.g. leachates), so this field is required at both the sample and test level. |
| 4 | sample_type_code | Text(20) | | Y | Code which distinguishes between different types of samples. For example, normal field samples must be distinguished from laboratory method blank samples, etc. IRPIMS-style sample type codes (see table X01-SA) are understood by Chem, and other valid sample |
| 5 | sample_source | Text(10) | | Y | This field identifies where the sample came from, either **Field** or **Lab**. |
| 6 | parent_sample_code | Text(40) | | N | The value of "sys_sample_code" that uniquely identifies the sample that was the source of this sample. For example, the value of this field for a duplicate sample would identify the normal sample of which this sample is a duplicate. Required in the laboratory EDD for all laboratory "clone" samples (e.g., spikes and duplicates). Field duplicates may be submitted blind to the laboratory, so this field is not required in the laboratory EDD for field "clones". Must be blank for samples which have no parent (e.g., normal field samples, LCS samples, method blanks, etc.). |

## EQuIS Chemistry 4 File Import Format (EFWEDD)

| Pos# | Field Name | DataType | PK | Required | Field Definition |
|---|---|---|---|---|---|
| 7 | sample_delivery_group | Text(10) | | N | Sample delivery group as defined by Chem project manager. This is an optional field for the laboratory EDD unless otherwise specified by the Chem project manager. |
| 8 | sample_date | Date | | N | Date sample was collected (in MM/DD/YY format for EDD). |
| 9 | sample_time | Time | | N | Time of sample collection in 24-hr (military) HH:MM format. |
| 10 | sys_loc_code | Text(20) | | N | Sample collection location. |
| 11 | start_depth | Double | | N | Beginning depth (top) of soil sample. This is an optional field for the laboratory EDD unless otherwise specified by the Chem project manager. |
| 12 | end_depth | Double | | N | Ending depth (bottom) of soil sample. This is an optional field for the laboratory EDD unless otherwise specified by the Chem project manager. |
| 13 | depth_unit | Text(15) | | N | Unit of measurement for the sample begin and end depths. IRPIMS-style unit of measurement codes (see table X03) are recognized by Chem; other codes may be allowed by the Chem project manager. This is an optional field for the laboratory EDD unless otherwise specified by the Chem project manager. |
| 14 | chain_of_custody | Text(15) | | N | Chain of custody identifier. A single sample may be assigned to only one chain of custody. This is an optional field for laboratory EDD unless otherwise specified by the Chem project manager. |
| 15 | sent_to_lab_date | Date | | N | Date sample was sent to lab (in MM/DD/YY format for EDD). Not included in the laboratory EDD. |
| 16 | sample_receipt_date | Date | | N | Date that sample was received at laboratory (in MM/DD/YY format for EDD). |
| 17 | sampler | Text(30) | | N | Name or initials of sampler. Not included in the laboratory EDD. |
| 18 | sampling_company_code | Text(10) | | N | Name or initials of sampling company (no controlled vocabulary). Not included in the laboratory EDD. |
| 19 | sampling_reason | Text(30) | | N | Optional reason for sampling. No controlled vocabulary is enforced. Not included in the laboratory EDD. |

## EQuIS Chemistry 4 File Import Format (EFWEDD)

| Pos# | Field Name | DataType | PK | Required | Field Definition |
|---|---|---|---|---|---|
| 20 | sampling_technique | Text(40) | | N | Sampling technique (no controlled vocabulary). Not included in the laboratory EDD. |
| 21 | task_code | Text(10) | | N | Code used to identify the task under which the field sample was retrieved. This is an optional field for laboratory EDD unless otherwise specified by the Chem project manager. |
| 22 | collection_quarter | Text(5) | | N | Quarter of the year sample was collected (e.g., "1Q96") Not included in the laboratory EDD. |
| 23 | composite_yn | Text(1) | | N | Boolean field used to indicate whether a sample is a composite sample. Not included in the laboratory EDD. |
| 24 | composite_desc | Text(255) | | N | Description of composite sample (if composite_yn is YES). Not included in the laboratory EDD. |
| 25 | sample_class | Text(10) | | N | Navy sample class code. Not included in the laboratory EDD. |
| 26 | custom_field_1 | Text(255) | | N | Custom sample field |
| 27 | custom_field_2 | Text(255) | | N | Custom sample field |
| 28 | custom_field_3 | Text(255) | | N | Custom sample field |
| 29 | comment | Text(255) | | N | Sample comments as necessary (optional). |
| 30 | sample_receipt_time | Text(5) | | N | Time of lab receipt sample in 24-hr (military) HH:MM format |

## Sample Import Format

| Pos# | Field Name | DataType | PK | Required | Field Definition |
|---|---|---|---|---|---|
| 1 | sys_sample_code | Text(40) | PK | Y/K | Unique sample identifier. Each sample must have a unique value, including spikes and duplicates. Laboratory QC samples must also have unique identifiers. The laboratory and the EQuIS Chemistry user have considerable flexibility in the methods they use to derive and assign unique sample identifiers, but uniqueness throughout the database is the only restriction enforced by EQuIS Chemistry. |
| 2 | sample_type_code | Text(20) | | Y | Code which distinguishes between different types of sample. For example, normal field samples must be distinguished from laboratory method blank samples, etc. IRPIMS-style sample type codes (see table X01) are understood by EQuIS Chemistry, and other valid sample types can be added by the |

## EQuIS Chemistry 4 File Import Format (EFWEDD)

| Pos# | Field Name | DataType | PK | Required | Field Definition |
|---|---|---|---|---|---|
| | | | | | EQuIS Chemistry user. Field sample types (e.g., field duplicates, field blanks, etc.) might be submitted blind to the laboratory; in such cases the laboratory may report all field samples as if they were all normal field samples. The laboratory is not required to export data for a spike if a spike duplicate is exported (unless the EQuIS Chemistry project manager requests all spikes). |
| 3 | sample_matrix_code | Text(10) | | Y | Code which distinguishes between different types of sample matrix. For example, soil samples must be distinguished from ground water samples, etc. IRPIMS-style sample matrix codes (see table X02) are understood by EQuIS Chemistry, and other valid sample types can be added by the EQuIS Chemistry user. The matrix of the sample as analyzed may be different from the matrix of the sample as retrieved (e.g. leachates), so this field is required at the sample level. |
| 4 | sample_source | Text(10) | | Y | Must be either "Field" for field samples or "Lab" for internally generated laboratory QC samples. No other values are allowed. For example, a matrix spike duplicate sample would be a "Lab" sample, while its parent (i.e., the field sample it was derived from) would be a "Field" sample. |
| 5 | parent_sample_code | Text(40) | | N | The value of "sys_sample_code" that uniquely identifies the sample that was the source of this sample. For example, the value of this field for a duplicate sample would identify the normal sample of which this sample is a duplicate. Required in the laboratory EDD for all laboratory "clone" samples (e.g., spikes and duplicates). Field duplicates may be submitted blind to the laboratory, so this field is not required in the laboratory EDD for field "clones". Must be blank for samples which have no parent (e.g., normal field samples, LCS samples, method blanks, etc.). This field must be filled out for those samples which have "parents". |
| 6 | comment | Text(255) | | N | Sample comments as necessary (optional). |
| 7 | sample_date | Date | | N | Date of sample collection in MM/DD/YY format. Must be blank for laboratory samples. |
| 8 | sample_time | Text(5) | | N | Time of sample collection in 24-hr (military) HH:MM format. Must be blank for laboratory samples. |
| 9 | sample_receipt_date | Date | | N | Date that sample was received at laboratory in MM/DD/YY format. Must be blank for laboratory samples. |

## EQuIS Chemistry 4 File Import Format (EFWEDD)

| Pos# | Field Name | DataType | PK | Required | Field Definition |
|---|---|---|---|---|---|
| 10 | sample_delivery_group | Text(10) | | N | Sample delivery group as defined by EQuIS Chemistry project manager. This is an optional field for the laboratory EDD unless otherwise specified by the EQuIS Chemistry project manager. Must be blank for laboratory samples. |
| 11 | standard_solution_ source | Text(20) | | N | Relevant only for laboratory-generated samples. Textual description of the source of standard solutions as needed for certain laboratory samples (e.g., LCS). Optional as far as the EQuIS Chemistry database is concerned, although it could possibly be required from the laboratory for certain projects. Must be blank for field samples. |
| 12 | sample_receipt_time | Text(5) | | N | Time that sample was received at laboratory in 24-hr (military) HH:MM format. Must be blank for laboratory samples. |

## Test Import Format

| Pos# | Field Name | DataType | PK | Required | Field Definition |
|---|---|---|---|---|---|
| 1 | sys_sample_code | Text(40) | PK | Y/K | Unique sample identifier. Each sample must have a unique value, including spikes and duplicates. Laboratory QC samples must also have unique identifiers. The laboratory and the EQuIS Chemistry user have considerable flexibility in the methods they use to derive and assign unique sample identifiers, but uniqueness throughout the database is the only restriction enforced by EQuIS Chemistry. |
| 2 | lab_anl_method_ name | Text(35) | PK | Y/K | Laboratory analytic method name or description. A controlled vocabulary (i.e., list of valid method names) is not required for the laboratory EDD unless otherwise specified by the EQuIS Chemistry project manager. The method name should be sufficient to reflect operation of the laboratory. For example both "SW8080-pest" and "SW8080-PCB" may be necessary to distinguish between laboratory methods, while "SW8080" may not provide sufficient detail. |
| 3 | analysis_date | Date | PK? | Y/K? | Date of sample analysis in MM/DD/YY format. May refer to either beginning or end of the analysis as required by EQuIS Chemistry project manager. This field is not always required, but most users will want it. |
| 4 | analysis_time | Text(5) | PK? | Y/K? | Time of sample analysis in 24-hr (military) HH:MM format. May refer to either beginning or |

## EQuIS Chemistry 4 File Import Format (EFWEDD)

| Pos# | Field Name | DataType | PK | Required | Field Definition |
|---|---|---|---|---|---|
| | | | | | end as required by EQuIS Chemistry project manager. This field might be required, depending on the test primary key used by the EQuIS Chemistry user. Note that this field, combined with the "analysis_date" field is used to distinguish between retests and reruns (if reported). Please ensure that retests have "analysis_date" and/or "analysis_time" different from the original test event (and fill out the test_type field as needed). |
| 5 | total_or_dissolved | Text(1) | PK? | Y/K? | If required, then it must be either "T" for total [metal] concentration, "D" for dissolved or filtered [metal] concentration, or "N" for organic (or other) constituents for which neither "total" nor "dissolved" is applicable. This field might be required, depending on the test primary key used by the EQuIS Chemistry user. |
| 6 | column_number | Text(2) | PK? | Y/K? | If required, then it must be either "1C" for first column analyses, "2C" for second column analyses, or "NA" for analyses for which neither "1C" nor "2C" is applicable. Second column data may not be required, depending on the needs identified by the EQuIS Chemistry project manager, in which case all results may be reported as "NA". However, if any "2C" tests are reported, then there must be corresponding "1C" tests present also. Also, laboratories typically can report which of the two columns is to be considered "primary". This distinction is handled by the "reportable_result" field in the result table. This field might be required, depending on the test primary key used by the EQuIS Chemistry user. |
| 7 | test_type | Text(10) | PK? | Y/K? | Type of test. Valid values include "initial", "reextract", and "reanalysis". |
| 8 | lab_matrix_code | Text(10) | | N | Code which distinguishes between different type of sample matrix. For example, soil samples must be distinguished from ground water samples, etc. IRPIMS-style sample matrix codes (see table X02) are understood by EQuIS Chemistry, and other valid sample types can be added by the EQuIS Chemistry user. The matrix of the sample as analyzed may be different from the matrix of the sample as retrieved (e.g. leachates), so this field is available at both the sample and test level. |
| 9 | analysis_location | Text(2) | | N | If required, then it must be either "FI" for field instrument or probe, "FL" for mobile field laboratory analysis, or "LB" for fixed-based laboratory analysis. |

## EQuIS Chemistry 4 File Import Format (EFWEDD)

| Pos# | Field Name | DataType | PK | Required | Field Definition |
|---|---|---|---|---|---|
| 10 | basis | Text(10) | | N | If required, then it must be either "Wet" for wet-weight basis reporting, "Dry" for dry-weight basis reporting, or "NA" for tests for which this distinction is not applicable. The EQuIS Chemistry project manager may require that all results must be reported under a particular basis. |
| 11 | container_id | Text(30) | | N | Sample container identifier. This is an optional field for the laboratory EDD unless otherwise specified by the EQuIS Chemistry project manager. |
| 12 | dilution_factor | Single | | N | Effective test dilution factor. |
| 13 | prep_method | Text(35) | | N | Laboratory sample preparation method name or description. A controlled vocabulary (i.e., list of valid method names) is not required for the laboratory EDD unless otherwise specified by the EQuIS Chemistry project manager. The method name should be sufficient to reflect operation of the laboratory (see analysis method discussion). |
| 14 | prep_date | Date | | N | Date of sample preparation in MM/DD/YY format. May refer to either beginning or end as required by EQuIS Chemistry project manager. |
| 15 | prep_time | Text(5) | | N | Time of sample preparation in 24-hr (military) HH:MM format. May refer to either beginning or end as required by EQuIS Chemistry project manager. |
| 16 | leachate_method | Text(15) | | N | Laboratory leachate generation method name or description. A controlled vocabulary (i.e., list of valid method names) is not required for the laboratory EDD unless otherwise specified by the EQuIS Chemistry project manager. The method name should be sufficient to reflect operation of the laboratory (see analysis method discussion). |
| 17 | leachate_date | Date | | N | Date of leachate preparation in MM/DD/YY format. May refer to either beginning or end as required by EQuIS Chemistry project manager. |
| 18 | leachate_time | Text(5) | | N | Time of leachate preparation in 24-hr (military) HH:MM format. May refer to either beginning or end as required by EQuIS Chemistry project manager. |
| 19 | lab_name_code | Text(10) | | N | Unique identifier of the laboratory as defined by the EQuIS Chemistry project manager. |
| 20 | qc_level | Text(10) | | N | Data validation QC level. This is an optional field |

## EQuIS Chemistry 4 File Import Format (EFWEDD)

| Pos# | Field Name | DataType | PK | Required | Field Definition |
|---|---|---|---|---|---|
| | | | | | for the laboratory EDD unless otherwise specified by the EQuIS Chemistry project manager. EQuIS Chemistry does not enforce a controlled vocabulary on the values of this field, although a list of valid values may optionally be provided by the EQuIS Chemistry project manager. |
| 21 | lab_sample_id | Text(20) | | N | Laboratory LIMS sample identifier. Required. If necessary, a field sample may have more than one LIMS lab-sample-id (maximum one per each test event). |
| 22 | percent_moisture | Text(5) | | N | Percent moisture of the sample portion used in this test; this value may vary from test to test for any sample. Numeric format is "NN.MM", i.e., 70.1% could be reported as "70.1" but not as "70.1%". This is an optional field for the laboratory EDD unless otherwise specified by the EQuIS Chemistry project manager. |
| 23 | subsample_amount | Text(14) | | N | Amount of sample used for test. This is an optional field for the laboratory EDD unless otherwise specified by the EQuIS Chemistry project manager. |
| 24 | subsample_amount_unit | Text(15) | | N | Unit of measurement for subsample amount. IRPIMS-style unit of measurement codes (see table X02) are recognized by EQuIS Chemistry; other codes may be allowed by the EQuIS Chemistry project manager. This is an optional field for the laboratory EDD unless otherwise specified by the EQuIS Chemistry project manager. This is an optional field for the laboratory EDD unless otherwise specified by the EQuIS Chemistry project manager. |
| 25 | analyst_name | Text(30) | | N | Name or initials of laboratory analyst. This is an optional field for the laboratory EDD unless otherwise specified by the EQuIS Chemistry project manager. |
| 26 | instrument_id | Text(50) | | N | Instrument identifier. This is an optional field for the laboratory EDD unless otherwise specified by the EQuIS Chemistry project manager. |
| 27 | comment | Text(255) | | N | Comments about the test as necessary. This is an optional field for the laboratory EDD unless otherwise specified by the EQuIS Chemistry project manager. |

## EQuIS Chemistry 4 File Import Format (EFWEDD)

| Pos# | Field Name | DataType | PK | Required | Field Definition |
|---|---|---|---|---|---|
| 28 | preservative | Text(50) | | N | Sample preservative used. |
| 29 | final_volume | Text(15) | | N | The final amount of the sample after sample preperation. |
| 30 | final_volume_unit | Text(15) | | N | The unit of measure that corresponds to the final_amount. |

## Result Import Format

| # | Field Name | Type | PK | Required | Field Definition |
|---|---|---|---|---|---|
| 1 | sys_sample_code | Text(40) | PK | Y/K | Unique sample identifier. Each sample must have a unique value, including spikes and duplicates. Laboratory QC samples must also have unique identifiers. The laboratory and the EQuIS Chemistry user have considerable flexibility in the methods they use to derive and assign unique sample identifiers, but uniqueness throughout the database is the only restriction enforced by EQuIS Chemistry. |
| 2 | lab_anl_method_name | Text(35) | PK | Y/K | Laboratory analytic method name or description. A controlled vocabulary (i.e., list of valid method names) is not required for the laboratory EDD unless otherwise specified by the EQuIS Chemistry project manager. The method name should be sufficient to reflect operation of the laboratory. For example both "SW8080-pest" and "SW8080-PCB" may be necessary to distinguish between laboratory methods, while "SW8080" may not provide sufficient detail. |
| 3 | analysis_date | Date | PK? | Y/K? | Date of sample analysis in MM/DD/YY format. May refer to either beginning or end of the analysis as required by EQuIS Chemistry project manager. This field is not always required, but most users will want it. |
| 4 | analysis_time | Text(5) | PK? | Y/K? | Time of sample analysis in 24-hr (military) HH:MM format. May refer to either beginning or end as required by EQuIS Chemistry project manager. This field might be required, depending on the test primary key used by the EQuIS Chemistry user. Note that this field, combined with the "analysis_date" field is used to distinguish between retests and reruns (if reported). Please ensure that retests have "analysis_date" and/or "analysis_time" different from the original test event (and fill out the test_type field as needed). |

## EQuIS Chemistry 4 File Import Format (EFWEDD)

| # | Field Name | Type | PK | Required | Field Definition |
|---|---|---|---|---|---|
| 5 | total_or_dissolved | Text(1) | PK? | Y/K? | If required, then it must be either "T" for total [metal] concentration, "D" for dissolved or filtered [metal] concentration, or "N" for organic (or other) constituents for which neither "total" nor "dissolved" is applicable. This field might be required, depending on the test primary key used by the EQuIS Chemistry user. |
| 6 | column_number | Text(2) | PK? | Y/K? | If required, then it must be either "1C" for first column analyses, "2C" for second column analyses, or "NA" for analyses for which neither "1C" nor "2C" is applicable. Second column data may not be required, depending on the needs identified by the EQuIS Chemistry project manager, in which case all results may be reported as "NA". However, if any "2C" tests are reported, then there must be corresponding "1C" tests present also. Also, laboratories typically can report which of the two columns is to be considered "primary". This distinction is handled by the "reportable_result" field in the result table. This field might be required, depending on the test primary key used by the EQuIS Chemistry user. |
| 7 | test_type | Text(10) | PK? | Y/K? | Type of test. Valid values include "initial", "reextract", and "reanalysis". |
| 8 | cas_rn | Text(15) | PK | Y | Chemical Abstracts Registry Number for the parameter if available. Otherwise use the IRPIMS PARLABEL. Other chemical identifier codes may be allowed by the EQuIS Chemistry project manager. |
| 9 | chemical_name | Text(60) | | Y | Chemical name is used only in review of EDD. The cas-rn field is the only chemical identity information actually imported in EQuIS Chemistry. |
| 10 | result_value | Text(20) | | N | Analytic result reported at an appropriate number of significant digits. May be blank for non-detects. |
| 11 | result_error_delta | Text(20) | | N | Error range applicable to the result value; typically used only for radiochemistry results. This is an optional field for the laboratory EDD unless otherwise specified by the EQuIS Chemistry project manager. |
| 12 | result_type_code | Text(10) | | Y | Must be either "TRG" for a target or regular result, "TIC" for tentatively identified compounds, "SUR" for surrogates, "IS" for internal standards, or "SC" for spiked compounds. Not all of these result types may be required, depending on the needs of the EQuIS Chemistry project manager. |

## EQuIS Chemistry 4 File Import Format (EFWEDD)

| # | Field Name | Type | PK | Required | Field Definition |
|---|---|---|---|---|---|
| 13 | reportable_result | Text(10) | | Y | Must be either "Yes" for results which are considered to be reportable, or "No" for other results. This field has many purposes. For example, it can be used to distinguish between multiple results where a sample is retested after dilution. It can also be used to indicate which of the first or second column result should be considered primary. The proper value of this field in both of these two examples should be provided by the laboratory (only one result should be flagged as reportable). Also, the EQuIS Chemistry project manager can also use this field as needed. For example, benzene may be detected by several test methods requested for a sample, all but one can be flagged as not reportable if desired. |
| 14 | detect_flag | Text(2) | | Y | Maybe either "Y" for detected analytes or "N" for non-detects. At the request of the EQuIS Chemistry project manager, other valid values may be used as necessary. These include "TR" for trace (above detection limit but below the quantitation limit) or ">" and "<" for tests such as flash point. Note that "<" must not be used to indicate non-detects (use "N" for non-detects instead). |
| 15 | lab_qualifiers | Text(7) | | N | Qualifier flags assigned by the laboratory. This is an optional field for the laboratory EDD unless otherwise specified by the EQuIS Chemistry project manager. EQuIS Chemistry does not enforce a controlled vocabulary on the values of this field, although a list of valid values may optionally be provided by the EQuIS Chemistry project manager. |
| 16 | organic_yn | Text(1) | | N | If required, then it must be either "Y" for organic constituents or "N" for inorganic constituents. |
| 17 | method_detection_limit | Text(20) | | N | Method detection limit. This is an optional field for the laboratory EDD unless otherwise specified by the EQuIS Chemistry project manager. |
| 18 | reporting_detection_limit | Text(20) | | N | Detection limit that reflects conditions such as dilution factors and moisture content. Required for all results for which such a limit is appropriate. |
| 19 | quantitation_limit | Text(20) | | N | Concentration level above which results can be quantified with confidence. It must reflect conditions such as dilution factors and moisture content. Required for all results for which such a limit is appropriate. This is an optional field for the laboratory EDD unless otherwise specified by the EQuIS Chemistry project manager. |
| 20 | result_unit | Text(15) | | Y | units of measurement for the result. IRPIMS-style unit of measurement codes (see table X02) are |

## EQuIS Chemistry 4 File Import Format (EFWEDD)

| # | Field Name | Type | PK | Required | Field Definition |
|---|---|---|---|---|---|
| | | | | | recognized by EQuIS Chemistry; other codes may be allowed by the EQuIS Chemistry project manager. |
| 21 | detection_limit_unit | Text(15) | | N | units of measurement for the detection limit(s). IRPIMS-style unit of measurement codes (see table X02) are recognized by EQuIS Chemistry; other codes may be allowed by the EQuIS Chemistry project manager. |
| 22 | tic_retention_time | Text(8) | | N | Retention time in seconds for tentatively identified compounds. This is an optional field for the laboratory EDD unless otherwise specified by the EQuIS Chemistry project manager. |
| 23 | result_comment | Text(255) | | N | Result specific comments. |
| 24 | qc_original_conc | Text(14) | | N | The concentration of the analyte in the original (unspiked) sample. Might be required for spikes and spike duplicates (depending on user needs). Not necessary for surrogate compounds or LCS samples (where the original concentration is assumed to be zero). |
| 25 | qc_spike_added | Text(14) | | N | The concentration of the analyte added to the original sample. Might be required for spikes, spike duplicates, surrogate compounds, LCS and any spiked sample (depending on user needs). |
| 26 | qc_spike_measured | Text(14) | | N | The measured concentration of the analyte. Use zero for spiked compounds that were not detected in the sample. Might b required for spikes, spike duplicates, surrogate compounds, LCS and any spiked sample (depending on user needs). |
| 27 | qc_spike_recovery | Text(14) | | N | The percent recovery calculated as specified by the laboratory QC program. Always required for spikes, spike duplicates, surrogate compounds, LCS and any spiked sample. Report as percentage multiplied by 100 (e.g., report "120%" as "120"). |
| 28 | qc_dup_original_conc | Text(14) | | N | The concentration of the analyte in the original (unspiked) sample. Might be required for spike or LCS duplicates only (depending on user needs). Not necessary for surrogate compounds or LCS samples (where the original concentration is assumed to be zero). |
| 29 | qc_dup_spike_added | Text(14) | | N | The concentration of the analyte added to the original sample. Might be required for spike or LCS duplicates, surrogate compounds, and any spiked and duplicated sample (depending on user needs). Use zero for spiked compounds that were not detected in the sample. Required for spikes, spike duplicates, |

## EQuIS Chemistry 4 File Import Format (EFWEDD)

| # | Field Name | Type | PK | Required | Field Definition |
|---|---|---|---|---|---|
| | | | | | surrogate compounds, LCS and any spiked sample. Also complete the qc_spike_added field. |
| 30 | qc_dup_spike_measured | Text(14) | | N | The measured concentration of the analyte in the duplicate. Use zero for spiked compounds that were not detected in the sample. Might be required for spike and LCS duplicates, surrogate compounds, and any other spiked and duplicated sample (depending on user needs). Also complete the qc_spike_measured field. |
| 31 | qc_dup_spike_recovery | Text(14) | | N | The duplicate percent recovery calculated as specified by the laboratory QC program. Always required for spike or LCS duplicates, surrogate compounds, and any other spiked and duplicated sample. Also complete the qc_spike_recovery field. Report as percentage multiplied by 100 (e.g., report "120%" as "120"). |
| 32 | qc_rpd | Text(8) | | N | The relative percent difference calculated as specified by the laboratory QC program. Required for duplicate samples as appropriate. Report as percentage multiplied by 100 (e.g., report "120%" as "120"). |
| 33 | qc_spike_lcl | Text(8) | | N | Lower control limit for spike recovery. Required for spikes, spike duplicates, surrogate compounds, LCS and any spiked sample. Report as percentage multiplied by 100 (e.g., report "120%" as "120"). |
| 34 | qc_spike_ucl | Text(8) | | N | Upper control limit for spike recovery. Required for spikes, spike duplicates, surrogate compounds, LCS and any spiked sample. Report as percentage multiplied by 100 (e.g., report "120%" as "120"). |
| 35 | qc_rpd_cl | Text(8) | | N | Relative percent difference control limit. Required for any duplicated sample. Report as percentage multiplied by 100 (e.g., report "120%" as "120"). |
| 36 | qc_spike_status | Text(10) | | N | Used to indicate whether the spike recovery was within control limits. Use the "*" character to indicate failure, otherwise leave blank. Required for spikes, spike duplicates, surrogate compounds, LCS and any spiked sample. |
| 37 | qc_dup_spike_status | Text(10) | | N | Used to indicate whether the duplicate spike recovery was within control limits. Use the "*" character to indicate failure, otherwise leave blank. Required for any spiked and duplicated sample. |

## EQuIS Chemistry 4 File Import Format (EFWEDD)

| # | Field Name | Type | PK | Required | Field Definition |
|---|---|---|---|---|---|
| 38 | qc_rpd_status | Text(10) | | N | Used to indicate whether the relative percent difference was within control limits. Use the "*" character to indicate failure, otherwise leave blank. Required for any duplicated sample. |

## Batch Import Format

| # | Field Name | Column Datatype | PK | Required | Field Definition |
|---|---|---|---|---|---|
| 1 | sys_sample_code | Text(40) | PK | Y/K | Unique sample identifier. Each sample must have a unique value, including spikes and duplicates. Laboratory QC samples must also have unique identifiers. The laboratory and the EQuIS Chemistry user have considerable flexibility in the methods they use to derive and assign unique sample identifiers, but uniqueness throughout the database is the only restriction enforced by EQuIS Chemistry. |
| 2 | lab_anl_method_name | Text(35) | PK | Y/K | Laboratory analytic method name or description. A controlled vocabulary (i.e., list of valid method names) is not required for the laboratory EDD unless otherwise specified by the EQuIS Chemistry project manager. The method name should be sufficient to reflect operation of the laboratory. For example both "SW8080-pest" and "SW8080-PCB" may be necessary to distinguish between laboratory methods, while "SW8080" may not provide sufficient detail. |
| 3 | analysis_date | Date | PK? | Y/K? | Date of sample analysis in MM/DD/YY format. May refer to either beginning or end of the analysis as required by EQuIS Chemistry project manager. This field is not always required, but most users will want it. |
| 4 | analysis_time | Text(5) | PK? | Y/K? | Time of sample analysis in 24-hr (military) HH:MM format. May refer to either beginning or end as required by EQuIS Chemistry project manager. This field might be required, depending on the test primary key used by the EQuIS Chemistry user. Note that this field, combined with the "analysis_date" field is used to distinguish between retests and reruns (if reported). Please ensure that retests have "analysis_date" and/or "analysis_time" different from the original test event (and fill out the test_type field as needed). |
| 5 | total_or_dissolved | Text(1) | PK? | Y/K? | If required, then it must be either "T" for total [metal] concentration, "D" for dissolved or filtered [metal] concentration, or "N" for organic (or other) constituents for which neither "total" nor "dissolved" is applicable. This field might be required, depending |

## EQuIS Chemistry 4 File Import Format (EFWEDD)

| # | Field Name | Column Datatype | PK | Required | Field Definition |
|---|---|---|---|---|---|
| | | | | | on the test primary key used by the EQuIS Chemistry user. |
| 6 | column_number | Text(2) | PK? | Y/K? | If required, then it must be either "1C" for first column analyses, "2C" for second column analyses, or "NA" for analyses for which neither "1C" nor "2C" is applicable. Second column data may not be required, depending on the needs identified by the EQuIS Chemistry project manager, in which case all results may be reported as "NA". However, if any "2C" tests are reported, then there must be corresponding "1C" tests present also. Also, laboratories typically can report which of the two columns is to be considered "primary". This distinction is handled by the "reportable_result" field in the result table. This field might be required, depending on the test primary key used by the EQuIS Chemistry user. |
| 7 | test_type | Text(10) | PK? | Y/K? | Type of test. Valid values include "initial", "reextract", and "reanalysis". |
| 8 | test_batch_type | Text(10) | PK | Y | Lab batch type. Valid values include "Prep", "Analysis", and "Leach". Additional valid values may optionally be provided by the EQuIS Chemistry project manager. This is a required field for all batches. |
| 9 | test_batch_id | Text(20) | | Y | Unique identifier for all lab batches. Must be unique within EQuIS Chemistry database. For example, the same identifier can not be used for a prep batch and an analysis batch. The EQuIS Chemistry project manager and the laboratory have the flexibility to devise a scheme to ensure unique values of this field. The EQuIS Chemistry project manager will determine which, if any, batch types are to be required in the EDD. |

## EQuIS Chemistry 4 File Import Format (EFWEDD)

### EDD File To EQuIS Chemistry Table Distribution

EFW2FSample

| EQuIS Chemistry Table | Field | Field# | Required by EQuIS | Reference Table/Values |
|---|---|---|---|---|
| dt_sample | Sys_Sample_Code | 1 | T | |
| (parent table) | Sample_Name | 2 | F | |
| | Sample_Matrix_Code | 3 | T | rt_matrix |
| | Sample_Type_Code | 4 | T | rt_sample_type |
| | Sample_Source | 5 | T | Field or Lab (set by Import) |
| | Parent_Sample_Code | 6 | F | |
| | Sample_Class | 25 | F | |
| | Custom_Field_1 | 26 | F | |
| | Custom_Field_2 | 27 | F | |
| | Custom_Field_3 | 28 | F | |
| | Comment | 29 | F | |
| dt_field_sample | Sys_Sample_Code | 1 | T | dt_sample |
| (parent table) | Sample_Delivery_Group | 7 | F | |
| | Sample_Date | 8 | F | |
| | Sample_Time | 9 | F | |
| | Sys_Loc_Code | 10 | F | |
| | Start_Depth | 11 | F | |
| | End_Depth | 12 | F | |
| | Depth_Unit | 13 | F | rt_unit |
| | Chain_of_Custody | 14 | F | |
| | Sent_to_Lab_Date | 15 | F | |
| | Sample_Receipt_Date | 16 | F | |
| | Sampler | 17 | F | |
| | Sampling_Company_Code | 18 | F | |
| | Sampling_Reason | 19 | F | |
| | Sampling_Technique | 20 | F | |
| | Task_Code | 21 | F | |
| | Collection_Quarter | 22 | F | |
| | Composite_YN | 23 | F | |
| | Composite_Desc | 24 | F | |
| | Sample_Receipt_Time | 30 | F | |
| dt_lab_sample (parent table) | Sys_Sample_Code (Field OR Lab sample will be created, depending on Sample Type | 1 | T | dt_sample |
| dt_location | sys_loc_code | 10 | F | |
| dt_task | task_code | 21 | F | |
| dt_chain_of_custody | chain_of_custody | 14 | F | |

## EQuIS Chemistry 4 File Import Format (EFWEDD)

EFW2LabSMP

| EQuIS Chemistry Table | Field | Field# | Required by EQuIS | Reference Table/Values |
|---|---|---|---|---|
| dt_sample | Sys_Sample_Code | 1 | T | |
| (primary table) | Sample_Type_Code | 2 | T | rt_sample_type |
| | Sample_Matrix_Code | 3 | T | rt_matrix |
| | Sample_Source | 4 | F | Field or Lab (set by Import) |
| | Parent_Sample_Code | 5 | F | dt_sample |
| | Comment | 6 | F | |
| dt_field_sample | Sys_Sample_Code | 1 | T | dt_sample |
| (child table) | Sample_Date | 7 | F | |
| | Sample_Time | 8 | F | |
| | Sample_Receipt_Date | 9 | F | |
| | Sample_Delivery_Group | 10 | F | |
| | Sample_Receipt_Time | 12 | F | |
| dt_lab_sample | Sys_Sample_Code | 1 | T | dt_sample |
| (child table) | Standard_Solution_Source (Field OR Lab sample will be created, depending on Sample Type) | 11 | F | |

EFW2LabTST

| EQuIS Chemistry Table | Field | Field# | Required by EQuIS | Reference Table/Values |
|---|---|---|---|---|
| dt_sample | Sys_Sample_Code | 1 | T | |
| | Sample_Source | n/a | | Field or Lab (set by Import) |
| dt_test | Sys_Sample_Code | 1 | T | dt_sample |
| (primary table) | Lab_Anl_Method_Name | 2 | T | rt_anl_mthd_var<br>rt_std_analytic_method |
| | Analysis_Date | 3 | opt. Key fld | |
| | Analysis_Time | 4 | opt. Key fld | |
| | Total_Or_Dissolved | 5 | opt. Key fld | T, D or N |
| | Column_Number | 6 | opt. Key fld | (may be set as Default) |
| | Test_Type | 7 | opt. Key fld | rt_test_type |
| | Lab_Matrix_Code | 8 | F | rt_matrix |
| | Analysis_Location | 9 | F | FI, FL or LB |
| | Basis | 10 | F | Wet, Dry or NA |
| | Container_Id | 11 | F | |
| | Dilution_Factor | 12 | F | |
| | Lab_Prep_Method_Name | 13 | F | rt_prep_mthd_var<br>rt_std_prep_method |
| | Prep_Date | 14 | F | |
| | Prep_Time | 15 | F | |
| | Leachate_Method | 16 | F | |
| | Leachate_Date | 17 | F | |
| | Leachate_Time | 18 | F | |
| | Lab_Name_Code | 19 | F | rt_subcontractor |

## EQuIS Chemistry 4 File Import Format (EFWEDD)

| EQuIS Chemistry Table | Field | Field# | Required by EQuIS | Reference Table/Values |
|---|---|---|---|---|
| dt_test | QC_Level | 20 | F | |
| (continued) | Lab_Sample_Id | 21 | F | |
| | Percent_Moisture | 22 | F | |
| | Subsample_Amount | 23 | F | |
| | Subsample_Amount_Unit | 24 | F | rt_unit |
| | Analyst_Name | 25 | F | |
| | Instrument_Id | 26 | F | |
| | Comment | 27 | F | |
| | Preservative | 28 | F | |
| | Final_Volume | 29 | F | |
| | Final_Volume_Unit | 30 | F | rt_unit |

EFW2LabRES

| EQuIS Chemistry Table | Field | Field# | Required by EQuIS | Reference Table/Values |
|---|---|---|---|---|
| dt_test | Sys_Sample_Code | 1 | T | dt_sample |
| (parent table) | Lab_Anl_Method_Name | 2 | T | rt_anl_mthd_var<br>rt_std_analytic_method |
| | Analysis_Date | 3 | opt. Key fld | |
| | Analysis_Time | 4 | opt. Key fld | |
| | Total_Or_Dissolved | 5 | opt. Key fld | T, D or N |
| | Column_Number | 6 | opt. Key fld | (may be set as Default) |
| | Test_Type | 7 | opt. Key fld | rt_test_type |
| dt_result | Sys_Sample_Code | 1 | T | dt_sample |
| (primary table) | Lab_Anl_Method_Name | 2 | T | rt_anl_mthd_var<br>rt_std_analytic_method |
| | Analysis_Date | 3 | opt. Key fld | |
| | Analysis_Time | 4 | opt. Key fld | |
| | Total_Or_Dissolved | 5 | opt. Key fld | T, D or N |
| | Column_Number | 6 | opt. Key fld | (may be set as Default) |
| | Test_Type | 7 | opt. Key fld | rt_test_type |
| | Cas_Rn | 8 | T | rt_analyte |
| | Result_Value | 10 | F | |
| | Result_Error_Delta | 11 | F | |
| | Result_Type_Code | 12 | F | rt_result_type |
| | Reportable_Result | 13 | F | Yes or No |
| | Detect_Flag | 14 | F | Y, N, TR or < |
| | Lab_Qualifiers | 15 | F | |
| | Organic_YN | 16 | F | Y or N |
| | Method_Detection_Limit | 17 | F | |
| | Reporting_Detection_Limit | 18 | F | |
| | Quantitation_Limit | 19 | F | |
| | Result_Unit | 20 | F | rt_unit |
| | Detection_Limit_Unit | 21 | F | |
| | TIC_Retention_Time | 22 | F | |
| | Result_Comment | 23 | F | |
| | QC_Original_Conc | 24 | F | |
| | QC_Spike_Added | 25 | F | |

## EQuIS Chemistry 4 File Import Format (EFWEDD)

| EQuIS Chemistry Table | Field | Field# | Required by EQuIS | Reference Table/Values |
|---|---|---|---|---|
| dt_result | QC_Spike_Measured | 26 | F | |
| (continued) | QC_Spike_Recovery | 27 | F | |
| | QC_Dup_Original_Conc | 28 | F | |
| | QC_Dup_Spike_Added | 29 | F | |
| | QC_Dup_Spike_Measured | 30 | F | |
| | QC_Dup_Spike_Recovery | 31 | F | |
| | QC_RPD | 32 | F | |
| | QC_Spike_LCL | 33 | F | |
| | QC_Spike_UCL | 34 | F | |
| | QC_RPD_CL | 35 | F | |
| | QC_Spike_Status | 36 | F | |
| | QC_Dup_Spike_Status` | 37 | F | |
| | QC_Rpd_Status | 38 | F | |
| none | Chemical_Name | 16 | F | |

EFW2LabBCH

| EQuIS Chemistry Table | Field | Field# | Required by EQuIS | Reference Table/Values |
|---|---|---|---|---|
| dt_test | Sys_Sample_Code | 1 | T | dt_sample |
| (parent table) | Lab_Anl_Method_Name | 2 | T | rt_anl_mthd_var<br>rt_std_analytic_method |
| | Analysis_Date | 3 | opt. Key fld | |
| | Analysis_Time | 4 | opt. Key fld | |
| | Total_Or_Dissolved | 5 | opt. Key fld | T, D or N |
| | Column_Number | 6 | opt. Key fld | (may be set as Default) |
| | Test_Type | 7 | opt. Key fld | rt_test_type |
| dt_test_batch | Test_Batch_Type | 8 | T | rt_test_batch_type |
| (Child table) | Test_Batch_Id | 9 | T | |
| dt_test_batch_assign | Sys_Sample_Code | 1 | T | dt_sample |
| (Subsidiary table) | Lab_Anl_Mthd_Name | 2 | T | rt_anl_mthd_var<br>rt_std_analytic_method |
| | Analysis_Date | 3 | opt. Key fld | |
| | Analysis_Time | 4 | opt. Key fld | |
| | Total_Or_Dissolved | 5 | opt. Key fld | T, D or N |
| | Column_Number | 6 | opt. Key fld | (may be set as Default) |
| | Test_Type | 7 | opt. Key fld | rt_test_type |
| | Test_Batch_Type | 8 | T | |
| | Test_Batch_Id | 9 | T | |

## EQuIS Chemistry 4 File Import Format (EFWEDD)

### Table X01 - Sample Types

| Sample_type_code | Sample_type_desc |
|---|---|
| AB | Ambient Conditions Blank |
| BD | Blank Spike Duplicate |
| BS | Blank Spike |
| BSD | Blank Spike and Duplicate considered as one sample |
| EB | Equipment Blank |
| FD | Field Duplicate |
| FR | Field Replicate |
| FS | Field Spike |
| KD | Known (External Reference Material) Duplicate |
| LB | Lab Blank |
| LR | Lab Replicate |
| MB | Material Blank |
| MS | Lab Matrix Spike |
| MSD | Lab Matrix Spike and Spike Duplicate pair considered as one sample |
| N | Normal Environmental Sample |
| RB | Material Rinse Blank |
| RD | Regulatory Duplicate |
| RM | Known (External Reference Material) |
| SD | Lab Matrix Spike Duplicate |
| TB | Trip Blank |

## EQuIS Chemistry 4 File Import Format (EFWEDD)

### Table X02 - Matrix Codes

| Matrix_code | Matrix_desc |
|---|---|
| AA | Ambient Air |
| AD | Drilling Air |
| AE | Air, Vapor Extraction Well Effluent |
| AQ | Air Quality Control Matrix |
| CA | Cinder-Ash |
| CF | Fly Ash Cinder |
| DC | Drill Cuttings |
| GE | Gaseous Effluent (Stack Gas) |
| GL | Headspace of Liquid Sample |
| GS | Soil Gas |
| LA | Aqueous Phase of a Multiple Phase Liquid or Solid Sample |
| LC | Liquid Condensate |
| LD | Drilling Fluid |
| LE | Liquid Emulsion |
| LF | Floating/Free Product on Groundwater Table |
| LH | Free-Flowing, or Liquid Waste Containing Less Than 0.5% Dry Solids |
| LM | Multiple Phase Liquid Waste Sample |
| LO | Organic Liquid |
| LV | Liquid from Vadose Zone |
| MH | Hazardous Multiple Phase Waste |
| SB | Bentonite |
| SC | Cement |
| SD | Drill Cuttings, Solid Matrix |
| SE | Sediment (Associated with Surface Water) |
| SF | Filter Sandpack |
| SH | Solid Waste Containing greater than or equal to 0.5% Dry Solids |
| SL | Sludge |
| SM | Water Filter (Solid Material used to filter Water) |
| SN | Miscellaneous Solid Materials - Building Materials |
| SO | Soil |
| SP | Casing (PVC, Stainless Steel, Cast Iron, Iron Piping, etc.) |
| SQ | Soil/Solid Quality Control Matrix |
| SR | Water Filter Residue (Solid that gets filtered out of Water) |
| SS | Scrapings |
| ST | Solid Waste |
| SW | Swab or Wipe |
| TA | Animal Tissue |
| TP | Plant Tissue |
| TQ | Tissue Quality Control Matrix |
| U | Unknown |
| W | Water |
| WA | Drill Cuttings, Aqueous Matrix |
| WC | Drilling Water (Used for Well Construction) |
| WD | Well Development Water |
| WE | Estuary |
| WG | Ground Water |
| WH | Equipment Wash Water, i.e., Water used for Washing |

 

## EQuIS Chemistry 4 File Import Format (EFWEDD)

| | |
|---|---|
| WL | Leachate |
| WO | Ocean Water |
| WP | Drinking Water |
| WQ | Water Quality Control Matrix |
| WS | Surface Water |
| WV | Water From Vadose Zone |
| WW | Waste Water |
| WZ | Special Water Quality Control Matrix |

## EQuIS Chemistry 4 File Import Format (EFWEDD)

### Table X03 - Unit of Measure

| Reported_unit | Unit_desc |
|---|---|
| %v/v | percent by volume |
| 1/s | per second |
| acre ft | acre feet |
| acres | acres |
| admi color | admi (american dye manufacturers institute) color units |
| bars | bars |
| cfs | cubic feet per second |
| cfu/100ml | colony forming units per 100 milliliters |
| cfu/g | colony forming units per gram |
| cfu/ml | colony forming units per milliliters |
| cm | centimeters |
| cm/hr | centimeters per hour |
| cm/sec | centimeters per second |
| cm/yr | centimeters per year |
| cm2/sec | square centimeters per second |
| colf/100ml | coliform bacteria per 100 milliliters |
| colf/g | coliform bacteria per gram |
| color unit | color unit |
| day | days |
| deg c | degrees celsius |
| deg c/hr | degrees celsius per hour |
| deg f | degrees fahrenheit |
| digits | number of digits to the right of the decimal point |
| dollars | dollars |
| dpy | drums per year |
| dynes/cm | dynes per centimeter |
| fibers/l | fibers per liter |
| ft | feet |
| ft candles | foot candles |
| ft msl | feet above mean sea level |
| ft/day | feet per day |
| ft/in | feet per inch |
| ft/min | feet per minute |
| ft/sec | feet per second |
| ft2 | square feet |
| ft2/day | square feet per day (cubic feet/day-foot) |
| ft2/min | feet squared per minute (for units of transmissivity) |
| ft3 | cubic feet |
| ft3/yr | cubic feet per year |
| g/cc | grams per cubic centimeter |
| g/g | grams per gram |
| g/kg | grams per kilogram |
| g/l | grams per liter |
| g/m2/yr | grams per square meter per year |
| g/ml | grams per milliliter |
| gal | gallons |
| gal/min | gallons per minute |

 

## EQuIS Chemistry 4 File Import Format (EFWEDD)

| | |
|---|---|
| gpd | gallons per day |
| gpd/ft | gallons per day per foot |
| gpd/ft2 | gallons per day per foot squared |
| gpm/ft | gallons per minute per foot |
| gpy | gallons per year |
| hrs | hours |
| hrs/day | hours per day |
| in | inches |
| in(hg) | inches of mercury |
| in/day | inches per day |
| in/ft | inches per foot |
| in/hr | inches per hour |
| in/in | inches per inch |
| in/wk | inches per week |
| in2/ft | square inches per foot |
| jcu | jackson candle units |
| jtu | jackson turbidity units |
| kg/1000gal | kilograms per 1000 gallons |
| kg/batch | kilograms per batch |
| kg/day | kilograms per day |
| kg/m3 | kilogram per meter cubed |
| kg/m3/s | kilogram per meter cubed per second |
| kg/s | kilogram per second |
| km2 | square kilometers |
| knots | knots |
| lb/1000lb | pounds per thousand pounds |
| lb/barrel | pound per barrel |
| lb/in2 | pounds per square inch |
| lb/ton | pounds per ton |
| lbs | pounds |
| lbs/day | pounds per day |
| lbs/mon | pounds per month |
| lbs/yr | pounds per year |
| m | meter |
| m/day | meters per day |
| m/s | meter per second |
| m2 | meter squared |
| m2/s | meter squared per second |
| m3 x 10(6) | meter cubed (in millions) |
| m3/kg | meter cubed per kilogram |
| m3/s | meter cubed per second |
| meq/100g | milliequivalents per 100 grams |
| mg/100cm2 | Milligrams per 100 square centimeters |
| mg/flt | Milligrams per filter |
| mg/g | Milligrams per gram |
| mg/kg | milligrams per kilogram |
| mg/l | milligrams per liter |
| mg/m2 | milligrams per square meter |
| mg/m2/day | milligrams per meter squared per day |
| mg/m3 | milligrams per cubic meter (ppbv) |

## EQuIS Chemistry 4 File Import Format (EFWEDD)

| | |
|---|---|
| mg/ml | milligrams per milliliter |
| mgal | million gallons |
| mgd | millions of gallons per day |
| mgdo/l | milligrams dissolved oxygen per liter |
| mgm | millions of gallons per month |
| mgy | millions of gallons per year |
| mile2 | square miles |
| miles | miles |
| mill ft3 | million feet cubed |
| millivolts | millivolts |
| min | minutes |
| ml | milliliter |
| ml/l | milliliter per liter |
| mm | millimeter |
| mm/m2/hr | millimeter per meter squared per hour |
| mm/yr | millimeter per year |
| mmhos/cm | milliohms (mmhos) per centimeter |
| mol % | mole percent |
| mon | month |
| mph | miles per hour |
| mpn/100ml | most probable number per 100 ml |
| ms/cm | microsiemens per centimeter |
| naut.mile | nautical mile |
| ng/100cm2 | nanograms per 100 square centimeters |
| ng/g | nanograms per gram |
| ng/kg | nanogram per kilogram |
| ng/l | nanogram per liter |
| ng/m3 | nanogram per cubic meter |
| ng/ml | nanograms per milliliter |
| none | no unit of measure |
| ntu | nephelometric turbidity units |
| pcf | pounds per cubic foot |
| pci/g | picocuries per gram |
| pci/l | picocuries per liter |
| pci/ml | picocuries per milliliters |
| per loss | percent loss |
| percent | percent |
| pg/g | picogram per gram |
| pg/kg | picograms per kilogram |
| pg/l | picogram per liter |
| pg/m3 | picograms per cubic meter |
| pg/ul | picograms per microliter |
| ph units | ph units |
| ppb | parts per billion |
| ppbv | parts per billion by volume |
| ppm | parts per million |
| ppmv | parts per million by volume |
| pptv | parts per trillion by volume |
| psf | pounds per square foot |
| psi | pounds per square inch |

## EQuIS Chemistry 4 File Import Format (EFWEDD)

| | |
|---|---|
| s | second |
| t.o.n. | threshold order number |
| tons/acre | tons per acre |
| tons/day | tons per day |
| ug/100cm2 | micrograms per 100 square centimeters |
| ug/cm2 | microgram per square centimeters |
| ug/g | micrograms per gram |
| ug/kg | micrograms per killogram |
| ug/l | micrograms/liter |
| ug/m3 | micrograms per cubic meter |
| ug/yr | micrograms per year |
| um/sec | micrometer per second |
| umhos/cm | umhos per centimeter |
| upy | units per year |

## EQuIS Chemistry 4 File Import Format (EFWEDD)

### Revision History

Version 11e – 08/23/2004

- Removed statement that file naming should be in DOS 8.3 format.

Version 11d – 02/16/2004

- Added reference to rt_test_type in EFW2LabTST and EFW2LabRES formats
- Changed sample_type_code from Text(10) to Text(20) in EFW2FSample format.

Version 11c - 11/1/2001

- Changed rt_lab to rt_subcontractor
- Added EFW2Fsample tab to the EFWEDD01.xls spreadsheet template

Version 11b

- Expanded Null field example
- Removed values in result_value column for LCS example.
- Added sample_receipt_time to list of sample optional fields, sample table field description, and EQuIS Chemistry Table Distribution.
- Added preservative, final_volume, and final_volume_unit to list of test optional fields, test table field description, and EQuIS Chemistry Table Distribution.

Version 11a

- Added EDD file to EQuIS Chemistry table distribution map

Version 11

- Added ability to use tab-separated ASCII format as an option. This is a relaxation of the specification.
- Mentioned ability to load files separately (rather than as a group).
- Defined the term "controlled vocabulary" and provided a simple example.
- Added consideration of the need for analysis-date and test-type to Step 1.
- Clarified discussion of the need for QC fields. Basically, most users will need only the calculated recovery fields for QC result rows. The fields which contain spike concentrations added or measure are not always needed, depending on user needs. However, the calculated recoveries are very important for QC, and should always be present.
- Moved this revision section to the end of the document.
- Indicated that analysis-date is an optional member of the test-level primary key, but that most users will want it. This is a relaxation of the specification.

Version 10 - 9/24/1997

- Added several examples.

Version 9 - 7/18/1997

- Corrected numerous spelling errors.
- Clarified language: changed "not null" to "required" for fields that are always required in the EDD.
- Clarified language: In the discussion of optional test level fields, optional key fields may be left blank rather than filled with an asterisk (removed contradictory instructions in previous draft).
- Corrected error on cas_rn column width (should be 15 instead of 75).
- Corrected apparent contradiction in lab_matrix_code definition: this field was not flagged as "required", but the text of the indicated that it was required. This is an optional field.

## EQuIS Chemistry 4 File Import Format (EFWEDD)

- Clarified language: changed "Must be..." to "If required, then it must be..." for the following optional or sometimes optional fields: total-or-dissolved, column-number, analysis-location, basis, and organic-yn.

Version 8 - 6/6/1997

- Increased analysis and prep method name field from 15 to 35 characters. This is a relaxation of the specification.
- Clarified discussion of optional test level fields.
- Increased comment fields to 255 characters. This is a relaxation of the specification.
- Added field position number (#) to tables for clarity.

Version 7

- The test_type valid values for the test, result, and batch level definitions specified below were changed to conform to the 10-character limit: "initial", "reextract", and "reanalysis".
- The cas_rn field was moved to position 8 in the result file definition below.
- The test-batch-type field was moved to position 8 in the batch file definition below.
- Moved the cas_rn field in the enclosed Access MDB file to position 8 in the result table.
- Moved the test-batch-type field in the enclosed Access MDB file to position 8 in the batch table (from position 2).

Version 6

- Included “not null” information for those fields which must always be filled out.
- Clarified and corrected certain field definitions: analysis_time, total_or_dissolved, total-or-dissolved, and column-number
- Corrected datatype error for chemical_name and test_type fields.
- Expanded discussion below for “optional” fields

Version 5

- Version 4 included a surrogate key approach for the test table that paralleled the structure of the project database. Upon further reflection, this seems to have been an error - it may be difficult for laboratories to prepare surrogate key values. Version 5 removed the surrogate key in test by using data columns to be the primary key, which means these columns are also propagated down to the result table. The current Version 6 does not include the mistaken surrogate key approach.

 

# 5 Appendix B

## *EarthSoft EDD Format Definition*
## *EQuIS Chemistry Simple Import Formats*

# EarthSoft - EDD Format Definition

### EQuIS Chemistry Simple Import Formats

EDD Version 3.1, 30 March 2004
Document Version 3.4, 30 March 2004
Prepared by EarthSoft, Inc.
Spreadsheet Template: EZ Formats.xls

## Table of Contents



### Introduction

The purpose of this document is to describe the 'simple' import templates and formats available in EQuIS Chemistry. The Electronic Data Deliverable, or EDD, referred to is EZ Formats.xls. This Microsoft Excel spreadsheet contains 3 tabs, each with a format for importing various data into different parts of the EQuIS Chemistry data structure. Each template has a corresponding import format available by the same name in the EQuIS Chemistry General Import module. It should be noted that, technically, the EDD is simply a data format. EarthSoft distributes the format as a Microsoft Excel document, but it could be created in Lotus or any other spreadsheet. Ultimately, the files that are actually imported into EQuIS Chemistry must be saved from the EDD as text (.txt) or comma-delimited (.csv) files, terminated with a carriage return.

In the following tables, fields with **Y** in the **Req** column are required but are not part of the key. Fields with **Y/K** in the **Req** column are part of the key and are used to determine the uniqueness of the row in the EDD file. A **/K?** indicates that the field may be part of the import's key if it is set up for the project as a required field. This applies to key fields in the dt_test table that are set in the System Administration module's Project Maintenance function, when the project is created.

All data to be imported into EQuIS Chemistry must be stored in an ASCII file using the following standard format. The data fields may be separated from each other by either tabs or commas. Whichever separator is used must be used consistently throughout the given EDD file. If commas are used, then each data field must be enclosed in double quotes (“). Data fields with no information may be represented by two tabs (or commas). For example, if “Analysis Date” has no value and commas are used, the record might look like this:

“12345”,,”12:50”,”MSD”,”2222”,... (and so on)

Maximum length of the field is listed under “DataType” column. If the information is less than the maximum length, do not pad the record with spaces. In the example above, even though “Project Number” can accommodate up to 20 characters, only 5 characters are included in the record.

# EarthSoft - EDD Format Definition

## EQuIS Chemistry Simple Import Formats

Each record must be terminated with a carriage return. The file can be produced using any software with the capability to create ASCII files. Date is reported as MM/DD/YY or MM/DD/YYYY (month/day/year) and time as HH:MM (hour:minute). Time uses a 24 hour clock, thus 3:30 p.m. will be reported as 15:30.

Lookup table indicates the use of controlled values contained in the listed table. In EQuIS the actual table name will have a prefix of **rt_**.

Questions about this document or the EZ Formats EDD may be referred to the EarthSoft Help Desk at **help@earthsoft.com**.

## EQuIS Chemistry Simple Import Formats

## EQuIS_UST Import Format

**Strict adherence to the specifications in this document is mandatory.**

| Pos# | Field Name | DataType | Req. | Lookup Table | Description |
|---|---|---|---|---|---|
| 1 | sys_sample_ code | Text40 | Y/K | | Unique sample identifier. Each sample must have a unique value, including spikes and duplicates. Laboratory QC samples must also have unique identifiers. The laboratory and the EQuIS Chemistry user have considerable flexibility in the methods they use to derive and assign unique sample identifiers, but uniqueness throughout the database is the only restriction enforced by EQuIS Chemistry. |
| 2 | sample_type_ code | Text20 | Y | sample_type | Code which distinguishes between different types of samples. For example, normal field samples must be distinguished from laboratory method blank samples, etc. |
| 3 | sample_matrix_ code | Text10 | Y | matrix | Code which distinguishes between different types of sample matrix. For example, soil samples must be distinguished from ground water samples, etc. |
| 4 | sample_date | Date | N | | Date sample was collected (in MM/DD/YYYY format for EDD). |
| 5 | sample_time | Text5 | N | | Time of sample collection in 24-hr (military) HH:MM format. |
| 6 | sys_loc_code | Text20 | N | | Soil boring or well installation location.<br><br>* Field should be null if field QC sample (e.g., field blank, trip blank, etc.) |
| 7 | lab_name_code | Text20 | Y | subcontractor | Unique identifier of the laboratory. |
| 8 | lab_anl_ method_name | Text35 | Y/K | anl_mthd_var | Laboratory analytic method name or description. The method name should be sufficient to reflect operation of the laboratory. For example both "SW8080-pest" and "SW8080-PCB" may be necessary to distinguish between laboratory methods, while "SW8080" may not provide sufficient detail. |
| 9 | analysis_date | Date | Y/K? | | Date sample was analyzed (in MM/DD/YYYY format for EDD). |
| 10 | test_type | Text10 | Y/K? | test_type | Type of test. Typical values may include initial, reextract, reanalysis, dilution1, dilution2, etc. |
| 11 | lab_sample_id | Text20 | Y | | Unique sample Id internally assigned by the |

## EQuIS Chemistry Simple Import Formats

| Pos# | Field Name | DataType | Req. | Lookup Table | Description |
|---|---|---|---|---|---|
| | | | | | laboratory. |
| 12 | basis | Text10 | Y | | Enter “Wet” for wet-weight basis reporting, “Dry” for dry-weight basis reporting, or “NA” for tests which this distinction is not applicable. |
| 13 | cas_rn | Text15 | Y/K | analyte | Unique analyte identifier. Use assigned CAS number when one is identified for an analyte.<br><br>Tentatively Identified Compounds (TICs) are not assigned a standard CAS number. The laboratory is required to assign a UNIQUE identifier for each TIC. The unique identifier must be placed in this field. Since retention time for TICs are unique per sample and sample analysis method, this information is the recommended value to use as the unique identifier. |
| 14 | chemical_name | Text60 | Y | | Name of analyte or parameter analyzed. |
| 15 | result_value | Text20 | N | | Must only be a numeric value. It is stored as a string of characters so that significant digits can be retained. Must be identical with values presented in the hard copy. Analytical result is reported left justified. It may be blank for non-detects. |
| 16 | result_unit | Text15 | Y | unit | This format assumes that the result value and detect limit have the same units. |
| 17 | detect_flag | Text2 | Y | | Enter “Y” for detected analytes or “N” for non-detected analytes. |
| 18 | reporting_ detection_limit | Text20 | Y | unit | Must only be a numeric value. Use the value of the Reported Detection Limit (RDL), Practical Quantitation Limit (PQL), or Contract Required Quantitation Limit. Value is stored as a string to retain significant figures. Unit of measure must be identical with the “Result Unit” field. |
| 19 | lab_qualifiers | Text7 | N | qualifiers | Qualifier flags assigned by the laboratory. This is an optional field for the laboratory EDD unless otherwise specified by the EQuIS project manager. EQuIS does not enforce a controlled vocabulary on the values of this field, although a list of valid values may optionally be provided by the EQuIS project manager. |
| 20 | result_comment | Text20 | N | | Result comment. |

 

## EQuIS Chemistry Simple Import Formats

EDD File To EQuIS Table Distribution

| EQuIS Table | Field | Field# | Required by EQuIS | Reference Table/Values |
|---|---|---|---|---|
| dt_result | sys_sample_code | 1 | T | |
| | lab_anl_method_name | 8 | T | rt_anl_method_var |
| | analysis_date | 9 | T | |
| | cas_rn | 13 | T | rt_analyte |
| | result_value | 15 | F | |
| | result_unit | 16 | T | rt_unit |
| | detect_flag | 17 | T | |
| | reporting_detection_limit | 18 | T | |
| | lab_qualifiers | 19 | F | rt_qualifiers |
| | result_comment | 20 | F | |
| dt_test | sys_sample_code | 1 | T | |
| | lab_name_code | 7 | T | rt_subcontractor |
| | lab_anl_method_name | 8 | T | rt_anl_method_var |
| | analysis_date | 9 | F | |
| | test_type | 10 | T | rt_test_type |
| | lab_Sample_id | 11 | T | |
| | basis | 12 | T | |
| dt_test_batch_ assign | sys_sample_code | 1 | T | |
| | lab_anl_method_name | 8 | T | rt_anl_method_var |
| | analysis_date | 9 | F | |
| dt_sample | sys_sample_code | 1 | T | |
| | sample_type_code | 2 | T | rt_sample_type |
| | sample_matrix_code | 3 | T | rt_matrix |
| dt_field_sample | sys_sample_code | 1 | T | |
| | sample_date | 4 | F | |
| | sample_time | 5 | F | |
| | sys_loc_code | 6 | F | |
| dt_lab_sample | sys_sample_code | 1 | T | |
| none | chemical_name | 14 | T | |

EQuIS_UST Revision History

Draft 1.0 (11/25/2002

- initial version

## EZ Result Import (EZEDD)

Version 1.2k, 3/30/2004
Provided by EarthSoft, Inc.

## EQuIS Chemistry Simple Import Formats

Spreadsheet Template: EZ Formats.xls (EZEDD Tab)
Former Title: Analytical Results - Electronic Data Transfer Format (EZEDD Format)

**Strict adherence to the specifications in this document is mandatory.**

| Pos# | Field Name | DataType | Req. | Lookup Table | Description |
|---|---|---|---|---|---|
| 1 | project_code | Text20 | N | | Unique identifier assigned to a project site or delivery order |
| 2 | sample_name | Text30 | Y | | This field contains the sample number as written in the Analysis Request and Chain of Custody (AR/COC) form sent to the laboratory with the field samples for analysis. This is a unique number assigned to each sample by sampling personnel.<br><br>It is critical to the operation of EQuIS (TM) that sample numbers appearing on the AR/COC form be identical with the entry in this field.<br><br>For laboratory blanks or samples, use the unique laboratory sample id. |
| 3 | sys_sample_code | Text40 | Y/K | | Uniquely identifies a field or lab sample. For field samples, use the Field Sample Id. For laboratory blanks or samples, the laboratory may use Lab Sample Id only if the Lab Sample Id is unique. Otherwise, the lab must come up with a way to generate a unique lab sample id to be entered in this field. |
| 4 | sample_date | Date | N | | Date sample was collected in the field in mm/dd/yyyy format. Date information must be identical with the date from the AR/COC form. Leave blank for lab samples. Year may be entered as yy. |
| 5 | sample_time | Text5 | N | | Time sample was collected in the field in hh:mm format (24-hour clock, e.g. 3:40 pm is 15:40). Time information must be identical with the time from the AR/COC form. Leave blank for lab samples. |
| 6 | analysis_ location | Text2 | Y | | Must be either "FI" for field instrument or probe, "FL" for mobile field laboratory analysis, or "LB" for fixed-based laboratory analysis. |
| 7 | lab_name_code | Text20 | Y | subcontractor | Laboratory that performed the analysis. |
| 8 | lab_sample_id | Text20 | Y | | Unique sample ID internally assigned by the laboratory. |

 

## EQuIS Chemistry Simple Import Formats

| Pos# | Field Name | DataType | Req. | Lookup Table | Description |
|---|---|---|---|---|---|
| 9 | sample_type_ code | Text20 | Y | sample_type | Specifies sample type. For field samples, enter N (regular environmental sample). Otherwise, use values listed in the sample type reference table. For example, normal field samples must be distinguished from laboratory method blank samples, etc. IRPIMS-style sample type codes are understood by EQuIS, and other valid sample types can be added by the EQuIS user. Field sample types (e.g., field duplicates, field blanks, etc.) might be submitted blind to the laboratory; in such cases the laboratory may report all field samples as if they were all normal field samples. The laboratory is not required to export data for a spike if a spike duplicate is exported (unless the EQuIS project manager requests all spikes). |
| 10 | lab_del_group | Text20 | N | | Tracking code used by the laboratory. Most commonly called Sample Delivery Group Id (SDG). |
| 11 | lab_batch_ number | Text20 | N | | Tracking number used by the laboratory to identify a group of samples analyzed in the same batch. This field, in conjunction with laboratory blank id, is used to link the relationship between field samples and laboratory blank and other QC samples. |
| 12 | lab_anl_ method_name | Text35 | Y/K | anl_mthd_var | Test method used in the analysis of the analyte. |
| 13 | cas_rn (CAS_Number) | Text15 | Y | analyte | Unique analyte identifier. Use assigned CAS number when one is identified for an analyte.<br><br>Tentatively Identified Compounds (TICs) are not assigned a standard CAS number. The laboratory is required to assign a UNIQUE identifier for each TIC. The unique identifier must be placed in this field. Since retention time for TICs are unique per sample and sample analysis method, this information is the recommended value to use as the unique identifier. |
| 14 | chemical_name | Text60 | Y | | Name of analyte or parameter analyzed. |
| 15 | result_value | Text20 | N | | Must only be a numeric value. It is stored as a string of characters so that significant digits can be retained. Must be identical with values presented in the hard copy. Analytical result is reported left justified. |

## EQuIS Chemistry Simple Import Formats

| Pos# | Field Name | DataType | Req. | Lookup Table | Description |
|---|---|---|---|---|---|
| | | | | | It may be blank for non-detects. |
| 16 | lab_qualifiers | Text7 | N | | Qualifier flags assigned by the laboratory. This is an optional field for the laboratory EDD unless otherwise specified by the EQuIS project manager. EQuIS does not enforce a controlled vocabulary on the values of this field, although a list of valid values may optionally be provided by the EQuIS project manager. |
| 17 | result_unit | Text15 | Y | unit | This format assumes that the result value and detect limit have the same units. |
| 18 | result_type_ code | Text10 | Y | result_type | Type of result (TIC, target analyte, etc.) |
| 19 | detect_flag | Text2 | Y | | Enter "Y" for detected analytes or "N" for non-detected analytes. |
| 20 | reporting_ detection_limit | Text20 | N | | Must only be a numeric value. Use the value of the Reported Detection Limit (RDL), Practical Quantitation Limit (PQL), or Contract Required Quantitation Limit. Value is stored as a string to retain significant figures.<br><br>Unit of measure must be identical with the "Result Unit" field. |
| 21 | dilution_factor | Single | N | | Must be a numeric entry. The factor by which the sample was diluted as part of the preparation process. If no dilution was done, enter the value 1. Value is stored as a string to retain significant figures. |
| 22 | sample_matrix_ code | Text10 | Y | matrix | Code which distinguishes between the different type of sample matrix. For example, soil samples must be distinguished from ground water samples, etc. IRPIMS-style sample matrix codes are understood by EQuIS, and other valid sample types can be added by the EQuIS user. The matrix of the sample as analyzed may be different from the matrix of the sample as retrieved (e.g., TCLP) but this EDD asks only for the matrix as sampled. |
| 23 | total_or_ dissolved | Text1 | N/K? | | Must be "T" for total metal concentration, "D" for dissolved or filtered metal concentration, or "N" for organic (or other) parameters for which neither "total" nor "dissolved" is applicable. |
| 24 | basis | Text10 | Y | | Enter "Wet" for wet-weight basis reporting, "Dry" for dry-weight basis reporting, or "NA" |

## EQuIS Chemistry Simple Import Formats

| Pos# | Field Name | DataType | Req. | Lookup Table | Description |
|---|---|---|---|---|---|
| | | | | | for tests for which this distinction is not applicable. |
| 25 | analysis_date | Date | N/K? | | Date sample was analyzed in mm/dd/yy format. |
| 26 | analysis_time | Text5 | N/K? | | Time sample was analyzed in hh:mm format (24-hour clock, e.g. 3:40pm is 15:40). |
| 27 | method_ detection_limit | Text20 | N | | Must be a numeric value. Use the Method Detection Limit (MDL) for Organic compounds, or the Instrument Detection Limit (IDL) for Inorganic compounds. The value is stored as a string of characters in order to retain significant digits. Unit of measure must be identical with the "Result Unit" field. |
| 28 | lab_prep_ method_nName | Text35 | N | prep_mthd_var | Description of sample preparation or extraction method. |
| 29 | prep_date | Date | N | | mm/dd/yy. This field, in conjunction with extraction time, is used to determine whether holding times for field samples have been exceeded. |
| 30 | prep_time | Text5 | N | | hh:mm. This field, in conjunction with extraction date, is used to determine whether holding times for field samples have been exceeded. |
| 31 | test_batch_id | Text20 | N | | Sample preparation batch number assigned by the laboratory. |
| 32 | result_error | Text20 | N | | Applicable only when reporting radiological sample results |
| 33 | TIC_retention_ time | Text8 | N | | For tentatively identified compounds. May be used in the CAS number field to identify individual TICs as long as each retention time per sample per method of analysis is unique. |
| 34 | qc_level | Text10 | N | | Laboratory QC level associated with the analysis |
| 35 | result_comment | Text255 | N | | Any comments related to the analysis. |
| 36 | parent_sample_ code | Text40 | N | | The value of "sys_sample_code" that uniquely identifies the sample that was the source of this sample. |

### EQuIS Chemistry Simple Import Formats

EDD File To EQuIS Table Distribution

| EQuIS Table | Field | Field# | Required by EQuIS | Reference Table/Values |
|---|---|---|---|---|
| dt_sample | Sample_Name | 2 | F | |
| (parent table) | Sys_Sample_Code | 3 | T | |
| | Sample_Type_Code | 9 | T | rt_sample_type |
| | Sample_Matrix_Code | 22 | T | rt_matrix |
| | Parent_Sample_Code | 36 | F | dt_sample |
| | Sample_Source | n/a | | Field or Lab (set by Import) |
| dt_field_sample | Sys_Sample_Code | 3 | T | dt_sample |
| (parent table) | Sample_Date | 4 | F | |
| | Sample_Time | 5 | F | |
| | Sample_Time | 9 | F | |
| dt_lab_sample | Sys_Sample_Code | 3 | T | dt_sample |
| (parent table) | (Field OR Lab sample will be created, depending on Sample Type | | | |
| dt_test | Sys_Sample_Code | 3 | T | dt_sample |
| (parent table) | Analysis_Location | 6 | F | FI, FL or LB |
| | Lab_Name_Code | 7 | F | rt_subcontractor |
| | Lab_Sample_Id | 8 | F | |
| | Lab_Anl_Method_Name | 12 | T | rt_anl_mthd_var<br>rt_std_analytic_method |
| | Dilution_Factor | 21 | F | |
| | Total_Or_Dissolved | 23 | F | T, D or N |
| | Basis | 24 | F | Wet, Dry, NA |
| | Analysis_Date | 25 | F | |
| | Analysis_Time | 26 | F | |
| | Lab_Prep_Method_Name | 28 | F | rt_prep_mthd_var<br>rt_std_prep_method |
| | Prep_Date | 29 | F | |
| | Prep_Time | 30 | F | |
| | QC_Level | 34 | F | |
| | Column_Number | n/a | F | (may be set as Default) |
| | Test_Type | n/a | F | rt_test_type |
| dt_result | Sys_Sample_Code | 3 | T | dt_sample |
| (primary table) | Lab_Anl_Method_Name | 12 | T | rt_anl_mthd_var<br>rt_std_analytic_method |
| | Cas_Rn | 13 | T | rt_analyte |
| | Result_Value | 15 | F | |
| dt_result | Lab_Qualifiers | 16 | F | |
| | Result_Unit | 17 | F | rt_unit |
| | Result_Type_Code | 18 | F | rt_result_type_code |
| | Detect_Flag | 19 | F | Y, N, TR or < |
| | Reporting_Detection_Limit | 20 | F | |
| | Total_Or_Dissolved | 23 | F | T, D or N |
| | Analysis_Date | 25 | F | |
| | Analysis_Time | 26 | F | |

## EQuIS Chemistry Simple Import Formats

| EQuIS Table | Field | Field# | Required by EQuIS | Reference Table/Values |
|---|---|---|---|---|
| | Method_Detection_Limit | 27 | F | |
| | Result_Error_Delta | 32 | F | |
| | TIC_Retention_Time | 33 | F | |
| | Result_Comment | 35 | F | |
| | Column_Number | n/a | F | (may be set as Default) |
| | Test_Type | n/a | F | rt_test_type |
| dt_test_batch_assign | Lab_Anl_Mthd_Name | 12 | T | rt_anl_mthd_var<br>rt_std_analytic_method |
| | Total_Or_Dissolved | 23 | F | T, D or N |
| | Analysis_Date | 25 | F | |
| | Analysis_Time | 26 | F | |
| | Test_Batch_Id | 31 | T | dt_test_batch |
| | Test_Type | n/a | F | rt_test_type |
| | Column_Number | n/a | F | (may be set as Default) |
| | Test_Batch_Type | n/a | F | rt_test_batch_type |
| dt_test_batch | Test_Batch_Id | 31 | T | |
| | Test_Batch_Type | n/a | T | rt_test_batch_type |
| none<br>(fields in EDD but not in EQuIS db) | Project_Code | 1 | F | can be checked by Import |
| | Lab_Del_Group | 10 | F | |
| | Lab_Batch_Number | 11 | F | |
| | Chemical_Name | 14 | F | |

### EZEDD Revision History

Draft 1.2k (3/30/2004)

- added parent_sample_code to the EZEDD format

Draft 1.2j (2/26/2002)

- changed sys_sample_code from Text20 to Text40
- changed sample_type from Text10 Text20
- changed sample_time from Time to Text5
- changed lab_name_code from Text10 to Text20
- changed analysis_time from Time to Text5
- changed prep_time from Time to Text5

Draft 1.2i (11/1/2001)

- replaced rt_lab with rt_subcontractor
- changed System_Sample_Code to Sys_Sample_Code
- changed Laboratory_Delivery_Group to Lab_Del_Group
- changed Laboratory_Batch_Name to Lab_Batch_Number
- changed Lab_Analysis_Method_Name to Lab_Anl_Method_Name
- changed Lab_Preparation_Method_Name to Lab_Prep_Method_Code
- changed Prep_Batch_Number to Test_Batch_ID

Draft 1.2h (12/28/1999)

- replaced EquIS references with EQuIS
- updated header/footer

## EQuIS Chemistry Simple Import Formats

Draft 1.2g (05/07/1998)
- ProjectCode is not required

Draft 1.2f (11/12/1997)
- added EDD File to EQuIS Table Distribution map

Draft 1.2e (10/01/1997)
- added Revision History section
- changed Result Qualifier to not be required
- added description to Result Qualifier
- format renamed to EZEdd from EFWDefault
- added Revision History section
- changed Result Qualifier to not be required
- added description to Result Qualifier

## ES Basic Import (ESBasic)

Version 1.0d, 2/26/2002
Provided by EarthSoft, Inc.
Spreadsheet Template: EZ Formats.xls (ESBasic tab)
Former Title: Analytical Results - Electronic Data Transfer Format (ES Basic Format)

This import format does not fully support Total_or_Dissolved or Column_Number as parts of the Test Key. If this data is typically received in your imports, then you most likely should not be using this import format. This import format does allow for setting Total_or_Dissolved and/or Column_Number for all rows by specifying a single default value. This might be a useful approach if you receive your data from other formats that do support those fields, but use this format occasionally.

**Strict adherence to the specifications in this document is mandatory.**

| Pos# | Field Name | DataType | Req. | Lookup Table | Description |
|---|---|---|---|---|---|
| 1 | sys_sample_ code | Text40 | Y/K | | Uniquely identifies a field or lab sample. For field samples, use the Field Sample Id. For laboratory blanks or samples, the laboratory may use Lab Sample Id only if the Lab Sample Id is unique, otherwise, the lab must come up with a way to generate unique lab sample id to be entered in this field. |
| 2 | sample_type_ code | Text20 | Y | sample_type | Specifies sample type. For field samples, enter N (regular environmental sample), otherwise, use values listed in the sample type reference table<br><br>For example, normal field samples must be distinguished from laboratory method blank samples, etc. IRPIMS-style sample type codes are understood by EQuIS, and other valid sample |

## EQuIS Chemistry Simple Import Formats

| Pos# | Field Name | DataType | Req. | Lookup Table | Description |
|---|---|---|---|---|---|
| | | | | | types can be added by the EQuIS user. Field sample types (e.g., field duplicates, field blanks, etc.) might be submitted blind to the laboratory; in such cases the laboratory may report all field samples as if they were all normal field samples. The laboratory is not required to export data for a spike if a spike duplicate is exported (unless the EQuIS project manager requests all spikes). |
| 3 | sample_matrix_ code | Text10 | Y | matrix | Code which distinguishes between different type of sample matrix. For example, soil samples must be distinguished from ground water samples, etc. IRPIMS-style sample matrix codes are understood by EQuIS, and other valid sample types can be added by the EQuIS user. The matrix of the sample as analyzed may be different from the matrix of the sample as retrieved (e.g., TCLP) but this EDD asks only for the matrix as sampled. |
| 4 | sample_date | Date | N | | Date sample was collected in the field in mm/dd/yyyy format. Date information must be identical with the date from the AR/COC form. Leave blank for lab samples. Year may be entered as yy. |
| 5 | sample_time | Text5 | N | | Time sample was collected in the field in hh:mm format (24-hour clock, e.g. 3:40 pm is 15:40). Time information must be identical with the time from the AR/COC form. Leave blank for lab samples. |
| 6 | sys_loc_code | Text20 | N | location | Sample collection location. |
| 7 | lab_name_code | Text20 | Y | subcontractor | Laboratory that performed the analysis. |
| 8 | lab_anal_ method_name | Text35 | Y/K | anl_mthd_var | Test method used in the analysis of the analyte. |
| 9 | analysis_date | Date | N/K? | | Date sample was analyzed in mm/dd/yy format. . |
| 10 | analysis_time | Text5 | N/K? | | Time sample was analyzed in hh:mm format (24-hour clock, e.g. 3:40pm is 15:40). |
| 11 | test_type | Text10 | N | test_type | Type of test. This field may be defaulted at import. |
| 12 | test_batch_id | Text20 | N | | Tracking number used by the laboratory to identify a group of samples analyzed in the same batch. This field, in conjunction with laboratory blank id, is used to link the relationship between field samples and laboratory blank and other QC |

## EQuIS Chemistry Simple Import Formats

| Pos# | Field Name | DataType | Req. | Lookup Table | Description |
|---|---|---|---|---|---|
| | | | | | samples. |
| 13 | lab_sample_id | Text20 | Y | | Unique sample Id internally assigned by the laboratory. |
| 14 | basis | Text10 | Y | | Enter “Wet” for wet-weight basis reporting, “Dry” for dry-weight basis reporting, or “NA” for tests which this distinction is not applicable. |
| 15 | lab_prep_ method_name | Text35 | N | prep_mthd_var | Description of sample preparation or extraction method. |
| 16 | prep_date | Date | N | | mm/dd/yy. This field, in conjunction with extraction time, is used to determine whether holding times for field samples have been exceeded. |
| 17 | prep_time | Text5 | N | | hh:mm. This field, in conjunction with extraction date, is used to determine whether holding times for field samples have been exceeded. |
| 18 | cas_rn (CAS_Number) | Text15 | Y/K | analyte | Unique analyte identifier. Use assigned CAS number when one is identified for an analyte.<br><br>Tentatively Identified Compounds (TICs) are not assigned a standard CAS number. The laboratory is required to assign a UNIQUE identifier for each TIC. The unique identifier must be placed in this field. Since retention time for TICs are unique per sample and sample analysis method, this information is the recommended value to use as the unique identifier. |
| 19 | chemical_name | Text60 | Y | | Name of analyte or parameter analyzed. |
| 20 | result_value | Text20 | N | | Must only be a numeric value. It is stored as a string of characters so that significant digits can be retained. Must be identical with values presented in the hard copy. Analytical result is reported left justified. It may be blank for non-detects. |
| 21 | result_unit | Text15 | Y | unit | This format assumes that the result value and detect limit have the same units. |
| 22 | detect_flag | Text2 | Y | | Enter “Y” for detected analytes or “N” for non-detected analytes. |
| 23 | detection_limit_ | Text20 | N | | Must only be a numeric value. Use the value of |

## EQuIS Chemistry Simple Import Formats

| Pos# | Field Name | DataType | Req. | Lookup Table | Description |
|---|---|---|---|---|---|
| | used | | | | the Reported Detection Limit (RDL), Practical Quantitation Limit (PQL), or Contract Required Quantitation Limit.<br><br>Value is stored as a string to retain significant figures.<br><br>Unit of measure must be identical with the "Result Unit" field. |
| 24 | lab_qualifiers | Text7 | N | | Qualifier flags assigned by the laboratory. This is an optional field for the laboratory EDD unless otherwise specified by the EQuIS project manager. EQuIS does not enforce a controlled vocabulary on the values of this field, although a list of valid values may optionally be provided by the EQuIS project manager. |
| 25 | comment | Text255 | N | | Any comments related to the analysis. |
| 26 | parent_sample_code | Text40 | N | | The value of "sys_sample_code" that uniquely identifies the sample that was the source of this sample. |

EDD File To EQuIS Table Distribution

| EQuIS Table | Field | Field# | Required by EQuIS | Reference Table/Values |
|---|---|---|---|---|
| dt_sample | Sys_Sample_Code | 1 | T | |
| (parent table) | Sample_Type_Code | 2 | T | rt_sample_type |
| | Sample_Matrix_Code | 3 | T | rt_matrix |
| | Parent_Sample_Code | 26 | F | |
| | Sample_Source | n/a | | Field or Lab (set by Import) |
| dt_field_sample | Sys_Sample_Code | 1 | T | dt_sample |
| (parent table) | Sample_Date | 4 | F | |
| | Sample_Time | 5 | F | |
| | Sys_Loc_Code | 6 | F | |
| dt_lab_sample | Sys_Sample_Code | 1 | T | dt_sample |
| (parent table) | (Field OR Lab sample will be created, depending on Sample Type | | | |
| dt_test | Sys_Sample_Code | 1 | T | dt_sample |
| (parent table) | Lab_Name_Code | 7 | F | rt_subcontractor |
| | Lab_Anl_Method_Name | 8 | T | rt_anl_mthd_var<br>rt_std_analytic_method |
| | Analysis_Date | 9 | F | |

### EQuIS Chemistry Simple Import Formats

| EQuIS Table | Field | Field# | Required by EQuIS | Reference Table/Values |
|---|---|---|---|---|
| | Analysis_Time | 10 | F | |
| | Test_Type | 11 | F | rt_test_type |
| | Lab_Sample_Id | 13 | F | |
| | Basis | 14 | F | Wet, Dry, NA |
| | Lab_Prep_Method_Name | 15 | F | rt_prep_mthd_var<br>rt_std_prep_method |
| | Prep_Date | 16 | F | |
| | Prep_Time | 17 | F | |
| | Total_Or_Dissolved | n/a | F | set to blank in Defaults. If it is part of key, it should be set to 'T' |
| | Column_Number | n/a | F | set to blank in Defaults. If it is part of key, it should be set to '1C' or 'PR' |
| | Analysis_Location | n/a | | set to 'LB' in Defaults |
| | Dilution_Factor | n/a | | set to '1' in Defaults |
| dt_result | Sys_Sample_Code | 1 | T | dt_sample |
| (primary table) | Lab_Anl_Method_Name | 8 | T | rt_anl_mthd_var<br>rt_std_analytic_method |
| | Analysis_Date | 9 | F | |
| | Analysis_Time | 10 | F | |
| | Test_Type | 11 | F | rt_test_type |
| | Cas_Rn | 18 | T | rt_analyte |
| | Result_Value | 20 | F | |
| | Result_Unit | 21 | F | rt_unit |
| | Detect_Flag | 22 | F | Y, N, TR or < |
| | Reporting_Detection_Limit | 23 | F | |
| | Lab_Qualifiers | 24 | F | |
| | Result_Comment | 25 | F | |
| | Total_Or_Dissolved | n/a | F | set to blank in Defaults. If it is part of key, it should be set to 'T' |
| | Column_Number | n/a | F | set to blank in Defaults. If it is part of key, it should be set to '1C' or 'PR' |
| dt_result | Result_Type_Code | n/a | | rt_result_type |
| dt_test_batch_assign | Sys_Sample_Code | 1 | T | dt_sample |
| | Lab_Anl_Method_Name | 8 | T | rt_anl_mthd_var<br>rt_std_analytic_method |
| | Analysis_Date | 9 | F | |
| | Analysis_Time | 10 | F | |
| | Test_Type | 11 | F | set to 'initial' in Defaults |
| | Test_Batch_Id | 12 | F | dt_test_batch |
| | Total_Or_Dissolved | n/a | F | set to blank in Defaults. If it is part of key, it should be set to 'T' |
| | Column_Number | n/a | F | set to blank in Defaults. If it is part of key, it should be set to '1C' or 'PR' |
| | Test_Batch_Type | n/a | | set to 'Analysis' in Defaults |
| dt_test_batch | Test_Batch_Id | 12 | F | |

### EQuIS Chemistry Simple Import Formats

| EQuIS Table | Field | Field# | Required by EQuIS | Reference Table/Values |
|---|---|---|---|---|
| | Test_Batch_Type | n/a | | set to 'Analysis' in Defaults |
| none | Chemical_Name | 19 | F | |

## EZ Formats (ESBasic) Revision History

Draft 1.0f (3/30/2004)
- added parent_sample_code to the ESBasic format

Version 1.0e (5/9/2003)
- Added EQuIS_UST import format
- Renamed *.doc and *.xls to EZ Formats

Version 1.0d (2/26/2002)
- changed Sys_Sample_Code from Text20 to Text40

Draft 1.0c (11/1/2001)
- changed System Sample Code to Sys_Sample_Code
- changed Location Code to Sys_Loc_Code
- replaced rt_lab with rt_subcontractor
- changed Analysis Batch Number to Test_Batch_ID
- changed Laboratory Sample ID to Lab_Sample_ID
- changed Preparation Method to Lab_Prep_Method_Name
- changed Laboratory_Batch_Name to Lab_Batch_Number
- changed Lab_Analysis_Method_Name to Lab_Anl_Method_Name

Draft 1.0b (12/29/1999)
- replaced references to EQuIS with EquIS
- updated Header/Footer
- fixed some formatting

Draft 1.0a (05/08/1998)
- added test_type as a part of the EDD file

Draft 1.0 (05/07/1998)
- cloned from EZEDD and simplified

 

# APPENDIX E
# NDEP DATA VALIDATION GUIDANCE

STATE OF NEVADA
Department of Conservation & Natural Resources
DIVISION OF ENVIRONMENTAL PROTECTION
Jim Gibbons, Governor
Allen Biaggi, Director
Leo M. Drozdoff, P.E., Administrator
NEVADA DIVISION OF ENVIRONMENTAL PROTECTION
protecting the future for generations

April 13, 2009

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Mr. Brian Spiller
Stauffer Management Co LLC
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Wilmington, DE 19850-6438

Mr. Craig Wilkinson
Titanium Metals Corporation
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Henderson, NV 89009

Re. **BMI Plant Sites and Common Areas Projects, Henderson, Nevada**
*Supplemental Guidance on Data Validation*

Dear Sirs and Madam:

All of the parties listed above shall be referred to as "the Companies" for the purposes of this letter. The Nevada Division of Environmental Protection (NDEP) provides supplemental guidance on data validation in Attachment A.

Please contact me with any questions (tel: 702-486-2850 x247; e-mail: brakvica@ndep.nv.gov).

Sincerely,

Brian A Rakvica, P.E.
Supervisor, Special Projects Branch
Bureau of Corrective Actions
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BAR:s

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# Attachment A

## NDEP Data Verification and Validation Requirements – Supplement April, 2009

This supplemental guidance combines all previous data verification and validation guidance associated with the BMI Complex and Common Areas work and also incorporates recent United States Environmental Protection Agency (USEPA) guidance into a single document. This document supersedes the prior NDEP guidance: May 3, 2006, *Guidance on Data Validation Procedures* (1), and February 23, 2007, *Additional Guidance on Data Validation Procedures* (2). It also incorporates the *Supplemental Guidance on Data Validation* (3), dated February 26 and March 19, 2009.

The new guidance that is incorporated here is based on the USEPA document, *Guidance for Labeling Externally Validated Laboratory Analytical Data for Superfund Use* (4), OSWER January, 2009. This new USEPA guidance is being incorporated into the verification and validation steps at the BMI Complex and Common Areas because it provides a consistent set of terms for each stage of data validation (DV). The prior BMI Complex and Common Areas DV guidance used terms based on the DRAFT *EPA Region 9 Superfund Data Evaluation/Validation Guidance* (5). This guidance has never been finalized since the 2001 draft.

New Guidance for Data Validation:

There are many terms used in verifying and validating environmental data that have an historical origin that are imprecise and in some cases outdated. These terms may be generally understood but no longer have a current reference point. The USEPA Guidance (1) incorporates terminology correlated with verification and validation steps that provide transparency and consistency in the DV process. For example, the new guidance categorizes DV Stages based upon sample specific and instrument specific quality control (QC). It provides explicit details as to what needs to be reported and what is to be validated at each Stage. There are differences between the analytical methods in the USEPA Contract Laboratory Program (CLP) Program (from which this new USEPA Guidance is derived) and the methods used at the BMI Complex and Common Areas (e.g. Resource Conservation and Recovery Act (RCRA) based), however, there is sufficient overlap such that the DV language is applicable to the BMI Complex and Common Areas methods and the use of the Stages language in this new USEPA guidance will be valuable to the BMI Complex and Common Areas quality assurance (QA) program.

This guidance does not propose any significant revisions with how data are validated, but we request use of the terminology in this new USEPA Guidance (4) as a common lexicon of terms to be used by the Companies when reporting validated data. Additional details are provided below describing how to use this new guidance for data collected at the BMI Complex and Common Areas.

We request that the Companies begin using the following Stages terminology in their Data Validation Summary Reports (DVSR) and electronic data deliverables (EDD) reports (where applicable):

Stages and Processes Used to Verify and Validate Lab Analytical Data:

Stage 1: Verification and validation based only on completeness and compliance of sample receipt conditions, sample characteristics, and basic analytical results

Stage 2A: Verification and validation based on completeness and compliance checks of sample receipt conditions and ONLY sample-related QC results

Stage 2B: Verification and validation based on completeness and compliance checks of sample receipt conditions and BOTH sample-related and instrument-related QC results

Stage 3: A verification and validation based on completeness and compliance checks of sample receipt conditions, both sample-related and instrument-related QC results, AND recalculation checks against the laboratory reported results

Stage 4: A verification and validation based on completeness and compliance checks of sample receipt conditions, both sample-related and instrument-related QC results, recalculation checks, AND the review of actual instrument outputs

The recommended minimum baseline checks that are to be followed for each stage of analytical data are shown in Appendix A of the USEPA Guidance. Using this new language, all data collected at the BMI Complex and Common Areas should be validated at least to Stage 2B . Also, items of particular note found in Appendix A of the USEPA Guidance (4) are identified below.

The QC acceptance criteria that are to be used in evaluation of the data will come from the NDEP Guidance [e.g. *Supplemental Guidance on Data Validation* (3)] along with Companies Work Plans, Quality Assurance Project Plans (QAPPs), standard operating procedures (SOPs), or Laboratory established criteria as described in the analytical methods. The origin of these criteria should be clearly documented in the data validation summary report (DVSR). For example, the DVSR should cite the document (e.g. SOP) that describes the specific acceptance criteria for continuing calibration.

For Requested Reporting Limits discussion in Section 1.1(5) of Appendix A of the USEPA Guidance (1). The Companies should ensure that the reporting limits are consistent with the NDEP Guidance *Detection Limits and Data Report* (December 3, 2008).

In addition, at least 10% of all data within a DVSR should be validated to Stage 4. Our 2006 guidance (1) on DV indicated this is calculated based on the number of data packages validated within a DVSR. To clarify, the criterion to use is calculated based on the total number of samples times the total number of analytical suites [e.g. semi-volatile organic compounds (SVOCs), radionuclides, organochlorine (OC) Pesticides]. If at least 10% of the samples with a similar number of analytical suites are chosen, this criterion is achieved.

**This Updated Guidance is consistent with the NDEP's May 3, 2006 Guidance**:

The requirement that all sample results be validated to Stage 2B and at least 10% are to be validated to Stage 4 is consistent with our prior guidance. Note that Stage 2B includes, among others items, the check of initial and continuing calibration information. Our guidance does not require 100% of this to be validated. Consistent with the previous guidance only a random check of 10-20% is required. The USEPA guidance uses the term Deuterated Monitoring Compound (DMC), which is analogous to a

surrogate compound as applied in most instances under the methods used at the BMI Complex and Common Areas. Also note that providing the reports specified in Stage 4 (instrument reports) in an electronic format for all results is requested to minimize the length of the DVSR hard copy reports.

At least 10% of all data are to be validated to Stage 4. Consistent with our previous guidance, only 10-20% of these samples need to have the recalculation checks (described in Stage 3 of the new USEPA guidance), and 5% of those samples should have the integration and mass spectrum match comparisons (described in Stage 4 of the new guidance). When calculating the percentage of data that need to be validated for recalculation and integration or mass spectrum matches, the algorithm is also based on the number of samples times the number of analytical suites. To meet this, choose a group of samples with a similar number of analytical suites and validate the appropriate percentage. The Companies are also encouraged to select data based upon historical results where a historically higher number of qualified data were observed.

**This Updated Guidance is consistent with the NDEP's February 23, 2007 Guidance**:

Validated data are to be provided in a summary report (hard copy and electronic format) along with a database (EDD) and laboratory reports (electronic format, include Chain-of-Custodies) for all samples validated. All laboratory reports should include a Case Narrative and other required reporting items consistent with the Nevada Laboratory Certification program. Any third party validation that was used to prepare the summary report should also be provided in electronic format. The database supplied with the summary report should only include the results that were validated (i.e., do not include historical data) and should also follow the *Guidance on Uniform Electronic Data Deliverables* (6). The data should also include the QC results (blanks, spikes, surrogates, etc) and other information desired by the Companies in separate database table(s). The EDD should specify the Stage of validation for each record in the validation level field. Please note that the revised EDD format is being developed by the NDEP based upon comments from the Companies. The revised EDD format will address this issue.

The following information is requested with the data validation summary reports:

- An Introduction with Purpose/Objective/Process. The report should describe the matrices sampled, along with the applicable sampling techniques or a reference to the exact work plan where this information can be found.
- Complete descriptions of the sensitivity indicator terms (sample quantitation limit (SQL), practical quantitation limit (PQL), quantitation limit (QL), etc.,) used in the report and EDD. See additional information on this topic in the NDEP *Guidance on Detection Limits and Data Reporting* (7), dated December 3, 2008.

- Details on the applicable samples and sample delivery group (SDG) identification numbers (IDs), that correspond to locations and sampling time, analyses performed (analytical suites), stage of validation performed (e.g.: 2B, 4). Any non-typical sampling or sample handling that was performed should be described (e.g. filtering).
- A data validation qualifier definition
- Reason codes that link results in the database to specific qualifier logic
- Data validation findings for each parameter based on the level of review. When non-conformances are identified they should be linked to the appropriate sample(s) and SDG.

When professional judgment is used to arrive at a decision, the logic should be clearly described. Please justify decisions (use of professional judgment) that don't follow the typical data validation algorithms.

- Evaluation of the Precision, Accuracy, Reproducibility, Comparability, Completeness, and Sensitivity (PARCCS) parameters
- Conclusions/Recommendations
- References
- The DVSRs should include tables that specify when a non-conformance has been identified during the data validation process. Providing these tables in both hardcopy and electronic (ideally in a spreadsheet or database format) will facilitate review of the DVSR and subsequent usability evaluation. These tables should be categorized by issue, for example, those samples qualified due to Laboratory Control Sample exceedances should be within the same table. Each table should specify the sample, SDG/lab package, the analyte(s), the data quality indicator and objective (e.g., % Recovery, Limits of 85-115%), the sample result(s) and the data validation qualifier(s). Both the qualifier based on this non-conformance issue and the overall qualifier applied to this datum should be provided to help understand the qualifiers supplied in the QC database table and EDD. This information is necessary to both properly evaluate the DVSR and will also facilitate data usability investigations. Each data quality indication, for example, percent recovery, percent difference, precision (relative percent difference (RPD)), area (for internal standards), raw level of blank value that is used to compare with analyte levels in the native samples, cooler temperature, holding time days and exceedance should be captured in these tables.

## References


1) NDEP *Guidance on Data Validation Procedures*. May 3, 2006.
2) NDEP *Additional Guidance on Data Validation Procedures*. February 23, 2007,
3) NDEP *Supplemental Guidance on Data Validation*. February 26 and March 19, 2009
4) USEPA *Guidance for Labeling Externally Validated Laboratory Analytical Data for Superfund Use*, OSWER January, 2009. EPA 540-R-08-005.
5) USEPA *Region 9 Superfund Data Evaluation/Validation Guidance* (DRAFT). December 2001. R9QA/006.1.
6) NDEP *Guidance on Uniform Electronic Data Deliverables*. February 27, 2009 (revision pending).
7) NDEP *Guidance on Detection Limits and Data Reporting.* December 3, 2008.

ndep
NEVADA DIVISION OF ENVIRONMENTAL PROTECTION
protecting the future for generations

STATE OF NEVADA
Department of Conservation & Natural Resources
DIVISION OF ENVIRONMENTAL PROTECTION

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January 5, 2012

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Re: **BMI Plant Sites and Common Areas Projects, Henderson, Nevada**
*Revised Guidance on Qualifying Data due to Blank Contamination for the BMI Complex and Common Areas*

Dear Messrs.:

All of the parties listed above shall be referred to as "the Companies" for the purposes of this letter. Attachment A of this letter provides revised guidance regarding the censoring of data due to blank contamination and should be utilized in the review and reporting of censored data. Please note that Attachment A is also posted on NDEP's website at http://ndep.nv.gov/bmi/technical.htm under "Data Validation."

Please contact the undersigned with any questions at sharbour@ndep.nv.gov or 775-687-9332.

Sincerely,

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NDEP-Carson City Office
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# Attachment A

# Guidance on Qualifying Data due to Blank Contamination for the BMI Complex and Common Areas

## 1. Purpose

The purpose of this NDEP guidance document is to define rules for interpreting the effects of blank contamination on the reporting of sample concentrations. Previous to this NDEP guidance, NDEP rules for interpreting blank contamination were based on the USEPA National Functional Guidelines (NFG). Changes to the USEPA NFG for organic chemicals have led NDEP to reconsider how the effects of blank contamination should be interpreted. This NDEP guidance document first provides some background information that explains the evolution of USEPA and NDEP guidance, specifies new rules for interpreting blank contamination, and provides some examples of the types of data problems or issues that have been observed in datasets previously submitted for the BMI Complex and Common Areas.

## 2. Background

USEPA National Function Guidelines

Previous NDEP guidance specific to qualifying data due to blank contamination is found in the February 26, 2009 *Supplemental Guidance on Data Validation* (1) with additional clarification provided in the March 19, 2009 *Supplemental Guidance on Data Validation* (2). The February and March 2009 supplements were established by NDEP because of an updated version of the USEPA NFG for Superfund Organic Methods Data Review (3). The 2008 guidance from USEPA included a new algorithm for qualifying volatile organic chemical (VOC) results based on blank contamination, and the NDEP guidance extended this approach to semi-VOCs (SVOCs).

Historically the USEPA NFGs (4, 5) have defined a factor (e.g., 5X, 10X) that is used to determine whether sample results that are associated with blank contamination should be censored (reported as non-detects). Briefly the USEPA NFG rules report a sample result as detected if the sample concentration is greater than the blank concentration by some factor. Otherwise the result is reported as not detected. For some inorganic chemicals (5) this factor is 10X (see Table 4, page 17 for example). The 2008 (3) USEPA NFGs for Organic Methods Data Review revised this methodology for VOCs, eliminating the 5X and 10X rule, hence simplifying the rules for qualifying concentration data that are associated with blank contamination. However, the latest USEPA NFG for Inorganic Superfund Data Review (6) continues to use a complicated set of algorithms and multiplication factor rules to determine detection status of a sample concentration when there is blank contamination.

The USEPA NFGs indicate use of two sensitivity indicators, a method detection limit (MDL) and a contract required quantitation limit (CRQL). The CRQL is analogous to the practical quantitation limit (PQL), and the MDL is analogous to the sample quantitation limit (SQL) for the purposes of this NDEP guidance. The USEPA NFG rules depend upon the type of blank, and the concentrations of both blanks and samples compared to the associated MDL (SQL) and

CRQL (PQL). The multiplication factor (e.g., 5X, 10X) is used as described above, and if the sample result is reported as non-detect because of blank contamination, then it is reported at the CRQL (PQL), regardless of the actual concentration result.

NDEP Considerations

NDEP understands that the relative uncertainty around an analyte concentration is greater below the PQL, but does not believe using a single datum approach to decision-making, which is the basis of the USEPA NFG rules, is appropriate for the types of decisions encountered at the BMI Complex and Common Areas. Instead, background and risk-based decisions should be made based on all of the data, and complicated datum-specific rules that result in unnecessary censoring is inappropriate and can introduces bias into subsequent background comparisons and risk assessment.

NDEP considers two conditions that need to be considered when evaluating a single sample result (datum) for detection of an analyte in the presence of blank contamination. These conditions assume the blank and sample concentrations are both greater than the MDL/SQL (otherwise reporting of the sample result as a detect or as a non-detect at the SQL is clear) with:

1. One or more associated Blanks > Sample. The possible reasons are:
    a. The sample contains NO (significant) native analyte.
    b. The sample contains some percentage of native analyte.
2. The Sample > all associated Blanks. The possible reason is:
    a. The sample contains some percentage of native analyte.

For Case 1, the original USEPA NFG (4, 5) recommends censoring the sample result, in most cases at the CRQL (PQL). The presumption is that the majority, if not all, of the analyte in the native sample is from blank contamination. For a single datum such a simple and conservative decision might be reasonable, but NDEP does not regard this as reasonable in the context of data from a collection of samples. For the NDEP BMI Complex and Common Areas work, this sample result is rarely considered separately, but is used to understand the distribution of analyte concentration for background comparisons, comparison with applicable risk-based metrics, and estimation of exposure point concentrations. Following the original USEPA NFG (4, 5), the typical action is to censor at the PQL or perhaps at some multiple of this level (e.g. one half the PQL). This results in a biased distribution, which is often a high bias because most blank contamination is less than any PQL, and is often less than one half of the PQL. If instead, the sample concentration is reported, with an associated qualifier and reason code that explains the effect of blank contamination, then background and risk-based decisions can be made with better information. The reported concentration, the SQL and information about the associated blank contamination would be provided.

For Case 2, the original USEPA NFG (4, 5) also recommends censoring the sample result, unless the sample value is greater than the PQL with sufficient difference between the blank and sample values. The logic is that the sample value contains some amount of contamination, and is therefore only usable if there is sufficient confidence (some factor is used) that the native amount is (significantly) greater than the blank amount. If the sample result is less than the PQL, then the result can be censored at the PQL. Similar to Case 1, if the sample value as reported by the

laboratory is reported with an associated qualifier and reason code, then more complete information is provided for decision-making.

There are a few other considerations that are important when considering the effect of potential blank contamination. For example, blank concentrations need to be compared to sample concentrations on an equal basis. If dilution factors, different matrices (soil versus water), or sample weights and volumes complicate the comparison, the comparison will need to be performed on the raw data (e.g. counts, areas). In addition, it is recognized that some analytical techniques have a sensitivity that will pick up a fairly static level of background signal. These techniques include High Resolution Mass Spectrometry (e.g. HR/GC of PCB congeners, and dioxin/furans) and ICP-MS. This static background is not the typical laboratory contaminant case such as phthalates or methylene chloride. In most cases these static levels are much less than important risk-based metrics. However, there are cases where laboratories have prevalent contamination that is observed in blank samples, and that can significantly impact sample data reporting and subsequent background comparisons and risk assessment. Examples in the NDEP BMI Complex and Common Areas include formaldehyde and in some instances metals using ICP-AES.

## 3. Requirements

All environmental concentration data collected from native samples that have associated blanks data should be reviewed to identify if the native samples might have been contaminated. Sample data that are associated with blank contamination should not be censored for this quality control issue. However, during data validation the data should be qualified with an appropriate qualifier (e.g. J-flag, B-flag) and further characterized with an appropriate reason code and discussion if necessary. In cases where the same data are censored or rejected due to other quality assurance and control issues, this should be clear in the validation reports and electronic data deliverables (EDDs).

This is the required approach for organic, inorganic, and radionuclide measurement data. That is blank contamination must not be used alone to censor sample data. When blank contamination is associated with data, a qualifier and reason code should be applied in the data set (e.g. EDD). The potential impact of blank contamination should be discussed in the Uncertainty Analysis of the subsequent human health risk assessment (HHRA) report (or similar report). This should include a discussion of the potential impact of blank contamination on site date (e.g. high bias), background comparisons, and the HHRA. Also, the data used for HHRA will need to address all compounds associated with blank contamination issues. This needs to be first discussed in the Data Usability (DU) section of the HHRA and interpreted in the Uncertainty Analysis. These issues will be addressed via revisions to the NDEP's EDD guidance document.

These requirements apply to all new data reported for the BMI Complex and Common Areas. However, NDEP acknowledges that previously reported data have not followed these new NDEP requirements for reporting of data associated with blank contamination, and that some reports have been reviewed and approved based on previous requirements. NDEP does not require that historical data be subjected to the requirements specified here, but instead that previously validated data that are impacted by blank contamination will be discussed in the Uncertainty Analysis section of any report that uses such data. In so doing, a semi-quantitative comparison

of the potential differences between approaches taken previously and the requirements specified herein will be described and explained in the Uncertainty Analysis section of any report that uses such data. The requirements specified herein will be applied to all data collected after June 2011.

NDEP further notes that the impact of addressing blank contamination issues following the requirement specified herein, or previous practice, are likely to be observed in background comparisons as well as risk assessment. A potential issue for background comparisons concerns censoring limits for reported data. This is particularly of concern because the background data were evaluated and reported using previous requirements for blank contamination. There are three possible outcomes – site concentrations for a chemical (metal or radionuclide) exceed background, do not exceed background, or cannot be determined. The latter outcome occurs if there are many non-detects in the data, and the SQLs for site and background data are different. In this case, the outcome of the background comparisons should be reported as not determined, and the chemical in question should be carried through to the HHRA.

**4. Reasoning behind Recommended change in Qualifying Data**

Censoring results in loss of data and therefore information. In cases where data quality indicators indicate sever bias, such as low spike recoveries, censoring is often justified. But in the case of blank contamination, the data should not be censored solely for this reason during data validation. Following the original USEPA NFGs (4, 5), censoring is performed *a priori*. This is before a complete understanding is gained of how the data will be used. By not censoring during the data validation step, but understanding the influence of blank contamination and including this information in the data usability evaluation, the full complement of data are still used and available for the decision making.

In many instances the approach taken when blank contamination is evident may have little influence on the ultimate decision(s). This is common when the concentrations of most samples are significantly greater than or less than any risk-based level of interest. Also, blank contamination is often insignificant with respect to the risk-based decisions that will be made. The most critical cases to consider are when the sensitivity of the analytical method is near background concentration levels or a risk-based comparison level (i.e., NDEP BCLs), and the blank contamination and sample concentrations are near the SQLs. For these cases, the full data set needs careful consideration to support a reasonable risk-based decision.

Many types of blank may be associated with a set of samples, including field, laboratory (calibration, preparation). It is impossible to associate a particular blank with a particular sample and it is possible that even though there is contamination of the blanks, this is not true for the samples. Recoveries of laboratory control spikes are one way to assess this. If the recovery is very close to the expected recovery, or even on the low side, any contamination in the blanks may not necessarily be associated with samples. USEPA guidance has always recommended comparing sample values against the highest blank in cases where more than one blank is associated with the sample. Since blank levels often change with time (continuing calibration blanks can show this) a more likely scenario is that blank contamination of samples is somewhat random.

There is an additional reason for not continuing to censor data due to blank contamination. This issue involves the relationship between the Companies and the commercial laboratories. When there are examples of blank contamination that are unexpected, the typical approach is to just censor the results and continue with the project. This provides no incentive for the laboratory to improve their operations. By not censoring the data, and considering the sample concentration data in the context of risk-based decisions, the impact of these laboratory practices will become more apparent and hopefully improved upon.

## 5. Example Data Sets

Results from a number of BMI Complex and Common Areas projects were reviewed to show instances where blank censoring has resulted in data sets that were impacted by blank censoring. In most instances the effect is to bias the data set high, since the censored level is greater than the sample reported (actual) value. In several cases large numbers of data were censored well above the original reported levels. Data from the BRC Mohawk Sub-Area Soil Investigation (Datasets 52, 52a, 52b) are provided as examples below.

Results from 83 samples collected at the Mohawk site for antimony were all adjusted to due blank contamination. The mean and median values of the actual reported samples (unadjusted) were 0.33 and 0.31 mg/kg respectively. In most cases the values were adjusted up to the quantitation limit of 1 mg/kg, in some cases higher. The resulting mean and median values are 1.33 and 1 respectively. This resulting shift in the distribution to these much higher central values impacts the comparison of this data set to background values. Data from the 2010 Background Soil Compilation Report, Table 2 shows both the censored (non-detect) data (mean and median values of 0.33 and 0.24 respectively) and detect data (mean and median values of 0.199 and 0.175 respectively) are below these adjusted Mohawk mean and median values.

For boron, the Mohawk uncensored mean and median values are 7.35 and 7.05 respectively with the censored values at 34.4 and 21.75. These censored values are well above the background levels for boron where the mean and median values in the detected data set are 7.85 and 6.6. Similar examples can be shown for mercury, thallium, molybdenum, and selenium.

In both cases, background comparisons might fail (suggest site concentrations are greater than background) because of censoring due to blank contamination, when, in fact, the background comparisons would not fail if blank contamination is addressed using the requirements specified herein.

Other more general concerns include uranium-235. U-235 exists naturally at very low activity concentrations compared with activity concentrations for other uranium isotopes of interest and compared to analytical sensitivity. However, following past practice, if there is blank contamination for U-235 sample results, then the result might be censored at a PQL, which is often around 1 pCi/g. This is much greater than the concentration levels in background samples, and can result in incorrect conclusions that uranium activity concentrations exceed background. Note also that NDEP requires that radionuclide data must not be censored for statistical analysis.

## 6. Required Changes

NDEP is recommending that instead of censoring any data due to blank contamination at the data validation step, the Companies should follow the same approach taken by the laboratories. That is, a qualifier should be applied to the associated data along with sufficient information to understand the level of contamination relative to that found in the samples. These data will therefore be assigned a qualifier with an associated reason code indicating blank contamination is associated with the results. These data will be carried through to the data usability and analysis process. By using a single common approach across all data sets where contamination is recognized but data are not censored during data validation, data comparability is more likely.

The impact of the blank contamination will be evaluated in the data usability analysis and considered within the context of any decisions in the Uncertainty Analysis sections of data or risk-based reports. Contamination will need to be considered on an equal basis and dilution factors, different matrices (soil versus water), or sample weights and volumes recognized.

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NEVADA DIVISION OF ENVIRONMENTAL PROTECTION
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Re: **BMI Plant Sites and Common Areas Projects, Henderson, Nevada**
*Guidance on Data Validation for Asbestos Data in Soils*

Dear Messrs.:

Enclosed in this letter is NDEP's *Data Validation Guidance for Asbestos Data in Soils for the Basic Management Incorporated (BMI) Complex and Common Areas* dated June 2012. Please note that this guidance is also posted on NDEP's website at http://ndep.nv.gov/bmi/technical.htm under "Data Validation".

Please contact the undersigned with any questions at sharbour@ndep.nv.gov or 775-687-9332.

Sincerely,

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# Data Validation Guidance for Asbestos Data in Soils for the Basic Management Incorporated (BMI) Complex and Common Areas

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**June 2012**

# Table of Contents

**List of Acronyms**

BMI - Basic Management, Incorporated
BRC - Basic Remediation Company
COC - Chain of Custody
DVSR - Data Validation Summary Report
EDXA - Energy Dispersive X-ray Analysis
ED - Electron Diffraction
IST - Isokinetic Sampling Tube
ME - Main Exit
MI - Midget Impinger
NDEP - Nevada Division of Environmental Protection
PCM - Phase Contrast Microscopy
SEM - Scanning Electron Microscopy
TEM - Transmission Electron Microscopy
USEPA - United State Environmental Protection Agency

## 1.0 Overview

The purpose of this document is to provide guidance for validating asbestos concentration data to ensure data integrity and evaluate data usability. This guidance is an expansion of the recommendations made in Appendix A of the NDEP (2011) technical guidance for asbestos related risk assessment. This asbestos data validation guidance has been developed in response to counting errors that have previously been found in reported asbestos data provided by the Companies that operate the BMI Complex and Common Areas. If the total number of asbestos structures reported by the Companies is less than the number found in laboratory reports, this is considered a fatal flaw according to BMI Complex and Common Areas Technical Review Guidance (NDEP, 2012). Additionally, the individual final reports for each asbestos sample have been found to include errors in the number of primary structure counts recorded, with respect to total structure counts. Consequently, this guidance document provides a step-by-step procedure that must be used by the Companies to verify the accurate reporting of asbestos laboratory results.

## 2.0 Introduction

Asbestos is the term used to describe a group of naturally occurring hydrated metal silicate minerals of fibrous habit (Berman and Crump, 2003), some of which have been found to cause serious health issues. Inhalation of asbestos fibers is associated with serious illnesses, such as lung cancer, mesothelioma and asbestosis. Consequently, potential exposure to the existing large quantities of asbestos products in public buildings and the natural presence of asbestos in large communities is of major concern to the scientific/medical community and the public (Berman and Crump, 2008a). For assessing health-related risks, collection, analysis and reporting of asbestos samples must be executed with little or no error. Additionally, the reported asbestos data from those samples should be verified via data validation to ensure accuracy.

### 2.1 Asbestos Mineral Types

Asbestos is generally considered as a description of 6 minerals that can be categorized into two types: chrysotile and amphibole. Chrysotile, which is from the serpentine mineral (magnesium silicate), is the most common type of asbestos. The 5 remaining minerals are all amphiboles (ferro-magnesium silicates) and are classified as crocidolite (fibrous reibeckite), amosite (fibrous grunerite), anthophyllite, tremolite and actinolite (Berman and Crump, 2003). The use of asbestos in commercial applications became widespread in the 19$^{th}$ century with chrysotile making up over 90% of its use (Berman and Crump, 2003). The toxicity of asbestos is considered based on its physical and chemical properties including fiber size, shape, and mineral type. Amphibole fibers are considered by some to be more potent than chrysotile fibers; it has been estimated that chrysotile potency for both mesothelioma and lung cancer is 0.0013 and 0.27 times, respectively, that for amphibole (Berman and Crump, 2003). However, the possibility that chrysotile and amphibole are equal in potency has not been completely discarded (Berman and Crump, 2003).

2.2 Asbestos Potency

There is continued debate about which fiber dimensions are most potent and contribute to specific disease endpoints. Berman and Crump (2001) reported that fibers longer than 5 μm and thinner than 0.5 μm are biologically active and have the potential to cause asbestos-related diseases. However, recent studies by Berman and Crump (2008a and 2008b) suggest that fibers longer than 10 μm and thinner than 0.4 μm may have the highest potency with respect to lung cancer and mesothelioma. Berman and Crump also suggest that fiber potency may increase with increasing length up to 20 μm or even 40 μm. Despite the ongoing debate, the USEPA interim guidelines (Berman and Crump, 2003) consider fibers longer than 10 μm and thinner than 0.4 μm to be most likely to cause asbestos-related disease. These fiber dimensions are used for calculating asbestos-related risk for the BMI Complex and related sub-areas (NDEP, 2011). It should be noted that the NDEP (2011) risk assessment guidance differs in approach from the USEPA (2008) *Framework for Investigating Asbestos-Contaminated Superfund Sites*, guidance that the USEPA considers as replacing or superseding the Berman and Crump (2003) USEPA interim guidance. The differences between the two approaches, regarding aspects such as sampling, analysis, counting and risk assessment calculations, are discussed in Appendix C of the NDEP (2011) guidance.

## 3.0 Data Validation

The following subsections describe the necessary components for validation of asbestos data and provide background for understanding the asbestos data validation process. Below, in Appendix I of this document, is a summarized step-by-step process for performing asbestos data validation.

3.1 Sample Receipt/Handling and Chain of Custody

A Chain of Custody (COC) record must accompany the samples throughout the shipping/handling and analysis. The COC record must provide the sample ID, sample collection date and time, analysis request, personnel contact information, who relinquished the samples and who received them. Additionally, a section for comments/instructions for the sampler can be completed if there are any issues during sample collection or to provide more specific instructions for sample analysis.

3.2 Sample Preparation and Analysis

Preparation and analysis of asbestos found in soil samples is the focus of this guidance, which is specific to the BMI Complex and Common Areas. USEPA Method 540-R-97-028, the reference method for this guidance, is employed by the Companies for analyzing releasable asbestos in soils. This method prepares samples via dust generation and utilizes transmission electron microscopy (TEM) for sample analysis. Although there are other methods for analyzing asbestos samples, such as phase contrast microscopy (PCM), midget impinger (MI) and scanning electron microscopy (SEM), TEM is the focus of this guidance. TEM is the preferred technique because

of its analytical capabilities to determine all of the asbestos characteristics that are associated with risk factors, such as mineral type, fiber size and shape.

### 3.2.1 Sample Preparation via Elutriator Method

The *Draft Modified Elutriator Method for the Determination of Asbestos in Soils and Bulk Material* (Berman and Kolk, 2000) was adapted from EPA Method 540-R-97-028 and includes changes that reduce analytical costs and refine the overall method. This adaptation is used by laboratories (such as EMSL Analytical, Inc.) that routinely analyze asbestos soil samples.

The elutriator method employs isokinetic sampling that will collect only the asbestos structures released from soils that are respirable. For sample preparation records, an elutriator prep worksheet must be provided that includes details such as sample weight (before and after drying), total dried sample weight fractions, tumbling speed, start and stop times, flow rate at the main exit (ME) and isokinetic sampling tube (IST) openings and filter IDs with pre- and post-weights. This information is used for determining the concentration of asbestos per gram of respirable dust ($S/g_{PM10}$), which must be listed on the final report sheet. Additionally, the rate of release of respirable dust can be calculated using the mass measurements of dust collected over time on the (main exit) ME filters. The mass percent of the respirable dust in the bulk sample can also be calculated from the mass measurements. The details for calculating the concentration, rate of release and mass percent are discussed at length in Section 10 of the modified elutriator method (Berman and Kolk, 2000).

### 3.2.2 Sample Analysis

For sample analysis, via TEM, a Bench Sheet Data report should be available for each sample. This report will list the sample ID, details about the TEM settings and a list of grids and their respective grid openings. For each grid opening, there will be notation about whether a structure was detected and details about the structure (e.g., dimensions and mineral type). The Bench Sheet Data will be used to verify the correct counting of the detected structures (asbestos and non-asbestos minerals). If a structure is detected, a Structure Sketch Sheet should be included where the identified structures are drawn by hand, or electronically if possible, to represent the image seen in the TEM view screen. If the detected structure is classified as an asbestos mineral, energy dispersive X-ray analysis (EDXA) and electron diffraction (ED) spectra are included to verify the mineral type. In some cases, the Photomicrograph Report (TEM image) is also included with the identified asbestos structures. The specific details for using the aforementioned laboratory reports are discussed in more detail below.

## 3.3 Structure Counting Criteria

The criteria used for counting asbestos structures is specific and only those fibers/structures meeting the criteria are considered in health-related risk assessments. The counting rules for EPA Method 540-R-97-028 follow ISO 10312:1995(E) (Chatfield, 1995), which is discussed below.

The following sections describe distinguishing which structures are considered the most relevant (i.e., potent) for health-related risk assessment, and discuss those structures that are excluded.

### 3.3.1 Asbestos Structures

Although the use of the term "fiber" has been used to encompass asbestos structures, there are several different types of structures that exist. These structures are well defined in ISO 10312:1995(E) (Chatfield, 1995). The four main structures are fiber, bundle, cluster (disperse and compact) and matrix (disperse and compact). According to the ISO 10312:1995(E) counting rules (Chatfield, 1995), these structures are defined as follows:

1) *fiber*- any particle with parallel or stepped sides that is at least 0.5 μm in length and has an aspect ratio of 5:1 or greater (note that some laboratories may use the historic definition that is a 3:1 ratio for comparison to historical optical measurements, also known as PCM equivalent),
2) *bundle*- group of attached fibers that are parallel,
3) *cluster*- aggregate of two or more randomly orientated fibers, with or without bundles,
4) *matrix*- one or more fibers or bundles that may be attached or somewhat concealed by a nonfibrous particle.

Each one of these four categories exists as a separate entity that is designated as a primary structure. Matrix and cluster primary structures can contain several structures (e.g., fibers and bundles) within them. For example, on a TEM grid opening one might identify a matrix primary structure that is comprised of two asbestos fibers, which are attached to or overlapping a group of nonfibrous particles. Individually identified structures within a primary structure are each counted and yield a total structure count for the sample.

### 3.3.2 Protocol Asbestos Structures (>5 μm in length; < 0.4 μm in diameter)

According to Berman and Kolk (2000), biologically relevant asbestos structures are those that are longer than 5 μm and thinner than 0.5 μm; structures satisfying these constraints are considered to be "protocol asbestos structures". However, a more recent report by Berman and Crump (2003) indicates that the diameter discrimination of a structure should be < 0.4 μm for risk assessment. For asbestos related risk assessments performed using NDEP (2011) guidance, the final report for each sample should **only include structures with diameters < 0.4 μm** because the dose-response coefficients (as mentioned below) used by NDEP (2011) guidance are specific to this diameter range. In addition to distinguishing structures by diameter for risk assessment, asbestos structures are also discriminated by length due to potency factors, as discussed below. For the purposes of this guidance, "protocol asbestos structures" will encompass both short and long protocol asbestos structures that are < 0.4 μm in diameter, as defined below, but only "long protocol asbestos structures" will be used to calculate asbestos related risk according to NDEP (2011) guidance.

3.3.2.1 Short Protocol Asbestos Structures (>5 µm, < 10µm in length; < 0.4 µm in diameter)

Protocol asbestos structures that are >5 µm, but ≤ 10 µm in length with a < 0.4 µm diameter are considered "short protocol asbestos structures" for the purpose of this guidance. The short protocol asbestos structures are recorded on the final report for each asbestos sample and are labeled as "asbestos structures >5 µm, ≤ 10 µm". However, the short protocol asbestos structures are not used for asbestos related risk calculations and are distinguished separately from "long" (> 10 µm in length) protocol asbestos structures because the "long" structures are considered to be more potent (Berman and Crump, 2003).

3.3.2.2 Long Protocol Asbestos Structures (> 10µm in length; < 0.4 µm in diameter)

Protocol asbestos structures that are > 10 µm in length with a < 0.4 µm diameter are defined as "long protocol asbestos structures". These are recorded on the final report for each asbestos sample and are labeled as "asbestos structures > 10 µm (Long)". Only long protocol asbestos structures are used when calculating asbestos related risk according to NDEP (2011) guidance. Structures meeting these dimension constraints are considered to be most likely to cause asbestos related diseases (Berman and Crump, 2003).

3.3.3 Structures Excluded from Risk Assessment

The asbestos sample analytical report will include the total protocol asbestos structures, but only a portion of them will be used for the asbestos health-related risk assessment. Regulated asbestos minerals include chrysotile and amphibole (tremolite, amosite, crocidolite, anthophyllite and actinolite). For inclusion in the asbestos risk assessment, these regulated mineral structures must also be > 10 µm in length and < 0.4 µm in diameter, as suggested by Berman and Crump (2003) for optimized dose-response coefficients. There are other minerals found in soil samples during asbestos analysis that are excluded from the risk assessment and include: non-asbestos minerals (e.g., apatite and talc) and non-regulated amphiboles (e.g., winchite, richterite and fluoro-edenite).

3.4 Fiber Mineral Identification

Identification of asbestos fibers or structures is achieved by evaluating the structure morphology and analyzing the sample with energy dispersive X-ray analysis (EDXA) and electron diffraction (ED). Note that only a specific level of classification for fiber identification can be obtained because of the nature of a sample (e.g., ED cannot be performed on non-crystalline material) and instrumentation limitations (e.g., grid positioning must be optimal for EDXA to be performed). These classification levels are discussed in detail in Tables D.1 and D.2 and Figures D.2 and D.4 of ISO 10312:1995(E) (Chatfield, 1995). The methods used for identifying asbestos fibers are briefly discussed below.

### 3.4.1 Morphology

Fiber morphology is based on two types of classification: 1) tubular and 2) non-tubular morphology. Fibers that are identified as having tubular morphology are suspected to be chrysotile, whereas non-tubular fibers are suspected to be amphibole. Once a fiber is suspected to be chrysotile or amphibole based on tubular morphology, ED and EDXA can be utilized to further classify the structure and thus confirm if it is either chrysotile or ampibole.

### 3.4.2 Electron Diffraction (ED)

ED, which is commonly found on TEM instruments, is used to analyze the crystalline structure of a solid using electron diffraction (i.e., interference) patterns. Section D.4.1 of ISO 10312:1995(E) (Chatfield, 1995) describes the features of the electron diffraction pattern that are used to identify chrysotile structures. Additionally, Figure D.3 of this same section shows an image of the electron diffraction pattern for chrysotile. Confirmation of amphibole presence can only be obtained by quantitative interpretation of zone-axis ED patterns (Chatfield, 1995). Figure D.1 of ISO 10312:1995(E) (Chatfield, 1995) shows an example zone-axis ED pattern and Sections D.3.2 and D.4.2 further discuss identification of amphibole fibers with ED.

### 3.4.3 Energy Dispersive X-ray Analysis (EDXA)

EDXA, which is commonly found on TEM instruments, is utilized to determine the elemental composition of a sample. According to Section 3.11 of ISO 10312:1995(E) (Chatfield, 1995), the nominal elemental composition of chrysotile is $Mg_3(Si_2O_5)(OH)_4$, but the exact composition in natural chrysotile can deviate from this where Si may be substituted by Al or Mg may be substituted by Fe(II), Fe(III), Ni, Mn, or Co. Additionally, ISO 10312:1995(E) (Chatfield, 1995) defines the nominal elemental composition for amphiboles as $A_{0-1}B_2C_5T_8O_{22}(OH, F, Cl)_2$ where A = K, Na; B = Fe(II), Mn, Mg, Ca, Na; C = Al, Cr, Ti, Fe(II), Fe(III), Mg; T = Si, Al, Cr, Fe(III), Ti; and some of these elements can be substituted by Li, Pb or Zn.

EDXA can provide both qualitative and quantitative analysis. Sections D.2.3, D.4.1 and D.4.2 of ISO 10312:1995(E) (Chatfield, 1995) further discuss EXDA measurements of chrysotile and amphibole fibers. For quantitative EDXA of chrysotile, Section D.4.1 (Chatfield, 1995) indicates that there are only two elements (Si and Mg) that are important and those two should be the prominent peaks (with appropriate area ratio) with minimal peaks from the other elements. Due to the 5 types of regulated amphibole minerals and the variations that may exist in chemical composition, EDXA of amphibole fibers is not as straightforward. However, Sections D.2.3 and D.4.2 of ISO 10312:1995(E) (Chatfield, 1995) provide some guidance for EXDA measurements and reference spectra can be found in the literature (Hayashi *et al.*, 1978).

## 3.5 Verification of Quality Controls and Quality Assurance

Section 12 of USEPA Method 540-R-97-028, Section 9.7 of ISO 10312:1995(E) (Chatfield, 1995) and Section 11 of Berman and Kolk (2000) discuss the quality assurance and quality

control requirements for asbestos sampling and analysis. These requirements are briefly discussed below.

### 3.5.1 Blanks

Berman and Kolk (2000), in an adaption of USEPA Method 540-R-97-028, recommend that the following blanks be collected routinely while employing their method: filter lot blanks, laboratory blanks, field blanks, method blanks, equipment blanks, and conditioning filters. The details for generating these blanks are specified in Section 11.1 of Berman and Kolk (2000), and criteria listed there for those blanks is summarized as follows:

- Filter lot blanks: 2 filters tested from each lot of 50; contamination should not exceed 0.2 structures/mm$^2$; only filters that meet this criterion can be used for sample analysis;
- Laboratory blanks: frequency not listed; ensure that laboratory air is in compliance or analysis halts until the issue is addressed; criterion not specified but reference is made to Section 10.6 of Chatfield and Burman (1990), which also does not specify the criterion; NDEP recommends that contamination does not exceed 0.2 structures/mm$^2$ similar to filter lot blanks;
- Field blanks: QC criterion is to be project specific; Chatfield (1995) recommends at least one field blank is processed with each sample batch and NDEP recommends that contamination does not exceed 0.2 structures/mm$^2$ similar to filter lot blanks;
- Method blanks: one per 20 samples analyzed; contamination must not exceed 0.2 structures/mm$^2$;
- Equipment blanks: interchangeable with method blanks, specifically should be used when issues exist with washed sand; no criteria listed but one should default to those for method blanks since they are considered interchangeable with equipment blanks;
- Conditioning filters: collected at the start of each run; no criteria specified other than these blanks should be used for troubleshooting if issues arise.

The results for the above-mentioned blanks must be reported to NDEP with the applicable field sample results.

### 3.5.2 Duplicates and Replicates

For duplicates and replicates, Berman and Kolk (2000) advise that 5-10% of field samples should have a spatial duplicate and that 100% of the field samples should be duplicate pairs, where only 2-3% are randomly selected to be analyzed by the laboratory. Additionally, Berman and Kolk (2000) state that the acceptable relative percent difference (%RPD) between duplicates is < 50%. If the %RPD is greater than acceptable, then replicate counts should be performed on chosen samples by different analysts. If re-analysis is not possible, the results for the duplicate pair should be flagged to indicate the lack of precision and the potential to affect data usability. Note, soil samples are naturally heterogeneous, which could affect the reproducibility of duplicate results.

### 3.5.3 Inter-Laboratory Assessments

BRC SOP-12 (2010) states that soil samples will be analyzed for asbestos using procedures consistent with the modified elutriator method developed by Berman and Kolk (2000). Because asbestos counting can be subjective, Berman and Kolk (2000) recommend that at least two different laboratories analyze the asbestos samples. If this recommendation is followed, then this can be accomplished by exchanging blind field replicates between two or more laboratories to compare counting results. The percentage of samples to be verified by other laboratories is not specified in Berman and Kolk (2000), but given the concerns expressed in Berman and Kolk (2000), NDEP recommends 5-10% of the collected samples be re-analyzed by an independent laboratory when inter-laboratory assessments are included in the sampling plan. NDEP also recommends targeting a %RPD of no greater than 50% when inter-laboratory replicates are analyzed.

### 3.5.4 Analytical Sensitivity Requirements

Analytical sensitivity represents the amount of airborne asbestos structures per gram of respirable dust ($S/g_{PM10}$) or the amount of asbestos structures per liter of air (S/l). The calculation for analytical sensitivity is shown in Section 8 of the ISO 10312:1995(E) (Chatfield, 1995). The purpose of the analytical sensitivity is to try to encompass the range of asbestos concentrations that are of concern for asbestos related risk assessment. Berman and Kolk (2000) suggest that an analytical sensitivity of $3 \times 10^6$ $S/g_{PM10}$ will encompass most of these concentrations and is adequate for most studies where protocol amphibole structures are suspected. However, they also suggest that a sensitivity of $5 \times 10^7$ $S/g_{PM10}$ may be sufficient in cases where only chrysotile structures are suspected due to their lower potency compared to amphibole structures. Based on the desired analytical sensitivity and experimental parameters (e.g., volume of air sampled, etc.), the number of grid openings required to be analyzed to achieve this sensitivity can be calculated using equation Section 8 of the ISO 10312:1995(E) (Chatfield, 1995), as mentioned above.

### 3.5.5 Limit of Detection

Chatfield (1995) defines the limit of detection as the upper limit for a Poisson distribution with a 95% confidence interval where there is a zero structure count. However, NDEP (2011) risk assessment guidance does not use this definition. Instead, a detect is defined as one or more counts of asbestos structures within a sample. A non-detect result is defined as zero structures observed or counted within a sample.

## 3.6 Commentary Write-Up For Asbestos Data Validation

Basic Remediation Company (BRC) has developed a standard operating procedure (SOP) for reviewers to follow (BRC, 2009) when reviewing and validating concentration data. This SOP is specific to traditional chemical analyses, such as organic and inorganic, and does not necessarily apply to asbestos-related data. The BRC SOP also explains the use of validation qualifiers. Presently, no data qualifiers have been employed for reported asbestos concentrations. Due to the

possibility of sample contamination, e.g., from the laboratory or field equipment, data validation qualifiers must used when appropriate. Data qualifiers are important in situations where there is blank contamination such as a laboratory or field blank that could affect the outcome of samples collected with the contaminated blank. Additionally, disagreement in results between duplicate samples could indicate issues within field and laboratory processes that could adversely affect data quality. Replicate and inter-lab results should also be assessed and if necessary qualifiers applied. At a minimum the validation report should discuss any non-conformance with respect to blanks, replicates, and inter-lab results and the possible affect on the data quality and usability. It is important to note that qualified data could still be used in subsequent calculations, such as a risk-assessment, but the qualifiers would clarify any possible influences that the data may have on decision-making.

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**Appendix I: Steps for Validating Reported Asbestos Data**

1. **Document Retrieval:** Retrieve final laboratory report, raw laboratory data (bench sheet data, structure sketches, elutriator prep of samples, ED and EDXA files), COC information and the electronic data deliverable (EDD) for all asbestos samples. The laboratory reports should also include all QC samples, such as the blanks described in Section 3.5.1, duplicates and replicates described in Section 3.5.2, and inter-laboratory replicates, if any, described in Section 3.5.3. Note: an EDD may not be available in all cases. In those cases, there should be a summary table for the asbestos data within the written report.
2. **Verify COC:** Compare the samples reported with any Chain-of-Custody (COC) information and ensure that they are consistent, e.g., confirm sampling names, dates and locations match up. The COC must provide the sample ID, sample collection date and time, analysis request, personnel contact information, who relinquished the samples and who received them. Note any issues that may have been recorded on the COC paperwork.
3. **Verify Methods:** Verify that the method being used for sample preparation and analysis is documented on laboratory reports in a manner that can be easily traced to the official document from the USEPA or other applicable source. For asbestos analysis in soil samples, laboratories should be following the modified elutriator method (Berman and Kolk, 2000), which is an adaptation of the USEPA Method 540-R-97-028. Both of these methods are relevant, but the modified elutriator method updates the USEPA Superfund Method.
4. **Verify Sample List:** Verify that the sample names on the laboratory raw data match up with the written report and/or the EDD. Batch identifier information should also be reported with each sample.
5. **Verify Analytical Sensitivity:** Verify that the analytical sensitivity reported for each sample meets the Sampling and Analysis or Work Plan specifications. Analytical sensitivity units should be consistent with the method, (e.g. $S/g_{PM10}$).
6. **Sample Preparation Sheets:** If any field or lab preparation technique was performed this must be reported. Ensure any mechanical steps used in laboratory sample preparation are included in the reports such as drying and splitting. Documentation of sample preparation must be provided in an elutriator prep worksheet that includes details such as sample weight (before and after drying), total dried sample weight fractions, tumbling speed, start and stop times, flow rate at the ME and IST openings and filter IDs with pre and post weights. From this data, the laboratory can calculate the concentration of asbestos per gram of respirable dust ($S/g_{PM10}$), which is listed on the final report sheet as "Conc." The mass percent or the amount of respirable dust in the bulk sample can also be calculated from the mass measurements. The details for calculating the concentration, rate of release and mass percent are discussed at length in Section 10 of the modified elutriator method (Berman and Kolk, 2000). Examples of typical mass curves, which are included with the elutriator prep worksheet, can be found in Section 11.2 of USEPA

Method 540-R-97-028 and can be used for comparison to the mass curves shown for each sample.

7. **Sample Analysis Sheets:** The Bench Sheet Data report, which details TEM results, must be available for each sample. This report must list the sample ID, details about the TEM settings and a list of grids and their respective grid openings. For each grid opening, there can be notation about whether a structure was detected and details about the structure (e.g., dimensions and mineral type). The Bench Sheet Data will be used for subsequent steps to verify the correct counting of the detected structures. If a structure is detected, a Structure Sketch Sheet must be included where the identified structures are drawn by hand to represent what is seen in the TEM view screen. If the detected structure is classified as an asbestos mineral, energy dispersive X-ray analysis (EDXA) and electron diffraction (ED) spectra must be included to verify the mineral type. And in some cases, the Photomicrograph Report (TEM image) will also be included with the identified asbestos structures.
8. **Know the Code:** These steps cannot provide all the details that are needed for properly identifying asbestos data on Bench Sheet Data reports. One should become acquainted with the types of primary structures discussed in Section 3.3.1 of this guidance and the codes or abbreviations used to identify them. More complete details, including examples of primary structures, can be found in Annex C of ISO 10312:1995(E) (Chatfield, 1995). For convenience, some of the "structure type" codes are:
    - Primary Structures: F = fiber, B = bundle, MD = matrix diffuse, MC = matrix compact, CD = cluster diffuse, CC = compact cluster;
    - Total Structures within Primary Structures: MF = matrix fiber, MB = matrix bundle, MR = matrix residual, CF = cluster fiber, CB = cluster bundle, CR = cluster residual.

    The primary structure codes MD, MC, CD and CC will be followed by a two-digit number. The first digit is the estimated total number of fibers and bundles in the structure and can range from 1 to 9, or 'I+" if there are more 9 fibers or bundles. The second digit is the total number of fibers and bundles longer than 5 □m within the structure.
9. **Count the Number of Protocol Asbestos Structures:** Find the Bench Sheet Data report (lists fiber types, dimensions and grid openings; EMSL ones are typically in a table format with alternating row colors of blue and white) for all of the samples and focus on them one at time. Looking at the Bench Sheet Data report, find the column listed as "Total" under "Structure Number". This column will sequentially number the total structures found in the sample. Note that this sheet will assign a number to all minerals found, even those that do not qualify as protocol asbestos structures (e.g., NAM or non-asbestos mineral). Verify that the codes (see Step 8 above) used for describing the structures (e.g., MD11) are consistent with the hand-drawn structures on the Structure Sketch Sheet. Next, identify the column "Mineral Type" and only look for chrysotile and amphibole (tremolite, amosite, crocidolite, anthophyllite and actinolite) structures. Then,

count all of the chrysotile and amphibole (total structures) that are >5 □m in length and < 0.4 □m in diameter; this will give the total protocol asbestos structures. Now separate the total count into chrysotile and amphibole structures since they are reported separately. The last step for this count is to count the number of primary structures in which the total structures were found. The primary structure numbers are listed under the column "Structure Type" – "Primary". For every total structure there should be one primary structure, but each primary structure can have several structures within it. Note that only primary structures > 5 μm in length and < 0.4 μm in width will be considered "countable" primary structures that will appear in the final report. Verify the determined counts with those recorded in the final and written reports.

10. **Count the Number of Short Protocol Asbestos Structures:** This will separate out the number of protocol structures that are "short" and not included in the risk assessment. Similar to step 8, look at the Bench Sheet Data report and find the column listed as "Total" under "Structure Number". Now count the chrysotile and amphibole (tremolite, amosite, crocidolite, anthophyllite and actinolite) total structures that are >5 □m, but ≤ 10□m in length and < 0.4 □m in diameter. This count will give the total number of short protocol asbestos structures. Now separate the total count into chrysotile and amphibole structures since they are reported separately. The last step is to count the number of primary structures in which the total structures were found. The primary structure numbers are listed under the column "Structure Type" – "Primary". For every total structure there should be one primary structure, but each primary structure can have several structures within it. Note that only primary structures > 5 μm in length and < 0.4 μm in width will be considered "countable" primary structures that will appear in the final report. Verify the determined counts with those recorded in the final and written reports.
11. **Count the Number of Long Protocol Asbestos Structures:** This will distinguish those structures that will be included in the risk assessment calculations. Similar to steps 8 and 9, look at the Bench Sheet Data report and find the column listed as "Total" under "Structure Number". Now count the chrysotile and amphibole (tremolite, amosite, crocidolite, anthophyllite and actinolite) total structures that are > 10□m in length and < 0.4 □m in diameter. This count will give the total number of short protocol asbestos structures. Now separate the total count into chrysotile and amphibole structures since they are reported separately. The last step for this count is to count the number of primary structures that the total structures were found in. The primary structure numbers are listed under the column "Structure Type" – "Primary". For every total structure there should be one primary structure, but each primary structure can have several structures within it. Note that only primary structures > 5 μm in length and < 0.4 μm in width will be considered "countable" primary structures and will appear on the final report. Verify the determined counts with those recorded in the final and written reports.

12. **Count the Number of Protocol Non-Asbestos Structures:** This step will count the structures that fall within the dimensions of a protocol asbestos structures, but are not classified as chrysotile or amphibole minerals. Similar to previous steps, look at the Bench Sheet Data report and find the column listed as "Total" under "Structure Number" and count the total non-asbestos structures (NAM or non-asbestos mineral) that are >5 □m length and < 0.4 □m in diameter. This count will give the total number of protocol non-asbestos structures. Similar to before, count the number of primary structures and verify that the NAM total and primary structure counts are reported correctly in the final and written reports.
13. **Verify Fiber Identification:** The laboratory should provide the data used for fiber identification, such as ED, EDXA and morphology from TEM images. However, all of these data are not always available for each fiber identification. Additionally, unless the reviewer has been sufficiently trained in interpreting these data, it will be difficult for the reviewer to verify the fiber identification. It is recommended that the reviewer refer to Sections 3.4.1 through 3.4.3 of this guidance for assistance in verifying fiber identification. If the reviewer suspects there might be an issue with how a fiber was identified, they should discuss this with the project manager for clarification.
14. **Verify Quality Controls:** Ensure that the proper blanks and field duplicates have been performed and meet the criteria specified in the method, which are summarized in Sections 3.5.1 and 3.5.2 of this guidance. Also, verify that 5-10% of the total samples have been sent to other, independent laboratories for count verifications and the data is reported. If the criteria for blanks, duplicates and inter-laboratory assessments are not met, this should be identified in the DVSR. At a minimum the validation report should discuss any non-conformance with respect to blanks, replicates, and inter-lab results and the possible affect on the data quality and usability.
15. **Examine the Final Laboratory Report Sheets:** The final laboratory report sheets typically have the name of the laboratory identifying the analysis and have summarized nearly all of the details included in the raw laboratory data. Looking at the final report for each sample, verify that the determined counts match those in the final report. Verify that the following is included on the final laboratory report: sample name, levels of analysis, magnification for fiber counting, aspect ratio used for fiber definition, mass of respirable dust on filter, area of the sample filter, number of grid openings analyzed, area of grid openings, dimensions used for counting, analyst name, dried sample weights, soil moisture, air flow rate through ME and IST openings, total elutriator flow rate, structure class, counts (primary and total), density, concentration, lower and upper detection limits, non-asbestos structures (primary and total) and a list of asbestiform amphibole present (ones that did not meet the dimension requirements or were non-regulated amphiboles).
16. **Comment Write-Up:** Summarize and formally write-up any issues that were found using the guidelines referenced in Section 3.6 of this document.

**From:** Weiquan Dong [mailto:wdong@ndep.nv.gov]
**Sent:** Wednesday, January 11, 2017 12:54 PM
**To:** Steve Clough <steve.clough@nert-trust.com>
**Cc:** James Carlton Parker <jcarltonparker@ndep.nv.gov>; James Dotchin <jdotchin@ndep.nv.gov>
**Subject:** DVSR/EDD for the data from NERT treatability studies

Steve,
We had a discussion about your question on DVSR/EDD for the data from NERT treatability studies. We want to have the data from NERT treatability studies validate at least Level 2A because the results from the treatability studies will be critical base for feasibility study. NDEP is revising the existing guidance for the data validation in the BMI region. The level 2A will be likely stablished in revised guidance. If you have any question about this issue, please let us know.
Thanks,
Weiquan

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**Sent:** Tuesday, March 07, 2017 12:29 PM
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**Subject:** Notification : Change in Groundwater Data Validation Guidance

All,
The NDEP-BISC and its contractor have reviewed the current groundwater data validation requirements for the BMI and Surrounding Areas to determine the need for the BMI Plant Sites and Common Areas Projects and other Industrial Sites (The Companies) to continue to use the higher validation rates for groundwater samples. The review of existing groundwater data determined that the higher data validation rates did not change the outcomes of any of the Reports or Projects although they did significantly increase costs for the Companies.

The result of this review will be a coming change in guidance for data validation. As this change will take some time to complete I wanted to get this to the Companies before the spring groundwater sampling takes place.

Please use this e-mail as NDEP authorization to deviate from the existing validation guidance posted on NDEP's website at http://ndep.nv.gov/bmi/technical.htm under "Data Validation" related to groundwater and surface water samples. The required data validation stage will now be 2A for all

groundwater and surface water sampling from March 1, 2017 forward. Please note that the data validation for soils has not changed and will remain the same.

Please contact me with any questions or comments about this notification, a letter will follow with an update to the guidance. Happy sampling.

Regards,
JD

**James (JD) Dotchin**
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# APPENDIX F
# QAPP ADDENDA REQUIREMENTS

## Appendix F. QAPP Addenda Requirements

A QAPP Addendum will be prepared for deviations to the *Quality Assurance Project Plan, Revision 2, Nevada Environmental Response Trust Site, Henderson, Nevada* (Ramboll Environ 2017) and when new sample collection tasks need to be added to the current QAPP. The following elements are required to be updated when a new data collection task is required that is not addressed in the current QAPP or a variance to the current QAPP is identified. The table below is provided as a template to complete a QAPP Addendum. Text in [ ] provides a description of the information that should be inserted.

***Title, Version and Approval/Sign-off:***

### Section 1. New Data Collection Task

| New Data Collection Task | QAPP Update |
|---|---|
| 1.1 Type of Collection Task | [List the data collection task i.e., remedial investigation, treatability, pilot study, etc.] |

| New Data Collection Task | QAPP Update |
|---|---|
| 1.1.2 Data Quality Objectives (DQOs) | [DQOs are task-specific, and may be referenced to the task work plan]<br>1. State the Problem<br>2. Identify the Goal of the Study<br>3. identify the Information Inputs<br>4. Define the Boundaries of the Study<br>5. Develop the Analytical Approach<br>6. Specify Performance of Acceptance Criteria<br>7. Develop the Detailed Plan for Obtaining Data |

| New Data Collection Task | QAPP Update |
|---|---|
| 1.1.3 Project Organization | [List individual assigned to project roles or roles not identified in the current QAPP. This can be accomplished by attaching a table to the QAPP Addendum.] |
| 1.2 Sampling Design | [Reference task-specific work plan.] |
| 1.2.1 Sampling Methods | [List sample collection procedures or refer to task-specific field sampling plan and or task-specific work plan. This can be accomplished by attaching a table to the QAPP Addendum.] |
| 1.2.2 Analytical Methods | [List sample containers, preservation, and holding times. This can be accomplished by attaching a table to the QAPP Addendum.] |
| 1.2.3 Field QC Procedures | [List any deviations for quality control requirements] |

## Section 2. Laboratory Requirements

| Laboratory Requirements | QAPP Update |
|---|---|
| 2.1 Name and Contact Information for Laboratory | [List new contact information] |
| 2.1.1 Analytical Methods & QC Requirements | [List of any new methods] |
| 2.2.2 Analytes, Reporting Limits, and Screening Criteria | [List new parameters or updates] |
| 2.2.3 QAMs and SOPs | [Attach as appendix to QAPP Addendum] |

## Section 3. Data Validation and Usability

| Validation Requirements | QAPP Update |
|---|---|
| 3.1 Stage of validation and review requirements | [List NDEP validation stage required] |
| 3.1.1 Data validation subcontractor or consultant responsible for data validation | [List subcontractor or role of person responsible for validation] |

| Validation Requirements | QAPP Update |
|---|---|
| 3.1.2 PARRC criteria | [List deviations for precision, accuracy, representativeness, completeness, comparability] |
| 3.1.3 Validation Guidance | [List any new validation guidance criteria required] |
| 3.1.4 Validation Qualifiers and Reason Codes | [List any new validation qualifiers and reason codes] |