Appendix A
Sampling and Analysis Plan

Delaware Sand & Gravel Superfund Site
New Castle County, Delaware

Submitted to:

United States Environmental Protection Agency
1650 Arch Street
Philadelphia, PA 19103 USA

Prepared by:

Golder Associates Inc.
200 Century Parkway, Suite C
Mt. Laurel, New Jersey 08054 USA
T: +1 856 793-2005

013-6052-014

March 2019

Revision 2

Distribution List

1 copy and electronic submittal – Debra Rossi - USEPA Region III

1 copy and electronic submittal – Christina Wirtz – DNREC

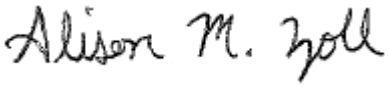
1 copy and 1 CD – Joe DiNunzio – AWC

1 copy and 1 CD – DS&G Trust

1 copy and electronic copy – Golder Associates

SIGNATURE PAGE

QUALITY ASSURANCE:

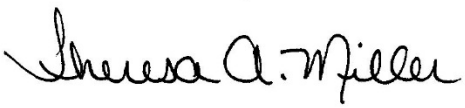


Alison Zoll, Project Chemist
Golder Associates Inc.

FIELD MANAGER:

Benjamin Reynolds, Senior Project Geologist
Golder Associates Inc.

SENIOR TECHNICAL REVIEW:



Theresa A. Miller, PG, LSP, Senior Consultant
Golder Associates Inc.

Table of Contents

1.0 INTRODUCTION 1

2.0 PROJECT BACKGROUND AND ADMINISTRATIVE INFORMATION 1

2.1 Problem Definition/Background 2

2.2 Project/Task Description 3

2.3 Project Organization 3

2.4 Special Training/Certification 5

2.5 Documents and Records 6

3.0 QUALITY OBJECTIVES AND CRITERIA 7

3.1 Precision 7

3.2 Accuracy 8

3.3 Representativeness 8

3.4 Comparability 8

3.5 Completeness 9

3.6 Sensitivity 9

4.0 DATA GENERATION AND ACQUISITION 10

4.1 Sampling Process Design 10

4.2 Locations, Frequencies and Matrices 10

4.2.1 Activities to Address Areas of Uncertainty 11

4.2.1.1 Target Capture Zones and Extraction Rates 11

4.2.1.2 Migration Between Hydrostratigraphic Units 12

4.2.1.3 Manganese and Other COCs in Groundwater 12

4.2.1.4 Extent of Contamination in UPCUTZ 12

4.2.2 Assessment for Enhanced LFExS Design 12

4.2.3 Potential LFG Migration 12

4.2.4 Ongoing Monitoring Activities 13

4.3 Sampling and Data Collection Methods 13

4.3.1 Drilling 13

4.3.1.1 Pre-Drilling Activities 13
4.3.1.2 Boring Advancement 14
4.3.1.3 Vertical Aquifer Profiling 15
4.3.1.4 Soil Sampling for Grain-Size Analysis 16
4.3.2 Monitoring and Extraction Well Installation and Development 17
4.3.2.1 Monitoring Well Installation 17
4.3.2.2 Extraction Well Installation 18
4.3.2.3 Well Development 18
4.3.2.4 Well Elevation and Location Survey 19
4.3.3 Well/Piezometer Sampling Procedures 19
4.3.3.1 Water Level Measurement Procedures 19
4.3.3.2 Vertical Flow Screening 19
4.3.3.3 Low-Flow Groundwater Sampling Procedures 20
4.3.3.4 Volume Average Purging Using Submersible Pumps 21
4.3.3.5 Volume Average Purging Using Bailers 22
4.3.3.6 Extraction Well Sampling 24
4.3.4 AWC Well Sampling 24
4.3.5 Collection of Split Samples 25
4.3.6 Aquifer Testing 25
4.3.6.1 Transducer Study - Response to AWC Pumping 25
4.3.6.2 UPA Pumping Tests and UPCUTZ Extraction Testing 26
4.4 Advancement and Screening of Barhole Probes 33
4.5 Decontamination 34
4.5.1 PFAS Decontamination 34
4.5.2 Drilling Equipment 35
4.5.2.1 Decontamination Pad Specifications 35
4.5.2.2 Decontamination Procedures 35
4.5.3 Non-Dedicated Sampling Equipment 35
4.5.4 Non-Dedicated Submersible Pump 36
4.6 Investigation-Derived Waste 36

4.6.1 Soil 36
4.6.2 Water 36
4.6.3 Disposable PPE 37
4.6.4 Labeling 37
4.7 Sample Handling and Custody 37
4.8 Analytical Methods 38
4.9 Quality Control 39
4.9.1 Field Duplicates 39
4.9.2 Trip Blanks 39
4.9.3 Rinsate Blanks 39
4.9.4 Field Blanks 39
4.9.5 MS/MSDs 40
4.9.6 Internal QC Samples 40
4.10 Instrument/Equipment Testing, Inspection, and Maintenance 40
4.11 Instrument Calibration and Frequency 41
4.11.1 Field Calibration 41
4.11.2 Photovac Microtip PID 41
4.11.2.1 Operational Information 41
4.11.2.2 Instrument Calibration and Frequency 42
4.11.3 Multi-Gas Meter 43
4.11.3.1 Operational Information 43
4.11.3.2 Instrument Calibration and Frequency 44
4.11.4 Laboratory Calibration 44
4.12 Inspection/Acceptance of Supplies and Consumables 44
4.13 Non-direct Measurements 44
4.14 Data Management 45
5.0 ASSESSMENT AND OVERSIGHT 45
5.1 Assessments and Response Actions 45
5.1.1 Peer Review 46

5.1.2 Audits 46
5.1.3 Field/Sampling Audit 46
5.1.4 Laboratory Audits 46
5.1.5 Data Quality Assessment 46
5.2 Reports to Management 46
5.3 Evaluation of Tentatively-Identified Compounds 47
6.0 DATA VALIDATION AND USABILITY 47
6.1 Data Review, Verification, and Validation 47
6.2 Verification and Validation Methods 48
6.3 Reconciliation with User Requirements 48
7.0 ACRONYMS AND ABBREVIATIONS 50
8.0 REFERENCES 52

TABLES

Table A-1 Data Quality Objectives
Table A-2 Decision Thresholds / Action Levels
Table A-3 List of and Rationale for Proposed Monitoring Well Locations
Table A-4 Proposed Wells and Areas of Uncertainty Matrix
Table A-5 Anticipated Construction Details for Proposed Monitoring and Extraction Wells
Table A-6 Site Monitoring Program Schedule (Tables A-6A through A-6D)
A - Semi-Annual Water Level Monitoring Locations
B - Monitoring Point Construction Information
C - Semi-Annual and Annual Monitoring Program
D - Monthly, Bi-Monthly and Quarterly Monitoring Program
Table A-7 Monitoring Locations for Enhanced LFExS Design
Table A-8 Pumping Test Locations and Purposes
Table A-9 VOC Analysis - Groundwater Measurement Performance Criteria
Table A-10 SVOC Analysis - Groundwater Measurement Performance Criteria
Table A-11 Inorganic Analysis - Groundwater Measurement Performance Criteria
Table A-12 Major Cations and Anions – Groundwater Measurement Performance Criteria
Table A-13 PFAS Analysis - Groundwater Measurement Performance Criteria
Table A-14 Reference Limits – Groundwater
Table A-15 Analytical Requirements – Groundwater
Table A-16 Analytical SOP References - Groundwater
Table A-17 MS/MSD Data Summary Requirements – Groundwater

Figures

Figure A-1 Site Location Map
Figure A-2 Conceptual Stratigraphic Column
Figure A-3 Proposed Monitoring Well Locations – DDA and Well PW-1(U) Area
Figure A-4 Proposed Monitoring Well Locations - Downgradient of Well PW-1(U)
Figure A-5 Proposed Enhanced LFExS Monitoring
Figure A-6 Aquifer Testing – Transducer Locations
A - Ongoing Transducer Study
B - Steps 1 and 2 - Existing Well PW-1(U) Shut Down and Re-Start
C - Step 3 - Proposed Extraction Well DDA-10/12-US-EXTR
D - Step 4 - Proposed Extraction Well DDA-05-TZ-EXTR
E - Step 5 - Proposed Extraction Well DDA-06-TZ-EXTR
F - Step 6 - Proposed Extraction Well UPA-01-US-EXTR
G - Step 7 - Proposed Extraction Well MW-18/34-US-EXTR
H - Step 8 - Existing Well P-6
Figure A-7 Barhole Probe Investigation Area

Attachments

Attachment A Standard Operating Procedures (SOPs) for PFAS Monitoring Events
SOP-1: General Field Methods for PFAS Sampling
SOP-2: PFAS Program Monitoring Well Purging and Sampling Protocols
SOP-3: Quality Assurance / Quality Control Protocols for PFAS Sampling Programs
Attachment B Low-Flow Groundwater Purge/Sample Field Information Form
Attachment C Volume Average Groundwater Purge/Sample Field Information Form
Attachment D Barhole Probe Advancement and Monitoring Form
Attachment E TestAmerica
E-1 - Laboratory Quality Manual
E-2 - Laboratory Standard Operating Procedures
Attachment F Eurofins
F-1 - Laboratory Quality Manual
F-2 - PFAS Standard Operating Procedure (Redacted)
Attachment G TRC - Laboratory Quality Manual
Attachment H Acronyms and Abbreviations used in Tables, Figures, and Attachments
Attachment I Standard Operating Procedures for Drilling and Groundwater Sampling

1.0 INTRODUCTION

Golder Associates Inc. (Golder) prepared this Sampling and Analysis Plan, Revision 2 (SAP-Rev 2) on behalf of Delaware Sand & Gravel (DS&G) Remedial Trust (DS&G Trust) for the DS&G Superfund Site located approximately two miles south of New Castle, Delaware (Site; see Figure A-1). This SAP-Rev 2 is for use with the Pre-Design Investigation Work Plan, Revision 2 (PDI WP-Rev 2) dated March 2019 and for the ongoing groundwater monitoring performed at this Site. A description of the comments and responses associated with the original SAP (August 2018; Golder, 2018c) and SAP-Rev 1 (January 2019; Golder, 2019) are provided in Section 1 of the PDI WP-Rev 2. The PDI WP field activities commenced on December 4, 2018 under the PDI WP (Revision 0; Golder, 2018c) based on the USEPA's Partial Approval of the PDI WP on November 30, 2018 (USEPA, 2018g).

This SAP-Rev 2 includes the following proposed activities and quality assurance (QA) protocols:

- Soil boring advancement via roto-sonic techniques with Push-Ahead™ sampling for aquifer profiling, and installation of monitoring wells in the Columbia Aquifer, the Upper Potomac Confining Unit Transition Zone (UPCUTZ), and the upper and lower sand of the Upper Potomac Aquifer (UPA) (see Figure A-2 and PDI WP-Rev 2 Section 6.3.1)
- Monitoring well development, purging and sampling techniques, and associated QA protocols for groundwater analyses including per- and polyfluoroalkyl substances (PFAS) (see PDI WP-Rev 2 Section 6.3.3)
- Aquifer testing associated with design of the Selected Remedy (see additional information in Section 2 and in the PDI WP-Rev 2 Section 6.3.4.2)
- Assessment for potential landfill gas (LFG) migration to areas with potential future receptors (see PDI WP-Rev 2 Section 6.3.5)

The following sections of this SAP-Rev 2 present the requirements necessary to implement data collection activities in accordance with United States Environmental Protection Agency (USEPA) Requirements for Quality Assurance Project Plans (QAPP) EPA QA/R-5 (March 2001). More specifically:

- Section 2 - Project Background and Administrative Information
- Section 3 - Quality Objectives and Criteria
- Section 4 - Data Generation and Acquisition
- Section 5 - Assessment and Oversight
- Section 6 - Data Validation and Usability

2.0 PROJECT BACKGROUND AND ADMINISTRATIVE INFORMATION

This SAP-Rev 2 was prepared in support of the PDI WP-Rev 2; therefore, a brief project background including a description of the additional assessments requested by the USEPA and required to support the design of the Selected Remedy is provided in this section. Refer to the PDI WP-Rev 2 for additional details on these activities, the Selected Remedy and the Conceptual Site Model (CSM). This section also provides information regarding project organization, training, documents and records that are necessary for the execution of this SAP-Rev 2.

2.1 Problem Definition/Background

The DS&G Trust is preparing to conduct the PDI for collection of the data that will be used to design the Selected Remedy for the Site. The Final Feasibility Study Revision 1 (Final FS Rev 1; Golder, 2016a) was submitted to the USEPA on May 25, 2016. The USEPA approved the Final FS Rev 1 via letter dated on July 5, 2016 (USEPA, 2016a), issued the Proposed Remedial Action Plan (PRAP) on September 7, 2016 (USEPA, 2016b) identifying Alternative C from the Final FS Rev 1 as the Selected Remedy, and held a public meeting on September 21, 2016 to present the PRAP. On December 12, 2017, the USEPA issued the Record of Decision-Amendment 2 (ROD-A2; USEPA, 2017d) for the DS&G Site, and on May 22, 2018, the DS&G Trust and the USEPA signed an "Administrative Settlement Agreement and Order on Consent for Remedial Design" (RD AOC); effective date May 29, 2018). The Statement of Work (SOW) associated with the RD AOC "sets forth the procedures and requirements for implementing the Work, which consists of: (1) a Preliminary Design Investigation (PDI), and (2) a Remedial Design (RD) for the components of the Remedial Action (RA)" (USEPA, 2018f).

The Selected Remedy is intended to address the groundwater at the Site which is impacted with volatile organic compounds (VOCs; primarily benzene), semi-volatile compounds (SVOCs; bis(2-chloroethyl)ether and 1,4-dioxane), and metals (primarily iron and manganese). Regionally, the groundwater is impacted with PFAS, predominantly perfluorooctanoic acid (PFOA) and perfluorooctane sulfonate (PFOS).

The ongoing semi-annual monitoring program has been conducted under the Feasibility Study Work Plan Revision 2 (FSWP Rev 2; Golder, 2011) with monitoring wells added in 2012 and 2013 after performance of additional investigation activities as documented in the semi-annual monitoring reports. Annual PFAS monitoring began in 2016, and was performed in October 2016 and April 2017 under the Perfluorinated Chemicals (PFCs) Work Plan (PFCs WP; Golder, 2016b) as approved by the USEPA on September 23, 2016 (USEPA, 2016c). On November 13, 2017, the USEPA requested via email (USEPA, 2017c) addition of sampling and analysis of the Columbia Aquifer and UPCUTZ locations to the PFCs WP. To address this request, the PFAS – Well Purging, Groundwater Sampling and Analysis Program For 2018 dated February 7, 2018 (PFAS 2018 WP; Golder, 2018a) was prepared and submitted to the USEPA for review and approval. The USEPA provided comments on the PFAS 2018 WP via letter dated April 26, 2018 (USEPA, 2018d). These comments are addressed in this SAP-Rev 2.

As presented in the PDI WP-Rev 2, the DS&G Site is adjacent to another Superfund site, the Army Creek Landfill (ACL) Superfund Site, and the Sites are located upgradient of Artesian Water Company's (AWC's) Llangollen wellfield (see CSM in PDI WP-Rev 2 Section 3 for additional information). There are ongoing monitoring activities at the adjacent ACL Site and AWC's Llangollen wellfield which overlap with the ongoing monitoring activities at the DS&G Site. Due to the proximity of the DS&G Site, ACL Site and the AWC wellfield, and the commingled nature of the plumes migrating from the Sites toward AWC's Llangollen wellfield, the USEPA requested addition of cations and anions to the DS&G monitoring program via email dated February 1, 2018 (USEPA, 2018c) consistent with its request for the ACL Site's monitoring program via email dated January 19, 2018 (USEPA, 2018a) with a subsequent letter dated January 24, 2018 (USEPA, 2018b).

As discussed with and agreed to by the USEPA via email dated May 18, 2018 (USEPA, 2018e), the revisions (PFAS 2018 WP comments) and additions (major cations and anions annually) to DS&G's ongoing groundwater monitoring program have been consolidated and are documented in this SAP for ease of review and consistency in methodologies. The SAP (Revision 0 dated August 2018) was followed for DS&G's semi-annual monitoring program in October 2018, and the SAP-Rev 2 will be followed for DS&G's semi-annual monitoring programs beginning in April 2019.

2.2 Project/Task Description

The primary objective of this SAP is to present monitoring, assessment and data analysis procedures designed to implement the data collection programs associated with the PDI and ongoing groundwater monitoring. The resulting data will be used to: 1) address areas of uncertainty associated with groundwater impacts at the Site prior to initiation of the Selected Remedy design, and 2) revise the ongoing semi-annual groundwater monitoring program.

The Data Quality Objectives (DQOs) for the PDI activities described in the PDI WP-Rev 2 are summarized in Table A-1 and the Decision Thresholds/Action Levels are presented in Table A-2. The DQO process as it relates to the Measurement Performance Criteria is described in Section 3.

To address the areas of uncertainty from the FS and collect additional information needed for the Remedial Design (RD), the following tasks will be performed:

- Additional monitoring wells will be installed (see Tables A-3, A-4 and A-5, Figures A-3 and A-4, and PDI WP-Rev 2 Section 6.3.1)
- Water levels and groundwater samples will be collected from new and existing wells (see Tables A-6A through A-6D, Figures A-3 and A-4, and PDI WP-Rev 2 Section 6.3.3)
- Soil samples will be collected for sieve analysis (see Section 4.3.1.4 and PDI WP-Rev 2 Section 6.3.2)
- Groundwater samples will be collected for analysis from a larger subset of Drum Disposal Area (DDA) wells (see Table A-7. Figure A-5 and PDI WP-Rev 2 Section 6.2.5)
- Aquifer testing will be performed (see Table A-8, Figures A-6A through A-6H, and PDI WP-Rev 2 Section 6.3.4)
- Barhole probes will be advanced (see Figure A-7 and PDI WP-Rev 2 Section 6.3.5)

Additional information on these activities is presented in Section 4.

2.3 Project Organization

The lead regulatory agency for the Site is the USEPA with involvement of the State of Delaware Department of Natural Resources and Environmental Control (DNREC). Doug Sutton, PhD, PE is the DS&G Trust Chairperson and Project Coordinator retained by the DS&G Trust, and Golder is the Supervising Contractor retained by the DS&G Trust. The following summarizes the organizations involved in this project and distribution for documents:

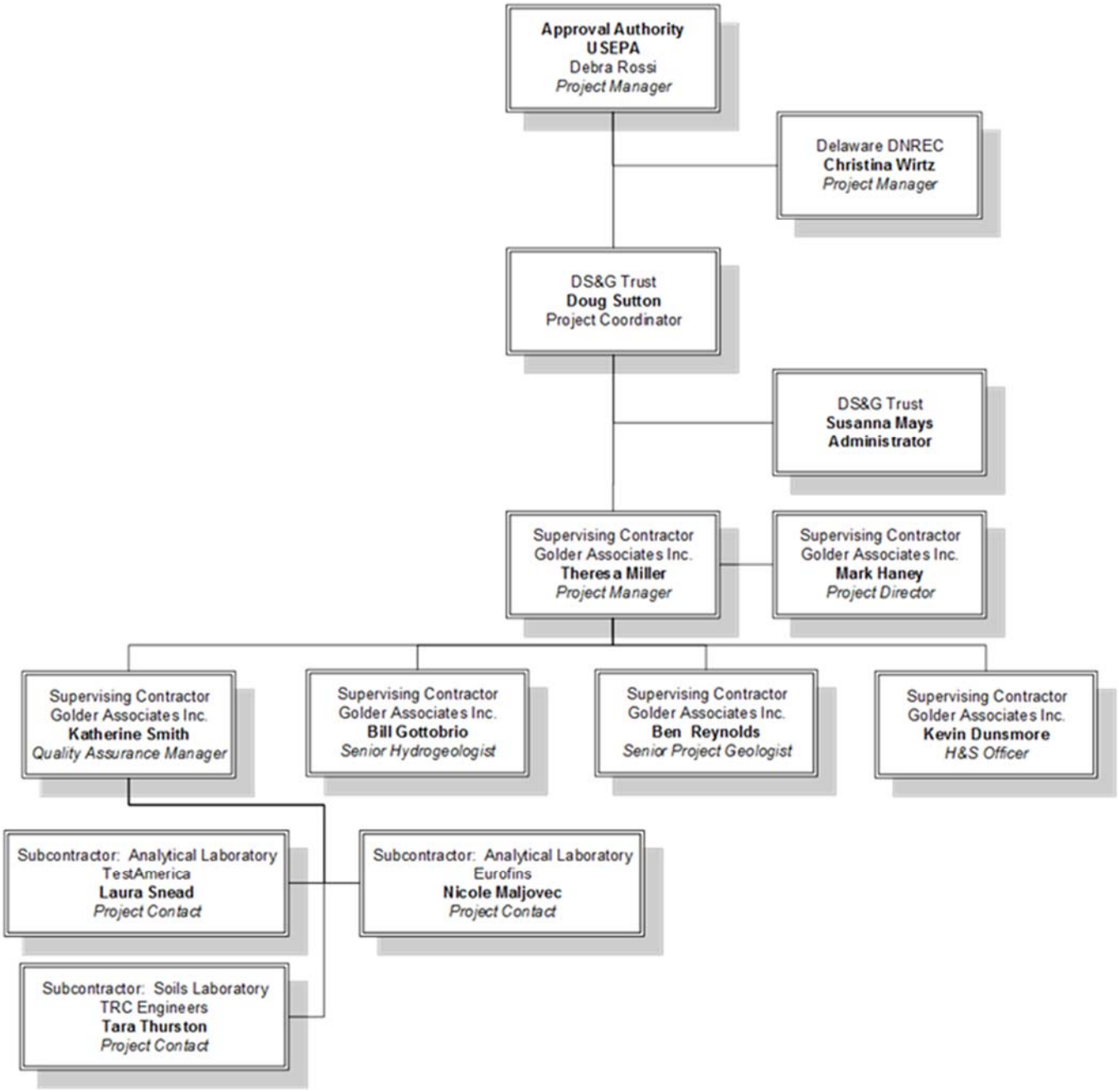
SAP Recipients	Organization
Debra Rossi	USEPA Region III Project Manager
Christina Wirtz	Delaware DNREC Project Manager
Doug Sutton	Chairperson and Project Coordinator, DS&G Trust
Susanna Mays	Administrator, DS&G Trust
Theresa A. Miller	Golder, Lead for Supervising Contractor
Benjamin Reynolds	Golder, Field Coordinator for Supervising Contractor
Katherine Smith	Golder, QA/QC Manager for Supervising Contractor
Joe DiNunzio	Artesian Water Company

The Project Team Organization Chart is shown below. It should be noted that these individuals have primary responsibility for the project, although other individuals may be involved. The chain-of-communication shown below will be followed throughout the project.

Specifically, the above-listed project personnel roles are as follows:

- USEPA Project Manager and USEPA Project Coordinator – Debra Rossi, responsible for regulatory reviews, Agency approvals and coordination between the DS&G Trust and the USEPA
- Delaware DNREC Project Manager – Christina Wirtz, responsible for regulatory reviews, Agency approvals and coordination between the DS&G Trust and the DNREC
- DS&G Trust Chairperson and Project Coordinator - Doug Sutton, represents owner and responsible for project decisions and coordination between the USEPA and Golder, as necessary
- Golder Project Director - Mark Haney, responsible for senior technical review and project direction within Golder
- Golder Project Manager and Lead for Supervising Contractor - Theresa Miller, primary point-of-contact between DS&G Trust and Golder, responsible for technical direction, overall coordination and management between Supervising Contractor and the DS&G Trust
- Golder Senior Project Geologist - Benjamin Reynolds, responsible for supervision of field data collection and management of field activities, interpretation of field data and report preparation
- Golder Senior Hydrogeologist - William Gottobrio, responsible for development, execution and data interpretation associated with aquifer testing activities
- Golder Quality Assurance (QA) Manager - Katherine Smith, environmental chemist responsible for laboratory and sampling subcontractor coordination, and data quality reviews and assessments
- Golder Health and Safety Officer - Kevin Dunsmore, Regional Health and Safety Advisor responsible for the review and coordination for project health and safety plans, staff training and conducting field health and safety audits

Analytical chemistry services are anticipated to be provided by TestAmerica of Edison, New Jersey (TestAmerica Edison), Eurofins Lancaster Laboratories, Inc. (Eurofins) of Lancaster, Pennsylvania, and TRC Companies Inc. (TRC) of Mount Laurel, New Jersey. If use of a different laboratory is required to satisfy project needs, the laboratory quality manual and the laboratory standard operating procedures will be forwarded to the USEPA for review prior to use of the laboratory. Field services will be provided by Golder.



2.4 Special Training/Certification

Golder project team members with appropriate experience, technical skills and training will be selected to perform the project tasks. The subcontractors selected for laboratory analysis were selected based on qualifications and experience of the subcontractor to perform the required work. The subcontractors will meet the general requirements of the USEPA Region III to perform these tasks. Golder's project personnel responsible for data collection and data quality reviews will be trained in relevant procedures and analytical methodologies.

2.5 Documents and Records

The organizations and their personnel listed in the SAP Distribution List will receive the most current approved version of this SAP. This SAP includes the revision number and date and will be updated as needed based on changes in Site conditions and/or applicable regulatory requirements. The revised SAP will be distributed to the USEPA, DNREC, DS&G Trust and AWC. The QAPP portion of the SAP will include revisions/updates (if applicable) every 3 to 5 years.

Golder will maintain electronic copies of all laboratory deliverables as part of the project file. A copy of these electronic deliverables will be incorporated into the Site chemical database. No printed reports of the laboratory data will be maintained, because multiple copies of the portable document format (PDF) report version will be maintained and backed up (by Golder as well as each subcontracted laboratory). Golder will retain chain-of-custody (COC) forms, field documentation forms, including sample field information forms and field notebooks. Golder will file and maintain data and other records (including interim progress reports) in an accessible location on its premises or in an off-site secured file storage facility for a period of at least 5 years.

For laboratory analytical data, PDF deliverables will be produced from the laboratory in a standard reduced deliverable format and the electronic data deliverables (EDD) will be in the EQuIS 4-file and Microsoft Excel database format. At the request of the USEPA (USEPA, 2018d), the PFAS data will be provided to the USEPA and DNREC in the EQuIS 4-file EDD format and Microsoft Excel database format.

The laboratory report format for all analytical data analyses performed by the selected laboratory will consist of the items listed below.

- Case Narrative:
  - Date of issuance
  - Laboratory analysis performed
  - Work order batch number
  - Numbers of samples and respective matrices
  - Quality control (QC) procedures utilized and also references to the acceptance criteria
  - Laboratory report contents
  - Project name and number
  - Condition of samples ‘as-received’
  - Discussion of whether or not sample holding times were met
  - Any deviations from intended analytical strategy
  - Any deviations or modifications of the laboratory standard operating procedures (SOPs)
  - Discussion of technical problems or other observations which may have created analytical difficulties
  - Discussion of any laboratory QC checks which failed to meet project criteria and the corrective actions pursued

  - Signature of the Laboratory QA Officer or designee
- Chemistry Data Package:
  - COC documentation
  - Case narrative for each analyzed batch of samples
  - Summary page indicating dates of analyses for samples and laboratory QC checks
  - Cross referencing of laboratory sample to project sample identification numbers
  - Description of data qualifiers to be used
  - Sample preparation and analyses for samples
  - Sample results (results between the method detection limit (MDL) and quantitation limit (QL) will be reported as estimated values)
  - QC summary package including the results of laboratory control samples (LCSs), matrix spike/matrix spike duplicates (MS/MSDs), interference check samples, serial dilutions, laboratory duplicates, and method blanks
  - EDD containing the results for field and QC samples. The PFAS data will be provided to the USEPA and DNREC in the EQuIS EDD format.

3.0 QUALITY OBJECTIVES AND CRITERIA

This section describes the approach to the Measurement Performance Criteria which uses data quality indicators expressed as precision, accuracy, representativeness, comparability, completeness, and sensitivity (PARCCS). Where possible, acceptance criteria are specified to help delineate minimum acceptability levels for use of data in the overall decision-making process.

3.1 Precision

Precision refers to the degree to which repeated measurements are similar to one another. It measures the agreement (reproducibility) among individual measurements, obtained under prescribed similar conditions. Measurements that are precise are in close agreement with one another.

Field precision is assessed through the collection and measurement of field duplicates. A field duplicate sample is defined as two or more representative portions taken from the same sampling location, homogenized, split and submitted for identical analyses.

Precision in the laboratory is assessed through the calculation of relative percent differences (RPDs) between sample results. The RPD is calculated according to the following formula:

$$RPD = \frac{2 \times |Amount\ in\ Sample\ 1 - Amount\ in\ Sample\ 2|}{(Amount\ in\ Sample\ 1 + Amount\ in\ Sample\ 2)} \times 100$$

Precision control limits for all analyses are provided in the Measurement Performance Criteria, Tables A-9 through A-13. The precision control limits provided are based on the laboratory QC limits, which are routinely re-

evaluated following the procedures in the laboratory quality assurance policies and the requirements of the analytical methods. Should the laboratory QC limits change between the submission of this SAP and the sample analyses, the limits in place at the time of sample analysis will be used to evaluate the data and will be reported with the data usability summary.

3.2 Accuracy

Accuracy is the degree of agreement between an observed value and an accepted reference or true value. The accuracy measurement is generally determined by the percent recovery (%R) of a known value. Accuracy as %R is determined by the following equation:

$$\%R = \frac{(\text{Amount in Spiked Sample} - \text{Amount in Sample})}{\text{Known Amount Added}} \times 100$$

Accuracy in the field is assessed through the use of equipment rinsate and trip blanks to assess the potential of cross contamination. In addition, field accuracy is assessed by the adherence to all sample handling, preservation, and holding time criteria.

Laboratory accuracy and bias will be assessed through the analysis of standard reference materials (SRMs), LCSs, MS/MSDs, surrogate compounds, and the determination of the %R for these measurements. General accuracy control limits are provided in the Measurement Performance Tables, Tables A-9 through A-13. Where accuracy criteria are not met, data will be qualified as either estimated (minor deviation from accuracy criteria) or rejected (major deviation from accuracy criteria). Data qualified as rejected should not be used for decision making purposes.

3.3 Representativeness

Representativeness expresses the degree to which data accurately and precisely represents a characteristic of a population, a parameter variation at a sampling point, a process condition, or an environmental condition within a defined spatial and/or temporal boundary.

Representativeness is dependent upon the proper design of the sampling program and will be satisfied by ensuring that the PDI WP-Rev 2 is followed and that proper sampling techniques are used. The sampling program was designed to provide data representative of Site conditions. During development of this program, consideration was given to historical activities, existing analytical data, physical setting and processes. Using the proper analytical procedures, appropriate methods, meeting sample holding times and meeting QC criteria for each parameter affirms representativeness in the laboratory. An additional assessment of representativeness will be made through field duplicates. While field duplicates are primarily used to assess precision, they also indicate sample homogeneity and therefore the representativeness of the data to the Site.

3.4 Comparability

Comparability is an expression of the confidence with which one data set can be compared to another. Comparability of data is achieved by ensuring Site-wide sample collection and analyses follow the same protocol. Comparability depends upon the proper design of the sampling program and will be satisfied by following the PDI WP-Rev 2, SOPs, and using the proper sampling techniques. The field manager will routinely oversee field activities and verify compliance with field sampling procedures identified in the PDI WP-Rev 2.

Analytical data are comparable when similar analytical methods are used. Appropriate laboratory personnel will review and have a working knowledge of the laboratory SOPs to be used during the analysis of samples for the

investigation. Additionally, the laboratory QA Manager will review data generated, determine compliance with method requirements, and attest that QA objectives are met.

3.5 Completeness

Completeness is a measure of the amount of valid data obtained from a measurement system compared to the amount of data that was expected under normal conditions. Data are considered valid and complete if QC elements have met the criteria established in this SAP. Qualified data may be considered usable and will be considered complete on a case by case basis.

Following completion of the analytical testing, the percent completeness will be calculated by the following equation:

$$\textit{Completeness} = \frac{\textit{(number of valid measurements)}}{\textit{(number of measurements planned)}} \times 100$$

The laboratory and field completeness goal for this project is greater than 85 percent. Field measurements not collected from a specified location, or samples not collected due to environmental conditions, will be identified in the report. Data qualified by the laboratory or data reviewer as estimated is usable and therefore considered complete; however, data qualified as rejected are not usable and do not count toward completeness goals.

3.6 Sensitivity

Sensitivity is defined as the capability of a method or instrument to discriminate between measurement responses representing different levels of a variable of interest. Two measurement responses of interest in assessing sensitivity are the MDL and the QL. The MDL is defined as the minimum concentration of a substance that can be identified, measured and reported with a 99 percent confidence that the substance concentration is greater than zero, for a specific matrix containing the substance. The MDLs are determined as outlined in 40 CFR Part 136. The QL is defined as the level of measurement that can be reliably achieved within specified limits of precision and accuracy during routine laboratory operations. The QLs are generally 2 to 5 times greater than the MDLs.

The sensitivity for field measurements will be determined, in part, by the limitations of field instrumentation as described in the manufacturer's manual and specific field measurement SOPs. Other factors that will influence sensitivity include matrix and environmental conditions.

The MDL and QL requirements for this project are identified in Table A-14. The laboratory will verify analytical QLs defined by a point on the calibration curve which is below the stated QL. Additionally, the laboratory will provide MDL studies for each compound upon request by the USEPA and/or Golder's QA officer. Should the laboratory MDL or QL change between the submission of this SAP and the sample analyses, the limits in place at the time of sample analysis will be used to evaluate the data and reported with the data usability summary.

4.0 DATA GENERATION AND ACQUISITION

This section provides the details regarding the field sampling program including: drilling/soil boring advancement, monitoring well installation and development, groundwater sampling and analysis, equipment calibration and decontamination, handling of investigation-derived waste (IDW), and data management. The DQOs (see Table A-1) and the Decision Thresholds/Action Levels (see Table A-2) for the PDI activities were used in development of these methods. Changes and/or additions to the activities and/or processes in this SAP may be necessary due to changed conditions in the field. The USEPA will be informed of these changes and approvals will be sought where necessary.

4.1 Sampling Process Design

As stated in Section 6.2 of the PDI WP-Rev 2, there are areas of uncertainty associated with the updated CSM as presented in the Final FS Rev 1 that require investigation prior to design of the Selected Remedy. To align these areas of uncertainty with the activities that will be performed to address them, the areas of uncertainty were consolidated and are described under the following subsections: 1) target capture zones and extraction rates; 2) migration between hydrostratigraphic units; 3) extent of contamination in the UPCUTZ; 4) source and extent of UPA manganese impacts; and 5) potential LFG migration. Also, prior to design of the enhanced low-flow extraction system (LFExS), a larger subset of wells within the DDA will be sampled to evaluate the extent of impacts within the DDA. This data from larger set of DDA wells will assist in evaluating the placement of additional extraction wells for the enhanced system.

As presented in Section 4.2, the activities necessary to address these uncertainties include:

- Boring advancement with vertical aquifer profiling (VAP) and soil sampling for grain-size analysis (see PDI WP-Rev 2 Section 6.3.1)
- Monitoring well installation and development (see PDI WP-Rev 2 Section 6.3.1)
- Groundwater monitoring (see PDI WP-Rev 2 Section 6.3.3)
- Aquifer testing (see PDI WP-Rev 2 Section 6.3.4)
- Advancement and screening of barhole probes (see PDI WP-Rev 2 Section 6.3.5)

In addition, there are ongoing activities[1] at the Site which are included in this SAP. These activities include monitoring of the Site-wide groundwater, the LFExS, and extraction well PW-1(U).

All work will be conducted following health and safety protocols, the Health and Safety Plan[2] (HASP; Appendix B of the PDI WP-Rev 2) and the Emergency Response Plan (ERP; Attachment A of the HASP included in Appendix B of the PDI WP-Rev 2).

4.2 Locations, Frequencies and Matrices

This section summarizes the sampling locations, frequencies and matrices detailed in the PDI WP-Rev 2 for the areas of uncertainty and the ongoing Site-wide groundwater monitoring program. The monitoring wells installed as part of the PDI will be incorporated into the ongoing groundwater monitoring program; therefore, the ongoing

[1] There are also ongoing activities related to LFG monitoring and LFG mitigation system operations, maintenance and monitoring (OM&M) which are included in the HASP with the OM&M Plan for the LFG Mitigation System (Golder, 2018b).
[2] Golder's HASP is a Health, Safety and Environment Plan (HASeP).

groundwater monitoring list has been updated and is presented after the PDI WP-Rev 2 information for addressing the areas of uncertainty.

4.2.1 Activities to Address Areas of Uncertainty

4.2.1.1 Target Capture Zones and Extraction Rates

As discussed in Section 3 of the Final FS Rev 1 and in Section 6.2.1 of the PDI WP-Rev 2, prior to design and implementation of the Selected Remedy, PDI activities are needed to confirm the target capture zones for the proposed extraction wells and address remaining areas of uncertainty coming out of the FS process. Table A-3 provides the list of proposed well locations and the primary and secondary purposes of the proposed PDI activities for these locations, and Table A-4 provides a matrix illustrating which uncertainties will be addressed by the proposed well locations. The proposed locations of these wells are shown in Figures A-3 and A-4. Following a baseline monitoring event, the new wells will be sampled semi-annually as described in Section 4.2.3 below.

As presented in the Final FS Rev 1, the proposed activities for the PDI to assist with design of the Selected Remedy target capture zones and extraction rates will require a phased approach and will consist of the following general sequence of activities:

- Phase 1 – Advance borings and install monitoring wells using Rotosonic drilling methods with VAP, collect soil samples for grain-size analysis to assist with extraction well design, and perform baseline sampling (after well development) to confirm target capture zones (see PDI WP-Rev 2 Sections 6.3.1 and 6.3.3).
- Phase 2 – Perform aquifer testing in the potential extraction well locations to evaluate the modeled extraction rate, evaluate the connection between the hydrostratigraphic units, and evaluate the nature/orientation of the "feature" used in the groundwater flow model to simulate the localized hydraulic mounding in the well P-6 area (see PDI WP-Rev 2 Section 6.3.4).
- Phase 3 – Update the numerical groundwater flow model including the geologic model using the observations from Phases 1 and 2 and recalibrate the model using hydraulic data from aquifer testing of the new extraction wells and the adjustments to AWC's Llangollen wellfield described in Section 6.3.4.1 of the PDI WP-Rev 2. Using the updated and recalibrated model, develop design extraction rates for the potential remedy wells, refine the capture zones of those wells, and evaluate the capture of the plume provided by AWC's Llangollen wellfield under various operating conditions (see PDI WP-Rev 2 Section 6.3.7).
- Phase 4 – Perform additional assessment (assumed to be similar to Phases 1 through 3), if necessary, to evaluate target capture zones, design extraction rates and/or areas of uncertainty not adequately addressed in Phases 1, 2, or 3. As requested by the USEPA in its PDI WP-Rev 1 comments, "Phase 4 activities may include, but shall not be limited to, determination of the extent of contamination north of the DDA slurry wall, evaluation of the potential for impacts to surface water due to discharge from the Columbia Aquifer to Army Creek downstream of Army Creek Landfill surface water and sediment sampling location A, and installation of additional monitoring wells to confirm capture at the Llangollen well field." (USEPA, 2019)

The methodologies for these activities are presented in Section 4.3. Additional details about these activities are included in Sections 6.2 and 6.3 of the PDI WP-Rev 2. The SAP-Rev 2 provides the details required to perform Phases 1 and 2, and the PDI WP-Rev 2 describes the evaluation included as Phase 3. If Phase 4 activities are necessary, then an addendum to the PDI WP-Rev 2 and SAP-Rev 2 will be prepared to address those activities.

4.2.1.2 Migration Between Hydrostratigraphic Units

As discussed in Section 6.2.2 of the PDI WP-Rev 2, the interaction between the UPCUTZ and UPA upper sand will be evaluated, including aquifer testing and groundwater monitoring to assess the area of greatest contaminant mass flux from the UPCUTZ to the UPA. Tables A-3 and A-4 indicate which wells will be used in this evaluation, and Figures A-3 and A-4 show these locations.

As part of the PDI activities, the connection between the upper and lower sand units of the UPA between and downgradient of the Sites will be evaluated. Tables A-3 and A-4 indicate which wells will be used in this evaluation, and Figures A-3 and A-4 show these locations. The methodologies for these activities are presented in Section 4.3 of the SAP and discussion of the groundwater evaluation is provided in Section 6.3 of the PDI WP-Rev 2.

4.2.1.3 Manganese and Other COCs in Groundwater

As described in Section 6.2.3 of the PDI WP-Rev 2, there is migration of manganese and other contaminants of concern (COCs) to the area between the DS&G and ACL Sites and increasing manganese concentrations have been observed in AWC's Llangollen wellfield. Also, metals were detected in temporary Columbia Aquifer wells in 2011. These areas of uncertainty will be evaluated through installation of monitoring wells, and semi-annual groundwater monitoring. Tables A-3 and A-4 indicate which wells will be used in this evaluation, and Figures A-3 and A-4 show these locations. The methodologies for these activities are presented in Section 4.3. Additional discussion of these groundwater evaluations is provided in Section 6.2.3 of the PDI WP-Rev 2.

4.2.1.4 Extent of Contamination in UPCUTZ

As discussed in Section 6.2.4 of the PDI WP-Rev 2, the extraction wells associated with the Selected Remedy are expected to mitigate migration of the groundwater impacts within the UPCUTZ; however, additional investigation activities in the UPCUTZ are required prior to design of the extraction systems. Tables A-3 and A-4 indicate which wells will be used in this evaluation, and Figures A-3 and A-4 show these locations. The methodologies for these activities are presented in Section 4.3. Additional discussion of these groundwater evaluations is provided in Section 6.2.4 of the PDI WP-Rev 2.

4.2.2 Assessment for Enhanced LFExS Design

Prior to design of the enhanced LFExS (ELFExS), a coincident set of groundwater samples from wells and vapor points screened within the DDA water table will be collected. A similar monitoring event was performed in October 2008 prior to the design of the existing LFExS. Table A-7 indicates which locations will be monitored for this evaluation, and Figure A-5 shows these locations. The methodologies for these activities are presented in Section 4.3.

4.2.3 Potential LFG Migration

As indicated above, there is an LFG Mitigation System in operation between the existing receptors (Cirillo Brothers' Office Building, Vincent Dell'Aversano's residence and the DS&G Treatment Building) and the Inert Area and Grantham South. In the ROD-A2 (USEPA, 2017d), the USEPA included delineation of areas outside the influence of the LFG Mitigation System and beyond the boundary of the Inert Area and the Grantham South Area impacted by subsurface migration of landfill gas in order to ensure that institutional controls are in place to prevent potential future exposure to all areas where Site-related vapor intrusion adversely affects indoor air.

A barhole probe investigation will be performed to assess these areas. Figure A-7 shows the proposed investigation area. The methodologies for these activities are presented in Section 4.3. Additional discussion of this evaluation is provided in Section 6.2.6 of the PDI WP-Rev 2.

4.2.4 Ongoing Monitoring Activities

Groundwater is monitored on a semi-annual basis in accordance with the SAP Rev 2 included in FSWP Rev 2 dated October 21, 2011 (prior to 2011, groundwater was monitored in accordance with Revision #1 Sampling and Analysis Plan Addendum dated June 15, 2009). Groundwater was monitored for PFAS in October 2016 and April 2017 in accordance with the PFCs Sampling Plan (Golder, 2016b). The monitoring program includes points for monitoring performance and discharge of the LFExS at the DDA and well PW-1(U), as well as routine groundwater monitoring at and downgradient of the Site.

Samples collected during the semi-annual monitoring events will be analyzed for the parameters indicated on Table A-6C, which include VOCs with low-level 1,4-dioxane as needed, SVOCs with low-level BCEE as needed, dissolved iron, dissolved manganese and total Target Analyte List (TAL)[3] metals. Well PW-1(U) and the LFExS effluent (New Castle County [NCC] sewer discharge monitoring points) will be analyzed for discharge permit parameters (total metals including arsenic, cadmium, chromium, copper, lead, molybdenum, nickel, selenium, zinc, and mercury, and general chemistry parameters including ammonia, total cyanide, biochemical oxygen demand, and total suspended solids).

During one monitoring event, coincident with an ACL monitoring event, samples will be collected for analysis of major cations and anions (i.e., calcium, magnesium, sodium, potassium, ammonia, nitrate, nitrite, ferrous iron, bicarbonate, chloride, sulfate, and sulfide; see Table A-6C).

Additional groundwater monitoring is performed at four downgradient wells (MW-26N, UPA-03D, AWC-E1, and AWC-E2) approximately every two months (see Table A-6D) due to their location upgradient of AWC's production well G-3R. AWC samples its operating production wells monthly or quarterly (see Table A-6D) depending on its monitoring and reporting requirements. Semi-annual groundwater monitoring as well as the monthly, bi-monthly, quarterly and annual monitoring by the Trust and/or AWC will continue as presented on Tables A-6A through A-6D at the locations shown on Figures A-3 and A-4. The methodologies for these activities are presented in Section 4.3. These data will continue to be evaluated and reported as part of the semi-annual monitoring reports. No changes are proposed to the format and/or content of the current semi-annual reports.

4.3 Sampling and Data Collection Methods

Golder's Standard Operating Procedures for drilling and groundwater sampling are included in Appendix I at the request of the USEPA; however, the information in this section was developed specific to the activities to be performed at the DS&G Site for the RD AOC SOW. As such, the methods presented below should be followed in the event of a difference between Golder's SOPs and this SAP-Rev 2.

4.3.1 Drilling

4.3.1.1 Pre-Drilling Activities

Soil borings will be advanced using Rotosonic drilling methods. Rotosonic drilling is a dual-cased system that employs high frequency vibration to obtain continuous core samples of unconsolidated formations and many

[3] TAL metals include aluminum, arsenic, barium, beryllium, calcium, chromium, cobalt, copper, iron, lead, magnesium, manganese, nickel, potassium, sodium, vanadium, and zinc.

consolidated formations (including bedrock), and/or to advance casing for well construction and other purposes. Rotosonic rigs have been used successfully in the area of the Site. A driller licensed by the State of Delaware will be utilized for the work. The Delaware-licensed driller will obtain drilling permits for the monitoring wells.

Prior to any ground disturbance or at the proposed drilling locations, the following activities will be performed for the drilling locations to avoid subsurface utilities:

1) The drilling locations will be pre-marked out and Miss Utility will be contacted to mark out utilities on public properties;

2) Available Site drawings and public utility information will be reviewed to locate utilities on private and public properties; and

3) Private utility locating service will be contracted to perform ground-penetrating radar (GPR) and/or electromagnetic (EM) surveys

Due to the known presence of utilities on private property that are undetectable using GPR and EM (polyvinyl chloride [PVC] water lines) within the vicinity of the proposed drilling locations, the drilling locations along Grantham Lane will be cleared using soft- dig procedures to 4 feet below ground surface (bgs) before drilling.

Because water will be needed for drilling and VAP sampling (see Section 4.3.1.3) will be performed during drilling, a sample of the drilling water will be collected for analysis of VOCs, SVOCs and PFAS at commencement of the drilling activities. It is assumed that drilling water will be supplied from a fire hydrant on the Cirillo property, consistent with previous practices for drilling activities at the Site. Additional samples of drilling water will be collected for analysis if/when the source of drilling water is changed.

PFAS-compliant materials (as certified by the manufacturer), including drilling fluids and tooling lubricants, will be used during drilling activities. If information related to PFAS compliance of a material is not available, the driller may be asked to change materials used, if possible, or a material sample or rinse sample of equipment (if applicable) will be collected for analysis of PFAS.

4.3.1.2 Boring Advancement

The anticipated drilling sequence is as follows:

- Core barrel advancement (using limited fluids, air, or muds)
- Casing override (potable water minimally used between outer and inner casing)
- Core barrel and geologic sample retrieval
- Repeat core advancement

The proposed borings (see Section 4.3.2, Tables A-3 through A-5, and Figures A-3 and A-4) will be advanced using Rotosonic drilling techniques which will allow for a collection of continuous soil core. It is recommended that the first boring installed in the location of a proposed UPA upper and lower sand well pair be advanced to the top of the Middle Potomac Confining Unit (MPCU; see Figure A-2) and be completed with a UPA lower sand well. To avoid the potential for communication between the UPA upper sand and UPA lower sand wells within a pair, the separate borings will be advanced such that the wells will be installed in borings that are separated from each other by between 15 and 25 feet horizontally.

For each proposed boring identified in Section 4.3.2, an 8-inch to 12-inch diameter (dependent on anticipated screened lithologic unit), threaded, permanent steel isolation casing will be installed to prevent mixing between potential monitoring intervals. Boreholes will be advanced according to the sequence above. Once the Upper Potomac Confining Unit (UPCU; clay layer) is encountered (confirmed by recovered drill cuttings), the permanent steel isolation casing will override the drill casing and be advanced 2 feet into the clay layer. The annular space between the permanent isolation casing and override casing will then be pressure tremie-grouted to the ground surface and will remain in-place. Grout will be allowed to set for a minimum of 24 hours before resuming drilling and the borehole will be advanced through the isolation casing. Upon completion of the boring/monitoring well, the override casing will be retrieved and the remaining annular space between the well casing and isolation casing will be grouted to the surface.

If the UPCU is absent, the isolation casing will be grouted into a finer-grained (lower conductivity) interval of the UPCUTZ. If both the UPCU and UPCUTZ are not observed during drilling, the isolation casing will be grouted approximately 2 feet into the top of the UPA upper sand as observed in the field based on lithologic changes in recovered soil cores during drilling. If the borehole is advanced beyond the desired isolation interval, the borehole will be backfilled with bentonite chips and hydrated.

Upon curing of the grout, the boring will be advanced to the Upper Potomac Dividing Clay (UPDC). Once the UPDC (clay layer) is encountered, an 8-inch to 10-inch diameter, threaded, permanent steel isolation casing will be advanced 2 feet into the clay layer. The isolation casing will then be pressure tremie-grouted to the ground surface. During grouting, the isolation casing will be recovered at a rate that ensures that the base of the casing remains below the tremied-grout surface. Grout will be allowed to set for a minimum of 24 hours before resuming drilling. If the UPDC is not encountered (i.e., no lithologic separation between the UPA upper and lower sand is observed), then there is no need for isolation casing between the UPA upper sand and UPA lower sand units for a UPA lower sand well, and the boring will be advanced until the top of the MPCU is encountered.

Recovered soil core in sample bags will be marked to verify proper orientation and sequence. The recovered core will be photo-documented, field screened for evidence of impacts from organic compounds using a photoionization detector (PID), and soil lithology will be logged by the Golder field geologist.

The drill rig and all drilling and sampling equipment coming in contact with subsurface soils will be decontaminated in accordance with procedures contained in Section 4.4.

4.3.1.3 Vertical Aquifer Profiling

During advancement of the profiling boring in each location through the UPCUTZ and the UPA upper and lower sand, the groundwater will be sampled using the Push-Ahead™ groundwater sampling system (for vertical aquifer profiling) used with sonic drilling methods. This system is used to collect representative groundwater samples directly from the interval of interest without requiring purging; however, the interval will be purged as described below. The sampler consists of a 2 7/8-inch inner diameter drill rod threaded onto a carbide-tipped point with flanges and sampling ports. The sampling ports remain closed until the sampler is advanced to the desired sampling interval, then the ports are opened by rotation of the drill rod.

For intervals that produce water, groundwater samples will be collected across a 2-foot interval for each 10-foot advance from the top of the UPCUTZ to the base of the hydrostratigraphic unit(s) under evaluation. Once a soil core and the core barrel are removed, the Push-Ahead™ sampler will be inserted through the outer casing to the bottom of the boring, then the sampler will be sonically driven beyond the bottom of the boring into undisturbed material (up to 10 feet in advance of the outer casing).

After the presence of water in the sampler is verified using a water level indicator, groundwater samples will be collected by lowering a submersible pump with dedicated polyethylene tubing and purging from the Push-Ahead™ sampler. For each 2-foot sampling interval, field parameters will be measured with a water quality meter and recorded on a field sampling form. The interval will be purged until the water quality parameters are similar to historic water quality parameters in existing wells screened within the same unit in the vicinity of the borehole. A multi-gas detector (VRAE or equivalent) reading will be collected directly from the pump discharge.

After collection of the groundwater sample, the drill rod with the Push-Ahead™ sampler will then be removed and the core barrel will be placed into the boring to continue drilling. The sampler will be decontaminated prior to collecting a sample from the next interval.

The groundwater sampling and boring advancement will continue through the UPA to the base of the lower sand. The groundwater samples will be collected from 10-foot intervals from the top of the UPCUTZ to the base of the UPA and analyzed for VOCs and SVOCs by TestAmerica of Edison, New Jersey with expedited (24-hour tor 48-hour) turnaround.

4.3.1.4 Soil Sampling for Grain-Size Analysis

During advancement of borings, one soil sample from each of five locations (UPA-101-LS; UPA-105[A or B]; UPA-107;UPA-108[A, B or C]; and DDA-20) will be collected from the hydrostratigraphic unit proposed for extraction (UPA upper sand or UPA lower sand) to support the design of the extraction well filter packs (see PDI WP-Rev 2, Tables A-3 through A-5, and Figures A-3 and A-4). Soil samples will be collected from an interval in the UPA upper sand or lower sand that corresponds to the desired screened interval of that proposed well. The desired screen interval will be based on the same criteria presented in Section 4.3.2 for monitoring well installation and in consideration of the coarsest interval within the unit.

The samples will be collected and placed in laboratory-supplied containers and then submitted under COC protocols to TRC for sieve analysis. The filter pack and well screen design will be based on the results of the sieve analysis.

In accordance with Design and Installation of Monitoring Wells by Striggow dated February 13, 2008 (USEPA SESDGUID-101-R0; USEPA, 2008), the selection of the design for the filter pack and well screen, will consider the following factors:

1) Select the well screen slot openings that will retain 90 percent of the filter pack material.

2) The filter pack material should be of the size that minimizes head losses through the pack and also prevents excessive sediment (sand, silt, clay) movement into the well.

3) A filter material of varying grain sizes in not acceptable because the smaller particles fill the spaces between the larger particles thereby reducing the void space and increasing resistance to flow; therefore, filter material of the same grain size and well-rounded is preferred.

4) The filter pack design is based on the gradation of the finest aquifer materials being analyzed.

For further guidance on specifications for well design, American Society for Testing and Materials (ASTM) Standard D5092 (ASTM, 2016) will also be considered.

Additional information regarding installation of proposed extraction wells is provided in Section 4.3.2.2 and details regarding the proposed well construction will be provided to the USEPA for review and approval prior to installation of the extraction wells.

4.3.2 Monitoring and Extraction Well Installation and Development

A driller licensed by the DNREC will be utilized for the work. The driller will obtain drilling permits for the monitoring wells prior to commencement of the drilling activities.

4.3.2.1 Monitoring Well Installation

As described in Section 4.3.1.2 (see Tables A-3, A-4 and A-5, and Figures A-3 and A-4), the proposed monitoring wells will be installed in borings advanced using Rotosonic drilling techniques. It is recommended that the first boring/well installed in the location of a proposed UPA upper and lower sand well pair be advanced to the top of the MPCU and be completed with a UPA lower sand well. Wells will be installed in borings that are separated from each other by between 15 and 25 feet horizontally. For locations where monitoring wells will be installed in both the UPCUTZ and the UPA upper sand well, the UPCUTZ well will be installed 15 feet upgradient, where possible, of the UPA upper sand well to allow for a degree of separation between the UPCUTZ and the UPA upper sand wells during aquifer testing.

The 5- to 7-foot screened interval for each well will be determined based on the following:

- Interval with the highest VOC and SVOC concentrations in groundwater, as indicated by the VAP groundwater sampling
- Interval with the highest PID readings within the saturated portions of the hydrostratigraphic unit under evaluation
- Interval with visual and/or olfactory evidence of impacts as observed during drilling
- Interval with lithology that is consistent with the unit of interest (i.e., Columbia Aquifer, UPCUTZ, UPA upper sand, or UPA lower sand) and which will produce water for sampling

In the absence of impact evidence, the well will be screened across interval with the coarsest material within the saturated portions of the hydrostratigraphic unit under evaluation (proposed well locations).

Prior to well construction within the advanced borehole, the USEPA will be provided with a draft annotated boring log indicating the proposed well screen interval for their review and approval. Due to time constraints associated with drilling activities, a quick response/approval (within two business days) from the USEPA will be necessary.

For locations where the proposed well screen interval is shallower than the drilled depth of the borehole, the borehole will be backfilled/tremied with bentonite chips to within 6 inches to 1 foot of the base of the well. This practice is intended to avoid creation of a “filter pack” in the overdrilled borehole beneath the base of the constructed well.

Monitoring wells will be constructed of either 2-inch, 4-inch, or 6-inch diameter[4], 5- to 7-foot long, 0.010-inch (No. 10-slot) polyvinyl chloride (PVC) screen and appropriate lengths of 2-inch, 4-inch, or 6-inch diameter, 10-foot long flush–threaded (with a non-Teflon seal) PVC riser pipe. A sand (filter) pack comprised of clean, washed, acid-

[4] The proposed well diameters are included in the PDI WP-Rev 2 and may be altered based on observations in the field during boring advancement, results of VAP sampling, and/or consideration for use as an extraction well during aquifer testing.

resistant, #1-sized silica sand will be placed in the annulus from within 6-inches to 1-foot below and to 2 feet above the well screen interval. The sand pack will be poured and continuously sounded until it extends to at least 2 feet above the top of the screened interval. A minimum of 0.5-foot thick #00 filter pack sand will be placed on top of the #1 sand pack, followed by a 2-foot thick seal of bentonite pellets tremied on top of the sand, and completed by a pressure tremie-grouted cement/5% bentonite slurry. Surface casings and concrete pads will be placed around each well, no sooner than 24 hours following well grouting. Surface completion, either flush-mount road boxes or standpipes with steel protective casings, will be dependent on the location of the monitoring well.

4.3.2.2 Extraction Well Installation

As described in Section 4.3.1.2 (see Tables A-3, A-4 and A-5, and Figures A-3 and A-4), the proposed extraction wells will be installed in borings advanced using Rotosonic drilling techniques. Wells will be installed in borings that are separated from each other by between 15 and 25 feet horizontally. For locations where monitoring and extraction wells will be installed in both the UPCUTZ and the UPA upper sand, the UPCUTZ well will be installed 15 feet upgradient of the UPA upper sand well to allow for a degree of separation between the UPCUTZ and the UPA upper sand wells during aquifer testing.

The screened interval for each well will be determined based on the following:

- Interval with the highest VOC and SVOC concentrations in groundwater, as indicated by the VAP groundwater sampling
- Interval with the highest PID readings within the saturated portions of the hydrostratigraphic unit under evaluation
- Interval with visual and/or olfactory evidence of impacts as observed during drilling
- Interval with coarsest material (for UPCUTZ, interval most likely to produce water)

In addition, grain-size analysis results will be used to design the well screen. It is anticipated that extraction wells will be constructed of either 6-inch or 8-inch diameter, stainless-steel screen, and appropriate lengths of 6-inch or 8-inch diameter stainless-steel riser pipe. Other specifications regarding well construction (e.g., filter pack material, slot size, screen length) will be based on sieve analysis and field observations. Prior to extraction well construction, the USEPA will be provided with the following for their review and approval: 1) an addendum to the PDI WP-Rev 2 addressing specifics of the extraction well construction (well materials, screen length, slot size, and filter pack design, surface completion, etc.), and 2) boring log annotated with criteria used in determining the recommended well screen interval.

4.3.2.3 Well Development

All new wells will be developed prior to the initiation of any groundwater sampling. Wells will be developed using surge blocks and continuous cycles of over-pumping and recovery until relatively clear water is produced, and field parameters (pH, specific conductance and turbidity) stabilize indicating good hydraulic communication with the surrounding water bearing zone. Field parameters will be measured with a calibrated water quality meter. Because these wells will be sampled for PFAS, PFAS-free extraction equipment and high-density polyethylene (HDPE) tubing will be used during development.

4.3.2.4 Well Elevation and Location Survey

Monitoring wells will be surveyed by a surveyor licensed in the State of Delaware after installation for location and elevation including ground surface, top of PVC and top of steel casing elevations. The survey datum will be consistent with existing datum used for the Site as follows: horizontal datum is Delaware State Plan Coordinate System North American Datum (NAD) 1983; and vertical datum is National Geodetic Vertical Datum (NGVD) 1929.

4.3.3 Well/Piezometer Sampling Procedures

After well construction and development, the new monitoring wells will be allowed to stabilize and equilibrate with the aquifer for a minimum of two weeks prior to sampling. Due to the extremely low method detection limits associated with PFAS analysis and the numerous potential sources of trace concentrations of PFAS, detailed operating procedures to reduce the potential for cross contamination and false positive sample results were developed for the groundwater monitoring program. As included in Attachment A, PFAS sampling activities will be performed in accordance with the general methods and procedures described in *SOP–1: General Field Methods for PFAS Sampling Programs*.

Tables A-6A through A-6D indicates which locations will be monitored and the monitoring method, and Figures A-3 and A-4 show the locations.

4.3.3.1 Water Level Measurement Procedures

Depth to water measurements should be collected for all wells indicated on Table A-6A within a time period (not to exceed 48 hours) that is not interrupted by severe changes in barometric pressure (+/- greater than 1 inch of mercury) or by precipitation events. The synoptic water level measurements will be performed AFTER collection of groundwater samples due to the inclusion of PFAS as an analyte at the Site.

Depth to water will be measured in each monitoring well to the nearest 0.01-foot using an electronic depth-indicating sounder. All groundwater measurements will be made in reference to a control point of known elevation at the top of the well casing. If a total depth measurement is necessary, to confirm well construction information for example, it will be taken after any scheduled sample collection to minimize potential cross-contamination and disturbance to sediments, which may have accumulated in the bottom of the well.

The water level will be decontaminated between locations using certified PFAS-free de-ionized (DI) water.

4.3.3.2 Vertical Flow Screening

Approximately two weeks prior to purging and sampling wells MW-26N and MW-34 (screened across both the UPA upper and lower sands), an electromagnetic or spinner flow-meter will be used for a one-time event to log the vertical flow within the wells, and adjustments to proposed low-flow purging and sampling depths will be made if necessary (see PDI WP-Rev 2 Section 6.3.3 and Table A-6B).

A vertical flow meter will be deployed in wells with long-screen intervals to assess the ambient direction and velocity of flow within the screen interval of the well. This assessment will be performed for wells screened across different lithologies (particularly wells screened across both the UPA upper and lower sands) at the Site to evaluate the migration between lithologic units, if any. The vertical flow meter will not be used to calculate horizontal hydraulic conductivity and/or determine the hydraulic conductivity distribution of the screened interval.

To screen the well, the flow meter will be lowered to the base of the well and the well will be allowed to equilibrate for 10 minutes. Screening measurements will be made from the base of the well to one foot above the top of the

screened interval at 1-foot increments for wells with 20 feet or less of screen and at 2-foot increments for wells with more than 20 feet of screen. The readings will be recorded, tabulated and plotted for evaluation.

4.3.3.3 Low-Flow Groundwater Sampling Procedures

Groundwater samples will generally be collected using the low-flow purge and sampling technique[5] consistent with previous work at the Site[6]. Prior to sampling, each monitoring well will be purged using a dedicated or decontaminated 2-inch submersible pump (Grundfos RediFlo™, Proactive™ or equivalent) and HDPE tubing dedicated to each well. The pump intake will be placed consistent with past sampling depths (see Table A-6C). Wells are typically purged at a rate of approximately 200 to 500 milliliters per minute (ml/min) with a goal of less than 0.1 meters (0.3 feet) of drawdown during purging. Best efforts will be made to minimize well drawdown by adjusting the flow when necessary and frequently monitoring the water level during purging. A minimum of 3 feet of water will be maintained over the pump intake at all times to avoid the risk of entrainment of air and pump overheating.

For the PFAS groundwater monitoring event, Golder will purge and sample each well using low-flow sampling procedures in accordance with this SAP and *SOP-2: PFAS Program Monitoring Well Purging and Sampling Protocols* (see Attachment A) prior to groundwater sample collection.

During purging, field parameters (temperature, pH, oxidation-reduction potential [ORP; measured using a platinum electrode], turbidity, specific conductance and dissolved oxygen [DO]) will be monitored with a Horiba U-52 instrument (or equivalent). Measurements will be collected using a flow-through cell device to minimize sample exposure to the atmosphere. All measurements will be recorded on the Low-Flow Groundwater Purge/Sample Field Information Form (see Attachment B) or other equivalent low-flow purge and sampling forms. Measurements will be collected approximately every 5 minutes. For water quality stabilization purposes, measurements will not be evaluated for stabilization until at least one tubing volume (including pump and flow through cell volume) have been purged from the well[7]. Once removal of one tubing volume has been achieved, stabilization will be based on three consecutive readings within the following ranges:

---

[5] The procedure is based upon either the USEPA Region II document entitled “Groundwater Sampling Procedure, Low Stress (Low Flow) Purging and Sampling” dated March 20, 1998 (USEPA, 1998b) and the USEPA document entitled “Low-Flow (Minimal Drawdown) Ground-Water Sampling Procedures” dated April 1996. (USEPA, 1996a)

[6] Golder notes that there are wells with screen lengths greater than 10-feet that are included in the sampling program … While flow-weighted average samples would be more representative of the interval across which the long-screen wells are screened, it should be recognized that long-screen wells were installed to monitor the entire aquifer thickness, but that plume thicknesses can be much less than the aquifer thickness; therefore, collecting samples from depths within the long-well screen consistent with the groundwater quality data in-hand is more useful in our evaluation than a flow-weighted average sample. Furthermore, the proposed low-flow purging and sampling approach presented in the PDI WP is applicable for collecting samples from discrete intervals to evaluate the contaminant plume elevation within the wells. Using a pumping rate slightly less than the yield of the well (which is tens of gallons per minute) would not provide discrete interval information, and could create turbulence in the well, entrain particulates in the samples, and strip VOCs from the samples. As stated in “Recommended Procedure for Low-flow Purging and Sampling of Groundwater Monitoring Wells, Bulletin No. QAD023” dated October 15, 1997, “[r]esearch conducted by Puls et al. (1992), Puls and Powell (1992), and Powell and Puls (1993) has shown that high-volume purging and sampling cause significant turbidity and suspended particulate artifacts that can result in biased-high metals results. Additionally, purging can cause pressure changes and bailing can cause aeration that can strip VOCs from the sample (Pennino, 1988). The use of low-flow pumping devices (preferably dedicated) for purging and sampling minimizes both the disturbance of water in well casing and the potential for mobilization of colloidal material (Barcelona et al., 1994). Low-flow purging with maintenance of water level in the well and stabilization of indicator parameters (especially turbidity) allows collection of groundwater samples that are more representative of conditions without filtering (U.S. EPA, 1993; Backhus et al., 1993).” (USEPA, 1997)

[7] This procedure is based on Ground-Water Sampling Guidelines for Superfund and RCRA Project Managers Ground-Water Sampling Guidelines for Superfund and RCRA Project Managers dated May 2002, written by Yeskis, Douglas and Zavala, Bernard (Yeskis, Zavala, 2002).

- Temperature:+/- 10%
- pH:+/- 0.1 Standard Units
- Conductivity: +/- 3%
- ORP: +/- 10 millivolts (mV)
- DO: +/- 10% (or +/- 0.1 milligrams per liter [mg/L] if less than 1.0 mg/L)
- Turbidity:+/- 10% (or three consecutive readings below 50 Nephelometric Turbidity Units [NTUs])

Once purging is complete, the discharge tubing will be disconnected from the flow-through cell and samples will be collected directly from the end of the discharge tubing. Certified-clean sample bottles, provided by the laboratory, will be filled by allowing the pump discharge to flow gently down the inside of the bottle with minimal agitation. Each pre-labeled bottle will be capped as it is filled. VOC samples will be collected first, at a flow rate of 100 to 250 ml/min, taking steps to eliminate all headspace in the vials. Immediately after filling, each VOC vial will be checked by inverting the vial and tapping the side of the vial to check for air bubbles. If air bubbles are discovered, the vial will be discarded and a new vial with be filled and checked for bubbles. The above procedure will continue until a minimum of two VOC vials per sample location are collected.

During the PFAS monitoring event, samples for PFAS analysis will be collected subsequent to VOC sample collection, otherwise samples for metals analyses will be collected subsequent to VOC sample collection. Consistent with Site practices over more than the last 10 years and to maintain a consistent data set, metals samples will be collected for analysis of total and dissolved metals. The filtered (dissolved) metals samples will be collected using an in-line 0.45-micron filter attached to the end of the discharge tubing without a flow-through cell in-place. While it is anticipated that wells will have a turbidity less than 10 NTUs (low-flow guidance allows for dissolved metals analysis if turbidity is greater than 10 NTUs), it is possible that entrained particles (metal flakes due to corrosion of old steel extraction well casings) will be included in the total metals samples. Therefore, total as well as dissolved metals samples should be collected and analyzed.

The samples will be preserved according to method-specific requirements, and promptly placed in a cooler with wet ice and maintained at approximately 4 degrees Celsius (°C). Following sampling, the samples will be shipped under COC procedures to the analytical laboratory(ies).

4.3.3.4 Volume Average Purging Using Submersible Pumps

Following low-flow purge and sampling during the April 2019 event, a subset of wells will be purged and sampled as a one-time event using volumetric averaging (3-well-volume purging) followed by sample collection (see PDI WP-Rev 2 Section 6.3.3 and Table A-6B). Prior to sampling, each monitoring well will be purged of 3 well volumes using a dedicated or decontaminated 2-inch submersible pump (Grundfos RediFlo™, Proactive™ or equivalent) and HDPE tubing dedicated to each well. The pump intake will be placed at the midpoint of the well screen.

Using the well construction information, the volume of standing water in each casing will be calculated using the following equation:

Standing Water Volume = ((Well depth) – (Water level))(Casing Volume/foot))*

Casing Volumes		
2-inch casing	**4-inch casing**	**6-inch casing**
0.163 gallons/foot	0.653 gallons/foot	1.47 gallons/foot

Wells are typically purged at a rate greater than one liter per minute (l/min). The wells for which this method will be used recharge quickly; therefore, a minimum of 3 feet of water will be maintained over the pump intake at all times to avoid the risk of entrainment of air and pump overheating.

During purging, field parameters (temperature, pH, oxidation-reduction potential (ORP; measured using a platinum electrode), turbidity, specific conductance and dissolved oxygen [DO]) will be monitored with a Horiba U-52 instrument (or equivalent). Measurements will be collected from the initial well volume of water, and following removal of each well volume. All measurements will be recorded on the Volume Average Groundwater Purge/Sample Field Information Form (Attachment C) and/or in field notebooks. This procedure will be repeated until at least 3 (minimum if field parameters meet stabilization criteria), but no more than 5 standing water volumes have been evacuated.

Once purging is complete, samples will be collected directly from the end of the discharge tubing. Certified-clean sample bottles, provided by the laboratory, will be filled by allowing the pump discharge to flow gently down the inside of the bottle with minimal agitation. Each pre-labeled bottle will be capped as it is filled. VOC samples will be collected first, at a flow rate of 100 to 250 ml/min, taking steps to eliminate all headspace in the vials. Immediately after filling, each VOC vial will be checked by inverting the vial and tapping the side of the vial to check for air bubbles. If air bubbles are discovered, the vial will be discarded and a new vial with be filled and checked for bubbles. The above procedure will continue until a minimum of two VOC vials per sample location are collected.

The samples will be preserved according to method-specific requirements, and promptly placed in a cooler with wet ice and maintained at approximately 4°C. Following sampling, the samples will be shipped under COC procedures to the analytical laboratory(ies).

The sample analytical results from the different methodologies will be tabulated along with their sampling depths to facilitate direct comparison of the information. The data will also be compared using an RPD calculation. The results will be included in the January to July 2019 Semi-Annual Monitoring Report for the Site. This one-time event activity is being conducted to determine that low-flow purging and sampling is appropriate for long-screen wells at the Site.

4.3.3.5 Volume Average Purging Using Bailers

Depending upon site specific conditions (e.g., the formation will not support the use of submersible pumps as groundwater recharge is too slow), then volume average purging will be utilized. The well will be purged of 3 to 5 well volumes and sampled using a dedicated or disposable bottom filling HDPE (non-Teflon) bailer. Using the well construction information, the volume of standing water in each casing will be calculated using the following equation:

Standing Water Volume = ((Well depth) – (Water level))*(Casing Volume/foot))

Casing Volumes		
2-inch casing	**4-inch casing**	**6-inch casing**
0.163 gallons/foot	0.653 gallons/foot	1.47 gallons/foot

Nylon well rope will be securely tied to the new or dedicated bailer. The bailer will be gently lowered into the water column in order to minimize disturbance. Once the bailer fills, it will be slowly pulled up. Field parameter readings (pH, DO, conductivity, temperature, ORP, and turbidity) will be collected from the initial bailer of water, and following removal of each well volume. All measurements will be recorded on the Volume Average Groundwater Purge/Sample Field Information Form (Attachment C) and/or in field notebooks. This practice will be repeated until one of the following occurs:

1) At least 3 (minimum if field parameters meet stabilization criteria), but no more than 5 standing water volumes have been evacuated.

2) The water level in the well drops to the top of the screened interval.

If the water level drops to the top of the screened interval using this method, the water will be allowed to recharge to 90-percent of its static condition, then purging can continue if necessary. The samples will be collected as soon as there is a sufficient recharge volume to fill the sample bottles without lowering the water level to the top of the screened interval. The bailer will be slowly lowered down the well into the top of the water column such that unnecessary disturbance to the groundwater sample does not take place.

Certified-clean sample bottles, provided by the laboratory, will be filled by allowing the water to flow out of the bottom of the bailer and gently down the inside of the bottle with minimal agitation. Each pre-labeled bottle will be capped as it is filled. VOC samples will be collected first, taking steps to eliminate all headspace in the vials. Immediately after filling, each VOC vial will be checked by inverting the vial and tapping the side of the vial to check for air bubbles. If air bubbles are discovered, the vial will be discarded and a new vial with be filled and checked for bubbles. The above procedure will continue until a minimum of two VOC vials per sample location are collected.

During the PFAS monitoring event, samples for PFAS analysis will be collected subsequent to VOC sample collection, otherwise samples for metals analyses will be collected subsequent to VOC sample collection. Consistent with Site practices over more than the last 10 years and to maintain a consistent data set, metals samples will be collected for analysis of total and dissolved metals. The filtered (dissolved) metals samples will be collected using an in-line 0.45-micron filter attached to the end of the bailer. While it is anticipated that wells will have a turbidity less than 10 NTUs (low-flow guidance allows for dissolved metals analysis if turbidity is greater than 10 NTUs), it is possible that entrained particles (metal flakes due to corrosion of old steel extraction well casings) will be included in the total metals samples. Therefore, total as well as dissolved metals samples should be collected and analyzed.

The samples will be preserved according to method-specific requirements, and promptly placed in a cooler with wet ice and maintained at approximately 4°C. Following sampling, the samples will be shipped under COC procedures to the analytical laboratory.

4.3.3.6 Extraction Well Sampling

Extraction wells will be sampled as close to the well source as possible. Wells that are on-line will be sampled by first discharging approximately 5 liters from the piping lines. Wells which are off-line must be activated, then allowed to discharge approximately 5 liters before sampling. It should be noted that during purging and sampling of the wells that are off-line, the well could run dry. In this instance, the well should be allowed to recharge. Once it has recharged to a level that is sufficient for pumping, the purging/sampling will be continued until all sample bottle ware has been filled. If a well is off-line and cannot be activated at the time of the monitoring event, then the well will be sampled during a subsequent monitoring event. Off-line AWC production wells will not be sampled.

Following purging, field parameters will be measured as grab samples with a Horiba U-22 instrument (or equivalent). All measurements will be recorded on the Low-Flow Groundwater Purge/Sample Field Information Form (see Attachment B) or other equivalent low-flow purge and sampling forms. Once purging is completed, samples will be collected directly from the port. Samples for VOC and light hydrocarbon samples will be collected first, by allowing the water to gently flow down the inside of the vial, taking steps to eliminate all headspace in the vials. Immediately after filling, each VOC vial will be checked by inverting the vial and tapping the side of the vial to check for air bubbles. If air bubbles are discovered, the vial will be discarded and a new vial with be filled and checked for bubbles. The above procedure will continue until a minimum of two VOC vials per sample location are collected.

During the PFAS monitoring event, samples for PFAS analysis will be collected subsequent to VOC sample collection, otherwise samples for metals analyses will be collected subsequent to VOC sample collection. Consistent with Site practices over more than the last 10 years and to maintain a consistent data set, metals samples will be collected for analysis of total and dissolved metals. The filtered (dissolved) metals samples will be collected using an in-line 0.45-micron filter attached to the end of the bailer. While it is anticipated that wells will have a turbidity less than 10 NTUs (low-flow guidance allows for dissolved metals analysis if turbidity is greater than 10 NTUs), it is possible that entrained particles (metal flakes due to corrosion of old steel extraction well casings) will be included in the total metals samples. Therefore, total as well as dissolved metals samples should be collected and analyzed.

The samples will be preserved according to method-specific requirements, and promptly placed in a cooler with wet ice and maintained at approximately 4°C. Following sampling, the samples will be shipped under COC procedures to the analytical laboratory.

4.3.4 AWC Well Sampling

AWC samples its operating production wells monthly or quarterly depending on its monitoring and reporting requirements. For the parameters and locations listed on Table A-6D, AWC will allow Golder to access and collect samples for analysis. Extraction wells will be sampled as close to the well source as possible. Wells that are on-line will be sampled by first discharging approximately 5 liters from the system piping. Off-line AWC production wells will not be sampled.

Following purging, field parameters will be measured as grab samples with a Horiba U-22 instrument (or equivalent). All measurements will be recorded on the Low-Flow Groundwater Purge/Sample Field Information Form (see Attachment B) or other equivalent low-flow purge and sampling forms. Once purging is completed and field parameters have been measured, samples will be collected directly from the port. Samples for VOC will be collected first, by allowing the water to gently flow down the inside of the vial, taking steps to eliminate all

headspace in the vials. Immediately after filling, each VOC vial will be checked by inverting the vial and tapping the side of the vial to check for air bubbles. If air bubbles are discovered, the vial will be discarded and a new vial with be filled and checked for bubbles. The above procedure will continue until a minimum of two VOC vials per sample location are collected.

During the PFAS monitoring event, samples for PFAS analysis will be collected subsequent to VOC sample collection, otherwise samples for metals analyses will be collected subsequent to VOC sample collection. Metals samples will be collected for analysis of total and dissolved metals. The filtered (dissolved) metals samples will be collected using an in-line 0.45-micron filter attached to the end of the bailer.

The samples will be preserved according to method-specific requirements, and promptly placed in a cooler with wet ice and maintained at approximately 4°C. Following sampling, the samples will be shipped under COC procedures to the analytical laboratory.

4.3.5 Collection of Split Samples

Pursuant to Paragraph 5.6(d)(6) of the RD AOC SOW, it is anticipated the USEPA's contractor (CDMSmith) will collect split samples during the next semi-annual groundwater monitoring event in which PFAS samples are collected for analysis (anticipated in October 2019) (USEPA, 2018i). As conducted previously at the Site, Golder will purge the monitoring wells using the procedures outlined in the SAP, then Golder will collect samples for analysis of PFAS in bottles supplied by Eurofins and CDMSmith will collect samples for PFAS analysis into bottles supplied by the laboratory that the USEPA intends to use for PFAS analysis. Golder and CDMSmith will alternate filling of bottles for PFAS analysis to acquire split samples and/or duplicate samples.

4.3.6 Aquifer Testing

After installation and development of the above-described monitoring wells, aquifer testing will be performed. The testing will be conducted between the DDA and the well PW-1(U) area and in two of the proposed extraction well locations (well UPA-01 area and wells MW-18/MW-34 area) downgradient of well PW-1(U) as required by the RD AOC SOW.

4.3.6.1 Transducer Study - Response to AWC Pumping

Ten transducers[8] are deployed between the DS&G Site and AWC's Llangollen wellfield (see Figure A-6A) to monitor aquifer response to wellfield operational changes between the DS&G and ACL Sites and in the areas where extraction wells for the Selected Remedy will be installed. Data from these transducers is periodically downloaded and will be used to update the groundwater model described below. Currently, the transducers are deployed in the following locations/wells:

- DDA to Well PW-1 Area – in well DDA-10-US

[8] As PFAS analytes are potential COPCs at the DS&G and ACL Sites, the following information is included herein. Through PFAS experience at this Site and at other sites, Golder is aware that PFAS may leach from the cabling associated with transducers despite being certified as "PFAS-free". Immediately after purchase and prior to deployment of the transducer in well AWC-6, Golder decontaminated the transducer and cabling (in accordance with our PFAS decontamination SOP for non-dedicated equipment), then collected an equipment/rinsate blank for the transducer and cabling and collected a sample of the unused portion of the decontamination fluids. The equipment/rinsate blank and the unused decontamination fluids sample were sent to Eurofins for analysis of PFAS via USEPA Method 537.1 Modified. PFAS analytes were not detected in the sample of unused decontamination fluids; however, the following PFAS analytes were detected in the equipment/rinsate blank at the indicated concentrations: PFOA at 28 nanograms/liter (ng/l); perfluoroheptanoic acid (PFHpA) at 1 ng/l (J-flagged concentration); perfluorononanoic acid (PFNA) at 2 ng/l (J-flagged concentration).

- Well UPA-01/RT-1-UP Area – in well UPA-01
- Well P-6 Area – in well UPA-101-US
- Downgradient of ACL's Eastern Lobe – in wells MW-40 and BW-2
- Upgradient of well AWC-G3-R – in wells MW-26N, UPA-02D, and AWC-E2
- AWC Wellfield – in well AWC-MW-6 (adjacent to production well AWC-G3R) and in former production well AWC-6 (until decommissioned in late 2017), then re-deployed in well DDA-16-US in August 2018

These transducers will remain in-place and the data will be periodically downloaded until the transducers are relocated to other wells as part of the aquifer testing for design of the Selected Remedy extraction wells. The data obtained from this transducer study will be evaluated, used in the updated groundwater flow model described in the PDI WP-Rev 2, and the evaluations and results will be included in the PDI Evaluation Report for submittal to and approval by the USEPA.

4.3.6.2 UPA Pumping Tests and UPCUTZ Extraction Testing

The aquifer testing scope includes pumping tests in UPA wells and extraction testing on UPCUTZ wells as presented on Table A-8 (this table also includes UPCUTZ extraction testing described in Section 4.3.4.3) with additional details as presented below. This information will be used to update the groundwater flow model (see PDI WP-Rev 2 Section 6.3.7). Extraction rates from production wells and wellfields that are operating within the domain of the groundwater model (e.g., Llangollen, Artisan's Village, Mid-Vale) will be acquired for the period prior to, and for the duration of, the pumping tests.

Pumping tests will be performed in series in five UPA upper sand wells (PW-1(U), DDA-10/12-US-EXTR, UPA-01-US-EXTR, MW-18/34-US-EXTR and P-6) and UPCUTZ wells (DDA-05-TZ-EXTR and DDA-06-TZ-EXTR) as shown on Figures A-3 and A-4, and A-6B through A-6H. The recommended duration of pumping for each step is presented below and in Table A-8; however, the duration will be re-evaluated as the testing proceeds and proposed changes will be discussed with the USEPA. Each pumping test will be followed by 48 to 72 hours of recovery prior to initiating the next phase of the pumping test. Due to regional fluctuations of the potentiometric elevations within the UPA and the UPCUTZ caused by pumping, recharge and tidal influences, observation of "full" recovery is unlikely; therefore, the groundwater elevation observed after the recovery period will be considered the baseline groundwater elevation for the subsequent pumping test. Recovery trends will be evaluated and drawdown data will be corrected to recovery trends.

Data reduction and evaluation will be performed as described in the PDI WP-Rev 2. The data obtained from this testing scope will be evaluated, design parameters will be developed for the UPA and UPCUTZ extraction wells included in the Selected Remedy, and the evaluations and results will be included in the PDI Evaluation Report for submittal to and approval by the USEPA.

The following information describes the series of activities for the UPA pumping tests:

<u>Step 1 - Deployment of Transducers, Shutdown of Extraction Well PW-1(U), and Baseline Water Level Monitoring</u>
The proposed scope of work for the deployment of transducers and baseline water level monitoring is as follows:

- Deploy data-logging In-Situ TROLL™ transducers/dataloggers in nearby UPCUTZ and UPA wells (see transducer locations shown on Figure A-6B and listed on Table A-8). All transducers used during the

pumping test will be vented such that changes in barometric pressure do not affect the water level measurements recorded by the data logger.

- Measure and record water levels manually prior to initiation of pumping test to verify transducer readings prior to initiation of pumping test. After initiation of pumping, measurements will be made at 4-hour intervals for first day, then once a day until end of test (frequency may be adjusted as needed).
- Measure and record water levels manually from the additional wells listed on Table A-8 prior to initiation of pumping test. After initiation of pumping, measurements will be made at 4-hour intervals for first day, then once a day until the end of the test (frequency may be adjusted as needed).
- Shut down extraction well PW-1(U) (currently extracting at approximately 30 to 35 gallons per minute [gpm]) for three days (72 hours) prior to initiation of pumping tests.

Step 2 – Extraction Well PW-1(U) Pumping Test / Restart at 15 gpm – The proposed scope of work for the well PW-1(U) pumping test is as follows:

- Deploy data-logging In-Situ TROLL™ transducers/dataloggers in existing extraction well PW-1(U) and in nearby wells as listed on Table A-8 and shown on Figure A-6B.
- Measure and record water levels manually from wells with transducers prior to initiation of pumping test to verify transducer readings. After initiation of pumping, measurements will be made at 4-hour intervals for first day, then once a day until the end of the test (frequency may be adjusted as needed).
- Measure and record water levels manually from the additional wells listed on Table A-8 prior to initiation of pumping test. After initiation of pumping, measurements will be made at 4-hour intervals for first day, then once a day until the end of the test (frequency may be adjusted as needed).
- Restart extraction well PW-1(U) at 15 gpm[9] to perform a constant rate pumping test. The extraction rate for well PW-1(U) will remain at 15 gpm for the duration of the aquifer testing (assumed 5 to 6-week testing period). The existing submersible pump in well PW-(U) will be used for the pumping test. The pump and well screen on extraction well PW-1(U) should be cleaned within a month prior to initiation of the pumping tests.
- Groundwater extracted during the pumping tests will be pumped to a fractionation (frac) / equalization tank for ease in maintaining a constant extraction rate. The flow/discharge rate from the well to the frac tank will be monitored using a flow meter. The discharge lines will be equipped with throttling valves installed before and after a totalizing flow meter. Groundwater from the frac tank will be discharged to the sanitary sewer system via the connection located near well PW-1(U).

[9] Well PW-1(U) will be operated at this reduced extraction rate (15 gpm) for the duration of the pumping tests for two reasons: 1) the discharge from well PW-1(U) to the POTW fluctuates by up to 5 gpm when operated at or around 30 to 35 gpm due to back pressure on the sewer discharge line caused by the intermittent discharge from the 10,000-gallon tank to which the LFExS discharges, and 2) the modeled extraction rate for well PW-1(U) as part of the Selected Remedy is 15 gpm.

Step 3 – Extraction Well DDA-10/12-US-EXTR Pumping Test – The proposed scope of work for the well DDA-10/12-US-EXTR pumping test is as follows:

- Deploy Grundfos submersible pump and data-logging transducer in proposed extraction well DDA-10/12-US-EXTR. The submersible pump will be sized appropriately to accommodate an anticipated pumping rate of 10 to 15 gpm.
- Deploy data-logging In-Situ TROLL™ transducers/dataloggers in nearby wells as listed on Table A-8 and shown on Figure A-6C.
- Measure and record water levels manually from wells with transducers prior to initiation of pumping test to verify transducer readings. After initiation of pumping, measurements will be made at 4-hour intervals for first day, then once a day until the end of the test (frequency may be adjusted as needed).
- Measure and record water levels manually from the additional wells listed on Table A-8 prior to initiation of pumping test. After initiation of pumping, measurements will be made at 4-hour intervals for first day, then once a day until the end of the test (frequency may be adjusted as needed).
- Conduct step test on proposed extraction well DDA-10/12-US-EXTR. The pumping rate will be limited such that the water level in the pumping well does not drop into the well screen interval during the test. Well development data and the results of previous pumping tests at the Site will be used to establish step test rates prior to initiating step testing in the field. It is assumed that the step test will include a maximum of three steps with a duration of approximately one hour per step.
- Conduct constant rate pumping test on proposed extraction well DDA-10/12-US-EXTR at proposed flow rate of 10 gpm for 72 hours (while 24 to 48 hours should be sufficient for this test, an evaluation of mass transfer from the UPCUTZ to the UPA upper sand will also be performed in this location as described below).
- To evaluate if pumping in the UPA upper sand can induce mass transfer from the UPCUTZ to the UPA upper sand, sampling will be conducted as follows:
  - Baseline samples will be collected from the proposed pumping test wells and adjacent wells during the semi-annual monitoring events in April and October 2019
  - Pumping well will be sampled at 24 hours, 48 hours, and 72 hours after the start of pumping
  - Samples collected will be analyzed for VOCs with low-level 1,4-dioxane (as needed) and SVOCs with low-level BCEE (as needed)
  - Samples from pumping wells will be grab samples from the pump discharge
- Groundwater extracted during the pumping tests will be pumped to a frac tank for ease in maintaining a constant extraction rate. The flow/discharge rate from the well to the frac tank will be monitored using a flow meter. The discharge lines will be equipped with throttling valves installed before and after a totalizing flow meter. Groundwater from the frac tank will be discharged to the sanitary sewer system via the connection located near well PW-1(U) or to the 10,000-gallon tank in the DS&G treatment building.

Step 4 – Extraction Well DDA-05-TZ-EXTR Testing – The proposed scope of work for the well DDA-05-TZ-EXTR extraction test is as follows:

- Deploy Grundfos submersible pump and data-logging transducer in proposed extraction well DDA-05-TZ-EXTR. The submersible pump will be sized appropriately to accommodate an anticipated pumping rate of 1 to 5 gpm.
- Deploy data-logging In-Situ TROLL™ transducers/dataloggers in nearby wells as listed on Table A-8 and shown on Figure A-6D.
- Measure and record water levels manually from wells with transducers prior to initiation of pumping test to verify transducer readings. After initiation of pumping, measurements will be made at 4-hour intervals for first day, then once a day until the end of the test (frequency may be adjusted as needed).
- Measure and record water levels manually from the additional wells listed on Table A-8 prior to initiation of pumping test. After initiation of pumping, measurements will be made at 4-hour intervals for first day, then once a day until the end of the test (frequency may be adjusted as needed).
- Conduct step test on proposed extraction well DDA-05-TZ-EXTR. The pumping rate will be limited such that the water level in the pumping well does not drop into the well screen interval during the test. Well development data and the results of previous pumping tests at the Site will be used to establish step test rates prior to initiating step testing in the field. It is assumed that the step test will include a maximum of three steps with a duration of approximately one hour per step.
- Conduct constant rate pumping test on proposed extraction well DDA-05-TZ-EXTR at proposed flow rate of 2 gpm for up to 48 hours.
- Groundwater extracted during the pumping tests will be pumped to a frac tank for ease in maintaining a constant extraction rate. The flow/discharge rate from the well to the frac tank will be monitored using a flow meter. The discharge lines will be equipped with throttling valves installed before and after a totalizing flow meter. Groundwater from the frac tank will be discharged to the sanitary sewer system via the connection located near well PW-1(U) or to the 10,000-gallon tank in the DS&G treatment building.

Step 5 – Extraction Well DDA-06-TZ-EXTR Testing – The proposed scope of work for the well DDA-06-TZ-EXTR extraction test is as follows:

- Deploy Grundfos submersible pump and data-logging transducer in proposed extraction well DDA-06-TZ-EXTR. The submersible pump will be sized appropriately to accommodate an anticipated pumping rate of 1 to 5 gpm.
- Deploy data-logging In-Situ TROLL™ transducers/dataloggers in nearby wells as listed on Table A-8 and shown on Figure A-6E.
- Measure and record water levels manually from wells with transducers prior to initiation of pumping test to verify transducer readings. After initiation of pumping, measurements will be made at 4-hour intervals for first day, then once a day until the end of the test (frequency may be adjusted as needed).
- Measure and record water levels manually from the additional wells listed on Table A-8 prior to initiation of pumping test. After initiation of pumping, measurements will be made at 4-hour intervals for first day, then once a day until the end of the test (frequency may be adjusted as needed).

- Conduct step test on proposed extraction well DDA-06-TZ-EXTR. The pumping rate will be limited such that the water level in the pumping well does not drop into the well screen interval during the test. Well development data and the results of previous pumping tests at the Site will be used to establish step test rates prior to initiating step testing in the field. It is assumed that the step test will include a maximum of three steps with a duration of approximately one hour per step.
- Conduct constant rate pumping test on proposed extraction well DDA-06-TZ-EXTR at proposed flow rate of 2 gpm for up to 48 hours.
- Groundwater extracted during the pumping tests will be pumped to a frac tank for ease in maintaining a constant extraction rate. The flow/discharge rate from the well to the frac tank will be monitored using a flow meter. The discharge lines will be equipped with throttling valves installed before and after a totalizing flow meter. Groundwater from the frac tank will be discharged to the sanitary sewer system via the connection located near well PW-1(U) or to the 10,000-gallon tank in the DS&G treatment building.

Step 6 - Extraction Well UPA-01-US-EXTR Pumping Test – The proposed scope of work for the well UPA-01-US-EXTR pumping test is as follows:

- Deploy Grundfos submersible pump and data-logging transducer in proposed extraction well UPA-01-US-EXTR. The submersible pump will be sized appropriately to accommodate an anticipated pumping rate of up to 60 gpm.
- Deploy data-logging In-Situ TROLL™ transducers/dataloggers in nearby wells as listed on Table A-8 and shown on Figure A-6F.
- Measure and record water levels manually from wells with transducers prior to initiation of pumping test to verify transducer readings. After initiation of pumping, measurements will be made at 4-hour intervals for first day, then once a day until the end of the test (frequency may be adjusted as needed).
- Measure and record water levels manually from the additional wells listed on Table A-8 prior to initiation of pumping test. After initiation of pumping, measurements will be made at 4-hour intervals for first day, then once a day until the end of the test (frequency may be adjusted as needed).
- Conduct step test on proposed extraction well UPA-01-US-EXTR. The pumping rate will be limited such that the water level in the pumping well does not drop into the well screen interval during the test. Well development data and the results of previous pumping tests at the Site will be used to establish step test rates prior to initiating step testing in the field. It is assumed that the step test will include a minimum of three steps with a duration of approximately one hour per step.
- Conduct constant rate pumping test on proposed extraction well UPA-01-US-EXTR at proposed flow rate of up to 60 gpm for 72 hours[10].
- Groundwater extracted during the pumping tests will be pumped to frac tanks for ease in maintaining a constant extraction rate. The flow/discharge rate from the well to the frac tanks will be monitored using a flow meter. The discharge lines will be equipped with throttling valves installed before and after a totalizing flow meter. Groundwater from the frac tanks will likely be piped and discharged directly to the sanitary sewer

[10] A 72-hour pumping test is required for this location per the RD AOC SOW.

system via a temporary connection[11] on Grantham Lane. If piping to the sanitary sewer system on Grantham Lane is not an option, then the water will either be transported to the area of the PW-1(U) discharge line and discharged to the sanitary sewer from that location or transported off-site to a licensed disposal facility.

Step 7 – Extraction Well MW-18/34-US-EXTR Pumping Test – The proposed scope of work for the MW-18/34-US-EXTR pumping test is as follows:

- Deploy Grundfos submersible pump and data-logging transducer in proposed extraction well MW-18/34-US-EXTR. The submersible pump will be sized appropriately to accommodate an anticipated pumping rate of up to 60 gpm.
- Deploy data-logging In-Situ TROLL™ transducers/dataloggers in nearby wells as listed on Table A-8 and shown on Figure A-6G.
- Measure and record water levels manually from wells with transducers prior to initiation of pumping test to verify transducer readings. After initiation of pumping, measurements will be made at 4-hour intervals for first day, then once a day until the end of the test (frequency may be adjusted as needed).
- Measure and record water levels manually from the additional wells listed on Table A-8 prior to initiation of pumping test. After initiation of pumping, measurements will be made at 4-hour intervals for first day, then once a day until the end of the test (frequency may be adjusted as needed).
- Conduct step test on proposed extraction well MW-18/34-US-EXTR. The pumping rate will be limited such that the water level in the pumping well does not drop into the well screen interval during the test. Well development data and the results of previous pumping tests at the Site will be used to establish step test rates prior to initiating step testing in the field. It is assumed that the step test will include a minimum of three steps with a duration of approximately one hour per step.
- Conduct constant rate pumping test on proposed extraction well MW-18/34-US-EXTR at proposed flow rate of up to 60 gpm for 72 hours[12].
- Groundwater extracted during the pumping tests will be pumped to frac tanks for ease in maintaining a constant extraction rate. The flow/discharge rate from the well to the frac tanks will be monitored using a flow meter. The discharge lines will be equipped with throttling valves installed before and after a totalizing flow meter. Groundwater from the frac tanks will likely be piped and discharged directly to the sanitary sewer system via a temporary connection[13] on Grantham Lane. If piping to the sanitary sewer system on Grantham Lane is not an option, then the water will either be transported to the area of the PW-1(U) discharge line and discharged to the sanitary sewer from that location or transported off-site to a licensed disposal facility.

[11] Details regarding a temporary sewer discharge connection (e.g., location, piping, volume, and permit) will need to be worked out with NCC.
[12] See footnote 10.
[13] See footnote 11.

Step 8 – Well P-6 Pumping Test – The proposed scope of work for the monitoring well P-6 pumping test is as follows:

- Deploy Grundfos submersible pump and data-logging transducer in existing well P-6. The submersible pump will be sized appropriately to accommodate an anticipated pumping rate of 10 to 20 gpm.
- Deploy data-logging In-Situ TROLL™ transducers/dataloggers in nearby wells as listed on Table A-8 and shown on Figure A-6H.
- Measure and record water levels manually from wells with transducers prior to initiation of pumping test to verify transducer readings. After initiation of pumping, measurements will be made at 4-hour intervals for first day, then once a day until the end of the test (frequency may be adjusted as needed).
- Measure and record water levels manually from the additional wells listed on Table A-8 prior to initiation of pumping test. After initiation of pumping, measurements will be made at 4-hour intervals for first day, then once a day until the end of the test (frequency may be adjusted as needed).
- Conduct step test on well P-6. The pumping rate will be limited such that the water level in the pumping well does not drop into the well screen interval during the test. Well development data and the results of previous pumping tests at the Site will be used to establish step test rates prior to initiating step testing in the field. It is assumed that the step test will include a maximum of three steps with a duration of approximately one hour per step.
- Conduct constant rate pumping test on well P-6 at proposed flow rate of 10 gpm for 72 hours (while 24 to 48 hours should be sufficient for this test, an evaluation of mass transfer from the UPCUTZ to the UPA upper sand will also be performed in this location as described below).
- To evaluate if pumping in the UPA upper sand can induce mass transfer from the UPCUTZ to the UPA upper sand, sampling will be conducted as follows:
  - Baseline samples will be collected from the proposed pumping test wells and adjacent wells during the semi-annual monitoring events in April and October 2019
  - Pumping well will be sampled at 24 hours, 48 hours, and 72 hours after the start of pumping
  - Samples collected will be analyzed for VOCs with low-level 1,4-dioxane (as needed) and SVOCs with low-level BCEE (as needed)
  - Samples from pumping wells will be grab samples from the pump discharge
- Groundwater extracted during the pumping tests will be pumped to a frac tank for ease in maintaining a constant extraction rate. The flow/discharge rate from the well to the frac tank will be monitored using a flow meter. The discharge lines will be equipped with throttling valves installed before and after a totalizing flow meter. Groundwater from the frac tank will be discharged directly to the sanitary sewer system via a temporary connection[14] on Grantham Lane.

[14] See footnote 11.

4.4 Advancement and Screening of Barhole Probes

Barhole probes will be advanced and screened to assess the potential for LFG migration to areas beyond the boundaries of the Inert Area and Grantham South where there are currently no gas monitoring wells or LFG Mitigation System wells. These areas are shown on Figure A-7. Measurements of % lower explosive limit (LEL) at each of the barhole probes will be reviewed in the field to determine whether additional barhole probe locations are needed to evaluate extent of methane migration.

The barhole probe monitoring program will be scheduled such that no work is performed during or within 12 hours after a heavy rainfall (i.e., 0.5 inches or more). Prior to use on-site and at the beginning of each day's use, the instruments will be calibrated following the manufacturer's standard operating procedures.

Prior to use at each sample point, background (ambient air) levels will be recorded. These measurements will be compared to the barhole probe and utility access point readings to determine if levels of carbon monoxide (CO) and oxygen (O_2) measured in the soil gas indicate possible short-circuiting (i.e., ambient air infiltration indicated by O_2 levels between 20 and 21% and CO at or near 0%). If soil gas levels indicate possible short-circuiting, efforts will be made to seal leaks, and if unsuccessful, a new barhole will be driven.

While monitoring with a combustible gas indicator at the ground surface, barholes will be driven into the ground to a depth of 2 to 3 ft-bgs using a steel rod attached to a sliding steel hammer. If refusal occurs at a depth shallower than 2 ft-bgs, then an adjacent sample location will be selected. Immediately following the removal of the steel rod from the probe hole, high density polyethylene tubing (dedicated for each sample location) will be inserted into the probe hole to a depth of approximately 2 ft-bgs. (Note that previous investigations indicate that hole collapse is not a significant concern in this area.) The tubing will be clamped off at the surface. The ground surface around the top of the probe will be tamped and bentonite powder will be used to form a seal around the tubing.

Following installation and prior to sampling, each probe hole will be purged by removing 1.5 probe hole volumes at a rate of 100 milliliters per minute (ml/min) using a Gillian GilAir sampling pump with low-flow module (or equivalent). The purge time required will be calculated in the field based on the length and diameter of the tubing[15]. Following purge, a Tedlar® bag will be connected to the low-flow pump and filled at a rate not to exceed 100 ml/min. Landfill gas measurements will be recorded using an RKI Eagle and/or a LandTech GEM with a methane %LEL sensor (or equivalent) from the Tedlar® bag. All measurements will be recorded on the Barhole Probe Monitoring Field Information Form (Attachment D) and/or in field notebooks.

In addition, during the first quarter 2019 LFG monitoring event, Golder will screen the small, slab-on-grade, 'office' building at the bottom of Grantham Lane near the Inert Area to evaluate the potential for LFG migration into the building. This monitoring will be conducted in accordance with the procedures in the September 2012 Revised Operation and Maintenance (O&M) Plan - Grantham South and Inert Area (Environmental Alliance [EA], 2012) and its Addendum-Revision 2 dated May 26, 2017 (Golder, 2017).

Following completion of the field activities, Golder will tabulate the data, update the figures to include monitored locations and %LEL readings, and prepare a letter report for submittal to the USEPA. The letter report will include recommendations for next steps and indicate areas for which use restrictions related to LFG migration are necessary.

[15] Each monitoring probe will be purged at a rate of approximately 100 ml/min and the anticipated diameter of the tubing is 0.25 inch, which holds a volume of 10 ml per foot. As such, each probe will be purged for 6 seconds for each foot of tubing installed.

4.5 Decontamination

Decontamination procedures in this section are intended for use by field personnel for cleaning sampling, drilling and other equipment in the field. Deviations from these procedures should be documented in the field records and investigative reports. Specifications for standard decontamination materials follow. These materials will be used, as appropriate, for non-dedicated equipment used during sample collection (e.g., pumps).

- Soap shall be a phosphate-free laboratory detergent such as Liquinox® or Alconox®. Use of other detergent must be documented in the field log books and investigative reports.
- Solvent shall be pesticide-grade isopropanol. Use of a solvent other than pesticide-grade isopropanol for equipment cleaning purposes must be justified and documented in field log books and investigation reports.
- Tap water may be used from any municipal water treatment system. Use of an untreated potable water supply is not an acceptable substitute for tap water.
- De-ionized water (DI) is tap water that has been run through a standard deionizing resin column. It is commercially available.
- Distilled water is tap water that has been distilled. It is commercially available.
- Analyte free water is tap water that has been treated with activated carbon and a standard deionizing resin column. At a minimum, the finished water should contain no constituents above the laboratory reporting limits that are being analyzed for as part of the remedial investigation.
- Other solvents may be substituted for a particular purpose if required. For example, removal of concentrated waste materials may require the use of either pesticide-grade hexane or petroleum ether. After the waste material is removed, the equipment must be subjected to the standard cleaning procedure. Because these solvents are not miscible with water, the equipment must be completely dry prior to use.

Solvents, laboratory detergent, and rinse waters used to clean equipment shall not be reused during field decontamination and shall be stored in Department of Transportation (DOT)-approved 55-gallon drums. These materials will be treated as IDW. See Section 4.6 for proper handling and disposal of these materials.

4.5.1 PFAS Decontamination

When decontaminating non-dedicated equipment used to sample PFAS, use the following procedure:

- Rinse thoroughly with Citranox® solution
- Rinse thoroughly with DI water (certified PFAS free)
- Rinse with methanol (certified PFAS free)
- Rinse with DI water
- Allow to air dry
- Submersible pumps used to sample PFAS, will be decontaminated at the monitoring well location to be sampled. Once the pump is decontaminated, it will be placed directly into the monitoring well and lowered to the sample interval to avoid accidental PFAS cross-contamination.

4.5.2 Drilling Equipment

The procedures in this section are to be used for all non-dedicated drilling equipment. All decontamination procedures in this section will be performed on a decontamination pad, constructed to the specifications in this section.

4.5.2.1 Decontamination Pad Specifications

Decontamination pads constructed for field cleaning of sampling and drilling equipment should meet the following minimum specifications:

- The pad should be constructed in an area known or believed to be free of surface contamination.
- Ideally, the pad should be located very close to a potable water source.
- The pad should not leak excessively.
- If possible, the pad should be constructed on a level, paved surface and should facilitate the removal of wastewater. This may be accomplished by either constructing the pad with one corner lower than the rest, or by creating a sump or pit in one corner or along one side. Any sump or pit should also be lined.
- Water should be removed from the decontamination pad as needed.
- A temporary pad should be lined with a water impermeable material. This material should be either easily replaced (disposable) or repairable.

At the completion of site activities, the decontamination pad should be deactivated. The pit or sump should be backfilled with the appropriate material designated by the site project field leader. No solvent rinsates will be placed on the pad. Solvent rinsates should be collected in separate containers for proper disposal.

4.5.2.2 Decontamination Procedures

The procedures in this section are to be employed prior to each use and prior to de-mobilization for all non-dedicated drilling equipment:

1) Clean with tap water and soap using a brush to remove obvious particulate matter and surface films.
2) Rinse thoroughly and power wash with potable water.
3) Rinse non-dedicated equipment that might contact samples with distilled water. If distilled water is not available, equipment should be allowed to completely dry.
4) Decontamination water should be containerized and as described in Section 4.6.

4.5.3 Non-Dedicated Sampling Equipment

The procedures in this section are to be employed prior to each use for all non-dedicated sampling equipment used to collect groundwater samples, with the exception of non-dedicated submersible pump:

1) Clean with tap water and soap using a brush to remove obvious particulate matter and surface films.
2) Rinse thoroughly with tap water.
3) Rinse thoroughly with deionized or distilled water.

4) Rinse thoroughly with solvent (pesticide-grade isopropanol) unless made of PVC or plastic. These items are not to be solvent rinsed.

5) Rinse thoroughly with analyte free water. If analyte free water is not available, equipment should be allowed to completely dry.

6) Decontamination water should be containerized and as described in Section 4.6.

4.5.4 Non-Dedicated Submersible Pump

Non-dedicated groundwater sampling equipment used for purging and sampling (such as a submersible pump) will be decontaminated prior to sampling each well. The submersible pump will not be removed from the well between purging and sampling operations. The pump and tubing (including support cable and electrical wires that are in contact with the sample) will be decontaminated by the procedure described below. It should be noted that the outside of the pump will be decontaminated consistent with the procedure described above. In addition, decontamination fluids will be pumped from buckets through the pump as follows:

1) Flush the pump with potable water to remove any sediment that may be trapped in the pump.

2) Flush the pump with a weak, non-phosphate detergent solution (approximately 5 gallons).

3) Flush the pump with tap water to remove all the detergent solution. Generous amounts of tap water (at least 3 pump volumes) should be used to ensure that detergent and any sediment that may be trapped in the pump does not remain in the pump.

4) Flush the pump with deionized or distilled water (during PFAS monitoring events, use certified PFAS-free DI water).

4) Flush the pump with isopropyl alcohol (during PFAS monitoring events, use certified PFAS-free methanol). Use sparingly to minimize presence of this decontamination fluid in the samples.

5) Flush the pump with analyte-free water (during PFAS monitoring events, use certified PFAS-free DI water). Generous amounts of water (at least three pump volumes) should be used to remove as much of the isopropyl alcohol (or methanol) as practical.

6) Decontamination water should be containerized and as described in Section 4.6.

4.6 Investigation-Derived Waste

IDW generated during remedial investigation field activities include: soil, decontamination water and solvent, purge water, well development water, and personal protective equipment (PPE). Each type of IDW will be handled as described below and stored on-Site until off-Site disposal arrangements are made:

4.6.1 Soil

All excess soil generated from drilling activities will be retained in 55-gallon drums and labeled as “Drill Cuttings”. Once a drum has been filled, it will be sealed, dated, numbered, labeled, and recorded in the field log book.

4.6.2 Water

All decontamination, purge, and well development water will be collected in five-gallon buckets, then transferred to 55-gallon drums and labeled as “IDW-Water”. Once a drum has been filled, it will be sealed, dated, numbered, labeled, and recorded in the field log book.

4.6.3 Disposable PPE

Disposable PPE generated during investigations will be retained in containers and labeled “PPE”. Once a drum has been filled, it will be sealed, dated and numbered, labeled, and recorded in the field log book.

4.6.4 Labeling

IDW container labels will include:

- Nature of the IDW (soil, purge water, etc.)
- ID of the well or wells that provided the IDW
- Date filled
- Container number, as recorded in the field log book

The drums will be staged on-Site. At the end of the field activities, IDW will be disposed of in accordance with applicable state and federal regulations.

4.7 Sample Handling and Custody

Table A-15 presents the types of analytical SOP reference, methods, containers, volumes, preservations, and hold times that are required for samples. The laboratory quality manuals (QMs) are provided in Attachments E-1, F-1, and G. Sample container labels will include the following information:

- Project (site) name
- Sample point identification number
- Date and time the sample was collected
- Preservative (if any)
- Analyses to be performed
- Initials of the sampler

Immediately after sample collection, sample bottles will be placed in a cooler with wet ice and completed COC form. The samples must be maintained at approximately 4° C after collection. COC forms will be completed and will accompany the samples at all times. The COC form and field log book should include:

- Sample identification number and matrix
- Project or site name or number
- Sampler’s name or initials
- Sample collection date and time (military time)
- Designation as a grab or composite sample
- Requested analysis
- Any special comments (i.e., samples will be filtered by laboratory upon receipt)

- Any preservatives added to the sample

When shipping samples to the laboratory, sample bottles and requested analyses should be noted on the COC form. The field team leader is responsible for sample handling and documentation requirements. One member of the sampling team should sign the COC form relinquishing custody to the laboratory. If using an overnight courier service, record the tracking number on the COC. The COC form should be sealed inside the shipping container with the samples. The paperwork should be sealed inside a plastic bag to prevent damage from water condensation. The courier does not need to sign the COC form if it is sealed within the shipping container using custody seals. Once samples are transported to the analytical laboratory, custodial responsibility is transferred to the laboratory.

4.8 Analytical Methods

The laboratories will perform sample analyses in accordance with the following USEPA method guidelines:

- VOCs[16] following USEPA SW-846 (USEPA, 1996b) Method 8260C VOCs by Gas Chromatography/Mass Spectrometry (GC/MS) (August 2006; USEPA, 2006)
- SVOCs following USEPA SW-846 Method 8270D Semivolatile Organic Compounds by GC/MS (January 1998; USEPA, 1998a)
- Total TAL metals, dissolved manganese, and dissolved iron following USEPA SW-846 Method 6010D Inductively Coupled Plasma-Atomic Emission Spectrometry (July 2014; USEPA, 2014) and SW-846 Method 7470A Mercury in Liquid Waste (Manual Cold-Vapor Technique) (September 1994; USEPA, 1994)
- PFAS following USEPA Method 537, Determination of Selected Perfluorinated Alkyl Acids in Drinking Water by Solid Phase Extraction and Liquid Chromatography/Tandem Mass Spectrometry (LC/MS/MS), Revision 1.1 (September 2009; USEPA, 2009), modified[17]
- Sulfide following Standard Method (SM) 4500-S2-E Sulfide, Iodometric Method (APHA, 2012)
- Alkalinity following SM 2320B, Titration Method (APHA, 2012)
- Nitrogen, ammonia following SM 4500 NH_3-H, Flow Injection analysis (APHA, 2005)
- Chloride, Nitrate, Nitrite, and Sulfate following USEPA Methods for Chemical Analysis of Water and Wastes (MCAWW) Method 300.0 Determination of Inorganic Anions by Ion Chromatography (USEPA, 1993), USEPA SW-846 (USEPA, 1996b) Method 9056A and SM 4110-B (APHA, 2012)
- Ferrous Iron following SM 3500-Fe-D Iron (APHA, 1992)

SOP documentation from TestAmerica and Eurofins[18] for each of these methods are included in Attachments E-2 and F-2, respectively. Please see Table A-16 - Analytical SOP References for the analytical group, reference number and title of each included SOP.

[16] The USEPA approved use of Method 8260 and 8260 SIM for 1,4-dioxane analysis via email dated October 31, 2012. (USEPA, 2012)

[17] As stated in an email from the USEPA dated December 18, 2018, "[b]ased on laboratory documents submitted to EPA for review in emails dated October 1 and November 27, 201[8], EPA approves the use of Eurofins' modified Method 537 for the analysis of groundwater. (USEPA, 2018h)

[18] Eurofin's SOPs for the PFAS method are confidential and proprietary, as is the case with other labs at this time, because it is a modified version of USEPA Method 537. Method 537 as written is strictly a drinking water method. A coversheet from Eurofins demonstrating that the

4.9 Quality Control

This section describes the various Quality Assurance/Quality Control (QA/QC) samples that will be collected in the field and analyzed in the laboratory and the frequency at which they will be performed. QA/QC samples which will be collected will consist of field duplicates, trip blanks, rinsate blanks, and MS/MSD (see Table A-17). As QA/QC sample requirements vary by analytical method, Tables A-9 through A-13 detail specific QC sample requirements. These QA/QC samples are described briefly in the following sections.

During the PFAS monitoring event, Golder will collect equipment blanks, field duplicates, field blanks, and trip blanks, as summarized on Table A-13 for QA/QC purposes. QA/QC samples will be collected in accordance with *SOP-3: Quality Assurance / Quality Control Protocols for PFAS Sampling Programs* (see Attachment A). These samples will be submitted to Eurofins for analysis of PFAS via USEPA Method 537 Revision 1.1 Modified.

4.9.1 Field Duplicates

Field duplicates will be collected at a frequency of one per 20 primary samples per matrix. Field duplicates are collected by sampling the same location twice, but are assigned a unique sample identification number. When collecting field duplicate samples, the sample containers for each analytical parameter should be filled for both the primary and duplicate sample before the jars for the next analytical parameter are filled.

4.9.2 Trip Blanks

Trip blanks will be collected at a frequency of one per shipping event for coolers containing VOCs and/or PFAS samples. Trip blanks are used to verify that the VOC and/or PFAS bottles and samples are not contaminated in transit between the laboratory to the Site, while on Site, and from the Site back to the laboratory. The laboratory will supply pre-prepared trip blanks. Trip blanks should accompany the VOC and/or PFAS samples throughout the event from collection through shipment to the laboratory and are recorded on the COC along with the primary samples. Trip blanks are shipped along with each cooler that contains aqueous VOC samples. For the PFAS monitoring event, see SOP-3 (Attachment A) for additional requirements.

4.9.3 Rinsate Blanks

Rinsate blanks are collected for all required analyses at a frequency of one per day per type of non-dedicated sampling equipment which comes in contact with the sample. Rinsate blanks are used to verify that decontamination of field equipment was sufficient. Rinsate blanks are prepared in the field using laboratory-supplied demonstrated analyte-free water. The water is poured over and through each type of sampling equipment and collected in labeled laboratory supplied bottles. Rinsate blanks are recorded on the COC along with the primary samples. For the PFAS monitoring event, see SOP-3 (Attachment A) for additional requirements.

4.9.4 Field Blanks

As described in SOP-3 (Attachment A), field personnel shall submit of one field blank per day of sampling. Field blanks shall consist of PFAS-free water containerized in an HDPE sample container filled at the laboratory prior to beginning the field program. Field blank sample containers shall be opened during the collection of a sample and the laboratory-supplied, PFAS-free water contained therein shall be poured directly into a laboratory-supplied

SOP exists, as well as their more generalized boiler plate documents outlining the analysis summary and PFAS collection considerations are included as Attachment F-2. Additional information regarding Eurofins' modified PFAS method was provided to the USEPA for review and approval as noted above.

HDPE sample container, then resealed. Field blank container lids shall remain in the hand of field personnel until replaced on the sample container. Sample container labels shall be completed as described above.

4.9.5 MS/MSDs

MS/MSDs are collected for all required analyses at a frequency of one per 20 primary samples per matrix. MS/MSD samples are prepared and run by the laboratory to verify the effectiveness of sample preparation procedures in measuring chemicals of interest from the matrix material. Additional sample volume is collected from a location and submitted to the laboratory for analysis. MS/MSD samples are recorded on the COC along with the primary samples. For the PFAS monitoring event, see SOP-3 (Attachment A) for additional requirements.

4.9.6 Internal QC Samples

Internal QC checks have been developed to help ensure accuracy and precision during field sampling and measurement as well as laboratory analysis. Field checks will be performed regularly. Laboratory QC checks will be performed in accordance with the specific analytical methods.

Field measurements will be made in duplicate at a frequency of one in twenty measurements taken. These duplicate measurements must agree +/-20 percent. If the duplicate measurements do not meet this criterion, the instrument will be recalibrated and the measurements will be retaken. Field measurements will be recorded in the field notebooks or field information forms and later entered into summary tables.

Details of the internal QC checks utilized by the laboratory will be found in each specific laboratory QM and the published analytical methods. Laboratory QC samples will be analyzed at a frequency of one per twenty analytical samples or at a frequency dictated by the methods. These QC samples will be used to determine if results may have been affected by field activities or procedures used in sample transportation or if matrix interferences are an issue. Assessment of laboratory QC will take into account the PARCCS criteria specified in Tables A-9 through A-13.

Applicable statistics will be calculated following the laboratory SOPs, which can be found in Attachments E-2 and F-2, and Section 3-Quality Objectives and Criteria. The laboratories routinely re-evaluate QC criteria using the procedures in their respective laboratory QMs. Analytical data that fall outside QC criteria will be qualified as discussed in Section 5-Data Validation and Usability.

4.10 Instrument/Equipment Testing, Inspection, and Maintenance

Preventive maintenance of equipment is essential if project resources are to be utilized in a cost-effective manner. Preventive maintenance will sustain the accuracy of measurement systems, minimize downtime, and provide inventory control of critical spare parts, backup systems, and other necessary equipment. The field sampling team will maintain an inventory of replacement parts for field instruments, and will routinely perform preventive maintenance or repair. Spare parts that often require replacement will be kept on hand at the Site during field activities. The preventive maintenance approach for equipment used in field for sampling, monitoring, and testing includes checking batteries and electrodes, checking condition of meters, checking sample bottles for cleanliness and breakage, and that a reasonable supply of bottles, batteries, probes, calibration solution, and supplies are on-hand to avoid unnecessary delays in the field.

Preventive maintenance of laboratory equipment and hardware are described in specific sections of each laboratory QM included in Attachments E-1, F-1, and G. TestAmerica discusses these procedures in Section 20 of their laboratory QM. More than one instrument is generally available for each type of analysis in case the initial

instrument malfunctions or does not meet the required measurement criteria. Laboratory personnel or qualified manufacturer representatives will perform preventive maintenance and repair. The laboratory will retain logbooks documenting preventative maintenance and repair for each instrument.

4.11 Instrument Calibration and Frequency

4.11.1 Field Calibration

The calibration and maintenance of field equipment will be the responsibility of the field sampling team. Field instruments, such as meters for measuring field parameters, will be standardized/calibrated in accordance with the manufacturers' recommendations against National Institute of Standards and Technology (NIST) traceable standards, where appropriate. During sampling, calibration checks will occur at a minimum of two times a day (beginning of each day and at least every four hours of operation). Appropriate calibration records will be maintained in project field log books, groundwater sample field information forms, or on calibration forms. A minimum of a two-point calibration will be performed for each parameter being calibrated. The field team leader is responsible for ensuring that calibrations are properly performed at the appropriate frequency.

4.11.2 Photovac Microtip PID

A MiniRAE 3000 (or equivalent) will be used to monitor VOC concentrations in ambient air during intrusive field activities (i.e., groundwater sampling and well installation). The MiniRAE 3000 is a microprocessor controlled PID. The instrument normally operates with a 10.6 electron volt (eV) lamp; however, 9.8 and 11.7 eV lamps are available as options. The detector is capable of measuring concentrations down to about 1 part per million (ppm) sensitivity for certain compounds. An 11.7 eV lamp will be used on the PID as gross screen for VOCs since the primary VOCs at the Site have good responses to the 11.7 eV lamp. The PID cannot be used to identify unknown substances, it can only quantify/estimate VOC vapors. Winds and high humidity will affect measurement readings. Foggy or high humidity conditions can cause condensation on the lamp, thus affecting measurements.

4.11.2.1 Operational Information

The instrument will be taken into the field fully charged and operated according to manufacturer’s instructions. Turn the instrument on by pressing the “MODE” key for one second and release. The pump will start and the message "Warming up now, please wait" will be displayed for about one minute.

The MiniRAE 3000 has two operation modes:

- Search – With the instrument in Search Mode, it only samples when the use activates sampling.
- Hygiene – the instrument is programmed to operate in Hygiene Mode as its default. This provides the most commonly needed features while requiring the fewest parameter adjustments.

The MiniRAE is factory calibrated with standard calibration gas, and is programmed with default alarm limits and will be field-calibrated using Isobutylene gas. The keypad is used to set up and calibrate the MiniRAE. The instrument must be calibrated against a dynamic standard in order to display concentrations in units equivalent to parts per million by volume (ppmv). Clean outdoor air is suitable as ‘zero gas’. Isobutylene should be used as the calibration gas and calibration should be conducted in a well-ventilated clean air environment. Note that cylinders of compressed gas must be handled with care.

4.11.2.2 Instrument Calibration and Frequency

Following is a description of the calibration procedure:

- Ensure that you are in an area with clean air, away from any exhaust or other potential vapor sources
- Press [N-] and [MODE] simultaneously for 3 seconds to enter Programming Mode
- Press [Y+] to select “Calibrate/Select Gas” menu item
- Press [Y+] to select “Zero Cal?” The display will then read “Apply zero gas”
- Press [Y+] the display will read “Zeroing” for 30 seconds
- While the MiniRAE is collecting the fresh air calibration, prepare the span gas:
- Attach a Tedlar® bag to the canister of Isobutylene via tubing and regulator
- Open the bag valve
- Open the gauge valve. DO NOT ALLOW TEDLAR BAG TO OVERFILL
- When bag is filled, close regulator valve, then close bag valve
- When zero span is completed, attach Tedlar bag to MiniRAE via tubing
- Press [Y+] to select “Span Cal?”
- When display reads “Apply gas now!”, open bag valve. Display will then show “wait …30” with a countdown timer while monitor performs the calibration.
- When done, the display should read calibrated value (for Isobutylene, this should be 100 ppm)
- The instrument is calibrated and ready for use

The MiniRAE will alarm for the following conditions:

- Measured gas concentration exceeds the programmed alarm limits, which for Isobutylene are:

Calibration Gas	Calibration Span Concentration		Low	High	Time Weighted Average (TWA)	Short Term Exposure Limit (STEL)
Isobutylene	100 ppm	Concentration	50	100	100	250
		Alarm	2 beeps/flashes per second	3 beeps/flashes per second	1 beep/flash per second	1 beep/flash per second

- Battery voltage falls below 4.4 V (there will then be approximately 20-30 minutes of operating time remaining)
- Ultraviolet (UV) lamp failure
- Pump stalls
- Datalog memory is full

4.11.3 Multi-Gas Meter

A VRAE, model PGM-7800, (or equivalent) will be used to take oxygen and LEL readings during soil boring advancement, drilling and vapor monitoring activities. The VRAE is a programmable multi gas monitor designed to provide continuous exposure monitoring of toxic gasses, oxygen and combustible gasses. It gives real time measurements and activates alarm signals whenever the exposure exceeds preset limits.

4.11.3.1 Operational Information

The instrument will be taken into the field fully charged and operated according to manufacturer's instructions. Prior to arriving at the Site, check all equipment for proper operation and to ensure all accessory materials are included, such as calibration gases and basic repair/maintenance supplies.

The instrument will be equipped with a rigid probe. Prior to use on-site and at the beginning of each day's use, the instrument will be calibrated following the manufacturer's standard operating procedures. Prior to use at each sample point, background combustible gas concentrations should be measured. These readings should be recorded at each sample location to compare to the vapor gas readings.

Turn the instrument on by pressing the "MODE" key for one second and release. The audio buzzer will beep once and the display will show "ON!"

The VRAE multi gas monitor offers three different user modes of operation:

- Text mode – Simplest mode of operation; the monitor displays the sensor name after the monitor is turned on.
- Display mode – Displays eight different readings: instantaneous gas concentration, sensor name, Peak, minimum, STEL, TWA, battery voltage and shut down voltage in volt, run time in hours and minutes and temperature in degree F. See the manufacturer's instructions for explanations of the different readings.
- Program mode – Allows the user to perform all the functions of the display mode, in addition to entering a programming mode. The programming mode enables the user to perform functions such as change sensor configurations, enter user information or calibrate the monitor, etc.

To switch between modes, press the [MODE] key one or more times until the desired display appears. See the manufacturer's instructions for explanations of the different readings.

The VRAE is factory calibrated with standard calibration gas and is programmed with default alarm limits as listed below.

Gas ppm	Cal Gas/ Balance	Unit	TWA	STEL	Low	High
CO	50/ Air	ppm	35	100	35	200
H_2S	25/ N_2	ppm	10	15	10	20
SO_2	5/ N_2	ppm	2	5	2	10
NO	25/ N_2	ppm	25	25	25	50
NO_2	5/ Air	ppm	1	1	1	10
Cl_2	10/ N_2	ppm	0.5	1	0.5	5
O_2	20.9/ N_2	%	-	-	19.5	23.5
CH_4	50/ Air	% LEL	-	-	10	20
CH_4	20/ N_2	% Vol	-	-	10	20
HCN	10/ N_2	ppm	5	5	5	50

Gas ppm	Cal Gas/ Balance	Unit	TWA	STEL	Low	High
NH_3	50/ N_2	ppm	25	35	25	50
PH_3	5/ N_2	ppm	0.3	1	1	2
ClO_2	0.5/ Air	ppm	0.1	0.3	0.2	0.5

4.11.3.2 Instrument Calibration and Frequency

Instrumentation calibration procedures are established in manufacturer's specifications. Instrument calibration will be performed by trained personnel using the approved written procedures. The VRAE can be calibrated in the programming mode. This is a two-point calibration process using "fresh air" and the standard reference gas (also known as Span Gas). First, a "fresh air" which contains 20.9% and no detectable, toxic or combustible gases is used to set the zero point for each sensor. Then a standard reference gas, which contains a known concentration of a given gas, is used to set the second point of reference. See the manufacturer's instructions for a detailed version of the calibration procedure.

4.11.4 Laboratory Calibration

Sample results should be within the calibration range of the instrument. Samples which do not contain concentrations of target analytes that exceed the instrument calibration range should be analyzed undiluted to achieve the lowest possible reporting limits. However, samples containing elevated levels of target analytes cannot be analyzed undiluted because the calibration range of the method would be exceeded. Such samples will require analysis at dilutions which would result in elevated reporting limits.

The major chemical analytical equipment used for this project are described in each laboratory's QM and the individual analytical methods, provided as Attachments E, F and G. Each laboratory's QM provides information regarding types of equipment used by the laboratory facility. Calibration procedures will follow published analytical methodologies. Each laboratory's QM references the specific methodologies or laboratory SOPs for calibration procedures. The laboratory will document sources for calibration material; for example, USEPA repository, Supelco© or equivalent. The laboratory QM also describes the procedures used to document equipment repair and maintenance.

4.12 Inspection/Acceptance of Supplies and Consumables

Sampling equipment will be inspected prior to use to ascertain proper operation and create a safe working environment. The laboratories chosen for this project have preventative maintenance and health and safety programs to ensure proper execution of project work.

4.13 Non-direct Measurements

Non-direct means of data acquisition refers to the use of non-measurement sources such as computer databases, spreadsheets, programs and literature files. DS&G does not intend to obtain information from non-measurement sources for decision-making regarding this project.

4.14 Data Management

Data collection during this project will be retained in an electronic format. Specific data management activities are as follows:

- Field sample collection forms:
  - Data will be transcribed from field forms or notebooks and tabulated, as appropriate, using a spreadsheet or database program.
  - Data entry will be checked to ensure no transcription errors occurred.
- COC forms:
  - COC forms will be reviewed by the field staff prior to sample submission to the laboratory to verify that the COC matches the cooler contents.
  - COC forms will also be reviewed after sample submission to the laboratory by the QA manager or designee to verify that the sampling plan is being followed.
- Laboratory sample receipt documentation:
  - The QA manager or designee will review the laboratory sample receipt documentation and compare to the COC. If discrepancies are found, the QA manager or designee will contact the field staff and laboratory to resolve any inconsistencies.
  - Communications concerning changes to the sample identifications and required analysis, including telephone memoranda and emails, will be saved to project files at Golder.
- Final chemistry analytical data documentation:
  - Analytical data packages will be verified internally by the laboratory performing the work for completeness prior to submittal to Golder.
  - The QA manager or designee will verify that the analytical data packages contain the information required for data validation upon receipt.
  - The data package elements required are described in Section 2.5 Documents and Records.
  - An electronic database, as well as validated qualifiers, will be kept on the project database, using Earthsoft's EQuIS database structure, at Golder. Database entries will be checked for correctness and completeness.

5.0 ASSESSMENT AND OVERSIGHT

5.1 Assessments and Response Actions

Assessment of activities or procedures will be the responsibility of the personnel performing such activities and procedures. For field measurements, the field team leader will be responsible for assessment while the laboratory analyst and sample custodian will be responsible for assessment within the laboratory. The assessment of activities or procedures must comply with the requirements specified in this SAP. Any deviation of a technical procedure or reference method must be noted within the appropriate logbook and, for laboratory analyses, in the Case Narrative of the analytical report.

Performance will be monitored in the field through the use of QC checks as previously discussed in Section 4.7. Performance will be monitored in the laboratory through the use of QC checks discussed in each laboratory QM and the PARCCS criteria presented in Tables A-9 through A-13.

As described in the guidance documents, assessment includes surveillance, peer review, management systems review, readiness review, technical systems audit, performance evaluation, data quality audit, and data quality assessment. The following assessment activities are planned:

- Peer review
- Technical systems audit
- Data quality assessment

5.1.1 Peer Review

Throughout the project, Golder will maintain a system of peer review by which generated data can be checked and verified. Data that are transcribed and tabulated will be checked for accuracy and completeness.

5.1.2 Audits

The QA/QC audit is an independent systematic on-site review of facilities, equipment, training procedures, record keeping, data validation, data management, and reporting aspects of the field and laboratory QA/QC program. Audits may be performed on field operations and sampling procedures, laboratory analyses and documentation.

5.1.3 Field/Sampling Audit

DS&G does not plan to conduct an audit of sampling activities. The field team leader will be responsible for following applicable quality assurance procedures described in this SAP.

5.1.4 Laboratory Audits

The laboratory will be expected to have a QA program whereby the QA department will routinely conduct internal audits. The laboratory QM discusses internal laboratory audits. DS&G does not anticipate performing audits of the laboratory during this project. If an external audit is deemed necessary by the USEPA, then the USEPA will consult with DS&G and Golder regarding an appropriate approach.

5.1.5 Data Quality Assessment

Analytical data will be assessed through a series of evaluation procedures. The details regarding data evaluation and validation are discussed in Section 6. Data quality assessments will be performed for the groundwater analytical data collected during the PDI activities and as part of the semi-annual monitoring reports for the Site. These assessments will be included in and distributed to the parties that receive the semi-annual monitoring reports, including but not limited to the USEPA, DNREC, and AWC.

5.2 Reports to Management

Timely quality assurance reports are necessary to the successful completion of this project. Quality assurance deficiencies in the field must be reported to the field team leader and Golder QA and Project Manager. Quality assurance deficiencies in the laboratory must be reported in a timely manner to laboratory and project management personnel. The laboratory's policies and procedures for reporting quality assurance activities to management are included in each laboratory’s QM and/or SOPs. Corrective actions for field and laboratory

activities will be reported to Golder's QA and Project Manager, the DS&G Trust's Project Coordinator, and, if necessary, the USEPA Project Manager.

5.3 Evaluation of Tentatively-Identified Compounds

In response to Golder's Technical Memorandum Re: Review of Tentatively Identified Compounds [TICs] in Groundwater dated December 21, 2016 (Golder, 2016c), the USEPA requested via email dated February 7, 2017 that "the DS&G Remedial Trust should perform a bi-annual [every two years] review of groundwater TICs and report the results, including any recommended additions to the TAL [target analyte list], to EPA." (USEPA, 2017b) Consistent with the previous TIC evaluations, the biennial reviews will include:

1) Summary of TICs for the previous two years' groundwater monitoring events (i.e., four semi-annual events performed in 2017 to 2018)

2) Comparison of the TICs to the USEPA's Superfund Organic Methods (SOM) analyte list

3) Comparison of estimated TIC concentrations to applicable standards and screening values

4) Recommendations for compounds to be added to the TAL for the Site

The next biennial review of groundwater TICs will be submitted to the USEPA in March 2019.

6.0 DATA VALIDATION AND USABILITY

6.1 Data Review, Verification, and Validation

The laboratory analytical data will be reviewed for completeness, QA/QC forms and holding times will be checked to ensure data quality. The data quality review will follow guidelines provided by USEPA Region III data validation guidance, which defers to the USEPA Contract Laboratory Program National Functional Guidelines for Superfund Organic and Inorganic Methods Data Review (USEPA, 2017a)[19], and professional judgment, where necessary. Project-specific DQOs are presented in Section 3. The laboratory will perform data reduction in accordance with the individual analytical methodologies used for this project. The laboratory QMs or SOPs will have more detailed information regarding the laboratory data reduction procedures.

In general, data reduction of field measurements will not be necessary because readings will be recorded in field notebooks or field forms directly from the field instruments. If reduction of data is necessary because units of measurement are not comparable (e.g., Fahrenheit vs. Celsius), then these conversions will be performed in the office using standard spreadsheet software. Field measurements will be tabulated using spreadsheet or data base software. Field measurements are anticipated to be recorded as follows:

- DO is to be recorded to the nearest 0.01 mg/L
- pH is to be recorded to the nearest 0.01 standard pH units
- Turbidity is to be recorded to the nearest 1 NTU

[19] Current USEPA National Functional Guidelines are shown. Data review will be performed in accordance with the most current versions of the guidance documents available at the time of data evaluation.

- ORP is to be recorded to the nearest 1 mV
- Specific conductance is to be recorded to the nearest 1 microSiemens per centimeter (uS/cm) or micromhos per centimeter (umhos/cm)
- Temperature is to be recorded to the nearest 0.1^{o}C

6.2 Verification and Validation Methods

Data validation techniques include screening, accepting, rejecting or qualifying data on the basis of specific quality control criteria for holding times, blank results, spike results, surrogates, and field duplicates. Data validation is a process whereby erroneous data may be identified prior to entering the project record. Data Verification of field measurements will be performed by field personnel in consultation with the QA and Project Manager. Field personnel will verify the field data through review of calibration and duplicate data readings. The data will be reviewed to determine if there are anomalous readings. Anomalies will be resolved immediately by means such as re-calibration or re-acquisition of the measurement.

For field samples associated with this project that are sent to a laboratory, the laboratory will produce data packages that will contain the information needed for formal validation of the data. Data will undergo a data evaluation process by which accuracy, precision and completeness are assessed.

The data will be evaluated based upon holding times, blank results, and QC results assessing accuracy and precision. Analytical data packages will be reviewed for completeness and QC summaries will be evaluated. Data review required for this project will be performed under the direction of the QA Manager.

If, based upon this data review, the QA Manager believes that a more extensive data validation should be performed, then a subset of the data will undergo full data validation. Data validation will be performed using the guidelines cited in Section 6.1 and the specific analytical methodologies or SOPs. PARCCS, as defined in Section 3, will be evaluated based upon field sampling documentation, adherence to sample hold times, and analysis of QC samples. Qualifiers will be applied to the data using the logic specified in the validation guidelines cited in Section 6.1, as well as Tables A-9 through A-13.

Qualified results will be reported for validated samples on the analytical reporting forms provided in the data packages or as data summary tables accompanying the laboratory deliverable data package. Qualified results, data packages and analytical results will be stored electronically in Golder's project files and will also be entered into the project database.

The PARCCS criteria and criteria specified in applicable guidelines may not always be achievable. The data validation guidelines provide directions for the determination of data usability. Qualified data can often provide useful information, although the degree of certainty associated with the result may not be as planned. Professional judgment, in conjunction with appropriate guidance documents, will be used to determine data usability.

6.3 Reconciliation with User Requirements

Throughout the project, Golder will determine if project DQOs are being met and assess whether the data that is being collected is sufficient and appropriate. Periodic evaluations of the sampling program will be made to determine if a change in frequency or analytical parameters is appropriate. Individuals making measurements throughout the process will also make assessments of whether the DQOs are being met.

Individuals making field measurements will determine whether or not field quality control criteria were met. The field QA/QC will be overseen by the field team leader. Corrective actions will be initiated in the field as necessary. This corrective action may include recalibration of instruments or use of a different type of instrument.

The analysts in the laboratory will determine if analytical QC criteria are achieved. Corrective action in the form of re-analysis or re-calibration may be warranted. Laboratory analytical data and field data will be assessed by a data validation specialist under the direction of the QA Manager to determine usability with regard to the DQOs.

As noted in the data validation guidelines, data may not always meet precision and accuracy requirements but may still be considered usable. The data will be assessed with regard to the project DQOs, and professional judgment used in conjunction with guidance documents will determine data usability.

Golder will assess collected data and ascertain whether objectives of the project are being met. The USEPA will be informed in writing of changes to the program that may be warranted.

7.0 ACRONYMS AND ABBREVIATIONS

%R	percent recovery
°C	degrees Celsius
ACL	Army Creek Landfill
APHA	American Public Health Association
ASTM	American Society for Testing and Materials
AWC	Artesian Water Company
BCEE	bis(2-chloroethyl)ether
bgs	below ground surface
CFR	Code of Federal Regulations
CO	carbon monoxide
COC	chain-of-custody
COCs	contaminants of concern
CSM	Conceptual Site Model
DDA	Drum Disposal Area
DI	de-ionized
DNREC	State of Delaware Department of Natural Resources and Environmental Control
DO	dissolved oxygen
DOT	Department of Transportation
DQOs	data quality objectives
DS&G	Delaware Sand & Gravel
EDD	electronic data deliverable
EM	electromagnetic
ERP	Emergency Response Plan
eV	electron volt
Fe	iron
Final FS Rev 1	Final Feasibility Study Revision 1
FS	Feasibility Study
FSWP	Feasibility Study Work Plan
GC/MS	gas chromatograph/mass spectrometer
gpm	gallons per minute
GPR	ground-penetrating radar
HASP	Health and Safety Plan
HDPE	high-density polyethylene
IDW	investigation-derived waste
LC/MS/MS	Liquid Chromatography/Tandem Mass Spectrometry
LCS	laboratory control sample
LEL	lower explosive limit
LFExS	low-flow extraction system
LFG	landfill gas
MCAWW	Methods for Chemical Analysis of Water and Wastes
MDL	method detection limit
mg/L	milligrams per liter
ml/min	milliliters per minute
MPCU	Middle Potomac Confining Unit
MS	matrix spike
MS/MSD	matrix spike/matrix spike duplicate
MSD	matrix spike duplicate
mV	millivolt
NAD	North American Datum
NCC	New Castle County
NGVD	National Geodetic Vertical Datum

NIST	National Institute of Standards and Technology
NTUs	Nephelometric Turbidity Units
O_2	oxygen
ORP	oxidation-reduction potential
PARCCS	Precision, Accuracy, Representativeness, Comparability, Completeness, and Sensitivity
PDF	portable document format
PDI	Pre-Design Investigation
PDI WP	Pre-Design Investigation Work Plan
PFAS	per- and poly-fluoroalkyl substances
PFAS 2018 WP	PFAS – Well Purging, Groundwater Sampling and Analysis Program for 2018
PFC	perfluorinated chemicals
PFCs WP	Perfluorinated Chemicals Work Plan
PFOA	perfluorooctanoic acid
PFOS	perfluorooctane sulfonate
PID	photoionization detector
PPE	personal protective equipment
ppm	parts per million
ppmv	parts per million by volume
PRAP	Proposed Remedial Action Plan
PVC	polyvinyl chloride
QA	Quality Assurance
QA/QC	Quality Assurance / Quality Control
QAPP	Quality Assurance Project Plan
QC	Quality Control
QL	quantitation limit
QM	Quality Manual
RA	Remedial Action
RD	Remedial Design
RD AOC	Administrative Settlement Agreement and Order on Consent for Remedial Design
ROD-A2	Record of Decision-Amendment 2
RPD	relative percent difference
SAP	Sampling and Analysis Plan
SOP	Standard Operating Procedures
SOW	Statement of Work
SRM	Standard Reference Material
STEL	short-term exposure limit
SVOCs	semi-volatile organic compounds
TAL	Target Analyte List
TRC	TRC Companies Inc.
TWA	time-weighted average
umhos/cm	micromhos per centimeter
UPA	Upper Potomac Aquifer
UPCU	Upper Potomac Confining Unit
UPCUTZ	Upper Potomac Confining Unit Transition Zone
UPDC	Upper Potomac Dividing Clay
uS/cm	microSiemens per centimeter
USEPA	United States Environmental Protection Agency
UV	ultraviolet
VAP	vertical aquifer profiling
VOCs	volatile organic compounds

8.0 REFERENCES

APHA, 1992. Standard Methods for the Examination of Water and Wastewater, 18th ed. Washington, DC, New York: American Public Health Association. 1992.

APHA, 2005. Standard Methods for the Examination of Water and Wastewater, 21st ed. Washington, DC, New York: American Public Health Association. 2005.

APHA, 2012. Standard Methods for the Examination of Water and Wastewater, 22nd ed. Washington, DC, New York: American Public Health Association. 2012.

ASTM, 2016. ASTM D5092/D5092M – 16, Standard Practice for Design and Installation of Groundwater Monitoring Wells, ASTM International, West Conshohocken, PA, 2016.

Backhus, D.A., J.N. Ryan, D.M. Groher, J.K. MacFarlane, and P. M. Gschwend, 1993. Sampling Colloids and Colloid-Associated Contaminants in Ground Water. Ground Water. V. 31, pp. 466-479.

Barcelona, M.J., H.A. Wehrmann, and M.D. Varljen, 1994. Reproducible Well Purging Procedures and VOC Stabilization Criteria for Ground-Water Sampling. Ground Water. V. 32, pp. 12-22.

Environmental Alliance, 2012. Revised Operation and Maintenance (O&M) Plan - Grantham South and Inert Area. September 2012.

Golder, 2011. Feasibility Study Work Plan, Revision 2, Delaware Sand & Gravel Superfund Site, New Castle, Delaware. October 21, 2011.

Golder, 2016a. Final Feasibility Study, Revision 1, Delaware Sand & Gravel Superfund Site, New Castle, Delaware. May 25, 2016.

Golder, 2016b. Perfluorinated Compounds – Well Purging, Groundwater Sampling and Analysis Program, Delaware Sand & Gravel Superfund Site, New Castle, Delaware. September 21, 2016.

Golder, 2016c. Technical Memorandum Re: Review of Tentatively Identified Compounds in Groundwater, Delaware Sand & Gravel Landfill Superfund Site, New Castle, Delaware. December 21, 2016.

Golder, 2017. Addendum to 2012 Revised O&M Plan – Revision 2, Grantham South and Inert Area, Delaware Sand & Gravel Landfill Superfund Site, New Castle, Delaware. May 26, 2017.

Golder, 2018a. Per- and Polyfluoroalkyl Substances – Well Purging, Groundwater Sampling and Analysis Program For 2018. February 7, 2018.

Golder, 2018b. Operations Monitoring & Maintenance Plan – LFG Mitigation System, Delaware Sand & Gravel Superfund Site, New Castle, Delaware. June 2018.

Golder, 2018c. Pre-Design Investigation Work Plan, Delaware Sand & Gravel Superfund Site, New Castle County, Delaware. August 17, 2018.

Golder, 2018d. Summary of Comments and Responses on Pre-Design Investigation and Remedial Design Work Plans, Delaware Sand & Gravel Superfund Site, New Castle County, Delaware. November 16, 2018.

Golder, 2018e. Response to USEPA Request for Alternate Purging and Sampling Method for Long-Screen Wells, Delaware Sand & Gravel Superfund Site, New Castle County, Delaware. December 7, 2018.

Golder, 2019. Sampling and Analysis Plan, Revision 1. Delaware Sand & Gravel Superfund Site, New Castle County, Delaware. January 31, 2019.

Pennino, J.D. 1988. There's No Such Thing as a Representative Ground Water Sample. Ground Water Monitoring Review. V 8, pp. 4-9.

Powell, R.M. and R.W. Puls. 1993. Passive Sampling of Ground-Water Monitoring Wells without Purging: Multilevel Well Chemistry and Tracer Disappearance. Journal Contaminant Hydrology. V.12, pp. 51-77.

Puls, R.W. and R.M. Powell. 1992. Acquisition of Representative Ground Water Quality Samples for Metals. Ground Water Monitoring Review. v. 12, pp. 167-176.

Puls, R.W., D.A. Clark, B. Bledsoe, R.M. Powell, and C.J. Paul. 1992. Metals in Ground Water: Sampling Artifacts and Reproducibility. Hazardous Waste and Hazardous Materials. V. 9, pp. 149-162.

USEPA, 1993. Chloride, Nitrate, Nitrite, and Sulfate following USEPA Methods for Chemical Analysis of Water and Wastes (MCAWW) Method 300.0 Determination of Inorganic Anions by Ion Chromatography. August 1993. Environmental Protection Agency, Environmental Monitoring Systems Laboratory, Cincinnati, Ohio, accessed at URL http://www.epa.gov/waterscience/methods/method/organics/

USEPA, 1994. EPA Method 7470A (SW-846): Mercury in Liquid Waste (Manual Cold-Vapor Technique), Revision 1. Washington, DC. September 1994.

USEPA, 1996a. Low-Flow (Minimal Drawdown) Ground-Water Sampling Procedures. April 1996.

USEPA, 1996b. Test Methods for Evaluating Solid Waste, Physical/Chemical Methods (SW-846): 3rd edition and its subsequent updates, Environmental Protection Agency, National Center for Environmental Publications, Cincinnati, Ohio, accessed at URL http://www.epa.gov/epaoswer/hazwaste/test/sw846.htm.

USEPA, 1997. Recommended Procedure for Low-flow Purging and Sampling of Groundwater Monitoring Wells, Bulletin No. QAD023. October 15, 1997.

USEPA, 1998a. EPA Method 8270D (SW-846): Semivolatile Organic Compounds by Gas Chromatography/Mass Spectrometry (GC/MS), Revision 4. Washington, DC. January 1998.

USEPA, 1998b. Groundwater Sampling Procedure, Low Stress (Low Flow) Purging and Sampling. March 20, 1998.

USEPA, 2006. EPA Method 8260C (SW-846): Volatile Organic Compounds by Gas Chromatography/Mass Spectrometry (GC/MS), Revision 3. Washington, DC. August 2006.

USEPA, 2008. Design and Installation of Monitoring Wells by Striggow, Brian (USEPA SESDGUID-101-R0) February 13, 2008.

USEPA, 2009. Method 537. Determination of Selected Perfluorinated Alkyl Acids in Drinking Water by Solid Phase Extraction and Liquid Chromatography/Tandem Mass Spectrometry (LC/MS/MS), Version 1.1. EPA/600/R-08/092. September 2009.

USEPA, 2012. Email from Debbie Rossi (USEPA) to Michael Barbara (DS&G Trust) providing approval for use of Method 8260 and 8260 SIM for 1,4-dioxane analysis. October 31, 2012.

USEPA, 2014. EPA Method 6010D (SW-846): Inductively Coupled Plasma-Atomic Emission Spectrometry, Revision 4. Washington, DC. July 2014.

USEPA, 2016a. Letter from Debbie Rossi (USEPA) to Doug Sutton (DS&G Trust) providing approval of the Final Feasibility Study – Revision 1. July 5, 2016.

USEPA, 2016b. Proposed Remedial Action Plan (PRAP), Delaware Sand & Gravel Superfund Site, New Castle, Delaware. September 7, 2016

USEPA, 2016c. Email from Debbie Rossi (USEPA) to Doug Sutton (DS&G Trust) providing approval of the PFC Work Plan. September 23, 2016.

USEPA, 2017a. USEPA Contract Laboratory Program National Functional Guidelines for Superfund Organic and Inorganic Methods Data Review. January 2017.

USEPA, 2017b. USEPA email approval of December 21, 2016 TIC evaluation and request for TIC evaluations every two years. February 6, 2017.

USEPA, 2017c. USEPA requested via email addition of sampling and analysis of the Columbia Aquifer and Upper Potomac Confining Unit Transition Zone (UPCUTZ) locations to the PFCs WP. November 13, 2017.

USEPA, 2017d. Amendment No. 2 to the 1988 Record of Decision for the Delaware Sand & Gravel Landfill Superfund Site, New Castle, Delaware. December 12, 2017.

USEPA, 2018a. Email from Debbie Rossi (USEPA) to Michael Sherrier (ACL Chairman) and Michael Harris (NCC) regarding addition of cations and anions to semi-annual groundwater monitoring event. January 19, 2018.

USEPA, 2018b. Letter from Rick Wilkins (USEPA) to Debbie Rossi (USEPA) regarding addition of cations and anions to semi-annual groundwater monitoring event. January 24, 2018.

USEPA, 2018c. Email from Debbie Rossi (USEPA) to Doug Sutton (DS&G Trust) requesting addition of cation and anions to semi-annual groundwater monitoring event. February 1, 2018.

USEPA, 2018d. Email from Debbie Rossi (USEPA) to Doug Sutton (DS&G Trust) providing comments on PFAS 2018 Work Plan. April 26, 2018.

USEPA, 2018e. Email from Debbie Rossi (USEPA) to Doug Sutton (DS&G Trust) providing agreement with inclusion of revised groundwater monitoring program in the PDI WP and SAP. May 18, 2018.

USEPA, 2018f. Administrative Settlement Agreement and Order on Consent for Remedial Design. May 22, 2018.

USEPA, 2018g. USEPA letter re: Partial Approval of the PDI WP with additional comments. November 30, 2018.

USEPA, 2018h. USEPA email re: Approval of Eurofins use of modified PFAS method. December 18, 2018.

USEPA, 2018i. USEPA email re: Collection of split samples for PFAS analysis. December 19, 2018.

USEPA, 2018j. USEPA email re: response to the Alternate Methods Memo. December 21, 2018.

USEPA, 2019. USEPA conditional approval of the PDI WP-Rev 1 and RDWP-Rev 1 via email. February 26, 2019.Yeskis, Zavala, 2002. Ground-Water Sampling Guidelines for Superfund and RCRA Project Managers Ground-Water Sampling Guidelines for Superfund and RCRA Project Managers written by Yeskis, Douglas and Zavala, Bernard. (EPA 542-S02-001). May 2002.

Golder and the G logo are trademarks of Golder Associates Corporation

\\mtlaurel\data\projects\2001 projects\013-6052 ds&g\post-aoc 2018-\reports-deliverables\pdi work plan\rev 2\sap revision 2.docx

Tables

TABLE A-1
DATA QUALITY OBJECTIVES
DELAWARE SAND & GRAVEL SUPERFUND SITE
NEW CASTLE COUNTY, DELAWARE

Investigation Activity	Matrix	Number of Locations	Parameters of Interest	Frequency of Monitoring	Purpose/Objective of Activity
Boring Advancement	Soil	33 proposed locations	Lithology; PID readings; visual and/or olfactory evidence of impacts; grain size	Once, during boring advancement	Collect definitive data via soil logging, PID readings and VAP for determining well screen intervals
Vertical Aquifer Profiling	Groundwater	Up to 14 boring locations	VOCs and SVOCs	Once, during boring advancement	Collect definitive data to determine most impacted groundwater intervals within the UPCUTZ, UPA upper sand and UPA lower sand across which to screen monitoring wells
Soil Sampling	Soil	One sample from each of 6 boreholes (DDA-05-TZ-EXTR, DDA-06-TZ-EXTR, DDA-10/12-US-EXTR, UPA-01-US-EXTR, UPA-101-LS, and MW-18/34-US-EXTR)	Grain size - sieve analysis	Once, during boring advancement	Collect definitive data for design of extraction well
Groundwater Monitoring	Groundwater	33 proposed wells	TCL VOCs + up to 20 TICs; TCL SVOCs + up to 10 TICs; low-level BCEE and 1,4-dioxane as needed; TAL total metals, dissolved iron, manganese, and cobalt, and field parameters (see note 3 below)	Once, approximately two weeks after well installation and development	Collect definitive data to define nature and extent of contamination
	Groundwater	82 existing wells, 33 proposed wells, and 2 new extraction wells	TCL VOCs + up to 20 TICs; TCL SVOCs + up to 10 TICs; low-level BCEE and 1,4-dioxane as needed; TAL total metals, dissolved iron, manganese, and cobalt, and field parameters (see note 3 below)	Semi-annually in April	Collect definitive data to define nature and extent of contamination
	Groundwater	37 existing wells, 33 proposed wells, and 2 new extraction wells	TCL VOCs + up to 20 TICs; TCL SVOCs + up to 10 TICs; low-level BCEE and 1,4-dioxane as needed; dissolved iron, manganese, and cobalt, and field parameters (see note 3 below)	Semi-annually in October	Collect definitive data to define nature and extent of contamination
	Groundwater	32 existing wells, and 33 proposed wells	Major cations and anions (see note 4 below)	Annually, first event coincident with ACL PFAS sampling event (either April or October)	Collect definitive data to evaluate cation/anion balance in groundwater
	Groundwater	37 existing wells and 5 new wells	PFAS	Annually, first event coincident with ACL PFAS sampling event (either April or October)	Collect definitive data to define nature and extent of contamination
	Groundwater	4 wells upgradient of AWC-G3R	Total and dissolved iron, manganese, and cobalt, and field parameters (see note 3 below)	Bi-monthly	Collect definitive data to evaluate trends
	Groundwater	Up to 4 AWC production wells	Total iron and manganese	Monthly (by AWC)	Collect definitive data to evaluate trends
	Groundwater	Up to 4 AWC production wells	BCEE, 1,4-Dioxane, PFAS and cobalt	Quarterly (by AWC)	Collect definitive data to evaluate trends
	Groundwater	Up to 53 DDA monitoring points	TCL VOCs + up to 20 TICs; TCL SVOCs + up to 10 TICs; low-level BCEE and 1,4-dioxane as needed	Once	Collect definitive data to define current nature and extent of contamination in DDA and design of ELFExS
Surveying	not applicable	New well locations	Ground and top of PVC elevation (wells only), northings and eastings	Once, after installation of new wells	Collect definitive data to provide location and elevation data for new well samples
Transducer Study	Groundwater	10 existing wells	Potentiometric heads	Continuous logging for approximately one year	Collect definitive data to evaluate potentiometric heads and changes over time due to AWC operations and other influences on the aquifer
UPA Pumping Tests	Groundwater	2 existing wells and 3 proposed wells	Potentiometric heads, VOCs, SVOCs and field parameters	24- to 72-hour test (duration dependent on location) followed by recovery period	Collect definitive data to evaluate potentiometric head changes, extent of capture zone and potentially sustainable extraction rates for UPA upper sand wells proposed as part of Selected Remedy
UPCUTZ Extraction Testing	Groundwater	2 proposed wells	Potentiometric heads	24-hour test for each location followed by recovery period	Collect definitive data to evaluate potentiometric head changes, extent of capture zone and potentially sustainable extraction rates for UPCUTZ low-flow extraction system proposed as part of Selected Remedy
LFG Barhole Probes	Soil Gas	Approximately 50 barhole probe locations	Methane, % lower explosive limit, oxygen, carbon dioxide, VOCs	Once, at time of barhole probe event	Collect definitive data to evaluate presence of LFG migration beyond that currently under mitigation

Notes:

1. The Target Compound List (TCL) for VOCs, SVOCs, metals, cations/anions and PFAS is provided in Table A-14.
2. The methodologies that will be used for analysis are listed in Table A-15.
3. Field parameters for groundwater monitoring include: water level, pH, temperature, specific conductivity, turbidity, dissolved oxygen, and oxidation-reduction potential.
4. Major cations and anions include: calcium, magnesium, potassium, sodium, ammonia, nitrate, nitrite, sulfate, sulfide, chloride, and bicarbonate.
5. See Attachment H for acronyms and abbreviations

Checked by: BAR
Reviewed by: TAM

TABLE A-2
DECISION THRESHOLDS / ACTION LEVELS
DELAWARE SAND & GRAVEL SUPERFUND SITE
NEW CASTLE COUNTY, DELAWARE

Investigation Activity - Goal		Matrix	Number of Locations	Parameters of Interest	Screening Values/Method	Decision Threshold / Action Level
Boring Advancement - Collect definitive data via soil logging, PID readings and VAP for determining well screen intervals		Soil	33 proposed locations	Lithology; PID readings; visual and/or olfactory evidence of impacts; grain size	PID readings > 10 ppmv; visual evidence of a sheen or impacts; petroleum and/or chemical odor	1) If the screening values are met or exceeded in a Proposed Monitoring Well Location, then a monitoring well will be screened across the 5-to 7-foot interval with the highest VOC/SVOC concentrations, PID readings, the most visual and/or olfactory evidence of impacts, and/or the most productive interval within the unit to be monitored (i.e., Columbia Aquifer, UPCUTZ, UPA upper sand or UPA lower sand). 2) If the screening values are NOT met in a Proposed Monitoring Well Location, then a monitoring well will be screened across the coarsest-grained 10-foot interval within the unit to be monitored.
Vertical Aquifer Profiling - Collect definitive data to determine most impacted groundwater intervals within the UPCUTZ, UPA upper sand and UPA lower sand across which to screen monitoring		Groundwater	Up to 14 boring locations	VOCs and SVOCs	ss-PRGs, MCLs, SMCLs and RSLs	
Soil Sampling - Collect definitive data for design of extraction well		Soil	One sample from each of 6 boreholes (DDA-05-TZ-EXTR, DDA-06-TZ-EXTR, DDA-10/12-US-EXTR, UPA-01-US-EXTR, UPA-101-LS, and MW-18/34-US-EXTR)	Grain size	Sieve analysis	1) If the screening values for a depth interval indicate that the interval will not produce sufficient flow for the extraction well, then a different depth interval will be chosen to determine the appropriate depth and well design parameters for extraction. 2) If the screening values for a depth interval indicate that the interval will produce sufficient flow for the extraction well, then those data and depth will be used to determine the appropriate well design parameters for extraction.
Groundwater Monitoring	Collect definitive data to define nature and extent of contamination	Groundwater	33 proposed wells	Baseline monitoring: TCL VOCs + up to 20 TICs; TCL SVOCs + up to 10 TICs; low-level BCEE and 1,4-dioxane as needed; TAL total metals, dissolved iron, manganese, and cobalt, and field parameters (see note 3 below)	ss-PRGs, MCLs, SMCLs and RSLs	1) If the screening values are exceeded in new monitoring wells, then location should be included in semi-annual monitoring program and additional investigation may be necessary. 2) If the screening values are NOT exceeded but are above method detection limits in new monitoring wells, then location should be included in semi-annual monitoring program. 3) If the parameters are not detected at monitoring locations, then discontinuation of monitoring at those locations should be considered.
			82 existing wells, 33 proposed wells, and 2 new extraction wells	Semi-annual (April) monitoring: TCL VOCs + up to 20 TICs; TCL SVOCs + up to 10 TICs; low-level BCEE and 1,4-dioxane as needed; TAL total metals, dissolved iron, manganese, and cobalt, and field parameters (see note 3 below)		
			37 existing wells, 33 proposed wells, and 2 new extraction wells	Semi-annual (October) monitoring: TCL VOCs + up to 20 TICs; TCL SVOCs + up to 10 TICs; low-level BCEE and 1,4-dioxane as needed; dissolved iron, manganese, and cobalt, and field parameters (see note 3 below)		
			32 existing wells, and 33 proposed wells	PFAS (annual monitoring)	HAL; RSL	1) If the screening values are exceeded, then annual monitoring will continue for that location and additional evaluation may be necessary. 2) If the screening values are NOT exceeded but are above method detection limits, then annual monitoring for that location may be discontinued. 3) If the parameters are not detected at monitoring locations, then PFAS monitoring at those locations should be discontinued.
	Collect definitive data to evaluate cation/anion balance in groundwater	Groundwater	37 existing wells and 5 new wells	Major cations and anions (see note 4 below; annual monitoring)	Cation/Anion Balance	1) If the cation/anion balance exceeds 20% difference, then additional monitoring of cations and anions and consideration of other potential cations and/or anions for analysis should be performed. 2) If the cation/anion balance has less than 10% difference, then additional monitoring of cations and anions should not be necessary.
	Collect definitive data to evaluate trends	Groundwater	4 wells upgradient of AWC-G3R	Total and dissolved iron, manganese, and cobalt, and field parameters (see note 3 below)	ss-PRGs, SMCLs and RSLs	Trends plots for these locations have been and will continue to be updated as new analytical data becomes available. Trend plots are routinely provided to and discussed with AWC. 1) If a trend plot indicates an increasing trend, then the location and parameter with the increasing trend will be reviewed carefully and brought to the attention of AWC and the USEPA as part of the semi-annual monitoring report. 2) If a trend plot indicates a stable or decreasing trend, then the trend will continue to be reviewed carefully and the trend plot will be included in the semi-annual monitoring report.
			Up to 4 AWC production wells	Total iron and manganese (monthly monitoring)		
			Up to 4 AWC production wells	BCEE, 1,4-Dioxane, PFAS and cobalt (quarterly monitoring)	ss-PRGs, MCLs, HAL (PFAS), and RSLs	
	Collect definitive data to define current nature and extent of contamination in DDA and design of ELFExS	Groundwater	Up to 53 DDA monitoring points	TCL VOCs + up to 20 TICs; TCL SVOCs + up to 10 TICs; low-level BCEE and 1,4-dioxane if needed (one-time monitoring)	ss-PRGs, HAL (PFAS), RSLs	1) If the screening values are exceeded, then that location will be considered for placement of an extraction well as part of the ELFExS. 2) If the screening values are not exceeded, then that location will be not considered for placement of an extraction well as part of the ELFExS.
Surveying - Collect definitive data to provide location and elevation data for new well samples		not applicable	New well locations	Ground and top of PVC elevation (wells only), northings and eastings	Comparison to previously surveyed points	1) If the survey elevations for a subset of existing points do not match previous elevations for those points, then datum will be reviewed and points resurveyed. 2) If the survey elevations for a subset of existing points match previous elevations for those points, then surveyed elevations for new points will be accepted.
Transducer Study - Collect definitive data to evaluate potentiometric heads and changes over time due to AWC operations and other influences on the aquifer		Groundwater	10 existing wells	Potentiometric heads	Groundwater flow modeling	1) If the data points recorded by a transducer are unstable or appear anomalous, then data points will not be used in the evaluations and to update the groundwater flow model. 2) If the data points recorded by a transducer are stable and appear consistent data recorded by other transducers, then data points will be used in the evaluations and to update the groundwater flow model.
UPA Pumping Tests - Collect definitive data to evaluate potentiometric head changes, extent of capture zone and potentially sustainable extraction rates for UPA upper sand wells proposed as part of Selected Remedy		Groundwater	2 existing wells and 3 proposed wells	Potentiometric heads, VOCs, SVOCs and field parameters	Groundwater flow modeling, comparison to ss-PRGs, RSLs and baseline data	1) If the data points recorded in the field and by the transducers appear anomalous, then data points will not be used in the evaluations and to update the groundwater flow model. 2) If the data points recorded in the field do not indicate any apparent errors and by a transducer appear consistent with data recorded by other transducers, then data points will be used in the evaluations and to update the groundwater flow model.
UPCUTZ Extraction Testing - Collect definitive data to evaluate potentiometric head changes, extent of capture zone and potentially sustainable extraction rates for UPCUTZ low-flow extraction system proposed as part of Selected Remedy		Groundwater	2 proposed wells	Potentiometric heads	Groundwater flow modeling	1) If the data points recorded in the field and by the transducers appear anomalous, then data points will not be used in the evaluations and to update the groundwater flow model. 2) If the data points recorded in the field do not indicate any apparent errors and by a transducer appear consistent with data recorded by other transducers, then data points will be used in the evaluations and to update the groundwater flow model.
LFG Barhole Probes - Collect definitive data to evaluate presence of LFG migration beyond that currently under mitigation		Soil Gas	Approximately 50 barhole probe locations	Methane, % lower explosive limit, oxygen, carbon dioxide, VOCs	% LEL at facility boundary	1) If the screening values are exceeded at the facility boundaries, then additional investigation in those areas may be necessary. 2) If the screening values are not exceeded at the facility boundaries, then additional investigation will not be necessary.

Notes:
1. The Target Compound List (TCL) for VOCs, SVOCs, metals, cations/anions and PFAS is provided in Table A-14.
2. The methodologies that will be used for analysis are listed in Table A-15.
3. Field parameters for groundwater monitoring include: water level, pH, temperature, specific conductivity, turbidity, dissolved oxygen, and oxidation-reduction potential.
4. Major cations and anions include: calcium, magnesium, potassium, sodium, ammonia, nitrate, nitrite, sulfate, sulfide, chloride, and bicarbonate.
5. See Attachment H for acronyms and abbreviations

Checked by: BAR
Reviewed by: TAM

TABLE A-3
LIST OF AND RATIONALE FOR PROPOSED MONITORING WELL LOCATIONS
DELAWARE SAND & GRAVEL SUPERFUND SITE
NEW CASTLE COUNTY, DELAWARE

Profiling Location ID	Location Description	Property Ownership	Primary Purpose	Secondary Purpose	Columbia Aquifer	UPCUTZ	UPA Upper Sand	UPA Lower Sand
Between the DDA and Well PW-1(U)								
DDA-18	Southwest of well pair DDA-16-TZ/DDA-16-US (south of well pair DGC-2S/DGC-2D)	Dell'Aversano	Confirmation of target capture zones and evaluation of modeled extraction rates for proposed UPCUTZ and well PW-1(U) systems	Performance monitoring of proposed UPCUTZ and PW-1(U) systems		X	X	
DDA-19	Southeast of well pair DDA-12-TZ/DDA-12-US and south of well DDA-10-US (no UPCUTZ present at well DDA-10-US)	Dell'Aversano	Confirmation of target capture zones and evaluation of modeled extraction rates for proposed UPCUTZ and DDA-10-US/DDA-12-US extraction systems	Performance monitoring of proposed UPCUTZ and DDA-10-US/DDA-12-US extraction systems		X	X	
DDA-20	Southeast of well DDA-10-US and northeast of wells DDA-12-US and PW-1(U)	Dell'Aversano	Confirmation of target capture zones in DDA-10/12-US area and evaluation of potential extraction rates for proposed for proposed UPCUTZ and DDA-10-US/DDA-12-US extraction systems	Performance monitoring of proposed UPCUTZ and DDA-10-US/DDA-12-US extraction systems		X	X	
Well P-6 Area								
101-2018	Adjacent to existing wells P-6, UPA-101-US and UPA-101-TZ	Cirillo Brothers	Three locations are needed for: 1) Evaluation of the nature/orientation of the "feature" used in the groundwater flow model to simulate the localized hydraulic mounding in the well P-6 area 2) Evaluation of the elevated manganese concentrations detected in temporary Columbia Aquifer well GMW-11 3) Improvement of the UPCUTZ characterization in this area 4) Assisting with confirmation of the modeled extraction rate and target capture zone for the proposed extraction well in this area	Migration between UPA upper and lower sand				X
102	North of well P-6 - between the Inert Area boundary and Grantham Lane	Dell'Aversano or New Castle County/Grantham Lane Right-of-Way		Assessment of potential presence of impacts in the Columbia Aquifer near the Inert Area, and the UPCUTZ and UPA upper and lower sand upgradient of well P-6	X	X	X	
103	Northwest of well P-6 - Along Grantham Lane and south of the Inert Area	New Castle County		Assessment of potential presence of impacts in the Columbia Aquifer near the Inert Area and UPA upper sand upgradient of wells MW-18, MW-34 and P-6	X	X	X	X
104	South of well P-6 - in Area of Attainment (AoA)	Cirillo Brothers		1) Performance monitoring of proposed extraction wells in the well P-6 and RT-1-UP/UPA-01 areas 2) Assessment of potential presence of impacts in the UPCUTZ and UPA upper and lower sand downgradient of well P-6	assumed dry	X	X	X
Well UPA-01 / RT-1-UP Area								
105A	Southeast of wells RT-1-UP and UPA-01 along Grantham Lane	Cirillo Brothers	Up to three locations are needed for: 1) Assessing the eastern extent of groundwater impacts in UPA-01 area 2) Assisting with evaluation of modeled extraction rate and target capture zone for a potential extraction well in the area of wells RT-1-UP/UPA-01 3) Evaluation of potential for connection between UPA upper and lower sand	Performance monitoring of proposed extraction well in the area of well UPA-01	assumed dry	X	X	X
105B	Southeast of wells RT-1-UP and UPA-01 along Grantham Lane	Cirillo Brothers			assumed dry	X	X	X
Well MW-18 / MW-34 Area								
106	Northwest of wells MW-18 and MW-34 - near toe of Grantham South	New Castle County	Assessment of the manganese impacts in the Columbia Aquifer and UPA upper and lower sand upgradient of wells MW-18 and MW-34	Evaluation of potential for connection between UPA upper and lower sand between the ACL Site and the wells MW-18/MW-34 area	X	X	X	X
107	South-southeast of wells MW-18 and MW-34 - in AoA	Cirillo Brothers	1) Assessment of the manganese impacts in the UPA upper and lower sand downgradient of wells MW-18 and MW-34 2) Assisting with evaluation of the modeled extraction rate for the proposed extraction well in this area	Performance monitoring of proposed extraction well in the area of wells MW-18 and MW-34	assumed dry	X	X	X
Well BW-2 Area								
Vertical Aquifer Profiling (VAP) Location 1 (one of three potential locations for 108)	North of well BW-2 - between wells MW-28 and MW-29 and along downgradient side of Army Creek Landfill (ACL) Site	New Castle County	Three locations are needed for: 1) Assessment of the impacts (e.g., 1,4-dioxane and manganese) to the well BW-2 area 2) Assessment of the potential presence of impacts in the UPA upper sand upgradient of well BW-2 3) Assisting with evaluation of the modeled extraction rate for the proposed extraction well in this area	Evaluation of potential for connection between UPA upper and lower sand between the ACL Site and the BW-2 area		X	X	X
VAP Location 2 (one of three potential locations for 108)	North of well BW-2 - downgradient of wells MW-28 and MW-29 and ACL Site	New Castle County						
VAP Location 3 (one of three potential locations for 108)	North of well BW-2 - further downgradient of wells MW-28 and MW-29 and ACL Site	New Castle County						

Notes:
1) This table is based on Final FS Rev 1 Appendix Table N-1
2) See Attachment H for acronyms and abbreviations

	Potential Well Counts				Totals
Total Proposed Wells	3	11	11	8	**33**
Total Contingent Wells	0	0	0	0	**0**
Total Potential Wells	**3**	**11**	**11**	**8**	**33**

Checked by: TAM
Reviewed by: BAR

TABLE A-4
PROPOSED WELLS AND AREAS OF UNCERTAINTY MATRIX
DELAWARE SAND & GRAVEL SUPERFUND SITE
NEW CASTLE COUNTY, DELAWARE

		Generalized Nature of Uncertainties							
Profiling Location ID	**Location Description**	**Target Capture Zone and Extraction Rate**	**Migration Between Hydrostratigraphic Units**	**UPCUTZ Extent of Contamination**	**Source and Extent of Manganese Impacts**	**Columbia Aquifer**	**UPCUTZ**	**UPA Upper Sand**	**UPA Lower Sand**
Between the DDA and Well PW-1(U)									
DDA-18	Southwest of well pair DDA-16-TZ/DDA-16-US (south of well pair DGC-2S/DGC-2D)	X	X	X			X	X	
DDA-19	Southeast of well pair DDA-12-TZ/DDA-12-US and south of well DDA-10-US (no UPCUTZ at well DDA-10-US)	X	X	X			X	X	
DDA-20	Southeast of well DDA-10-US and northeast of wells DDA-12-US and PW-1(U)	X		X			X	X	
Well P-6 Area									
101-2019	Adjacent to existing wells P-6, UPA-101-US and UPA-101-TZ	X	X						X
102	North of well P-6 - between the Inert Area boundary and Grantham Lane	X	X	X	X	X	X	X	X
103	Northwest of well P-6 - Along Grantham Lane and south of the Inert Area	X	X	X	X	X	X	X	
104	South of well P-6 - in Area of Attainment (AoA)	X	X	X	X	assumed dry	X	X	X
Well UPA-01 / RT-1-UP Area									
105A	Southeast of wells RT-1-UP and UPA-01 along Grantham Lane	X	X	X		assumed dry	X	X	X
105B	Southeast of wells RT-1-UP and UPA-01 along Grantham Lane	X	X	X		assumed dry	X	X	X
Well MW-18 / MW-34 Area									
106	Northwest of wells MW-18 and MW-34 - near toe of Grantham South	X	X	X	X	X	X	X	X
107	South-southeast of wells MW-18 and MW-34 - in AoA	X	X	X	X	assumed dry	X	X	X
Well BW-2 Area									
108	North of well BW-2 - downgradient of wells MW-28 and MW-29 and ACL Site	X	X	X	X		X	X	X

Notes:
1) This table is based on Final FS Rev 1 Appendix Table N-1
2) See Attachment H for acronyms and abbreviations

Checked by: BAR
Reviewed by: TAM

TABLE A-5
ANTICIPATED CONSTRUCTION DETAILS FOR PROPOSED MONITORING AND EXTRACTION WELLS
DELAWARE SAND & GRAVEL SUPERFUND SITE
NEW CASTLE COUNTY, DELAWARE

Well ID	Profiling Location ID	Purpose of Boring and Well	Unit	Anticipated Boring Depth (ft-bgs)	Diameter (inches)	Material	Anticipated Screen Length (feet)	Screen Slot Size (inches)	Anticipated Screen Interval (ft-bgs)	Anticipated Filter Pack Length (feet)
Between the DDA and Well PW-1(U)										
DDA-18-TZ	DDA-18	Monitoring Well	UPCUTZ	85	2	Schedule 40 PVC	5 to 7	0.010	TBD	10
DDA-18-US	DDA-18	VAP, Monitoring Well	UPA Upper Sand	100	2	Schedule 40 PVC	5 to 7	0.010	TBD	10
DDA-19-TZ	DDA-19	Monitoring Well	UPCUTZ	70	2	Schedule 40 PVC	5 to 7	0.010	TBD	10
DDA-19-US	DDA-19	VAP, Monitoring Well	UPA Upper Sand	105	2	Schedule 40 PVC	5 to 7	0.010	TBD	10
DDA-20-TZ	DDA-20	Monitoring Well	UPCUTZ	70	2	Schedule 40 PVC	5 to 7	0.010	TBD	10
DDA-20-US	DDA-20	VAP, Monitoring Well for DDA-10/12-US-EXTR	UPA Upper Sand	105	2	Schedule 40 PVC	5 to 7	0.010	TBD	10
DDA-10/12-US-EXTR	DDA-10/12-US-EXTR	Extraction Well	UPA Upper Sand	105	6	Stainless Steel	TBD	TBD	TBD	TBD
DDA-05-TZ-EXTR	former DDA-05	Extraction Well	UPCUTZ	65	6	Stainless Steel	TBD	TBD	TBD	TBD
DDA-06-TZ-EXTR	former DDA-06	Extraction Well	UPCUTZ	65	6	Stainless Steel	TBD	TBD	TBD	TBD
Well P-6 Area										
UPA-101-LS	101-2019	VAP only lower than UPA-101-US (2018), Monitoring Well	UPA Lower Sand	175	2	Schedule 40 PVC	5 to 7	0.010	TBD	10
CA-102	102	Monitoring Well	Columbia Aquifer	45	2	Schedule 40 PVC	5 to 7	0.010	TBD	10
UPA-102-TZ	102	Monitoring Well	UPCUTZ	80	2	Schedule 40 PVC	5 to 7	0.010	TBD	10
UPA-102-US	102	Monitoring Well	UPA Upper Sand	110	2	Schedule 40 PVC	5 to 7	0.010	TBD	10
CA-103	103	Monitoring Well	Columbia Aquifer	25	2	Schedule 40 PVC	5 to 7	0.010	TBD	10
UPA-103-TZ	103	Monitoring Well	UPCUTZ	74	2	Schedule 40 PVC	5 to 7	0.010	TBD	10
UPA-103-US	103	Monitoring Well	UPA Upper Sand	120	2	Schedule 40 PVC	5 to 7	0.010	TBD	10
UPA-103-LS	103	VAP, Monitoring Well	UPA Lower Sand	175	2	Schedule 40 PVC	5 to 7	0.010	TBD	10
UPA-104-TZ	104	Monitoring Well	UPCUTZ	80	2	Schedule 40 PVC	5 to 7	0.010	TBD	10
UPA-104-US	104	Monitoring Well	UPA Upper Sand	110	2	Schedule 40 PVC	5 to 7	0.010	TBD	10
UPA-104-LS	104	VAP, Monitoring Well	UPA Lower Sand	175	2	Schedule 40 PVC	5 to 7	0.010	TBD	10
Well UPA-01 / RT-1-UP Area										
UPA-105A-TZ	105A	Monitoring Well	UPA Upper Sand	80	2	Schedule 40 PVC	5 to 7	0.010	TBD	10
UPA-105A-US	105A	Monitoring Well for UPA-01-US-EXTR	UPA Upper Sand	105	2	Schedule 40 PVC	5 to 7	0.010	TBD	10
UPA-105A-LS	105A	VAP, Monitoring Well	UPA Lower Sand	175	2	Schedule 40 PVC	5 to 7	0.010	TBD	10
UPA-105B-TZ	105B	Monitoring Well	UPA Upper Sand	80	2	Schedule 40 PVC	5 to 7	0.010	TBD	10
UPA-105B-US	105B	Monitoring Well for UPA-01-US-EXTR	UPA Upper Sand	105	2	Schedule 40 PVC	5 to 7	0.010	TBD	10
UPA-105B-LS	105B	VAP, Monitoring Well	UPA Lower Sand	175	2	Schedule 40 PVC	5 to 7	0.010	TBD	10
UPA-01-US-EXTR	105B	Extraction Well	UPA Upper Sand	105	8	Stainless Steel	TBD	TBD	TBD	TBD
Well MW-18 / MW-34 Area										
CA-106	106	Monitoring Well	Columbia Aquifer	20	2	Schedule 40 PVC	5 to 7	0.010	TBD	10
UPA-106-TZ	106	Monitoring Well	UPCUTZ	35	2	Schedule 40 PVC	5 to 7	0.010	TBD	10
UPA-106-US	106	Monitoring Well	UPA Upper Sand	65	2	Schedule 40 PVC	5 to 7	0.010	TBD	10
UPA-106-LS	106	VAP, Monitoring Well	UPA Lower Sand	115	2	Schedule 40 PVC	5 to 7	0.010	TBD	10
UPA-107-TZ	107	Monitoring Well	UPCUTZ	80	2	Schedule 40 PVC	5 to 7	0.010	TBD	10
UPA-107-US	107	Monitoring Well for MW-18/34-US-EXTR	UPA Upper Sand	110	2	Schedule 40 PVC	5 to 7	0.010	TBD	10
UPA-107-LS	107	VAP, Monitoring Well	UPA Lower Sand	175	2	Schedule 40 PVC	5 to 7	0.010	TBD	10
MW-18/34-US-EXTR	107	Extraction Well	UPA Upper Sand	110	8	Stainless Steel	TBD	TBD	TBD	TBD
Well BW-2 Area										
UPA-108-TZ	108A, B or C	Monitoring Well	UPCUTZ	80	2	Schedule 40 PVC	5 to 7	0.010	TBD	10
UPA-108-US	108A, B or C	Monitoring Well	UPA Upper Sand	90	2	Schedule 40 PVC	5 to 7	0.010	TBD	10
UPA-108-LS	108A, B or C	VAP, Monitoring Well	UPA Lower Sand	125	2	Schedule 40 PVC	5 to 7	0.010	TBD	10

Notes:

1) This table is based on Final FS Rev 1 Appendix Table N-1

2) See Attachment H for acronyms and abbreviations

3) Anticipated boring depths are listed as a guide; however, actual boring depths will be dependent on field observations including lithologic changes.

4) Anticipated screen construction listed as "TBD" (to be determined) because the screen construction will be determined based on information to be collected in the field.

Checked by: TAM

Reviewed by: BAR

TABLE A-6A
SEMI-ANNUAL WATER LEVEL MONITORING LOCATIONS
DELAWARE SAND & GRAVEL SUPERFUND SITE
NEW CASTLE COUNTY, DELAWARE

Monitoring Point ID	Well Type/Purpose	Hydrogeologic Unit	Reference Elevation (feet MSL)
DDA Low-Flow Extraction System Wells			
B-4DR	Extraction - LFExS	Columbia Aquifer - Columbia Sand	30.15
BG-1	Extraction - LFExS	Columbia Aquifer	24.97
C-18D	Extraction - LFExS	Columbia Aquifer	25.41
C-19D	Extraction - LFExS	Columbia Aquifer	28.86
C-20D	Extraction - LFExS	Columbia Aquifer	32.57
C-2D	Extraction - LFExS	Columbia Aquifer - Columbia Sand and Basal Gravel	22.44
C-30	Extraction - LFExS	Columbia Aquifer	25.71
C-4D	Extraction - LFExS	Columbia Aquifer - Columbia Sand and Basal Gravel	22.74
DDA Monitoring Wells within Containment Area			
B-1D	Hydraulic gradient monitoring	Columbia Aquifer	27.40
B-2D	Monitoring near BG-1 and C-2D	Columbia Aquifer - Columbia Sand and Basal Gravel	28.60
B-3D	Monitoring near BG-1 and C-4D	Columbia Aquifer	31.21
C-1D	Monitoring along Northern Boundary	Columbia Aquifer - Columbia Sand and Basal Gravel	22.30
C-3D	Monitoring along Northern Boundary	Columbia Aquifer	22.66
C-5D	Hydraulic gradient monitoring	Columbia Aquifer	23.20
C-6	Hydraulic gradient monitoring	Columbia Aquifer	25.32
C-7	Hydraulic gradient monitoring	Columbia Aquifer	23.07
C-8	Hydraulic gradient monitoring	Columbia Aquifer	23.50
C-9	Hydraulic gradient monitoring	Columbia Aquifer	24.93
C-10	Hydraulic gradient monitoring	Columbia Aquifer	26.43
C-12	Hydraulic gradient monitoring	Columbia Aquifer	26.04
C-14	Hydraulic gradient monitoring	Columbia Aquifer	25.50
C-15D	Hydraulic gradient monitoring	Columbia Aquifer	23.53
C-16	Hydraulic gradient monitoring	Columbia Aquifer	24.01
C-17	Hydraulic gradient monitoring	Columbia Aquifer	24.80
C-21D	Hydraulic gradient monitoring	Columbia Aquifer	32.42
C-22D	Hydraulic gradient monitoring	Columbia Aquifer	33.38
C-22S	Hydraulic gradient monitoring	Columbia Aquifer	34.55
C-23	Hydraulic gradient monitoring	Columbia Aquifer	29.70
C-24	Hydraulic gradient monitoring	Columbia Aquifer	28.28
C-25	Hydraulic gradient monitoring	Columbia Aquifer	30.37
C-27	Hydraulic gradient monitoring	Columbia Aquifer	29.25
MHW-1M	Monitoring near C-20D	Base of Columbia Aquifer	29.83
MHW-1S	Monitoring near C-20D	Columbia Aquifer	29.83
PZ-11-EXT	Hydraulic gradient monitoring	Columbia Aquifer	23.27
PZ-11-INT-R	Hydraulic gradient monitoring	Columbia Aquifer	24.28
PZ-12-EXT	Hydraulic gradient monitoring	Columbia Aquifer	26.07
PZ-12-INT	Hydraulic gradient monitoring	Columbia Aquifer	24.77
PZ-2-EXT	Hydraulic gradient monitoring	Columbia Aquifer	25.49
PZ-2-INT	Hydraulic gradient monitoring	Columbia Aquifer	29.53
PZ-5-EXT	Hydraulic gradient monitoring	Columbia Aquifer	24.18
PZ-5-INT	Hydraulic gradient monitoring	Columbia Aquifer	24.40
PZ-6S	Monitoring near C-30 and Partition	Columbia Aquifer	28.01
DDA Monitoring Wells within Partition Area			
P-4D	Monitoring - Partition	Columbia Aquifer - UPCU, Columbia Clay, Basal Gravel	25.22
PZ-3 INT	Hydraulic gradient monitoring	Columbia Aquifer	21.56
PZ-4-EXT	Hydraulic gradient monitoring	Columbia Aquifer	23.98
PZ-4-INT-R	Hydraulic gradient monitoring	Columbia Aquifer	24.11
PZ-6N	Monitoring - Partition	Columbia Aquifer	27.55
DDA to PW-1(U) Monitoring Wells			
GA-101	Monitoring - Northern DDA Boundary	Columbia Aquifer	23.65
DGC-7C	Monitoring - Near Inert Area	Columbia Aquifer	29.65
DDA-01	Monitoring - Downgradient of DDA	UPA - Upper Sand	30.92
DDA-02	Monitoring - Downgradient of DDA	UPA - Upper Sand	29.57
DDA-03	Monitoring - Downgradient of DDA	UPA - Upper Sand	27.12
DDA-04	Monitoring - Well PW-1(U) Drawdown	UPA - Upper Sand	31.03
DDA-05	Monitoring - Downgradient of DDA	UPCU - Transition Zone	28.68
DDA-06	Monitoring - Downgradient of DDA	UPCU - Transition Zone	28.14
DDA-07-TZ	Monitoring - Beneath DDA	UPCU - Transition Zone	25.22
DDA-07-US	Monitoring - Beneath DDA	UPA - Upper Sand	25.44
DDA-08-TZ	Monitoring - Beneath DDA	UPCU - Transition Zone	26.20
DDA-08-US	Monitoring - Beneath DDA	UPA - Upper Sand	24.52
DDA-09-TZ	Monitoring - Downgradient of DDA	UPCU - Transition Zone	30.86
DDA-10-US	Monitoring - Downgradient of DDA	UPA - Upper Sand	24.30
DDA-11-LS	Monitoring - Downgradient of DDA	UPA - Lower Sand	31.38
DDA-11-US	Monitoring - Downgradient of DDA	UPA - Upper Sand	31.53
DDA-12-TZ	Monitoring - Downgradient of DDA	UPCU - Transition Zone	27.90
DDA-12-US	Monitoring - Downgradient of DDA	UPA - Upper Sand	28.27
DDA-13-TZ	Monitoring - Downgradient of DDA	UPCU - Transition Zone	29.90
DDA-14-TZ	Monitoring - Beneath DDA	UPCU - Transition Zone	30.32
DDA-15-TZ	Monitoring - Beneath DDA	UPCU - Transition Zone	33.98
DDA-15-US	Monitoring - Beneath DDA	UPA - Upper Sand	34.58
DDA-16-TZ	Monitoring - Downgradient of DDA	UPCU - Transition Zone	28.59
DDA-16-US	Monitoring - Downgradient of DDA	UPA - Upper Sand	29.05
DDA-17	Monitoring - Downgradient of DDA	UPA - Upper Sand	29.39
DGC-2D	Monitoring - West of DDA	UPA - Lower Sand	31.51
DGC-2S	Monitoring - West of DDA	UPA - Upper Sand	31.90
DGC-5	Monitoring - Northern DDA Boundary	UPCU - Transition Zone	15.14
DGC-7S	Monitoring - Near Inert Area	UPA - Upper Sand	29.74
MHW-1D	Monitoring - Beneath DDA	UPA - Upper Sand	29.99
MW-45	Monitoring - East of DDA	UPA - Upper and Lower Sand	25.85
PW-1(U)	Extraction - PW-1(U)	UPA - Upper Sand	31.38

TABLE A-6A
SEMI-ANNUAL WATER LEVEL MONITORING LOCATIONS
DELAWARE SAND & GRAVEL SUPERFUND SITE
NEW CASTLE COUNTY, DELAWARE

Monitoring Point ID	Well Type/Purpose	Hydrogeologic Unit	Reference Elevation (feet MSL)
PDI WP Proposed Wells			
DDA-05-TZ-EXTR	UPCUTZ - Future Extraction Well	UPCU - Transition Zone	TBD
DDA-06-TZ-EXTR	UPCUTZ - Future Extraction Well	UPCU - Transition Zone	TBD
DDA-18-TZ	Monitoring - West of Well PW-1(U)	UPCU - Transition Zone	TBD
DDA-18-US	Monitoring - West of Well PW-1(U)	UPA - Upper Sand	TBD
DDA-19-TZ	Monitoring - East of Well PW-1(U)	UPCU - Transition Zone	TBD
DDA-19-US	Monitoring - East of Well PW-1(U)	UPA - Upper Sand	TBD
DDA-20-TZ	Monitoring - Northeast of Well PW-1(U)	UPCU - Transition Zone	TBD
DDA-20-US	Monitoring - Northeast of Well PW-1(U)	UPA - Upper Sand	TBD
UPA-101-LS	Monitoring - Well P-6 Area	UPA - Lower Sand	TBD
CA-102	Monitoring - Inert Area	Columbia Aquifer	TBD
UPA-102-TZ	Monitoring - Well P-6 Area	UPCU - Transition Zone	TBD
UPA-102-US	Monitoring - Well P-6 Area	UPA - Upper Sand	TBD
CA-103	Monitoring - Inert Area	Columbia Aquifer	TBD
UPA-103-TZ	Monitoring - Well P-6 Area	UPCU - Transition Zone	TBD
UPA-103-US	Monitoring - Well P-6 Area	UPA - Upper Sand	TBD
UPA-103-LS	Monitoring - Well P-6 Area	UPA - Lower Sand	TBD
UPA-104-TZ	Monitoring - Well P-6 Area	UPCU - Transition Zone	TBD
UPA-104-US	Monitoring - Well P-6 Area	UPA - Upper Sand	TBD
UPA-104-LS	Monitoring - Well P-6 Area	UPA - Lower Sand	TBD
UPA-105A-TZ	Monitoring - Well UPA-101 Area	UPCU - Transition Zone	TBD
UPA-105A-US	Monitoring - Well UPA-101 Area	UPA - Upper Sand	TBD
UPA-105A-LS	Monitoring - Well UPA-101 Area	UPA - Lower Sand	TBD
UPA-105B-TZ	Monitoring - Well UPA-101 Area	UPCU - Transition Zone	TBD
UPA-105B-US	Monitoring - Well UPA-101 Area	UPA - Upper Sand	TBD
UPA-105B-LS	Monitoring - Well UPA-101 Area	UPA - Lower Sand	TBD
CA-106	Monitoring - Grantham South	Columbia Aquifer	TBD
UPA-106-TZ	Monitoring - Wells MW-18/MW-34 Area	UPCU - Transition Zone	TBD
UPA-106-US	Monitoring - Wells MW-18/MW-34 Area	UPA - Upper Sand	TBD
UPA-106-LS	Monitoring - Wells MW-18/MW-34 Area	UPA - Lower Sand	TBD
UPA-107-TZ	Monitoring - Wells MW-18/MW-34 Area	UPCU - Transition Zone	TBD
UPA-107-US	Monitoring - Wells MW-18/MW-34 Area	UPA - Upper Sand	TBD
UPA-107-LS	Monitoring - Wells MW-18/MW-34 Area	UPA - Lower Sand	TBD
UPA-108-TZ	Monitoring - Well BW-2 Area	UPCU - Transition Zone	TBD
UPA-108-US	Monitoring - Well BW-2 Area	UPA - Upper Sand	TBD
UPA-108-LS	Monitoring - Well BW-2 Area	UPA - Lower Sand	TBD
Downgradient DS&G Monitoring Locations			
DGC-8C	Monitoring - Inert Area	Columbia Aquifer	24.57
DGC-15**	Monitoring - Upgradient of Grantham South	Columbia Aquifer	42.48
DGC-8D	Monitoring - Inert Area	UPA - Lower Sand	24.41
DGC-8S	Monitoring - Inert Area	UPA - Upper Sand	24.61
DGC-10D	Monitoring - Eastern AoA Boundary	UPA - Lower Sand	41.77
DGC-10S	Monitoring - Eastern AoA Boundary	UPA - Upper Sand	41.92
DGC-11D	Monitoring - Eastern AoA Boundary	UPA - Upper Sand	38.93
DGC-11S	Monitoring - Eastern AoA Boundary	UPA - Upper Sand	38.54
RT-1-UP	Monitoring	UPA - Upper Sand	39.16
UPA-01	Monitoring	UPA - Upper Sand	35.76
UPA-02D	Monitoring	UPA - Lower Sand	44.15
UPA-02S	Monitoring	UPA - Upper Sand	44.37
UPA-03D	Monitoring - Eastern AoA Boundary	UPA - Lower Sand	30.14
UPA-101-TZ	Monitoring - Well P-6 Area	UPCU - Transition Zone	46.13
UPA-101-US	Monitoring - Well P-6 Area	UPA - Upper Sand	46.23
Downgradient NCC Monitoring Locations			
BW-1	Monitoring	UPA - Lower Sand	30.32
BW-2	Monitoring	UPA - Lower Sand	33.68
BW-3	Monitoring	UPA - Lower Sand	7.80
MW-18	Monitoring	UPA - Upper Sand	6.97
MW-22N	Monitoring - ACL Western Lobe	UPA - Lower Sand	51.68
MW-26N	Monitoring	UPA - Upper and Lower Sand	36.76
MW-28	Former Extraction - ACL Eastern Lobe	UPA - Upper and Lower Sand	20.37
MW-29	Former Extraction - ACL Eastern Lobe	UPA - Upper and Lower Sand	17.38
MW-31	Former Extraction - ACL Eastern Lobe	UPA - Upper and Lower Sand	13.77
MW-34	Monitoring	UPA - Upper and Lower Sand	7.20
MW-38N	Monitoring	UPA - Upper and Lower Sand	35.55
MW-40	Monitoring	UPA - Lower Sand	36.32
MW-49N	Monitoring	UPA - Upper and Lower Sand	51.41
P-4_UPA	Monitoring - ACL Western Lobe	UPA - Upper Sand	47.89
P-5L	Monitoring	UPA - Lower Sand	14.90
P-5U	Monitoring	UPA - Upper Sand	15.30
P-6_UPA	Monitoring	UPA - Upper Sand	43.06
RW-2	Former Extraction - ACL Eastern Lobe	UPA - Upper Sand	6.98
RW-5	Former Extraction - ACL Eastern Lobe	UPA - Upper Sand	33.09
RW-6	Former Extraction - ACL Eastern Lobe	UPA - Upper Sand	15.01
RW-10	Former Extraction - ACL Western Lobe	UPA - Upper Sand	8.67
AWC Wells			
AWC-E1	Former Production - Upgradient of AWC	UPA - Upper and Lower Sand	40.50
AWC-E2	Former Production - Upgradient of AWC	UPA - Upper and Lower Sand	34.00
AWC-J1	Former AWC Production Well	UPA - Upper and Lower Sand	11.58
AWC-K1	Former AWC Production Well	UPA - Lower Sand	29.56
AWC-MW-1	Monitoring near production wells	UPA - Lower Sand	10.34
AWC-MW-2	Monitoring near production wells	UPA - Upper Sand	10.50
AWC-MW-3	Monitoring near production wells	UPA - Lower Sand	9.65
AWC-MW-4	Monitoring near production wells	UPA - Lower Sand	12.49
AWC-MW-5	Monitoring near production wells	UPA - Upper Sand	12.49
AWC-MW-6	Monitoring near production wells	UPA - Lower Sand	18.58
AWC-2	Production well	UPA - Lower Sand	64.58
AWC-6R	Production well	UPA - Upper and Lower Sand	TBD
AWC-7	Production well	UPA - Upper and Lower Sand	47.98
AWC-G3R	Production well	UPA - Upper and Lower Sand	19.70
AWC-ASR	aquifer storage and recovery well	UPA - Lower Sand	10.70

Notes
1) Survey data provided by Delaware Sand & Gravel Remedial Trust
2) Survey data updated based on 12/5/2012, 12/11/2012, and 12/18/2013 surveys by Taylor, Wiseman, Taylor, where available.
3) ** = Perched water table
4) See Attachment H for acronyms and abbreviations

Checked by: MBS
Reviewed by: TAM

TABLE A-6B
MONITORING POINT CONSTRUCTION AND SAMPLING METHOD INFORMATION
DELAWARE SAND & GRAVEL SUPERFUND SITE
NEW CASTLE COUNTY, DELAWARE

Monitoring Point ID	Constructed Use	Inside Diameter (in)	Construction Material	Sounded Depth (ft-btoc)	Screened Interval (ft-bgs)	Screen Length (ft)	Filter Pack Interval (ft-bgs)	Filter Pack Material	Backfill Interval[2] (ft-bgs)	Backfill Material	Screened Unit	Surface Completion	Filter Pack Length (ft)	Purging and Sampling Method	Proposed Sampling Depth (ft-btoc)	Secondary Method for Comparison	Rationale for Comparison	Sample Collection Purpose
DDA Low-Flow Extraction System Wells																		
B-4DR	Extraction - LFExS	4	PVC	41.31	31-41	10	29-41	#2 Sand	41-42	#2 Sand	Columbia Aquifer - Columbia Sand	Standpipe	13	no purge - direct draw	NA	no	extraction well	System monitoring
BG-1	Extraction - LFExS	10	Stainless Steel	43.4	22-42	20	22-45	#2 Morie Sand	45-46	#2 Morie Sand	Columbia Aquifer	Concrete vault	24	no purge - direct draw	NA	no	extraction well	System monitoring
C-18D	Extraction - LFExS	4	PVC	35.07	31-37	6	30-37	#2 Morie Sand	37-37.5	#2 Morie Sand	Columbia Aquifer	Standpipe	6.5	no purge - direct draw	NA	no	extraction well	System monitoring
C-19D	Extraction - LFExS	4	PVC	38.86	38-43	5	37-43	#2 Morie Sand	43-43.5	#2 Morie Sand	Columbia Aquifer	Standpipe	5.5	no purge - direct draw	NA	no	extraction well	System monitoring
C-20D	Extraction - LFExS	4	PVC	50.91	43-48	5	42-48	#2 Morie Sand	48-48.5	#2 Morie Sand	Columbia Aquifer	Standpipe	6.5	no purge - direct draw	NA	no	extraction well	System monitoring
C-2D	Extraction - LFExS	4	PVC	32.93	29-40	11	28-40	#2 Morie Sand	40-40.5	#2 Morie Sand	Columbia Aquifer - Columbia Sand and Basal Gravel	Standpipe	11.5	no purge - direct draw	NA	no	extraction well	System monitoring
C-30	Extraction - LFExS	2	PVC	38.77	27-37	10	25-37	#2 Sand	37-40	#2 Sand	Columbia Aquifer	Standpipe	15	no purge - direct draw	NA	no	extraction well	System monitoring
C-4D	Extraction - LFExS	4	PVC	41.62	34-42	8	33-42	#2 Morie Sand	42-42.5	#2 Morie Sand	Columbia Aquifer - Columbia Sand and Basal Gravel	Standpipe	9.5	no purge - direct draw	NA	no	extraction well	System monitoring
DDA Monitoring Wells within Containment Area																		
B-2D	Vertical Bio-venting System	4	PVC	43.29	36-46	10	35-46	#2 Morie Sand	46-46.5	#2 Morie Sand	Columbia Aquifer - Columbia Sand and Basal Gravel	Standpipe	11.5	submersible - low flow	41	no	NA	System monitoring
B-3D	Vertical Bio-venting System	4	PVC	45.11	38-45	7	37-45	#2 Morie Sand	45-45.5	#2 Morie Sand	Columbia Aquifer	Standpipe	8.5	submersible - low flow	41	no	NA	System monitoring
C-1D	Vertical Bio-venting System	4	PVC	37.9	28-38	10	NR	NR	NR	NR	Columbia Aquifer - Columbia Sand and Basal Gravel	Standpipe	>10	submersible - low flow	33	no	NA	System monitoring
C-22S	Vertical Bio-venting System	4	Stainless Steel	39.13	30-38	8	29-38	#2 Morie Sand	38-38.5	#2 Morie Sand	Columbia Aquifer	Standpipe	9.5	submersible - low flow	36	no	NA	System monitoring
C-3D	Vertical Bio-venting System	4	PVC	43.75	31-44	13	30-44	#2 Morie Sand	44-44.5	#2 Morie Sand	Columbia Aquifer	Standpipe	14.5	submersible - low flow	38	no	NA	System monitoring
MHW-1M	Monitoring	4	Stainless Steel	46.15	40-45	5	Not Specified	Not Specified	Not Specified	Not Specified	Base of Columbia Aquifer	Standpipe	>5	submersible - low flow	43	no	NA	System monitoring
MHW-1S	Monitoring	4	Stainless Steel	37.52	30.2-35.2	5	Not Specified	Not Specified	Not Specified	Not Specified	Columbia Aquifer	Standpipe	>5	submersible - low flow	33	no	NA	System monitoring
PZ-11-EXT	Hydraulic gradient monitoring	2	PVC	42.94	37-42	5	35-42	#0 Sand	42-44	#0 Sand	Columbia Aquifer	Standpipe	7	submersible - low flow	29	no	NA	System monitoring
PZ-5-EXT	Hydraulic gradient monitoring	2	PVC	32.88	27-30	3	25-30	#0 Well Gravel	30-31	Not Specified	Columbia Aquifer	Standpipe	6	submersible - low flow	40	no	NA	System monitoring
PZ-6S	Hydraulic gradient monitoring	2	PVC	36.56	26-29	3	24-29	#0 Well Gravel	29-30	Not Specified	Columbia Aquifer	Standpipe	6	3x - submersible	27	no	NA	System monitoring
DDA Monitoring Wells within Partition Area																		
P-4D	Monitoring - Partition	4	PVC	38.12	26.5-36.5	10	25.5-36.5	#2 Morie Sand	36.5-37	#2 Morie Sand	Columbia Aquifer - UPCU, Columbia Clay, Basal Gravel	Standpipe	11.5	submersible - low flow	31	no	NA	LT monitoring/trends
PZ-4-INT-R	Hydraulic gradient monitoring	2	PVC	32.42	29-34	5	26-34	#1 Morie Sand	34-35	#1 Morie Sand	Columbia Aquifer	Standpipe	9	submersible - low flow	32	no	NA	LT monitoring/trends
PZ-6N	Hydraulic gradient monitoring	2	PVC	40.1	30-33	3	28-33	#0 Well Gravel	33-43.5	Not Specified	Columbia Aquifer	Standpipe	15.5	3x - submersible	31	no	NA	LT monitoring/trends
DDA to PW-1(U) Monitoring Wells																		
DDA-01	Monitoring - Downgradient of DDA	2	PVC	95.25	84-94	10	82-94	#1 Sand	94-97	#1 Sand	UPA - Upper Sand	Standpipe	15	submersible - low flow	89	no	NA	LT monitoring/trends
DDA-02	Monitoring - Downgradient of DDA	2	PVC	95.25	84-94	10	82-94	#1 Sand	94-97	#1 Sand	UPA - Upper Sand	Standpipe	15	submersible - low flow	89	no	NA	LT monitoring/trends
DDA-03	Monitoring - Downgradient of DDA	2	PVC	92.52	80-90	10	78-90	#1 Sand	90-92	#1 Sand	UPA - Upper Sand	Standpipe	14	submersible - low flow	85	no	NA	LT monitoring/trends
DDA-05	Monitoring - Downgradient of DDA	2	PVC	66.33	54-64	10	52-64	#1 Sand	64-94	#1 Sand to 65/Bentonite 65-94	UPCU - Transition Zone	Standpipe	13	submersible - low flow	59	no	NA	LT monitoring/trends
DDA-06	Monitoring - Downgradient of DDA	2	PVC	57.12	46-56	10	44-56	#1 Sand	56-94	#1 Sand to 57/Bentonite 57-94	UPCU - Transition Zone	Standpipe	13	submersible - low flow	51	no	NA	LT monitoring/trends
DDA-07-TZ	Monitoring - Beneath DDA	2	PVC	51.92	44-49	5	42-49	#1 Sand	49-50	#1 Sand to 50	UPCU - Transition Zone	Standpipe	8	submersible - low flow	47	no	NA	LT monitoring/trends
DDA-07-US	Monitoring - Beneath DDA	2	PVC	74.69	63-73	10	61-73	#1 Sand	73-84	#1 Sand to 74/Bentonite 74-84	UPA - Upper Sand	Standpipe	13	submersible - low flow	68	no	NA	LT monitoring/trends
DDA-08-TZ	Monitoring - Beneath DDA	2	PVC	61.45	49-59	10	47-59	#1 Sand	59-60	#1 Sand to 60	UPCU - Transition Zone	Standpipe	13	submersible - low flow	54	no	NA	LT monitoring/trends
DDA-08-US	Monitoring - Beneath DDA	2	PVC	74.23	62-72	10	60-72	#1 Sand	72-87	#1 Sand to 73/Bentonite 73-87	UPA - Upper Sand	Standpipe	13	submersible - low flow	67	no	NA	LT monitoring/trends
DDA-09-TZ	Monitoring - Downgradient of DDA	2	PVC	67.36	55-65	10	53-65	#1 Sand	65-107	#1 Sand to 66/Bentonite 66-107	UPCU - Transition Zone	Standpipe	13	submersible - low flow	67	no	NA	LT monitoring/trends
DDA-10-US	Monitoring - Downgradient of DDA	2	PVC	54.49	42-52	10	40-52	#1 Sand	52-86	#1 Sand to 53/Bentonite 53-86	UPA - Upper Sand	Standpipe	13	submersible - low flow	47	no	NA	LT monitoring/trends
DDA-11-LS	Monitoring - Downgradient of DDA	2	PVC	117.51	105-115	10	103-115	#1 Sand	115-117	#1 Sand to 116/Bentonite 116-117	UPA - Lower Sand	Standpipe	13	submersible - low flow	110	no	NA	LT monitoring/trends
DDA-11-US	Monitoring - Downgradient of DDA	2	PVC	88.12	75-85	10	73-85	#1 Sand	85-87	#1 Sand to 86/Bentonite 86-87	UPA - Upper Sand	Standpipe	13	submersible - low flow	80	yes - 3x purge	variations in lithology	LT monitoring/trends
DDA-12-TZ	Monitoring - Downgradient of DDA	2	PVC	56.21	39-54	15	37-54	#1 Sand	54-58	#1 Sand to 55/Bentonite 55-58	UPCU - Transition Zone	Standpipe	18	submersible - low flow	47	no	NA	LT monitoring/trends
DDA-12-US	Monitoring - Downgradient of DDA	4	PVC	79.11	67-77	10	65-77	#1 Sand	77-127	#1 Sand to 78/Bentonite 78-127	UPA - Upper Sand	Standpipe	13	submersible - low flow	72	no	NA	LT monitoring/trends
DDA-13-TZ	Monitoring - Downgradient of DDA	2	PVC	60.32	48-58	10	46-58	#1 Sand	58-97	#1 Sand to 59/Bentonite 59-97	UPCU - Transition Zone	Standpipe	13	submersible - low flow	53	no	NA	LT monitoring/trends
DDA-14-TZ	Monitoring - Beneath DDA	2	PVC	61.67	49-59	10	47-59	#1 Sand	59-97	#1 Sand to 60/Bentonite 60-97	UPCU - Transition Zone	Standpipe	13	submersible - low flow	54	no	NA	LT monitoring/trends
DDA-15-TZ	Monitoring - Beneath DDA	2	PVC	66.26	54-64	10	52-64	#1 Sand	64-67	#1 Sand to 65/Bentonite 65-67	UPCU - Transition Zone	Standpipe	13	submersible - low flow	59	no	NA	LT monitoring/trends
DDA-15-US	Monitoring - Beneath DDA	2	PVC	97.94	85-95	10	83-95	#1 Sand	95-97	#1 Sand to 96/Bentonite 96-97	UPA - Upper Sand	Standpipe	13	submersible - low flow	90	no	NA	LT monitoring/trends
DDA-16-TZ	Monitoring - Downgradient of DDA	2	PVC	61.29	51-59	8	49-59	#1 Sand	59-83	#1 Sand to 60/Bentonite 60-83	UPCU - Transition Zone	Standpipe	11	submersible - low flow	56	no	NA	LT monitoring/trends
DDA-16-US	Monitoring - Downgradient of DDA	4	PVC	75.53	63-73	10	61-73	#1 Sand	73-77	#1 Sand to 74/Bentonite 74-77	UPA - Upper Sand	Standpipe	13	submersible - low flow	68	no	NA	LT monitoring/trends
DDA-17	Monitoring - Downgradient of DDA	4	PVC	80.1	67-77	10	65-77	#1 Sand	77-78	#1 Sand	UPA - Upper Sand	Standpipe	13	submersible - low flow	72	no	NA	LT monitoring/trends
DGC-2D	Monitoring - West of DDA	4	PVC	117.32	105-115	10	103-115	#1 Morie Sand	115-150	#1 Morie Sand to 117/Bentonite Seal 117-120/Sand Backfill 120-150	UPA - Lower Sand	Standpipe	14	submersible - low flow	110	no	NA	LT monitoring/trends
DGC-2S	Monitoring - West of DDA	4	PVC	72.66	50-70	20	48-72	#1 Morie Sand	72-78	#1 Morie Sand to 72/Bentonite Seal 72-74/Cellar Backfill 74-78	UPA - Upper Sand	Standpipe	26	submersible - low flow	60	yes - 3x purge	long screen and change in lithology, target capture zone	LT monitoring/trends
DGC-5	Monitoring - Northern DDA Boundary	4	PVC	56.5	35-55	20	33-55	#1 Morie Sand	55-82	#1 Morie Sand to 56/Sand fill and cuttings 56-82	UPCU - Transition Zone	Standpipe	49	submersible - low flow	45	yes - 3x purge	long screen and change in lithology, target capture zone	Upgradient
DGC-7C	Monitoring - Near Inert Area	4	PVC	35.06	23-33	10	21-33	#1 Morie Sand	33-35	#1 Sand to 33.5/Bentonite 33.5-35	Columbia Aquifer	Standpipe	12.5	3x - submersible	28	no	NA	LT monitoring/trends
DGC-7S	Monitoring - Near Inert Area	4	PVC	83.07	60-80	20	58-80	#1 Morie Sand	80-82	#1 Morie Sand	UPA - Upper Sand	Standpipe	24	submersible - low flow	70	no	NA	LT monitoring/trends
GA-101	Monitoring - Northern DDA Boundary	2	PVC	31.22	22-28	6	20-28	#1 Sand	28-44.5	#1 Sand to 29/Bentonite 29-44.5	Columbia Aquifer	Standpipe	9	submersible - low flow	26	no	NA	LT monitoring/trends
MHW-1D	Monitoring - Beneath DDA	4	Stainless Steel	74.86	65-75	10	Not Specified	Not Specified	Not Specified	Not Specified	UPA - Upper Sand	Standpipe	>10	submersible - low flow	70	no	NA	LT monitoring/trends
PW-1(U)	Extraction - PW-1(U)	8	Stainless Steel	79.37	68-93	15	60-93,100-118	#1 Sand	Not Specified	Not Specified	UPA - Upper Sand	Standpipe	33	no purge - direct draw	NA	no	NA	System monitoring

TABLE A-6B
MONITORING POINT CONSTRUCTION AND SAMPLING METHOD INFORMATION
DELAWARE SAND & GRAVEL SUPERFUND SITE
NEW CASTLE COUNTY, DELAWARE

Monitoring Point ID	Constructed Use	Inside Diameter (in)	Construction Material	Sounded Depth (ft-btoc)	Screened Interval (ft-bgs)	Screen Length (ft)	Filter Pack Interval (ft-bgs)	Filter Pack Material	Backfill Interval[2] (ft-bgs)	Backfill Material	Screened Unit	Surface Completion	Filter Pack Length (ft)	Purging and Sampling Method	Proposed Sampling Depth (ft-btoc)	Secondary Method for Comparison	Rationale for Comparison	Sample Collection Purpose
Downgradient DS&G Monitoring Locations																		
DGC-8C	Monitoring - Inert Area	4	PVC	34.4	19-29	10	17-29	#1 Morie Sand	29-31	#1 Morie Sand	Columbia Aquifer	Standpipe	14	submersible - low flow	30	no	NA	LT monitoring/trends
DGC-8D	Monitoring - Inert Area	4	PVC	122.71	108-118	10	106-118	#1 Morie Sand	118-132	#1 Morie Sand/Sand fill and cuttings	UPA - Lower Sand	Standpipe	26	submersible - low flow	117	no	NA	LT monitoring/trends
DGC-8S	Monitoring - Inert Area	4	PVC	85.72	60-80	20	58-80	#1 Morie Sand	80-82	#1 Morie Sand	UPA - Upper Sand	Standpipe	24	submersible - low flow	75	no	NA	LT monitoring/trends
DGC-10D	Monitoring - Eastern AoA Boundary	4	PVC	138.4	128 - 138	10	126-138	#1 Morie Sand	138-172	#1 Morie Sand/Sand fill and cuttings	UPA - Lower Sand	Standpipe	46	submersible - low flow	133	yes - 3x purge	long screen and change in lithology, target capture zone	Eastern extent monitoring/trends
DGC-10S	Monitoring - Eastern AoA Boundary	4	PVC	115.4	93 - 113	20	91-113	#1 Morie Sand	113-115	Morie #1 Sand	UPA - Upper Sand	Standpipe	24	submersible - low flow	103	yes - 3x purge	long screen and change in lithology, target capture zone	Eastern extent monitoring/trends
DGC-11D	Monitoring - Eastern AoA Boundary	4	PVC	115	105 - 115	10	103-115	#1 Morie Sand	115-182	#1 Morie Sand/Sand fill and cuttings	UPA - Upper Sand	Standpipe	79	submersible - low flow	110	yes - 3x purge	long screen and change in lithology, target capture zone	Eastern extent monitoring/trends
DGC-11S	Monitoring - Eastern AoA Boundary	4	PVC	79.9	70 - 80	10	68-80	#1 Morie Sand	80-82	Morie #1 Sand	UPA - Upper Sand	Standpipe	14	submersible - low flow	75	no	NA	Eastern extent monitoring/trends
RT-1-UP	Monitoring	4	PVC	101.72	91-101	10	90-101	Not Specified	101-105	Not Specified	UPA - Upper Sand	Standpipe	15	submersible - low flow	100	no	NA	LT monitoring/trends
UPA-01	Monitoring	2	PVC	98.98	90-100	10	88-100	#1 Sand	100-172	# 1 Sand to 102/Bentonite 102-172	UPA - Upper Sand	Standpipe	14	3x - submersible	95	no	NA	LT monitoring/trends
UPA-02D	Monitoring	2	PVC	163.24	151-161	10	149-161	#1 Sand	161-177	#1 Sand to 162/Bentonite 162-177	UPA - Lower Sand	Standpipe	13	submersible - low flow	156	no	NA	LT monitoring/trends
UPA-02S	Monitoring	2	PVC	107.44	97-107	10	95-107	#1 Sand	Not Applicable	Not Applicable	UPA - Upper Sand	Standpipe	12	submersible - low flow	102	no	NA	LT monitoring/trends
UPA-03D	Monitoring - Eastern AoA Boundary	2	PVC	168.05	155-165	10	153-165	#1 Sand	165-187	#1 Sand to 166/Bentonite 166-187	UPA - Lower Sand	Standpipe	13	submersible - low flow	160	no	NA	LT monitoring/trends
UPA-101-TZ	Monitoring - Well P-6 Area	2	PVC	78.34	73-78	5	71-78	#1 Sand	78-79	#1 Sand to 79	UPCU - Transition Zone	Standpipe	8	submersible - low flow	75	no	NA	LT monitoring/trends
UPA-101-US	Monitoring - Well P-6 Area	4	PVC	114.17	101-111	10	99-111	#1 Sand	111-122	#1 Sand to 112/Bentonite 112-122	UPA - Upper Sand	Standpipe	13	submersible - low flow	106	no	NA	LT monitoring/trends
Downgradient NCC Monitoring Locations																		
BW-1	Monitoring	4	PVC	130.6	106.5 - 126.5	20	101-126.5	#2 Morie Sand	Not Applicable	Not Applicable	UPA - Lower Sand	Standpipe	25.5	submersible - low flow	126	yes - 3x purge	long screen and target capture zone	LT monitoring/trends
BW-2	Monitoring	4	PVC	143.1	105 - 125	20	100-125	#2 Morie Sand	125-126	Not Specified	UPA - Lower Sand	Standpipe	≤26	submersible - low flow	133	yes - 3x purge	long screen and target capture zone	LT monitoring/trends
MW-18	Monitoring	1	PVC	90.5	80 - 90	10	NR	NR	90-145	NR	UPA - Upper Sand	Standpipe	≥10	low flow - peristaltic	85	no	NA	LT monitoring/trends
MW-26N	Monitoring	4	PVC	167.41	108 - 168	60	105-168	#2 Morie Sand	Not Applicable	Not Applicable	UPA - Upper and Lower Sand	Standpipe	63	submersible - low flow	138	yes - 3x purge	long screen and across UPA US and LS	LT monitoring/trends
MW-28	Former Extraction - ACL Eastern Lobe	6	Stainless Steel	111.6	43 - 120	77	NR	NR	120-130	NR	UPA - Upper and Lower Sand	Standpipe	≥77	submersible - low flow	90	yes - perform by ACL	NA	LT monitoring/trends
MW-29	Former Extraction - ACL Eastern Lobe	6	Stainless Steel	110.5	34 - 113	79	NR	NR	126-113	NR	UPA - Upper and Lower Sand	Standpipe	≥79	submersible - low flow	126	yes - perform by ACL	NA	LT monitoring/trends
MW-31	Former Extraction - ACL Eastern Lobe	6	Stainless Steel	112.1	57 - 105	46	NR	NR	105-116	NR	UPA - Upper and Lower Sand	Standpipe	≥57	submersible - low flow	133	yes - perform by ACL	NA	LT monitoring/trends
MW-34	Monitoring	4	PVC	130.45	75 - 131.5	56.5	NR	NR	131.5-140	NR	UPA - Upper and Lower Sand	Standpipe	≥65	submersible - low flow	100	yes - 3x purge	long screen and across UPA US and LS	LT monitoring/trends
P-5L	Monitoring	4	PVC	138	126 - 136	10	104-126	Sand	126-136	Sand	UPA - Lower Sand	Standpipe	≥15	submersible - low flow	131	no	NA	LT monitoring/trends
P-5U	Monitoring	4	PVC	82.8	70 - 80	10	65-90	Sand	80-90	Sand	UPA - Upper Sand	Standpipe	≥22	submersible - low flow	75	no	NA	LT monitoring/trends
P-6_UPA	Monitoring	2	PVC	110.5	100 - 110	10	87-120	Sand	110-120	Sand	UPA - Upper Sand	Flush mount	33	submersible - low flow	105	no	NA	LT monitoring/trends
AWC Wells																		
AWC-E1	Former Production - Upgradient of AWC	10	Stainless Steel	150.24	122-142, 149-162	40	Not Specified	#2 Gravel Pack	175-200	Not Specified	UPA - Upper and Lower Sand	Aboveground Concrete	≥40	submersible - low flow	132, 156	no	NA	Risk
AWC-E2	Former Production - Upgradient of AWC	12	Stainless Steel	167.48	131-173	42	Not Specified	#2 Gravel Pack	189-205	Not Specified	UPA - Upper and Lower Sand	Aboveground Concrete	≥42	submersible - low flow	140, 165	no	NA	Risk
AWC-K1	Former AWC Production Well	18	Concrete	164.54	135-173	38	NR	NR	173-220	NR	UPA - Lower Sand	Concrete vault	≥38	submersible - low flow	160	no	NA	Eastern extent monitoring/trends
AWC-6R	Production well	12	Stainless Steel	not available	105-145	40	100-145	#2 Gravel	Not Specified	Not Specified	UPA - Upper and Lower Sand	Standpipe	≥40	no purge - direct draw	NA	no	extraction well	Risk
AWC-7	Production well	12	Stainless Steel	136.02	115-175	60	Not Specified	Not Specified	175-180	Not Specified	UPA - Upper and Lower Sand	Standpipe	≥65	no purge - direct draw	NA	no	extraction well	Risk
AWC-G3R	Production well	12	Stainless Steel	not available	102-157	55	98-157	#2 Well Gravel	157-162	Not Specified	UPA - Upper and Lower Sand	Standpipe	≥64	no purge - direct draw	NA	no	extraction well	Risk

Notes:

1) Information compiled from water level monitoring purge forms between 2012 and 2018 and historic boring logs.
2) All material between the drilled depth of the borehole to the bottom of screen/casing is considered backfill to maintain consistency among boring logs.
3) No Record (NR) indicates Golder does not have a monitoring well installation log for this location. Limited information provided is from summary tables in historical reports by others.
4) Not specified indicates available boring log and/or monitoring well installation log do not include this information.
5) See Attachment H for acronyms and abbreviations

Checked by: MBS
Reviewed by: TAM

TABLE A-6C
SEMI-ANNUAL AND ANNUAL MONITORING PROGRAM
DELAWARE SAND & GRAVEL SUPERFUND SITE
NEW CASTLE COUNTY, DELAWARE

Sample ID	Well Type/Purpose	Screened Unit	Screen Interval (ft-bgs)	Sampling Depth (ft-bgs)	Purging and Sampling Method	April Event	October Event	Once Per Year		FSWP Revision 2 (October 2011) Sampling Location	ACL Semi-Annual Monitoring Program	ACL Additional Investigation Work Plan (not including to-be-installed ACL wells, upgradient wells or gas vents)
						Routine Groundwater Monitoring		Cations and Anions	PFAS Monitoring			
						VOCs+II-1,4-dioxane, SVOCs+II-BCEE, d-Fe/Mn, TAL Metals, Ammonia	VOCs+II-1,4-dioxane, SVOCs+II-BCEE, d-Fe/Mn					
DDA Low-Flow Extraction System Wells												
B-4DR	Extraction - LFExS	Columbia	31-41	NA	no purge - direct draw	x	x	-	x*	Yes	-	-
BG-1	Extraction - LFExS	Columbia	22-42	NA	no purge - direct draw	x	x	-	-	Yes	-	-
C-18D	Extraction - LFExS	Columbia	31-37	NA	no purge - direct draw	x	x	-	x*	Yes	-	-
C-19D	Extraction - LFExS	Columbia	38-43	NA	no purge - direct draw	x	x	-	-	Yes	-	-
C-20D	Extraction - LFExS	Columbia	43-48	NA	no purge - direct draw	x	x	-	-	Yes	-	-
C-2D	Extraction - LFExS	Columbia	29-40	NA	no purge - direct draw	x	x	-	-	Yes	-	-
C-30	Extraction - LFExS	Columbia	27-37	NA	no purge - direct draw	x	x	-	-	Yes	-	-
C-4D	Extraction - LFExS	Columbia	34-42	NA	no purge - direct draw	x	x	-	-	Yes	-	-
DDA Monitoring Wells within Containment Area												
B-2D	Monitoring near BG-1 and C-2D	Columbia	36-46	41	submersible - low lfow	x	-	-	-	Yes	-	-
B-3D	Monitoring near BG-1 and C-4D	Columbia	38-45	41	submersible - low lfow	x	-	-	x*	Yes	-	-
C-1D	Monitoring along Northern Boundary	Columbia	28-38	33	submersible - low lfow	x	-	-	-	Yes	-	-
C-22S	Monitoring above Columbia Clay	Columbia	30-38	36	submersible - low lfow	x	-	-	-	Yes	-	-
C-3D	Monitoring along Northern Boundary	Columbia	31-44	38	submersible - low lfow	x	-	-	-	Yes	-	-
MHW-1M	Monitoring near C-20D	Columbia	40-45	43	submersible - low lfow	x	-	-	x*	Yes	-	-
MHW-1S	Monitoring near C-20D	Columbia	30.2-35.2	33	submersible - low lfow	x	-	-	-	Yes	-	-
PZ-6S	Monitoring near C-30 and Partition	Columbia	26-29	27	3x - bailer	x	-	-	-	Yes	-	-
DDA Monitoring Wells within Partition Area												
P-4D	Monitoring - Partition	Columbia	26.5-36.5	31	submersible - low lfow	x	-	-	-	Yes	-	-
PZ-4-INT-R	Monitoring - Partition	Columbia	29-34	32	submersible - low lfow	x	-	-	-	Yes	-	-
PZ-6N	Monitoring - Partition	Columbia	30-33	31	3x - bailer	x	-	-	-	Yes	-	-
DDA to PW-1(U) Monitoring Wells												
GA-101	Monitoring - Northern DDA Boundary	Columbia	22-28	26	submersible - low lfow	x	x	-	x*	Yes	-	-
PZ-5-EXT	Monitoring - Northern DDA Boundary	Columbia	27-30	29	submersible - low lfow	x	x	-	-	-		
PZ-11-EXT	Monitoring - Northern DDA Boundary	Columbia	37-42	40	submersible - low lfow	x	x	-	x*	Yes	-	-
DGC-7C	Monitoring - Near Inert Area	Columbia	23-33	28	3x - bailer	x	x	-	-	Yes	-	-
DDA-05	Monitoring - Downgradient of DDA	UPCUTZ	54-64	59	submersible - low lfow	x	-	x	-	Yes	-	-
DDA-06	Monitoring - Downgradient of DDA	UPCUTZ	46-56	51	submersible - low lfow	x	-	x	x*	Yes	-	-
DDA-07-TZ	Monitoring - Beneath DDA	UPCUTZ	44-49	47	submersible - low lfow	x	-	-	x**	added in 2012	-	-
DDA-08-TZ	Monitoring - Beneath DDA	UPCUTZ	49-59	54	submersible - low lfow	x	-	-	x*	added in 2012	-	-
DDA-09-TZ	Monitoring - Downgradient of DDA	UPCUTZ	55-65	67	submersible - low lfow	x	-	-	-	added in 2012	-	-
DDA-12-TZ	Monitoring - Downgradient of DDA	UPCUTZ	39-54	47	submersible - low lfow	x	-	-	x*	added in 2012	-	-
DDA-13-TZ	Monitoring - Downgradient of DDA	UPCUTZ	48-58	53	submersible - low lfow	x	-	-	-	added in 2012	-	-
DDA-14-TZ	Monitoring - Beneath DDA	UPCUTZ	49-59	54	submersible - low lfow	x	-	-	-	added in 2012	-	-
DDA-15-TZ	Monitoring - Beneath DDA	UPCUTZ	54-64	59	submersible - low lfow	x	-	-	x*	added in 2012	-	-
DDA-16-TZ	Monitoring - Downgradient of DDA	UPCUTZ	51-59	56	submersible - low lfow	x	-	-	x*	added in 2012	-	-
DGC-5	Monitoring - Northern DDA Boundary	UPCUTZ	35-55	45	submersible - low lfow	x	x	x	x	Yes	-	-
DGC-7S	Monitoring - Near Inert Area	UPCUTZ	60-80	70	submersible - low lfow	x	-	x	-	Yes	-	-
DDA-01	Monitoring - Downgradient of DDA	UPA-Upper Sand	84-94	89	submersible - low lfow	x	-	x	-	Yes	-	-
DDA-02	Monitoring - Downgradient of DDA	UPA-Upper Sand	84-94	89	submersible - low lfow	x	x	x	x	Yes	-	-
DDA-03	Monitoring - Downgradient of DDA	UPA-Upper Sand	80-90	85	submersible - low lfow	x	-	x	x	Yes	-	-
DDA-04	Head monitoring for PW-1(U)	UPA-Upper Sand	80-90	85	submersible - low lfow	-	-	-	-	-	-	-
DDA-07-US	Monitoring - Beneath DDA	UPA-Upper Sand	63-73	68	submersible - low lfow	x	-	-	x**	added in 2012	-	-
DDA-08-US	Monitoring - Beneath DDA	UPA-Upper Sand	62-72	67	submersible - low lfow	x	-	-	-	added in 2012	-	-
DDA-10-US	Monitoring - Downgradient of DDA	UPA-Upper Sand	42-52	47	submersible - low lfow	x	x	x	x	added in 2012	-	-
DDA-11-US	Monitoring - Downgradient of DDA	UPA-Upper Sand	75-85	80	submersible - low lfow	x	-	-	-	added in 2012	-	-
DDA-12-US	Monitoring - Downgradient of DDA	UPA-Upper Sand	67-77	72	submersible - low lfow	x	x	x	x	added in 2012	-	-
DDA-15-US	Monitoring - Beneath DDA	UPA-Upper Sand	85-95	90	submersible - low lfow	x	-	-	-	added in 2012	-	-
DDA-16-US	Monitoring - Downgradient of DDA	UPA-Upper Sand	63-73	68	submersible - low lfow	x	-	-	x*	added in 2012	-	-
DDA-17	Monitoring - Downgradient of DDA	UPA-Upper Sand	67-77	72	submersible - low lfow	x	-	-	-	added in 2012	-	-
DGC-2S	Monitoring - West of DDA	UPA-Upper Sand	50-70	60	submersible - low lfow	x	-	x	x*	Yes	-	-
DGC-2D	Monitoring - West of DDA	UPA-Lower Sand	105-115	110	submersible - low lfow	x	-	-	-	-	-	-
MHW-1D	Monitoring - Beneath DDA	UPA-Upper Sand	65-75	70	submersible - low lfow	x	x	x	-	Yes	-	-
PW-1(U)	Extraction - PW-1(U)	UPA-Upper Sand	68-93	NA	no purge - direct draw	x	x	x	x	Yes	-	-
DDA-11-LS	Monitoring - Downgradient of DDA	UPA-Lower Sand	105-115	110	submersible - low lfow	x	-	-	x**	added in 2012	-	-
MW-45	Monitoring	UPA-US and LS	110-145	-	submersible - low lfow	-	-	-	-	-	-	-

TABLE A-6C
SEMI-ANNUAL AND ANNUAL MONITORING PROGRAM
DELAWARE SAND & GRAVEL SUPERFUND SITE
NEW CASTLE COUNTY, DELAWARE

Sample ID	Well Type/Purpose	Screened Unit	Screen Interval (ft-bgs)	Sampling Depth (ft-bgs)	Purging and Sampling Method	April Event	October Event	Once Per Year		FSWP Revision 2 (October 2011) Sampling Location	ACL Semi-Annual Monitoring Program	ACL Additional Investigation Work Plan (not including to-be-installed ACL wells, upgradient wells or gas vents)
						Routine Groundwater Monitoring		Cations and Anions	PFAS Monitoring			
						VOCs+II-1,4-dioxane, SVOCs+II-BCEE, d-Fe/Mn, TAL Metals, Ammonia	VOCs+II-1,4-dioxane, SVOCs+II-BCEE, d-Fe/Mn					
NCC Sewer Discharge Monitoring Points												
PW-1(U) Discharge	Extraction - PW-1(U)	UPA-Upper Sand	NA	NA	no purge - direct draw	x	x	-	-	-	-	-
TTO	LFExS Combined Discharge	Columbia	NA	NA	no purge - direct draw	x	x	-	-	-	-	-
PDIWP Proposed Wells												
DDA-05-TZ-EXTR	UPCUTZ - Future Extraction Well	UPCUTZ	Proposed Well - Depths to be determined			x	x	-	-	-	-	-
DDA-06-TZ-EXTR	UPCUTZ - Future Extraction Well	UPCUTZ	Proposed Well - Depths to be determined			x	x	-	-	-	-	-
DDA-18-TZ	Monitoring - West of Well PW-1(U)	UPCUTZ	Proposed Well - Depths to be determined			x	x	x	-	-	-	-
DDA-18-US	Monitoring - West of Well PW-1(U)	UPA-Upper Sand	Proposed Well - Depths to be determined			x	x	x	-	-	-	-
DDA-19-TZ	Monitoring - East of Well PW-1(U)	UPCUTZ	Proposed Well - Depths to be determined			x	x	x	-	-	-	-
DDA-19-US	Monitoring - East of Well PW-1(U)	UPA-Upper Sand	Proposed Well - Depths to be determined			x	x	x	-	-	-	-
DDA-20-TZ	Monitoring - Northeast of Well PW-1(U)	UPCUTZ	Proposed Well - Depths to be determined			x	x	x	-	-	-	-
DDA-20-US	Monitoring - Northeast of Well PW-1(U)	UPA-Upper Sand	Proposed Well - Depths to be determined			x	x	x	-	-	-	-
UPA-101-LS	Monitoring - Well P-6 Area	UPA-Lower Sand	Proposed Well - Depths to be determined			x	x	x	-	-	-	-
CA-102	Monitoring - Inert Area	Columbia	Proposed Well - Depths to be determined			x	x	x	-	-	-	-
UPA-102-TZ	Monitoring - Well P-6 Area	UPCUTZ	Proposed Well - Depths to be determined			x	x	x	-	-	-	-
UPA-102-US	Monitoring - Well P-6 Area	UPA-Upper Sand	Proposed Well - Depths to be determined			x	x	x	-	-	-	-
CA-103	Monitoring - Inert Area	Columbia	Proposed Well - Depths to be determined			x	x	x	-	-	-	-
UPA-103-TZ	Monitoring - Well P-6 Area	UPCUTZ	Proposed Well - Depths to be determined			x	x	x	-	-	-	-
UPA-103-US	Monitoring - Well P-6 Area	UPA-Upper Sand	Proposed Well - Depths to be determined			x	x	x	x	-	-	-
UPA-103-LS	Monitoring - Well P-6 Area	UPA-Lower Sand	Proposed Well - Depths to be determined			x	x	x	-	-	-	-
UPA-104-TZ	Monitoring - Well P-6 Area	UPCUTZ	Proposed Well - Depths to be determined			x	x	x	-	-	-	-
UPA-104-US	Monitoring - Well P-6 Area	UPA-Upper Sand	Proposed Well - Depths to be determined			x	x	x	-	-	-	-
UPA-104-LS	Monitoring - Well P-6 Area	UPA-Lower Sand	Proposed Well - Depths to be determined			x	x	x	-	-	-	-
UPA-105A-TZ	Monitoring - Well UPA-101 Area	UPCUTZ	Proposed Well - Depths to be determined			x	x	x	-	-	-	-
UPA-105A-US	Monitoring - Well UPA-101 Area	UPA-Upper Sand	Proposed Well - Depths to be determined			x	x	x	x	-	-	-
UPA-105A-LS	Monitoring - Well UPA-101 Area	UPA-Lower Sand	Proposed Well - Depths to be determined			x	x	x	x	-	-	-
UPA-105B-TZ	Monitoring - Well UPA-101 Area	UPCUTZ	Proposed Well - Depths to be determined			x	x	x	-	-	-	-
UPA-105B-US	Monitoring - Well UPA-101 Area	UPA-Upper Sand	Proposed Well - Depths to be determined			x	x	x	-	-	-	-
UPA-105B-LS	Monitoring - Well UPA-101 Area	UPA-Lower Sand	Proposed Well - Depths to be determined			x	x	x	-	-	-	-
CA-106	Monitoring - Grantham South	Columbia	Proposed Well - Depths to be determined			x	x	x	-	-	-	-
UPA-106-TZ	Monitoring - Wells MW-18/MW-34 Area	UPCUTZ	Proposed Well - Depths to be determined			x	x	x	-	-	-	-
UPA-106-US	Monitoring - Wells MW-18/MW-34 Area	UPA-Upper Sand	Proposed Well - Depths to be determined			x	x	x	-	-	-	-
UPA-106-LS	Monitoring - Wells MW-18/MW-34 Area	UPA-Lower Sand	Proposed Well - Depths to be determined			x	x	x	-	-	-	-
UPA-107-TZ	Monitoring - Wells MW-18/MW-34 Area	UPCUTZ	Proposed Well - Depths to be determined			x	x	x	-	-	-	-
UPA-107-US	Monitoring - Wells MW-18/MW-34 Area	UPA-Upper Sand	Proposed Well - Depths to be determined			x	x	x	-	-	-	-
UPA-107-LS	Monitoring - Wells MW-18/MW-34 Area	UPA-Lower Sand	Proposed Well - Depths to be determined			x	x	x	-	-	-	-
UPA-108-TZ	Monitoring - Well BW-2 Area	UPCUTZ	Proposed Well - Depths to be determined			x	x	x	-	-	-	-
UPA-108-US	Monitoring - Well BW-2 Area	UPA-Upper Sand	Proposed Well - Depths to be determined			x	x	x	x	-	-	-
UPA-108-LS	Monitoring - Well BW-2 Area	UPA-Lower Sand	Proposed Well - Depths to be determined			x	x	x	x	-	-	-
Downgradient DS&G Monitoring Locations												
DGC-8C	Monitoring - Inert Area	Columbia	19-29	30	submersible - low lfow	x	-	-	-	-	-	-
DGC-15	Columbia Head Monitoring	Columbia	19-29	-	submersible - low lfow	-	-	-	-	-	-	-
AWC-E1	Former Production - Upgradient of AWC	UPA-Upper Sand	122-162	132	submersible - low lfow	-	-	x	x	Yes	-	-
AWC-E1	Former Production - Upgradient of AWC	UPA-Lower Sand	122-162	156	submersible - low lfow	-	-	x	x	-	-	-
AWC-E2	Former Production - Upgradient of AWC	UPA-Upper Sand	131-173	140	submersible - low lfow	-	-	x	x	Yes	-	-
AWC-E2	Former Production - Upgradient of AWC	UPA-Lower Sand	131-173	165	submersible - low lfow	-	-	x	x	-	-	-
DGC-10D	Monitoring - Eastern AoA Boundary	UPA-Lower Sand	128-138	133	submersible - low lfow	x	x	x	x	Yes	-	-
DGC-10S	Monitoring - Eastern AoA Boundary	UPA-Upper Sand	93-113	103	submersible - low lfow	x	x	x	x	Yes	-	-
DGC-11D	Monitoring - Eastern AoA Boundary	UPA-Upper Sand	105-115	110	submersible - low lfow	x	x	x	-	Yes	-	-
DGC-11S	Monitoring - Eastern AoA Boundary	UPA-Upper Sand	70-80	75	submersible - low lfow	x	x	x	-	Yes	-	-
DGC-8D	Monitoring - Inert Area	UPA-Lower Sand	108-118	117	submersible - low lfow	x	-	x	-	-	-	-
DGC-8S	Monitoring - Inert Area	UPA-Upper Sand	60-80	75	submersible - low lfow	x	-	x	-	-	-	-
RT-1-UP	Monitoring	UPA-Upper Sand	91-101	100	submersible - low lfow	x	x	x	x	Yes	-	-
UPA-01	Monitoring	UPA-Upper Sand	90-100	95	submersible - low lfow	x	x	x	x	Yes	-	-
UPA-02D	Monitoring	UPA-Lower Sand	151-161	156	submersible - low lfow	x	x	x	x	Yes	-	-
UPA-02S	Monitoring	UPA-Upper Sand	97-107	102	submersible - low lfow	x	-	x	x	Yes	-	-
UPA-03D	Monitoring - Eastern AoA Boundary	UPA-Lower Sand	155-165	160	submersible - low lfow	x	x	x	x	Yes	-	-
UPA-101-TZ	Monitoring - Well P-6 Area	UPCUTZ	73-78	75	submersible - low lfow	x	-	-	-	added in 2013	-	-
UPA-101-US	Monitoring - Well P-6 Area	UPA-Upper Sand	101-111	106	submersible - low lfow	x	-	-	x	added in 2013	-	-

TABLE A-6C
SEMI-ANNUAL AND ANNUAL MONITORING PROGRAM
DELAWARE SAND & GRAVEL SUPERFUND SITE
NEW CASTLE COUNTY, DELAWARE

Sample ID	Well Type/Purpose	Screened Unit	Screen Interval (ft-bgs)	Sampling Depth (ft-bgs)	Purging and Sampling Method	April Event	October Event	Once Per Year		FSWP Revision 2 (October 2011) Sampling Location	ACL Semi-Annual Monitoring Program	ACL Additional Investigation Work Plan (not including to-be-installed ACL wells, upgradient wells or gas vents)
						Routine Groundwater Monitoring		Cations and Anions	PFAS Monitoring			
						VOCs+II-1,4-dioxane, SVOCs+II-BCEE, d-Fe/Mn, TAL Metals, Ammonia	VOCs+II-1,4-dioxane, SVOCs+II-BCEE, d-Fe/Mn					
Downgradient NCC Monitoring Locations												
MW-18	Monitoring	UPA-Upper Sand	80 - 90	85	peristaltic	x	x	x	x	Yes	Annual (Oct)	-
MW-22N	Monitoring - ACL Western Lobe	UPA-Lower Sand	139 - 159	149	submersible - low lfow	-	-	-	-	-	Semi-Annual (Apr&Oct)	PFAS, Western Lobe
MW-22NU	Proposed Monitoring - ACL Western Lobe	UPA-Upper Sand	Proposed Well - Depths to be determined			-	-	-		-	-	PFAS, Western Lobe
MW-26N	Monitoring	UPA-US and LS	108 - 168	138	submersible - low lfow	x	x	x	x	Yes	Semi-Annual (Apr&Oct)	-
MW-28	Former Extraction - ACL Eastern Lobe	UPA-US and LS	40 - 120	50	submersible - low lfow	x	-	-	-	-	Annual (Oct)	PFAS
MW-29	Former Extraction - ACL Eastern Lobe	UPA-US and LS	34 - 113	39	submersible - low lfow	x	-	-	-	-	Annual (Oct)	PFAS
MW-31	Former Extraction - ACL Eastern Lobe	UPA-US and LS	59 - 105	75	submersible - low lfow	x	-	-	-	-	Annual (Oct)	PFAS
MW-34	Monitoring	UPA-US and LS	75-131.5	100	submersible - low lfow	x	x	x	x	Yes	-	-
MW-38N	Monitoring	UPA-US and LS	72 - 132	102	submersible - low lfow	-	-	-	-	-	-	-
MW-40	Monitoring	UPA-Lower Sand	110 - 140	125	submersible - low lfow	-	-	-	-	-	Annual (Oct)	PFAS
MW-49N	Monitoring	UPA-US and LS	72 - 132	135	submersible - low lfow	-	-	-	-	-	Semi-Annual (Apr&Oct)	-
MW-54	Monitoring	UPA-Upper Sand	40 - 50	TBD - no log	submersible - low lfow	-	-	-		-	-	PFAS
MW-56	Monitoring	UPA-Upper Sand	75 - 100	85	submersible - low lfow	-	-	-		-	-	PFAS
MW-58	Monitoring	UPA-Upper Sand	65 - 110	75 and 95	submersible - low lfow	-	-	-		-	-	PFAS
P-6	Monitoring	UPA-Upper Sand	100 - 110	105	submersible - low lfow	x	x	x	-	Yes	Semi-Annual (Apr&Oct)	-
BW-1	Monitoring	UPA-Lower Sand	106.5 - 126.5	126	submersible - low lfow	x	-	-	-	-	Annual (Oct)	PFAS
BW-2	Monitoring	UPA-Lower Sand	105 - 125	133	submersible - low lfow	x	-	-	-	-	Semi-Annual (Apr&Oct)	PFAS
BW-3	Monitoring	UPA-US and LS	50 - 135	92	submersible - low lfow	-	-	-	-	-	Annual (Oct)	PFAS
P-4	Monitoring - ACL Western Lobe	UPA-Upper Sand	115 - 125	120	submersible - low lfow	-	-	-	-	-	Annual (Oct)	PFAS, Western Lobe
P-4L	Proposed Monitoring - ACL Western Lobe	UPA-Lower Sand	Proposed Well - Depths to be determined			-	-	-		-	-	PFAS, Western Lobe
WL-1U	Proposed Monitoring - ACL Western Lobe	UPA-Upper Sand	Proposed Well - Depths to be determined			-	-	-		-	-	PFAS, Western Lobe
WL-1L	Proposed Monitoring - ACL Western Lobe	UPA-Lower Sand	Proposed Well - Depths to be determined			-	-	-		-	-	PFAS, Western Lobe
WL-2U	Proposed Monitoring - ACL Western Lobe	UPA-Upper Sand	Proposed Well - Depths to be determined			-	-	-		-	-	PFAS, Western Lobe
WL-2L	Proposed Monitoring - ACL Western Lobe	UPA-Lower Sand	Proposed Well - Depths to be determined			-	-	-		-	-	PFAS, Western Lobe
P-5L	Monitoring	UPA-Lower Sand	70 - 80	131	submersible - low lfow	x	x	x	-	-	-	-
P-5U	Monitoring	UPA-Upper Sand	126 - 136	75	submersible - low lfow	x	-	-	-	-	-	-
RW-10	Former Extraction - ACL Western Lobe	UPA-Upper Sand	77 - 102	90	submersible - low lfow	-	-	-	-	-	-	PFAS, Western Lobe
AWC Wells - only extraction wells which are pumping at the time of the event can be sampled												
AWC-2	Production Well	UPA-Lower Sand	122-160	NA	no purge - direct draw	-	-	-	by AWC qrtrly	-	-	Cations/anions once
AWC-6R	Production Well	UPA-US and LS	100-140	NA	no purge - direct draw	x	x	-	by AWC qrtrly	-	-	Cations/anions once
AWC-7	Production Well	UPA-US and LS	115-175	NA	no purge - direct draw	x	x	-	by AWC qrtrly	Yes	-	Cations/anions once
AWC-G3R	Production - Southern AoA Boundary	UPA-US and LS	102-157	NA	no purge - direct draw	x	x	-	by AWC qrtrly	Yes	-	Cations/anions once
AWC-K1	Monitoring - Eastern AoA Boundary	UPA-Lower Sand	135-173	160	submersible - low lfow	x	x	-	-	Yes	-	-

Notes:
1) "x" indicates location will be sampled for indicated parameter(s)
2) "-" indicates location will not be sampled for indicated parameters and/or location was not included as a FSWP Revision 2 sample location
3) List of cations and anions for analysis includes: calcium, magnesium, potassium, sodium, ammonia, nitrate, nitrite, sulfate, sulfide, chloride and bicarbonate.
4) * indicates EPA requested PFAS sampling location
5) ** indicates proposed additional PFAS sampling location based on EPA's April 26, 2018 email and Trust's August 2018 response
6) Frequency of "once per year" = annually; however, which semi-annual event (April or October) will depend on well installation date and ACL coordination
7) April monitoring event represents a is site-wide event and October monitoring event is limited to information needed for design
8) A synoptic round of water levels will be collected prior to sampling during each monitoring event.
9) AWC agreed to let the Golder sample AWC wells as part of semi-annual monitoring events beginning in October 2018.
10) Trip blanks will accompany each shipment of VOC samples (1 per day).
11) The following quality assurance/quality control (QA/QC) samples will be collected during each monitoring event at a rate of 1 per 20 primary samples: field duplicates, field equipment rinsate blanks, matrix spikes and matrix spike duplicates.
12) See Attachment H for acronyms and abbreviations
13) The LFExS discharge is monitored on a semi-annual basis in accordance with the New Castle County Wastewater Discharge Permit requirements. The samples are analyzed for Total Toxic Organics (TTO) VOCs, TTO SVOCs, TTO pesticides, polychlorinated biphenyls (PCBs), biological oxygen demand (BOD), Inductively Coupled Plasma Mass Spectroscopy (ICP MS) metals (arsenic, cadmium, chromium, copper, lead, molybdenum, nickel, selenium, and zinc), mercury (cold vapor atomic absorption; CVAA), ammonia, total suspended solids (TSS), total cyanide and pH.
14) The PW-1(U) system discharge is monitored on a semi-annual basis in accordance with the New Castle County Wastewater Discharge Permit requirements. The samples are analyzed for VOCs, SVOCs, BOD, ICP MS metals (arsenic, cadmium, chromium, copper, lead, molybdenum, nickel, selenium, and zinc), mercury (CVAA), ammonia, TSS, cyanide and

Checked by: MBS
Reviewed by: TAM

TABLE A-6D
MONTHLY, BI-MONTHLY AND QUARTERLY MONITORING PROGRAM
DELAWARE SAND & GRAVEL SUPERFUND SITE
NEW CASTLE COUNTY, DELAWARE

Sample ID	Well Type/Purpose	Screened Unit	Screen Interval (ft-bgs)	Sampling Depth (ft-bgs)	Monthly Events	Bi-monthly Events	Quarterly Events	
					Total Iron and Manganese	Total and Dissolved Iron, Manganese and Cobalt	Cobalt	PFAS
Downgradient DS&G Monitoring Locations								
AWC-E1	Former Production - Upgradient of AWC	UPA-Upper Sand	122-162	132	-	x	-	-
AWC-E1	Former Production - Upgradient of AWC	UPA-Lower Sand	122-162	156	-	x	-	-
AWC-E2	Former Production - Upgradient of AWC	UPA-Upper Sand	131-173	140	-	x	-	-
AWC-E2	Former Production - Upgradient of AWC	UPA-Lower Sand	131-173	165	-	x	-	-
UPA-03D	Monitoring - Eastern AoA Boundary	UPA-Lower Sand	155-165	160	-	x	-	-
MW-26N	Monitoring	UPA-US and LS	108-168	138	-	x	-	-
AWC Wells - only extraction wells which are pumping at the time of the event can be sampled								
AWC-2	Production Well	UPA-Lower Sand	122-160	NA	by AWC	-	by AWC	by AWC
AWC-6R	Production Well	UPA-US and LS	100-140	NA	by AWC	-	by AWC	by AWC
AWC-7	Production Well	UPA-US and LS	115-175	NA	by AWC	-	by AWC	by AWC
AWC-G3R	Production - Southern AoA Boundary	UPA-US and LS	102-157	NA	by AWC	-	by AWC	by AWC

Notes:
1) "x" indicates location will be sampled for indicated parameter(s)
2) "-" indicates location will not be sampled for indicated parameters and/or location was not included as a FSWP Revision 2 sample location
3) The following quality assurance/quality control (QA/QC) samples will be collected during each monitoring event at a rate of 1 per 20 primary samples: field duplicates, field equipment rinsate blanks, matrix spikes and matrix spike duplicates.
4) See Attachment H for acronyms and abbreviations

Prepared by: TAM
Checked by: BAR

TABLE A-7
MONITORING LOCATIONS FOR ELFExS DESIGN
DELAWARE SAND & GRAVEL SUPERFUND SITE
NEW CASTLE COUNTY, DELAWARE

Monitoring Point ID	Sampling Frequency	Well Type/Purpose	Hydrogeologic Unit
DDA Low-Flow Extraction System Wells			
B-4DR	Semi-Annual	Extraction - LFExS	Columbia Aquifer - Columbia Sand
BG-1	Semi-Annual	Extraction - LFExS	Columbia Aquifer
C-18D	Semi-Annual	Extraction - LFExS	Columbia Aquifer
C-19D	Semi-Annual	Extraction - LFExS	Columbia Aquifer
C-20D	Semi-Annual	Extraction - LFExS	Columbia Aquifer
C-2D	Semi-Annual	Extraction - LFExS	Columbia Aquifer - Columbia Sand and Basal Gravel
C-30	Semi-Annual	Extraction - LFExS	Columbia Aquifer
C-4D	Semi-Annual	Extraction - LFExS	Columbia Aquifer - Columbia Sand and Basal Gravel
DDA Monitoring Wells within Containment Area			
B-1D	Once prior to ELFExS design	Monitoring - Containment Area	Columbia Aquifer
B-2D	Reduce from Semi-Annual to Annual	Monitoring near BG-1 and C-2D	Columbia Aquifer - Columbia Sand and Basal Gravel
B-3D	Reduce from Semi-Annual to Annual	Monitoring near BG-1 and C-4D	Columbia Aquifer
C-10 (3)	Once prior to ELFExS design	Monitoring - Containment Area	Columbia Aquifer
C-11 (3)	Once prior to ELFExS design	Monitoring - Containment Area	Columbia Aquifer
C-12 (4)	Once prior to ELFExS design	Monitoring - Containment Area	Columbia Aquifer
C-13	Once prior to ELFExS design	Monitoring - Containment Area	Columbia Aquifer
C-14	Once prior to ELFExS design	Monitoring - Containment Area	Columbia Aquifer
C-15D	Once prior to ELFExS design	Monitoring - Containment Area	Columbia Aquifer
C-16	Once prior to ELFExS design	Monitoring - Containment Area	Columbia Aquifer
C-17	Once prior to ELFExS design	Monitoring - Containment Area	Columbia Aquifer
C-18S (3)	Once prior to ELFExS design	Monitoring - Containment Area	Columbia Aquifer
C-19S	Once prior to ELFExS design	Monitoring - Containment Area	Columbia Aquifer
C-1D	Reduce from Semi-Annual to Annual	Monitoring along Northern Boundary	Columbia Aquifer - Columbia Sand and Basal Gravel
C-20S	Once prior to ELFExS design	Monitoring - Containment Area	Columbia Aquifer
C-21D	Once prior to ELFExS design	Monitoring - Containment Area	Columbia Aquifer
C-22D	Once prior to ELFExS design	Monitoring - Containment Area	Columbia Aquifer
C-22S	Once prior to ELFExS design	Monitoring - Containment Area	Columbia Aquifer
C-23	Once prior to ELFExS design	Monitoring - Containment Area	Columbia Aquifer
C-24	Once prior to ELFExS design	Monitoring - Containment Area	Columbia Aquifer
C-25	Once prior to ELFExS design	Monitoring - Containment Area	Columbia Aquifer
C-27	Once prior to ELFExS design	Monitoring - Containment Area	Columbia Aquifer
C-28	Once prior to ELFExS design	Monitoring - Containment Area	Columbia Aquifer
C-29	Once prior to ELFExS design	Monitoring - Containment Area	Columbia Aquifer
C-2S (3)	Once prior to ELFExS design	Monitoring - Containment Area	Columbia Aquifer
C-3D	Reduce from Semi-Annual to Annual	Monitoring along Northern Boundary	Columbia Aquifer
C-4S	Once prior to ELFExS design	Monitoring - Containment Area	Columbia Aquifer
C-5D	Once prior to ELFExS design	Monitoring - Containment Area	Columbia Aquifer
C-6 (3)	Once prior to ELFExS design	Monitoring - Containment Area	Columbia Aquifer
C-7	Once prior to ELFExS design	Monitoring - Containment Area	Columbia Aquifer
C-8 (3)	Once prior to ELFExS design	Monitoring - Containment Area	Columbia Aquifer
C-9	Once prior to ELFExS design	Monitoring - Containment Area	Columbia Aquifer
MHW-1M	Reduce from Semi-Annual to Annual	Monitoring near C-20D	Base of Columbia Aquifer
MHW-1S	Reduce from Semi-Annual to Annual	Monitoring near C-20D	Columbia Aquifer
PZ-6S	Reduce from Semi-Annual to Annual	Monitoring near C-30 and Partition	Columbia Aquifer
DDA Monitoring Wells within Partition Area			
P-1D	Once prior to ELFExS design	Monitoring - Partition Area	Columbia Aquifer
P-2D	Once prior to ELFExS design	Monitoring - Partition Area	Columbia Aquifer
P-3	Once prior to ELFExS design	Monitoring - Partition Area	Columbia Aquifer
P-4D	Reduce from Semi-Annual to Annual	Monitoring - Partition Area	Columbia Aquifer - UPCU, Columbia Clay, Basal Gravel
P-5	Once prior to ELFExS design	Monitoring - Partition Area	Columbia Aquifer
P-6 (3)	Once prior to ELFExS design	Monitoring - Partition Area	Columbia Aquifer
P-7	Once prior to ELFExS design	Monitoring - Partition Area	Columbia Aquifer
P-8D	Once prior to ELFExS design	Monitoring - Partition Area	Columbia Aquifer
PZ-4-INT-R	Reduce from Semi-Annual to Annual	Hydraulic gradient monitoring	Columbia Aquifer
PZ-6N	Reduce from Semi-Annual to Annual	Hydraulic gradient monitoring	Columbia Aquifer

Notes:
1) See Attachment H for acronyms and abbreviations

Prepared by: TAM
Checked by: BAR

TABLE A-8
PUMPING TEST LOCATIONS AND PURPOSES
DELAWARE SAND & GRAVEL SUPERFUND SITE
NEW CASTLE COUNTY, DELAWARE

Pumping Test Locations	Location Description	Primary Assessment Purposes	Secondary Assessment Purposes	Pump Test Description and Duration	Proposed Transducer Locations (see note 1)		Additional Manual Water Level Measurements
Step 1: PW-1(U) Shutdown (from 30 gpm)	South of DDA along northern boundary of Inert Area	Pumping influence (hydraulics) on UPCUTZ and UPA between well PW-1(U) and the DDA	-	Shutdown of existing PW-1(U) extraction well, LFExS remains in operation, transducers record aquifer recovery over a 3-day (72-hour) period and establish a 'baseline' without well PW-1(U) operating. Confirm that AWC's Llangollen wellfield will be in typical operation during this period.	New wells: DDA-18-TZ DDA-18–US DDA-19-US DDA-20-TZ DDA-20-US	Existing wells: DDA-04(US) DDA-09-TZ DDA-02(US) DDA-10-US DDA-12-TZ DDA-16-US	DDA-13-TZ DGC-7S DDA-11-US DDA-11-LS DDA-12-US DDA-03(US)
Step 2: PW-1(U) Restart (at 15 gpm)	South of DDA along northern boundary of Inert Area	Evaluation of target capture zone and modeled extraction rate to develop design flow rate for continued operation of existing well PW-1(U)	-	Restart of existing extraction well PW-1(U) at lower extraction rate of 15 gpm. Transducers record aquifer response in the area of existing well PW-1(U). Pumping test performed for a 24-hour period. LFExS remains in operation, confirm that AWC's Llangollen wellfield will be in 'typical' operation during this period.	Same as Step 1 unless testing during Step 2 indicates transducers and/or additional water level measurements needed in other areas		
Step 3: DDA-10/12-US-EXTR	Southeast of DDA and northeast of well PW-1	Evaluation of target capture zone and modeled extraction rate to develop design flow rate for extraction system/well to the east of existing well PW-1(U)	Evaluation of: 1 - Migration between hydrostratigraphic units (vertical gradients) 2 - Mass transfer from UPCUTZ to UPA (chemistry)	Transducers record aquifer response in the area of DDA-10-US and DDA-12-US to extraction from DDA-10/12-US-EXTR at approximately 10 gpm. Step test performed to assess well efficiency and determine pumping test rate, followed by a 72-hour pumping test to evaluate sustainable extraction rate and potential for mass transfer from the UPCUTZ to UPA upper sand. Existing PW-1(U) extraction well is operating at 15 gpm, LFExS remains in operation, confirm that AWC's Llangollen wellfield will be in 'typical' operation during this period.	New wells: DDA-18-TZ DDA-18–US DDA-19-US DDA-20-TZ DDA-20-US	Existing wells: DDA-03(US) DDA-10-US DDA-12-TZ DDA-12-US DDA-13-TZ DDA-17(US) PW-1(U)	DDA-09-TZ DDA-06(TZ) DDA-15-TZ DDA-15-US
Step 4: DDA-05-TZ-EXTR	South of well DDA-05(TZ) within line of proposed UPCUTZ low-flow extraction system	Evaluation of target capture zones in DDA-05(TZ) area and potential extraction rates for proposed UPCUTZ low-flow extraction system	Future UPCUTZ low-flow extraction system well	Transducers record aquifer response in the UPCUTZ to extraction from DDA-05-TZ-EXTR. Step test performed to assess well efficiency and determine pumping test rate, followed by an up to 48-hour pumping test to evaluate sustainable extraction rate. Existing PW-1(U) extraction well is operating at 15 gpm, LFExS remains in operation, confirm that AWC's Llangollen wellfield will be in 'typical' operation during this period.	New wells: DDA-18-TZ	Existing wells: DDA-05(TZ) DDA-06(TZ) DDA-07-TZ DDA-15-TZ DDA-16-TZ DDA-16-US DDA-14-TZ DDA-02(US) DDA-13-TZ PW-1(U)	P-4D PZ-6S PZ-6S PZ-4-INTR
Step 5: DDA-06-TZ-EXTR	South of well DDA-06(TZ) within line of proposed UPCUTZ low-flow extraction system	Evaluation of target capture zones in DDA-06(TZ) area and potential extraction rates for proposed UPCUTZ low-flow extraction system	Future UPCUTZ low-flow extraction system well	Transducers record aquifer response in the UPCUTZ to extraction from DDA-05-TZ-EXTR. Step test performed to assess well efficiency and determine pumping test rate, followed by an up to 48-hour pumping test to evaluate sustainable extraction rate. Existing PW-1(U) extraction well is operating at 15 gpm, LFExS remains in operation, confirm that AWC's Llangollen wellfield will be in 'typical' operation during this period.	New wells: DDA-20-TZ	Existing wells: DDA-06(TZ) DDA-05(TZ) DDA-17(US) DDA-08-TZ DDA-15-TZ DDA-14-TZ DDA-12-TZ DDA-13-TZ DDA-10-US DDA-03(US) PW-1(U)	DDA-15-US MHW-1D DDA-08-US DDA-12-US
Step 6: UPA-01-US-EXTR	South of Inert Area, along Grantham Lane, near well UPA-01	Evaluation of target capture zone and modeled extraction rate to develop design flow rate for extraction system/well to the east of Wells UPA-01/RT-1-UP Area	Evaluation of potential for connection between UPA upper sand and UPA lower sand	Transducers record aquifer response in the area of existing well UPA-01 to extraction at up to 60 gpm from to be installed extraction well in the area of proposed well UPA-105A or UPA-105B (extraction well to be named UPA-01-US-EXTR). Step test performed to assess well efficiency and determine pumping test rate, followed by a 72-hour pumping test to evaluate sustainable extraction rate. Existing PW-1(U) extraction well is operating at 15 gpm, LFExS remains in operation, confirm that AWC's Llangollen wellfield will be in 'typical' operation during this period.	New Wells: DDA-19-US DDA-20-US UPA-105A-US UPA-105A-LS UPA-105B-US UPA-105B-LS UPA-102-US UPA-104-US	Existing Wells: UPA-01 PW-1(U) DDA-10-US DDA-12-TZ RT-1-UP P-6	Due to the distance between the extraction well area and monitoring wells that will not have transducers deployed, additional water levels measurements are not proposed at this time.
Step 7: MW-18/34-US-EXTR	South of Grantham South, likely along edge of NCC Reforestation Area, downgradient of wells MW-18 and MW-34	Evaluation of target capture zone and modeled extraction rate to develop design flow rate for extraction system/well to the southeast of Wells MW-18/MW-34 Area	Evaluation of potential for connection between UPA upper sand and UPA lower sand	Transducers record aquifer response in the area of existing wells MW-18 and MW-34 to extraction at up to 60 gpm from to be installed extraction well in the area of proposed well UPA-107-US (extraction well to be named MW18/34-US-EXTR). Step test performed to assess well efficiency and determine pumping test rate, followed by a 72-hour pumping test to evaluate sustainable extraction rate. Existing PW-1(U) extraction well is operating at 15 gpm, LFExS remains in operation, confirm that AWC's Llangollen wellfield will be in 'typical' operation during this period.	New Wells: UPA-107-US UPA-107-LS UPA-104-US UPA-104-LS CA-106 UPA-106-US UPA-106-LS UPA-103-US UPA-102-US UPA-102-LS UPA-108-US	Existing Wells: MW-18 MW-34 P-6 UPA-01 MW-29 MW-31	P-5U P-5L Due to the remote location of wells MW-18 and MW-34, additional water levels measurements are not proposed at this time.
Step 8: P-6 Area	East of Grantham South	Evaluation of target capture zone and modeled extraction rate to develop design flow rate for extraction system/well in the Well P-6 Area	Evaluation of: 1 - Modeled feature - location and orientation 2 - Potential for connection between hydrostratigraphic units (vertical gradients; UPCUTZ, UPA upper sand) 3 - Mass transfer between UPCUTZ and UPA upper sand (chemistry)	Transducers record aquifer response in the area of existing wells P-6, UPA-101-TZ and UPA-101-US to extraction from existing well P-6. Step test performed to assess well efficiency and determine pumping test rate, followed by a 72-hour pumping test to evaluate sustainable extraction rate and potential for mass transfer from the UPCUTZ to UPA upper sand. Existing PW-1(U) extraction well is operating at 15 gpm, LFExS remains in operation, confirm that AWC's Llangollen wellfield will be in 'typical' operation during this period.	New Wells: UPA-107-US UPA-107-LS UPA-104-US UPA-104-LS UPA-106-US UPA-106-LS CA-103 UPA-103-US UPA-102-US UPA-102-LS UPA-108-US	Existing Wells: MW-18 MW-34 UPA-01 RT-1-UP DGC-8S RW-6 MW-29 MW-31 UPA-101-TZ UPA-101-US	CA-102 Due to the remote location of well P-6 and the various uncertainties associated with groundwater flow to and from this well, many transducers will be deployed in this area. Additional water levels measurements are not proposed at this time.

Notes:
1) Transducer locations subject to change based on results of drilling and VAP sampling in the investigation areas.
2) See Attachment H for acronyms and abbreviations

Checked by: TAM
Reviewed by: WEG

TABLE A-9
VOC ANALYSIS - GROUNDWATER MEASUREMENT PERFORMANCE CRITERIA
DELAWARE SAND & GRAVEL SUPERFUND SITE
NEW CASTLE COUNTY, DELAWARE

Matrix	Groundwater
Analytical Group	VOCs + 10 TICs
Analytical Method	SW-846 8260C / SIM
Analytical Organization	TestAmerica

QC Sample:	Frequency/ Number	Method/SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator	Measurement Performance Criteria
Method Blank	1 per extraction batch	No results above QL.	If sufficient sample volume is available, reanalyze the samples.	Laboratory Analyst	Accuracy	No results above QL.
Method Blank	1 per extraction batch	None.	Qualify data as required.	Data Validator	Accuracy	No results above QL.
Rinsate Blank	1 rinsate blank per 20 samples, whenever field decontaminated equipment is used.	None.	Qualify data as required.	Data Validator	Accuracy	No results above QL.
Trip Blank	1 trip blank each day VOC samples are collected.	None.	Qualify data as required.	Data Validator	Accuracy	No results above QL.
Field Duplicates	1 per 20 samples	None.	Qualify data as required.	Data Validator	Precision	<40% RPD
Surrogate Spike	Every sample	Meets method criteria.	If sufficient sample volume is available, reanalyze the samples.	Laboratory Analyst	Accuracy	Surrogate recovery meets QC limits as specified in the method.
Surrogate Spike	Every sample	Meets method criteria.	Qualify data as required.	Data Validator	Accuracy	Surrogate recovery meets QC limits as specified in the method.
MS	1 per 20 samples	See Table A-17, when sample concentration is <4x the spike added.	When the recovery is outside of control limits and the sample result is < 4x the spike added, a post-digestion spike must be performed. An aliquot of the unspiked sample will be spiked at 2x the indigenous level or 2x the CRQL, whichever is greater.	Data Validator	Accuracy	See Table A-17, when sample concentration is <4x the spike added.
MSD	1 per 20 samples	See Table A-17, when sample concentration is <4x the spike added.	When the recovery is outside of control limits and the sample result is < 4x the spike added, a post-digestion spike must be performed. An aliquot of the unspiked sample will be spiked at 2x the indigenous level or 2x the CRQL, whichever is greater.	Data Validator	Accuracy	See Table A-17, when sample concentration is <4x the spike added.
MS/MSD %R	1 per 20 samples	See Table A-17	Qualify data as required.	Data Validator	Precision	See Table A-17

Notes:
1) See Attachment H for acronyms and abbreviations

Checked by: AMZ
Reviewed by: TAM

TABLE A-10
SVOC ANALYSIS - GROUNDWATER MEASUREMENT PERFORMANCE CRITERIA
DELAWARE SAND & GRAVEL SUPERFUND SITE
NEW CASTLE COUNTY, DELAWARE

Matrix	Groundwater and Surface Water
Analytical Group	SVOCs + 20 TICs
Analytical Method	SW-846 8270D / SIM
Analytical Organization	TestAmerica

QC Sample:	Frequency/ Number	Method/SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator	Measurement Performance Criteria
Method Blank	1 per extraction batch	No results above QL.	If sufficient sample volume is available, reanalyze the samples.	Laboratory Analyst	Accuracy	No results above QL.
Method Blank	1 per extraction batch	None.	Qualify data as required.	Data Validator	Accuracy	No results above QL.
Rinsate Blank	1 rinsate blank per 20 samples, whenever field decontaminated equipment is used.	None.	Qualify data as required.	Data Validator	Accuracy	No results above QL.
Field Duplicates	1 per 20 samples	None.	Qualify data as required.	Data Validator	Precision	<40% RPD
Surrogate Spike	Every sample	Meets method criteria.	If sufficient sample volume is available, reanalyze the samples.	Laboratory Analyst	Accuracy	Surrogate recovery meets QC limits as specified in the method.
Surrogate Spike	Every sample	Meets method criteria.	Qualify data as required.	Data Validator	Accuracy	Surrogate recovery meets QC limits as specified in the method.
MS	1 per 20 samples	See Table A-17, when sample concentration is <4x the spike added.	When the recovery is outside of control limits and the sample result is < 4x the spike added, a post-digestion spike must be performed. An aliquot of the unspiked sample will be spiked at 2x the indigenous level or 2x the CRQL, whichever is greater.	Data Validator	Accuracy	See Table A-17, when sample concentration is <4x the spike added.
MSD	1 per 20 samples	See Table A-17, when sample concentration is <4x the spike added.	When the recovery is outside of control limits and the sample result is < 4x the spike added, a post-digestion spike must be performed. An aliquot of the unspiked sample will be spiked at 2x the indigenous level or 2x the CRQL, whichever is greater.	Data Validator	Accuracy	See Table A-17, when sample concentration is <4x the spike added.
MS/MSD %R	1 per 20 samples	See Table A-17, when sample concentration is <4x the spike	Qualify data as required.	Data Validator	Precision	See Table A-17, when sample concentration is <4x the spike added.

Notes:
1) See Attachment H for acronyms and abbreviations

Checked by: AMZ
Reviewed by: TAM

TABLE A-11
INORGANICS ANALYSIS - GROUNDWATER MEASUREMENT PERFORMANCE CRITERIA
DELAWARE SAND & GRAVEL SUPERFUND SITE
NEW CASTLE COUNTY, DELAWARE

Matrix	Groundwater
Analytical Group	Inorganics
Analytical Method	SW-846 6010D / 6020A / 7470A
Analytical Organization	TestAmerica

QC Sample:	Frequency/ Number	Method/SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator	Measurement Performance Criteria
Rinsate Blank	1 rinsate blank per 20 samples, whenever field decontaminated equipment is used.	No results above IDL.	Qualify data as required.	Data Validator	Accuracy	No results above IDL.
ICP Interference Check Sample	1 at the beginning and end of each sample analysis run, or a minimum of twice per 8-hour shift, whichever is more frequent.	±20% of true value	Analysis terminated and affected samples reanalyzed.	Laboratory Analyst	Accuracy	±20% of true value
Initial Calibration Blank	Method specific	No results above IDL.	Qualify data as required.	Data Validator	Accuracy	No results above IDL.
Continuing Calibration Blank	1 per 10 samples	No results above IDL.	Reanalyze sample bracketed by compliant continuing calibration blank.	Data Validator	Accuracy	No results above IDL.
Preparation Blank	1 per extraction batch	No results above IDL.	Qualify data as required.	Data Validator	Accuracy	No results above IDL.
Field Duplicates	1 per 20 samples	none	Qualify data as required.	Data Validator	Precision	<40% RPD
MS	1 per 20 samples.	75-125% of true value, when sample concentration is <4x the spike added.	When the recovery is outside of control limits and the sample result is < 4x the spike added, a post-digestion spike must be performed. An aliquot of the unspiked sample will be spiked at 2x the indigenous level or 2x the CRQL, whichever is greater.	Data Validator	Accuracy	75-125% of true value, when sample concentration is <4x the spike added.
Post-Digestion spike	If warranted following MS analysis	75-125% of true value	Qualify data as required.	Data Validator	Accuracy	Minimum level of 10 times and a maximum of 100 times the lower limit of quantitation.
LCS	1 per 20 samples.	80-120% of true value.	Qualify data as required.	Data Validator	Accuracy	80-120% of true value.
Lab Duplicate	1 per 20 samples.	±20% of true value.	± CRDL when the sample value is < 5x CRDL, including when only one of the duplicate sample values is < 5x CRDL.	Data Validator	Accuracy	±20% of true value.
Initial Calibration Verification	Method specific	90-110% of true value for all analytes except mercury (90-120%) and cyanide (85-115%).	Qualify data as required.	Data Validator	Accuracy	90-110% of true value for all analytes except mercury (90-120%) and cyanide (85-115%).
Continuing Calibration Verification	1 per 10 samples	90-110% of true value for all analytes except mercury (90-120%) and cyanide (85-115%).	Reanalyze sample bracketed by compliant continuing calibration verification.	Data Validator	Accuracy	90-110% of true value for all analytes except mercury (90-120%) and cyanide (85-115%).
ICP Serial Dilution	When warranted.	When analyte concentration is minimally a factor of 50 above the IDL, an analysis of a 5-fold dilution must agree within 10% difference of the original results.	Qualify data as required.	Data Validator	Precision	When analyte concentration is minimally a factor of 50 above the IDL, an analysis of a 5-fold dilution must agree within 10% difference of the original results.

Notes:
1) See Attachment H for acronyms and abbreviations

Checked by: AMZ
Reviewed by: TAM

TABLE A-12
MAJOR CATIONS AND ANIONS - GROUNDWATER MEASUREMENT PERFORMANCE CRITERIA
DELAWARE SAND & GRAVEL SUPERFUND SITE
NEW CASTLE COUNTY, DELAWARE

Matrix	Groundwater
Analytical Group	Major Cations and Anions
Analytical Method	See Table A-15
Analytical Organization	TestAmerica

QC Sample:	Frequency/ Number	Method/SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator	Measurement Performance Criteria
Method Blank	1 per extraction batch	No results above QL.	If sufficient sample volume is available, reanalyze the samples.	Laboratory Analyst	Accuracy	No results above QL.
Method Blank	1 per extraction batch	None.	Qualify data as required.	Data Validator	Accuracy	No results above QL.
Continuing Calibration Blank	1 per 10 samples	No results above QL.	Reanalyze sample bracketed by compliant Continuing Calibration Blank.	Laboratory Analyst	Accuracy	No results above QL.
Continuing Calibration Verification	1 per 10 samples	90-110% of true value	Reanalyze sample bracketed by compliant Continuing Calibration Verification.	Laboratory Analyst	Accuracy	90-110% of true value
Field Duplicates	1 per 20 samples	None.	Qualify data as required.	Data Validator	Precision	<40% RPD
LCS	1 per extraction batch	Meets laboratory QC limits in SOPs, Attachment E-1.	Analysis terminated and affected samples reanalyzed, if additional sample volume is available.	Laboratory Analyst	Accuracy	Meets laboratory QC limits in SOPs, Attachment E-1.
LCS	1 per extraction batch	Meets laboratory QC limits in SOPs, Attachment E-1.	Qualify data as required.	Data Validator	Accuracy	Meets laboratory QC limits in SOPs, Attachment E-1.
Matrix Spike	1 per 20 samples	Meets laboratory QC limits in SOPs, Attachment E-1.	If the LCS meets acceptance criteria, no corrective action is required. Otherwise, if sufficient sample volume is available, re-extract and reanalyze the samples.	Laboratory Analyst	Accuracy	Meets laboratory QC limits in SOPs, Attachment E-1.
Matrix Spike	1 per 20 samples	Meets laboratory QC limits in SOPs, Attachment E-1.	Qualify data as required.	Data Validator	Accuracy	Meets laboratory QC limits in SOPs, Attachment E-1.

Notes:
1) See Attachment H for acronyms and abbreviations

Checked by: AMZ
Reviewed by: TAM

TABLE A-13
PFAS - GROUNDWATER MEASUREMENT PERFORMANCE CRITERIA
DELAWARE SAND & GRAVEL SUPERFUND SITE
NEW CASTLE COUNTY, DELAWARE

Matrix	Groundwater
Analytical Group	Per- and Polyfluoroalkyl Substances (PFAS)
Analytical Method	USEPA 537 Rev. 1.1, Modified
Analytical Organization	Eurofins

QC Sample:	Frequency/ Number	Method/SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator	Measurement Performance Criteria
Equipment/Rinsate Blank	1 equipment/rinsate blank per event for each set of non-dedicated, decontaminated field equipment used.	None.	Qualify data as required.	Data Validator	Accuracy	No results above QL.
Trip Blank	1 trip blank each day PFAS samples are collected.	None.	Qualify data as required.	Data Validator	Accuracy	No results above QL.
Field Blank	1 field blank each day PFAS samples are collected.	None.	Qualify data as required.	Data Validator	Accuracy	No results above QL.
Method Blank	1 per extraction batch	No results above QL.	If sufficient sample volume is available, reanalyze the samples.	Laboratory Analyst	Accuracy	No results above QL.
Method Blank	1 per extraction batch	None.	Qualify data as required.	Data Validator	Accuracy	No results above QL.
Continuing Calibration Verification	1 per 10 samples	70-130% of true value	Reanalyze sample bracketed by compliant Continuing Calibration Verification.	Laboratory Analyst	Accuracy	70-130% of true value
Field Duplicates	1 per 20 samples	None.	Qualify data as required.	Data Validator	Precision	<40% RPD
Surrogate Spike	Every sample	Meets method criteria.	If sufficient sample volume is available, reanalyze the samples.	Laboratory Analyst	Accuracy	Surrogate recovery meets QC limits as specified in the method.
Surrogate Spike	Every sample	Meets method criteria.	Qualify data as required.	Data Validator	Accuracy	Surrogate recovery meets QC limits as specified in the method.
LCS	1 per extraction batch	Meets laboratory QC limits in SOPs, Attachment F-1.	Analysis terminated and affected samples reanalyzed, if additional sample volume is available.	Laboratory Analyst	Accuracy	Meets laboratory QC limits in SOPs, Attachment F-1.
LCS	1 per extraction batch	Meets laboratory QC limits in SOPs, Attachment F-1.	Qualify data as required.	Data Validator	Accuracy	Meets laboratory QC limits in SOPs, Attachment F-1.
Matrix Spike	1 per 20 samples	Meets laboratory QC limits in SOPs, Attachment F-1.	If the LCS meets acceptance criteria, no corrective action is required. Otherwise, if sufficient sample volume is available, re-extract and reanalyze the samples.	Laboratory Analyst	Accuracy	Meets laboratory QC limits in SOPs, Attachment F-1.
Matrix Spike	1 per 20 samples	Meets laboratory QC limits in SOPs, Attachment F-1.	Qualify data as required.	Data Validator	Accuracy	Meets laboratory QC limits in SOPs, Attachment F-1.
MS/MSD %R	1 per 20 samples	See Table A-17	Qualify data as required.	Data Validator	Precision	See Table A-17

Notes:
1) See Attachment H for acronyms and abbreviations

Checked by: AMZ
Reviewed by: TAM

TABLE A-14
REFERENCE LIMITS - GROUNDWATER
DELAWARE SAND & GRAVEL SUPERFUND SITE
NEW CASTLE COUNTY, DELAWARE

Analytical Group	CAS	Analyte	Units	Achievable Laboratory Limits	
				MDLs	QLs
Cations/Anions	7664-41-7	Ammonia as N	mg/l	0.045	0.1
Cations/Anions	ALKB-C	Bicarbonate	mg/l	5	5
Cations/Anions	16887-00-6	Chloride	mg/l	0.078	0.12
Cations/Anions	Fe2+	Ferrous Iron	ug/l	120	400
Cations/Anions	14797-55-8-N	Nitrate as N	mg/l	0.0157	0.1
Cations/Anions	14797-65-0-N	Nitrite as N	mg/l	0.0191	0.12
Cations/Anions	14808-79-8	Sulfate as S04	mg/l	0.332	0.6
Cations/Anions	18496-25-8	Sulfide, total	mg/l	0.579	1
Inorganics	7429-90-5	Aluminum	ug/l	18.8	40
Inorganics	7440-36-0	Antimony	ug/l	0.399	2
Inorganics	7440-38-2	Arsenic	ug/l	0.734	2
Inorganics	7440-39-3	Barium	ug/l	1.16	4
Inorganics	7440-41-7	Beryllium	ug/l	0.245	0.8
Inorganics	7440-43-9	Cadmium	ug/l	0.808	2
Inorganics	7440-70-2	Calcium	ug/l	98.8	200
Inorganics	7440-47-3	Chromium	ug/l	2.3	4
Inorganics	7440-48-4	Cobalt	ug/l	1.6	4
Inorganics	7440-50-8	Copper	ug/l	1.99	4
Inorganics	7439-89-6	Iron	ug/l	51.1	120
Inorganics	7439-89-6	Iron - dissolved	ug/l	34.2	150
Inorganics	7439-92-1	Lead	ug/l	0.552	1.2
Inorganics	7439-95-4	Magnesium	ug/l	73.7	200
Inorganics	7439-96-5	Manganese	ug/l	2.88	8
Inorganics	7439-96-5	Manganese - dissolved	ug/l	0.987	15
Inorganics	7439-97-6	Mercury	ug/l	0.115	0.2
Inorganics	7440-02-0	Nickel	ug/l	2.36	4
Inorganics	7440-09-7	Potassium	ug/l	86.7	200
Inorganics	7782-49-2	Selenium	ug/l	5.35	10
Inorganics	7440-22-4	Silver	ug/l	0.591	2
Inorganics	7440-23-5	Sodium	ug/l	128	200
Inorganics	7440-28-0	Thallium	ug/l	0.157	0.8
Inorganics	7440-62-2	Vanadium	ug/l	1.11	4
Inorganics	7440-66-6	Zinc	ug/l	11.1	16
PFAS	2991-50-6	N-ethyl perfluorooctanesulfonamidoacetic acid (NEtFOSAA)	ng/l	1	3
PFAS	2355-31-9	N-methyl perfluorooctanesulfonamidoacetic acid (NMeFOSAA)	ng/l	1	3
PFAS	1763-23-1	Perfluorooctane sulfonate (PFOS)	ng/l	2	6
PFAS	375-73-5	Perfluorobutane sulfonate (PFBS)	ng/l	0.8	3
PFAS	335-76-2	Perfluorodecanoic acid (PFDA)	ng/l	0.5	2
PFAS	307-55-1	Perfluorododecanoic acid (PFDoA)	ng/l	0.5	2
PFAS	375-85-9	Perfluoroheptanoic acid (PFHpA)	ng/l	0.5	2
PFAS	355-46-4	Perfluorohexane sulfonate (PFHxS)	ng/l	1	3
PFAS	307-24-4	Perfluorohexanoic acid (PFHxA)	ng/l	0.6	2
PFAS	375-95-1	Perfluorononanoic acid (PFNA)	ng/l	0.6	2
PFAS	335-67-1	Perfluorooctanoic acid (PFOA)	ng/l	0.6	2
PFAS	376-06-7	Perfluorotetradecanoic acid (PFTA)	ng/l	0.5	2
PFAS	72629-94-8	Perfluorotridecanoic acid (PFTrDA)	ng/l	0.5	2
PFAS	2058-94-8	Perfluoroundecanoic acid (PFUnA)	ng/l	1	3
SVOCs	120-82-1	1,2,4-Trichlorobenzene	ug/l	1.26	2
SVOCs	95-50-1	1,2-Dichlorobenzene	ug/l	1.34	10
SVOCs	541-73-1	1,3-Dichlorobenzene	ug/l	2.03	10
SVOCs	106-46-7	1,4-Dichlorobenzene	ug/l	1.27	10
SVOCs	95-95-4	2,4,5-Trichlorophenol	ug/l	0.279	10
SVOCs	88-06-2	2,4,6-Trichlorophenol	ug/l	0.299	10
SVOCs	120-83-2	2,4-Dichlorophenol	ug/l	0.421	10
SVOCs	105-67-9	2,4-Dimethylphenol	ug/l	0.238	10
SVOCs	51-28-5	2,4-Dinitrophenol	ug/l	14.4	20
SVOCs	121-14-2	2,4-Dinitrotoluene	ug/l	0.997	2
SVOCs	606-20-2	2,6-Dinitrotoluene	ug/l	0.39	2
SVOCs	91-58-7	2-Chloronaphthalene	ug/l	1.18	10
SVOCs	95-57-8	2-Chlorophenol	ug/l	0.377	10

TABLE A-14
REFERENCE LIMITS - GROUNDWATER
DELAWARE SAND & GRAVEL SUPERFUND SITE
NEW CASTLE COUNTY, DELAWARE

Analytical Group	CAS	Analyte	Units	Achievable Laboratory Limits	
				MDLs	QLs
SVOCs	91-57-6	2-Methylnaphthalene	ug/l	1.1	10
SVOCs	95-48-7	2-Methylphenol	ug/l	0.259	10
SVOCs	88-74-4	2-Nitroaniline	ug/l	0.474	10
SVOCs	88-75-5	2-Nitrophenol	ug/l	0.747	10
SVOCs	91-94-1	3,3'-Dichlorobenzidine	ug/l	1.43	10
SVOCs	99-09-2	3-Nitroaniline	ug/l	0.962	10
SVOCs	534-52-1	4,6-Dinitro-2-Methylphenol	ug/l	13.3	20
SVOCs	101-55-3	4-Bromophenyl-Phenylether	ug/l	0.745	10
SVOCs	59-50-7	4-Chloro-3-Methylphenol	ug/l	0.575	10
SVOCs	106-47-8	4-Chloroaniline	ug/l	1.88	10
SVOCs	7005-72-3	4-Chlorophenyl-Phenylether	ug/l	1.28	10
SVOCs	106-44-5	4-Methylphenol	ug/l	0.235	10
SVOCs	100-01-6	4-Nitroaniline	ug/l	0.543	10
SVOCs	100-02-7	4-Nitrophenol	ug/l	0.689	20
SVOCs	83-32-9	Acenaphthene	ug/l	1.08	10
SVOCs	208-96-8	Acenaphthylene	ug/l	0.823	10
SVOCs	120-12-7	Anthracene	ug/l	0.634	10
SVOCs	56-55-3	Benzo(A)Anthracene	ug/l	0.592	1
SVOCs	50-32-8	Benzo(A)Pyrene	ug/l	0.405	1
SVOCs	205-99-2	Benzo(B)Fluoranthene	ug/l	1.14	2
SVOCs	191-24-2	Benzo(G,H,I)Perylene	ug/l	1.43	10
SVOCs	207-08-9	Benzo(K)Fluoranthene	ug/l	0.674	1
SVOCs	111-91-1	Bis(2-Chloroethoxy)Methane	ug/l	0.239	10
SVOCs	111-44-4	Bis(2-Chloroethyl)Ether	ug/l	0.295	1
SVOCs	117-81-7	Bis(2-Ethylhexyl)Phthalate	ug/l	1.7	2
SVOCs	80-05-7	Bisphenol A	ug/l	9.9	10
SVOCs	85-68-7	Butylbenzylphthalate	ug/l	0.854	10
SVOCs	105-60-2	Caprolactam	ug/l	0.684	10
SVOCs	86-74-8	Carbazole	ug/l	0.679	10
SVOCs	218-01-9	Chrysene	ug/l	0.907	2
SVOCs	53-70-3	Dibenzo(A,H)Anthracene	ug/l	0.72	1
SVOCs	132-64-9	Dibenzofuran	ug/l	1.1	10
SVOCs	84-66-2	Diethylphthalate	ug/l	0.976	10
SVOCs	131-11-3	Dimethylphthalate	ug/l	0.766	10
SVOCs	84-74-2	Di-N-Butylphthalate	ug/l	0.84	10
SVOCs	117-84-0	Di-N-Octylphthalate	ug/l	4.75	10
SVOCs	101-84-8	Diphenyl Ether	ug/l	1.21	10
SVOCs	206-44-0	Fluoranthene	ug/l	0.842	10
SVOCs	86-73-7	Fluorene	ug/l	0.912	10
SVOCs	118-74-1	Hexachlorobenzene	ug/l	0.396	1
SVOCs	87-68-3	Hexachlorobutadiene	ug/l	0.78	1
SVOCs	77-47-4	Hexachlorocyclopentadiene	ug/l	1.72	10
SVOCs	67-72-1	Hexachloroethane	ug/l	1.19	2
SVOCs	193-39-5	Indeno(1,2,3-Cd)Pyrene	ug/l	1.29	2
SVOCs	78-59-1	Isophorone	ug/l	0.798	10
SVOCs	121-69-7	N,N-Dimethylaniline	ug/l	0.909	1
SVOCs	91-20-3	Naphthalene	ug/l	1.13	10
SVOCs	98-95-3	Nitrobenzene	ug/l	0.567	1
SVOCs	100-61-8	N-Methylaniline	ug/l	0.48	5
SVOCs	621-64-7	N-Nitroso-Di-N-Propylamine	ug/l	0.43	1
SVOCs	86-30-6	N-Nitrosodiphenylamine	ug/l	0.891	10
SVOCs	87-86-5	Pentachlorophenol	ug/l	1.45	20
SVOCs	85-01-8	Phenanthrene	ug/l	0.58	10
SVOCs	108-95-2	Phenol	ug/l	0.56	5
SVOCs	129-00-0	Pyrene	ug/l	1.64	10

TABLE A-14
REFERENCE LIMITS - GROUNDWATER
DELAWARE SAND & GRAVEL SUPERFUND SITE
NEW CASTLE COUNTY, DELAWARE

Analytical Group	CAS	Analyte	Units	Achievable Laboratory Limits	
				MDLs	QLs
VOCs	71-55-6	1,1,1-Trichloroethane	ug/l	0.238	1
VOCs	79-34-5	1,1,2,2-Tetrachloroethane	ug/l	0.367	1
VOCs	79-00-5	1,1,2-Trichloroethane	ug/l	0.433	1
VOCs	75-34-3	1,1-Dichloroethane	ug/l	0.264	1
VOCs	75-35-4	1,1-Dichloroethene	ug/l	0.117	1
VOCs	526-73-8	1,2,3-Trimethylbenzene	ug/l	0.36	1
VOCs	95-63-6	1,2,4-Trimethylbenzene	ug/l	0.374	1
VOCs	107-06-2	1,2-Dichloroethane	ug/l	0.43	1
VOCs	78-87-5	1,2-Dichloropropane	ug/l	0.353	1
VOCs	108-67-8	1,3,5-Trimethylbenzene	ug/l	0.326	1
VOCs	123-91-1	1,4-Dioxane	ug/l	0.2	0.4
VOCs	78-93-3	2-Butanone	ug/l	1.85	5
VOCs	591-78-6	2-Hexanone	ug/l	2.9	5
VOCs	108-10-1	4-Methyl-2-pentanone	ug/l	2.73	5
VOCs	67-64-1	Acetone	ug/l	4.98	5
VOCs	71-43-2	Benzene	ug/l	0.428	1
VOCs	75-27-4	Bromodichloromethane	ug/l	0.343	1
VOCs	75-25-2	Bromoform	ug/l	0.536	1
VOCs	74-83-9	Bromomethane	ug/l	1	1
VOCs	75-15-0	Carbon disulfide	ug/l	0.155	1
VOCs	56-23-5	Carbon tetrachloride	ug/l	0.208	1
VOCs	108-90-7	Chlorobenzene	ug/l	0.377	1
VOCs	75-00-3	Chloroethane	ug/l	0.32	1
VOCs	67-66-3	Chloroform	ug/l	0.326	1
VOCs	74-87-3	Chloromethane	ug/l	0.143	1
VOCs	156-59-2	cis-1,2-Dichloroethene	ug/l	0.219	1
VOCs	10061-01-5	cis-1,3-Dichloropropene	ug/l	0.455	1
VOCs	110-82-7	Cyclohexane	ug/l	0.321	1
VOCs	124-48-1	Dibromochloromethane	ug/l	0.281	1
VOCs	75-43-4	Dichlorofluoromethane	ug/l	0.341	1
VOCs	60-29-7	Ethyl ether	ug/l	0.439	1
VOCs	100-41-4	Ethylbenzene	ug/l	0.298	1
VOCs	496-11-7	Indane	ug/l	0.345	1
VOCs	98-82-8	Isopropylbenzene	ug/l	0.336	1
VOCs	108-87-2	Methylcyclohexane	ug/l	0.258	1
VOCs	75-09-2	Methylene Chloride	ug/l	0.315	1
VOCs	1634-04-4	MTBE	ug/l	0.465	1
VOCs	103-65-1	N-Propylbenzene	ug/l	0.322	1
VOCs	100-42-5	Styrene	ug/l	0.415	1
VOCs	127-18-4	Tetrachloroethene	ug/l	0.249	1
VOCs	109-99-9	Tetrahydrofuran	ug/l	1.04	2
VOCs	108-88-3	Toluene	ug/l	0.379	1
VOCs	156-60-5	trans-1,2-Dichloroethene	ug/l	0.235	1
VOCs	10061-02-6	trans-1,3-Dichloropropene	ug/l	0.485	1
VOCs	79-01-6	Trichloroethene	ug/l	0.314	1
VOCs	75-01-4	Vinyl chloride	ug/l	0.171	1
VOCs	1330-20-7	Xylenes, Total	ug/l	0.654	2

Notes:

1) MDLs and QLs were based on QC limits as of October 21, 2010 and updated January 24, 2018 for analysis of samples following the various analytical methods as listed in Table A-15. TestAmerica Edison and Eurofins routinely re-evaluates QC criteria using the procedures in the laboratory QM.

2) See Attachment H for acronyms and abbreviations

Checked by: KS
Reviewed by: TAM

TABLE A-15
ANALYTICAL REQUIREMENTS - GROUNDWATER
DELAWARE SAND & GRAVEL SUPERFUND SITE
NEW CASTLE COUNTY, DELAWARE

PARAMETER	ANALYTICAL AND PREPARATION SOP REFERENCE[1]	METHODOLOGY	CONTAINER	MINIMUM SAMPLE VOLUME REQUIRED	PRESERVATION[6]	FIELD FILTERED	HOLD TIME[2]
Groundwater							
Ammonia	ED-WET-018	SM 4500 NH_3 H	1-500 ml polyethylene	500 ml	Cool $\leq 6^{o}$ C, H_2SO_4; pH<2	No	28 days
Bicarbonate	ED-WET-039	SM2320B	1-100 ml polyethylene	100 ml	Cool $\leq 6^{o}$ C	No	14 days
Chloride	ED-GCS-022	EPA 300.0	1-250 ml polyethylene	200 ml	Cool $\leq 6^{o}$ C	No	28 days
Ferrous Iron	ED-WET-057	SM 3500 FE D	1-125 ml polyethylene	50 ml	Cool $\leq 6^{o}$ C; HCl, pH<2	No	ASAP
TAL Metals (dissolved)	ED-MTP-003, ED-MT-004	SW-846 6010D	1-500 ml polyethylene	300 ml	Cool $\leq 6^{o}$ C; HNO_3, pH<2 after filter	Yes	180 days
TAL/RCRA[4] Metals	ED-MTP-003, ED-MT-017, ED-MT-029	SW-846 6020A/7470A	1-500 ml polyethylene	300 ml	Cool $\leq 6^{o}$ C; HNO_3, pH<2	No	180 days
Nitrate	ED-GCS-022	EPA 300.0	1-100 ml polyethylene	100 ml	Cool $\leq 6^{o}$ C	No	48 hours
Nitrite	ED-GCS-022	EPA 300.0	1-100 ml polyethylene	100 ml	Cool $\leq 6^{o}$ C	No	48 hours
PFAS	1-P-QM-WI-9039651	EPA 537 Rev 1.1, Modified	1-100 ml PFAS-free polyethylene; Teflon-free lids	100 ml	Cool $\leq 6^{o}$ C	No	14 days (extraction); 28 days (analysis)
Phosphate (ortho)	ED-GCS-022	EPA 300.0	1-100 ml polyethylene	100 ml	Cool $\leq 6^{o}$ C	No	48 hours
Sulfate	ED-GCS-022	EPA 300.0	1-100 ml polyethylene	100 ml	Cool $\leq 6^{o}$ C	No	28 days
Sulfide	ED-WET-069	SM 4500 S2 E	1-500 ml polyethylene	500 ml	Cool $<6^{o}$ C; ZnAc/NaOH, pH>9	No	7 days
SVOCs + 20 TICs	ED-ORP-002, ED-MSS-009	SW-846 8270D / SIM	2-1,000 ml amber glass	1,000 ml	Cool $\leq 6^{o}$ C	No	7/40 days[5]
VOCs + 10 TICs	ED-MSV-001, ED-MSV-014	SW-846 8260C / SIM	3-40 ml glass vial	3-40 ml	Cool $\leq 6^{o}$ C; HCl, pH<2	No	14 days

Notes:

1. Reference number from Table A-16.
2. Hold time based upon day of sample collection not Verified Time of Sample Receipt.
3. RCRA 8 Metals list includes: arsenic, barium, cadmium, chromium, lead, mercury, silver, selenium
4. The total metals holding time is 28 days for mercury, 180 days for all other metals.
5. The aqueous SVOC holding time is 7 days for extraction, 40 days from extraction to analysis.
6. The frequency of temperature blanks for cooler temperature verification during shipment is one blank per cooler.
7. See Attachment H for acronyms and abbreviations

Checked by: CLY
Reviewed by: TAM

TABLE A-16
ANALYTICAL SOP REFERENCES - GROUNDWATER
DELAWARE SAND & GRAVEL SUPERFUND SITE
NEW CASTLE COUNTY, DELAWARE

Reference Number	Title, Revision Date, and/or Number	Analytical Group
ED-MT-004	Trace Metals Analysis by Inductively Coupled Plasma Emission Spectroscopy by SW846 Method 6010B, 6010C & 6010D	Inorganics
ED-MSV-014	SW846 Method 8260C, Volatile Organic Compounds by Gas Chromatography/Mass Spectrometry (GC/MS)	VOCs
1-P-QM-WI-9039651	Determination of Selected Perfluorinated Alkyl Acids in Drinking Water by Solid Phase Extraction and LC/MS/MS, Revision 1.1, 9/2009	PFAS
ED-GCS-022	Anions by Ion Chromatography using EPA Method 300.0, SW846 9056A and SM 4110 B	Major Cations and Anions
ED-MSS-009	Semivolatile Organic Compounds by Gas Chromatography/Mass Spectrometery (GC/MS), SW846 Method 8270D	SVOCs
ED-MSV-001	Purge and Trap for Aqueous Samples, SW846 Method 5030B and 5030C	VOCs
ED-MT-017	Mercury Analysis for Water and Wastewater using EPA 245.1and SW846 7470A; Mercury in Drinking Water using EPA 245.1; Leeman Mercury Analyzer (Cold Vapor Technique)	Inorganics
ED-MT-029	Trace Metals Analysis for Water, Wastewater, Soil, Sediment and Leachate Samples by ICP-MS Method No(s). EPA Method 200.8 and SW-846 Method 6020	Inorganics
ED-MTP-003	Digestion of Water and Wastewater Samples for Analysis by ICP and ICP-MS, SW846 Method 3010A	Inorganics
ED-ORP-002	Extraction of Semi-Volatile Organic Compounds in Aqueous Samples and Leachates - Separatory Funnel, SW846 Method 3510C	SVOCs
ED-WET-018	Analysis of Ammonia in Water, Wastewater and Soil-Automated by Methods EPA 350.1, SM 4500-NH3 B plus G-11, SM 4500-NH3 B plus H-11, and QuikChem Method 10-107-06-1-C rev. 05-07-87	Major Cations and Anions
ED-WET-039	Analysis of Alkalinity in Water, Wastewater and Soil by Manual Titration or Auto-Titrator, Standard Method 2320 B-11	Major Cations and Anions
ED-WET-057	Analysis of Ferrous Iron in Aqueous Samples by Standard Methods 3500- Fe D - Discreet Analyzer	Major Cations and Anions
ED-WET-069	Analysis of Sulfide in Water, Titrimetric by Standard Methods 4500-S2 F and SW846 9034	Major Cations and Anions

Notes:
1) See Attachment H for acronyms and abbreviations

Checked by: CLY
Reviewed by: TAM

TABLE A-17
MS/MSD DATA SUMMARY REQUIREMENTS - GROUNDWATER
DELAWARE SAND & GRAVEL SUPERFUND SITE
NEW CASTLE COUNTY, DELAWARE

Compound	MS/MSD % R	MS/MSD RPD
Inorganic Aqueous Samples		
Aluminum	70-130	--
Antimony	70-130	--
Arsenic	70-130	--
Barium	70-130	--
Beryllium	70-130	--
Cadmium	70-130	--
Calcium	70-130	--
Chromium	70-130	--
Cobalt	70-130	--
Copper	70-130	--
Iron	70-130	--
Iron - dissolved	70-130	--
Lead	70-130	--
Magnesium	70-130	--
Manganese	70-130	--
Manganese - dissolved	70-130	--
Mercury	70-130	--
Nickel	70-130	--
Potassium	70-130	--
Selenium	70-130	--
Silver	70-130	--
Sodium	70-130	--
Thallium	70-130	--
Vanadium	70-130	--
Zinc	70-130	--
Cation/Anion Aqueous Samples		
Ammonia as N	65-135	--
Bicarbonate	85-115	--
Chloride	90-110	--
Ferrous Iron	90-110	--
Nitrate as N	90-110	--
Nitrite as N	90-110	--
Sulfate as S04	90-110	--
Sulfide, total	90-110	--

TABLE A-17
MS/MSD DATA SUMMARY REQUIREMENTS - GROUNDWATER
DELAWARE SAND & GRAVEL SUPERFUND SITE
NEW CASTLE COUNTY, DELAWARE

Compound	MS/MSD % R	MS/MSD RPD
PFAS Aqueous Samples		
N-ethyl perfluorooctanesulfonamidoacetic acid (NEtFOSAA)	70-130	30
N-methyl perfluorooctanesulfonamidoacetic acid (NMeFOSAA)	70-130	30
Perfluorooctane sulfonate (PFOS)	70-130	30
Perfluorobutane sulfonate (PFBS)	70-130	30
Perfluorodecanoic acid (PFDA)	70-130	30
Perfluorododecanoic acid (PFDoA)	70-130	30
Perfluoroheptanoic acid (PFHpA)	70-130	30
Perfluorohexane sulfonate (PFHxS)	70-130	30
Perfluorohexanoic acid (PFHxA)	70-130	30
Perfluorononanoic acid (PFNA)	70-130	30
Perfluorooctanoic acid (PFOA)	70-130	30
Perfluorotetradecanoic acid (PFTA)	70-130	30
Perfluorotridecanoic acid (PFTrDA)	70-130	30
Perfluoroundecanoic acid (PFUnA)	70-130	30
SVOC Aqueous Samples		
Phenol	12-110	42
2-Chlorophenol	27-123	40
1,4-Dichlorobenzene	36-97	28
N-Nitroso-di-n-propylamine	41-116	38
1,2,4-Trichlorobenzene	39-98	28
4-Chloro-3-methylphenol	23-97	42
Acenaphthene	46-118	31
4-Nitrophenol	10-80	50
2,4-Dinitrotoluene	24-96	38
Pentachlorophenol	9-103	50
Pyrene	26-127	31

TABLE A-17
MS/MSD DATA SUMMARY REQUIREMENTS - GROUNDWATER
DELAWARE SAND & GRAVEL SUPERFUND SITE
NEW CASTLE COUNTY, DELAWARE

Compound	MS/MSD % R	MS/MSD RPD
VOC Aqueous Samples		
1,1,1-Trichloroethane	75-125	30
1,1,2,2-Tetrachloroethane	74-120	30
1,1,2-Trichloroethane	78-120	30
1,1-Dichloroethane	77-123	30
1,1-Dichloroethene	74-123	30
1,2,3-Trimethylbenzene	70-130	30
1,2,4-Trimethylbenzene	78-122	30
1,2-Dichloroethane	76-121	30
1,2-Dichloropropane	77-123	30
1,3,5-Trimethylbenzene	80-120	30
1,4-Dioxane	10-150	30
2-Butanone	64-120	30
2-Hexanone	71-125	30
4-Methyl-2-pentanone	78-124	30
Acetone	39-150	30
Benzene	77-121	30
Bromodichloromethane	76-120	30
Bromoform	53-120	30
Bromomethane	10-150	30
Carbon disulfide	69-133	30
Carbon tetrachloride	70-132	30
Chlorobenzene	80-120	30
Chloroethane	52-150	30
Chloroform	80-120	30
Chloromethane	56-131	30
cis-1,2-Dichloroethene	80-120	30
cis-1,3-Dichloropropene	77-120	30
Cyclohexane	56-150	30
Dibromochloromethane	73-120	30
Dichlorofluoromethane	71-145	30
Ethyl ether	68-136	30
Ethylbenzene	80-120	30
Indane	80-120	30
Isopropylbenzene	80-123	30
Methylcyclohexane	61-145	30
Methylene Chloride	77-123	30
MTBE	79-122	30
N-Propylbenzene	80-123	30
Styrene	80-120	30
Tetrachloroethene	78-122	30
Tetrahydrofuran	79-122	30
Toluene	80-120	30
trans-1,2-Dichloroethene	79-120	30
trans-1,3-Dichloropropene	76-120	30
Trichloroethene	77-120	30
Vinyl chloride	62-138	30
Xylenes, Total	80-120	30

Notes:
1) See Attachment H for acronyms and abbreviations

Checked by: AMZ
Reviewed by: TAM

Figures

REFERENCE(S)

1. BASE MAP TAKEN FROM USGS 7.5 MINUTE QUADRANGLE OF WILMINGTON SOUTH, DELAWARE, DATED 2016.

0 1000 2000
1" = 2000' FEET

CLIENT
DELAWARE SAND & GRAVEL SUPERFUND SITE
NEW CASTLE COUNTY, DELAWARE

PROJECT
SAMPLING AND ANALYSIS PLAN

CONSULTANT
GOLDER

YYYY-MM-DD	2018-08-10
DESIGNED	BAR
PREPARED	GLS
REVIEWED	BAR
APPROVED	TAM

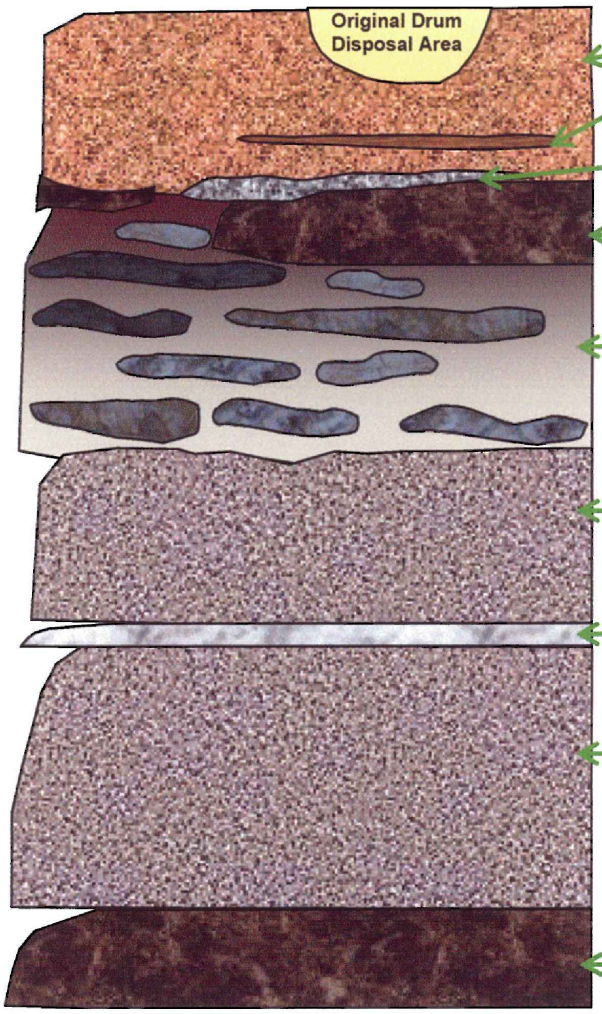
TITLE
SITE LOCATION MAP

PROJECT NO.	CONTROL	REV.	FIGURE
0136052.14	0004-001	0	A-1

IF THIS MEASUREMENT DOES NOT MATCH WHAT IS SHOWN, THE SHEET SIZE HAS BEEN MODIFIED FROM: ANSI A

Last Edited By: gsantell Date: 2018-08-09 Time:9:03:55 AM | Printed By: AMorales Date: 2018-08-13 Time:8:33:47 AM
Path: \\mtlaurel\cadd\Projects\DelawareSand&Gravel\DS&G_NewCastleDelaware\99_PROJECTS\0136052.14_Monitoring&Reporting\0004-SamplingAnalysisPlan\02_PRODUCTION\DWG\ | File Name: 0136052140004-001.dwg

Current Conceptual Site Model Lithologic Diagram



Columbia Sand – brown, tan, orange sandy aquifer unit.

Columbia Clay (not always present) – brown, tan clay unit located within the Columbia Sand.

Basal Gravel (not always present) – gravel and cobble unit with coarse sand.

Upper Potomac Confining Unit (UPCU) (not always present) – hard, competent, dry, mottled red-orange-yellow-white clay unit. Eroded in some areas by basal gravel (paleochannel).

UPCU Transition Zone (UPCUTZ) (not known to always be present) - Interbedded silt, clay, and sand, but generally fining upward sequence observed beneath and to south of Drum Disposal Area.

Upper Potomac Aquifer (UPA) – Upper Sand – generally a fine to medium sand unit in the Site area.

Upper Potomac Dividing Clay (UPDC) – Typically, a mottled clay unit, generally present in the Site area, but can be thin or interbedded with sand.

Upper Potomac Aquifer (UPA) – Lower Sand – generally a fine to coarse sand unit greater than 10 feet thick in the Site area.

Middle Potomac Confining Unit (MPCU) - competent, grey or mottled red-orange-yellow-white clay unit.

NOT TO SCALE

CLIENT
DELAWARE SAND & GRAVEL SUPERFUND SITE
NEW CASTLE COUNTY, DELAWARE

PROJECT
SAMPLING AND ANALYSIS PLAN

CONSULTANT
GOLDER

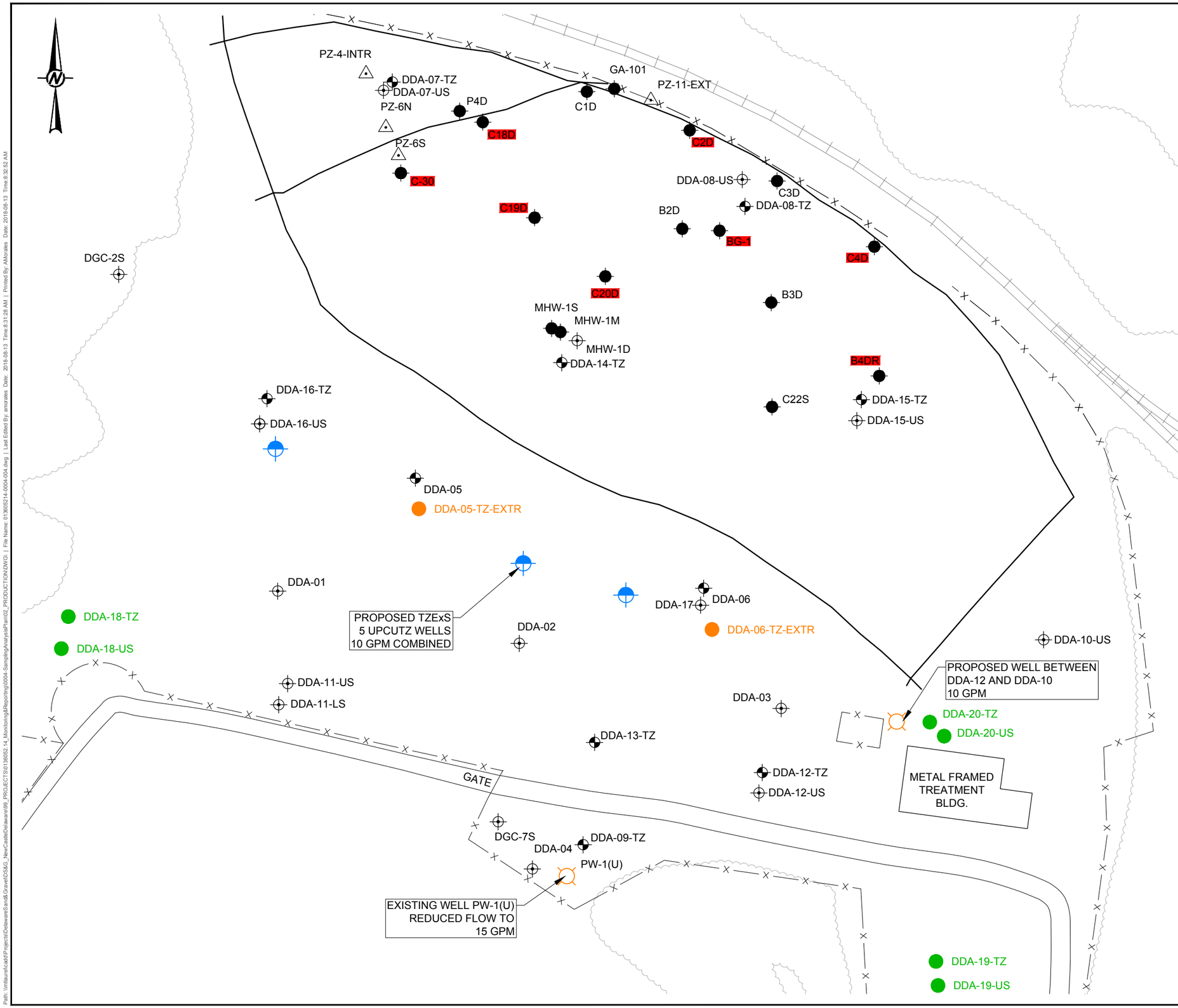
YYYY-MM-DD	2018-08-10
DESIGNED	MBS
PREPARED	GLS
REVIEWED	BAR
APPROVED	TAM

TITLE
CONCEPTUAL STRATIGRAPHIC COLUMN

PROJECT NO.	CONTROL	REV.	FIGURE
0136052.14	0004-003	0	A-2

Last Edited By: gsantell Date: 2018-08-09 Time:9:04:19 AM | Printed By: AMorales Date: 2018-08-13 Time:8:33:17 AM
Path: \\mtlaurel\cadd\Projects\DelawareSand&Gravel\DS&G_NewCastleDelaware\99_PROJECTS\0136052.14_Monitoring&Reporting\0004-SamplingAnalysisPlan\02_PRODUCTION\DWGs | File Name: 0136052\14-0004-003.dwg

IF THIS MEASUREMENT DOES NOT MATCH WHAT IS SHOWN, THE SHEET SIZE HAS BEEN MODIFIED FROM: ANSI A



LEGEND

—— x —— x ——	FENCELINE
	CENTERLINE OF SLURRY WALL AROUND DRUM DISPOSAL AREA
	RAILROAD
C19D	DDA LOW-FLOW EXTRACTION SYSTEM (LFExS) WELL
DGC-2S	MONITORING WELL SCREENED IN UPPER POTOMAC AQUIFER
DDA-05	UPPER POTOMAC CONFINING UNIT TRANSITION ZONE (UPCUTZ) MONITORING WELLS
	POTENTIAL UPCUTZ EXTRACTION WELLS (TZEXS)
	PROTENTIAL UPA EXTRACTION WELLS
	PROPOSED UPCUTZ EXTRACTION WELL
	PROPOSED MONITORING WELL

NOTE(S)

1. LOCATIONS OF WELLS AND BORINGS ARE BASED ON SURVEY BY TAYLOR WISEMAN & TAYLOR (REVISED 2012).

REFERENCE(S)

1. BASE MAP TAKEN FROM DIGITAL U.S.G.S. 7.5 MINUTE QUADRANGLES OF WILMINGTON SOUTH, DELAWARE/NEW JERSEY (DATED 1984), DELAWARE CITY, DELAWARE (DATED 1984), NEWARK EAST, DELAWARE (DATED 1984) AND SAINT GEORGES, DELAWARE (DATED 1984).

0 30 60
1" = 30' FEET

CLIENT
DELAWARE SAND & GRAVEL SUPERFUND SITE
NEW CASTLE COUNTY, DELAWARE

PROJECT
SAMPLING AND ANALYSIS PLAN

TITLE
PROPOSED MONITORING WELL LOCATIONS - DDA AND WELL PW-1(U) AREA

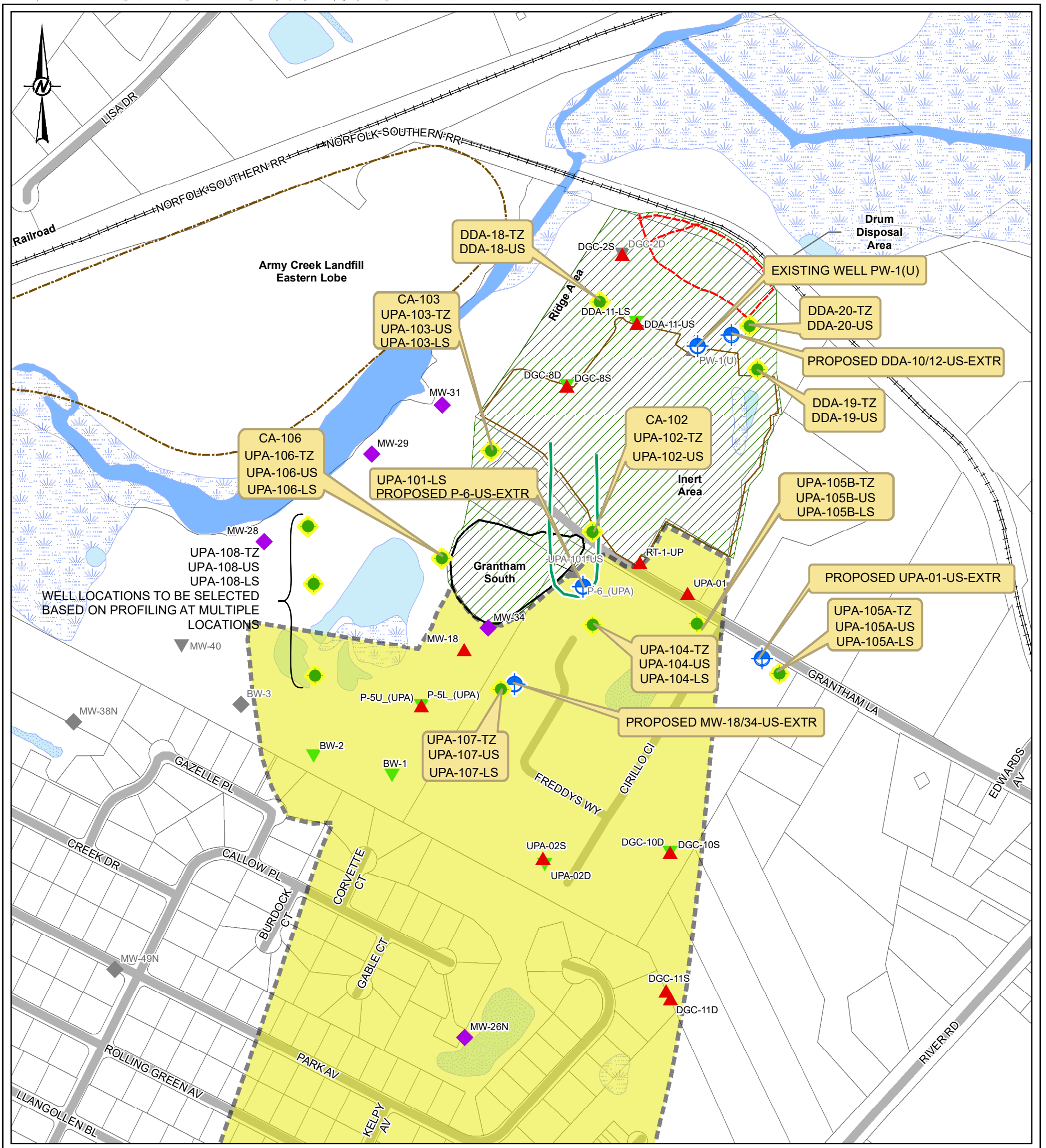
CONSULTANT
GOLDER

YYYY-MM-DD	2018-08-10
DESIGNED	MBS
PREPARED	GLS
REVIEWED	BAR
APPROVED	TAM

PROJECT NO.	CONTROL	REV.	FIGURE
0136052.14	0004-004	0	A-3

IF THIS MEASUREMENT DOES NOT MATCH WHAT IS SHOWN, THE SHEET SIZE HAS BEEN MODIFIED FROM: ANSI D

PATH: W:\Projects\DelawareSand&Gravel\DS&G_NewCastleDelaware\99_PROJECTS\0136052.14_Monitoring&Reporting\0004-SamplingAnalysisPlan\02_PRODUCTION\MXD\FIGURES\Rev0\013605214-0004-011.mxd PRINTED ON: 2018-08-17 AT: 12:16:06 PM



LEGEND

- Proposed Extraction Well
- Proposed Assessment Location
- Well screened in UPA Upper Sand included in the Proposed 2018 Monitoring Program
- Well screened in UPA Upper Sand not included in the Proposed 2018 Monitoring Program
- Well screened in UPA Lower Sand included in the Proposed 2018 Monitoring Program
- Well screened in UPA Lower Sand not included in the Proposed 2018 Monitoring Program
- Well screened across UPA Upper Sand and Lower Sand included in the Proposed 2018 Monitoring Program
- Well screened across UPA Upper Sand and Lower Sand not included in the Proposed 2018 Monitoring Program
- Modeled Feature in the Well P-6 area
- Area of Attainment - The source of contaminated groundwater detected in well BW-2 remains under evaluation
- Waste Management Area

REFERENCE(S)

1. BASE DATA FROM NEW CASTLE COUNTY DELAWARE, DEPARTMENT OF LAND USE, "EPARCEL VIEW MAP" WEB SITE GIS DATA DOWNLOAD. DATA ACQUIRED 01/18/2012.

0 400 800

1 " = 400 feet Feet

IF THIS MEASUREMENT DOES NOT MATCH WHAT IS SHOWN, THE SHEET SIZE HAS BEEN MODIFIED FROM: ANSI B

1 in

0

CLIENT
DELAWARE SAND & GRAVEL SUPERFUND SITE
NEW CASTLE COUNTY, DELAWARE

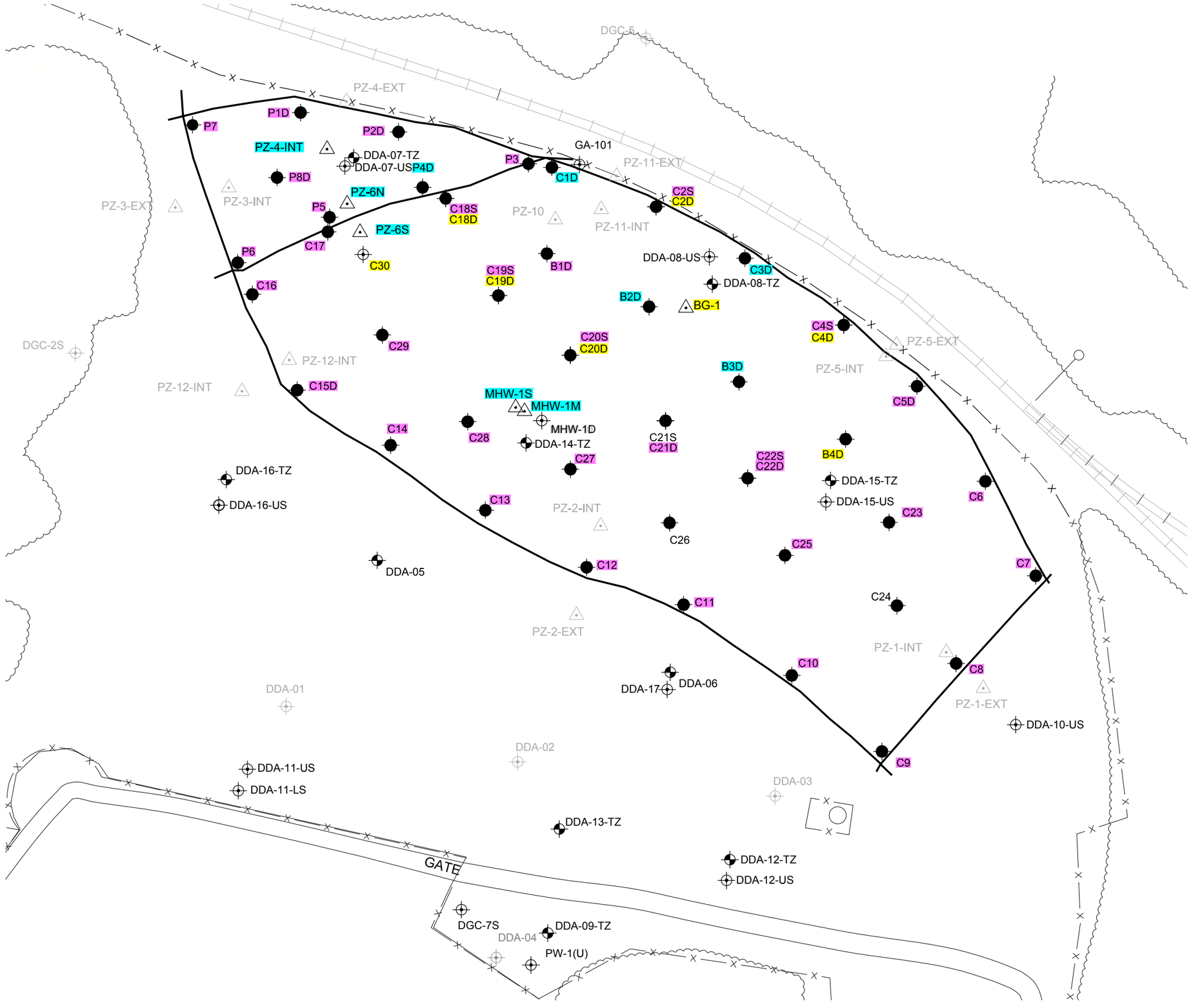
PROJECT
PRE-DESIGN INVESTIGATION
WORK PLAN

CONSULTANT
GOLDER

YYYY-MM-DD	01-08-2019
DESIGNED	MBS
PREPARED	AM
REVIEWED	BAR
APPROVED	

TITLE
PROPOSED MONITORING WELL LOCATIONS - DOWNGRADIENT OF WELL PW-1(U)

PROJECT NO.	CONTROL	REV.	FIGURE
013-6052.14	0004-011	0	A-4



LEGEND

	CENTERLINE OF SLURRY WALL AROUND DRUM DISPOSAL AREA
	RAILROAD
C1D	DDA VERTICAL SYSTEM WELLS
PZ-6S	DDA MONITORING WELLS, SAMPLED
PZ-10	DDA MONITORING WELLS, NOT SAMPLED
DGC-2S	MONITORING WELL SCREENED IN UPPER POTOMAC AQUIFER
DDA-02	PROPOSED MONITORING WELL IN UPPER POTOMAC AQUIFER
	SAMPLE ONCE PRIOR TO ELFExS DESIGN
	LOW-FLOW EXTRACTION SYSTEM WELLS - SAMPLE SEMI-ANNUALLY
	SAMPLE ANNUALLY

NOTE(S)

1. LOCATIONS OF WELLS AND BORINGS ARE BASED ON SURVEY BY TAYLOR WISEMAN & TAYLOR (REVISED 2012).

REFERENCE(S)

1. BASE MAP TAKEN FROM DIGITAL U.S.G.S. 7.5 MINUTE QUADRANGLES OF WILMINGTON SOUTH, DELAWARE/NEW JERSEY (DATED 1984), DELAWARE CITY, DELAWARE (DATED 1984), NEWARK EAST, DELAWARE (DATED 1984) AND SAINT GEORGES, DELAWARE (DATED 1984).

0 30 60
1" = 30' FEET

CLIENT
DELAWARE SAND & GRAVEL SUPERFUND SITE
NEW CASTLE COUNTY, DELAWARE

PROJECT
SAMPLING AND ANALYSIS PLAN

TITLE
MONITORING LOCATIONS FOR ENHANCED LFExS DESIGN

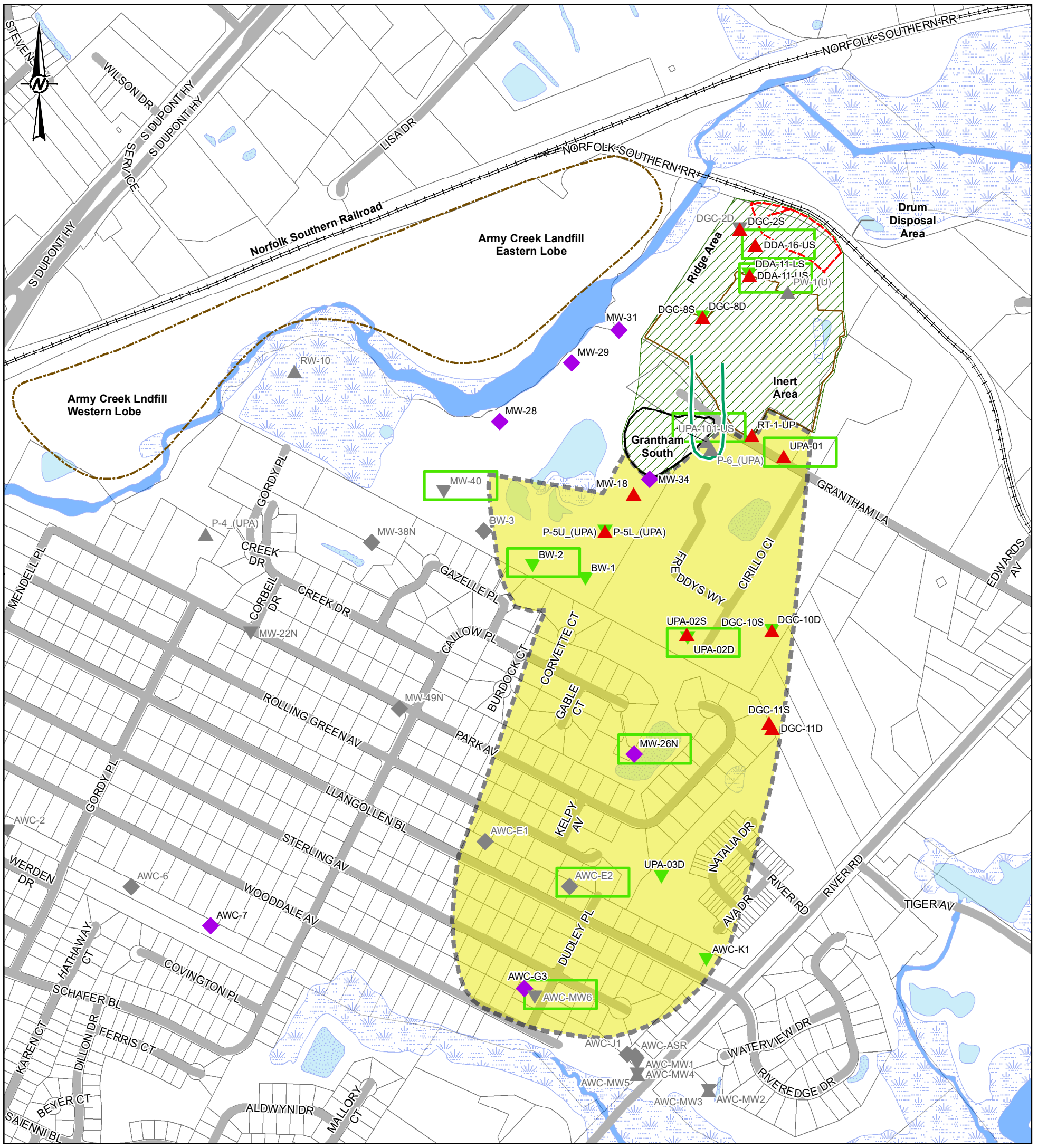
CONSULTANT
GOLDER

YYYY-MM-DD	2018-08-10
DESIGNED	MBS
PREPARED	GLS
REVIEWED	BAR
APPROVED	TAM

PROJECT NO.	CONTROL	REV.	FIGURE
0136052.14	0004-005	0	A-5

IF THIS MEASUREMENT DOES NOT MATCH WHAT IS SHOWN, THE SHEET SIZE HAS BEEN MODIFIED FROM: ANSI D

PATH: W:\Projects\DelawareSand&Gravel\DS&G_NewCastleDelaware\99_PROJECTS\0136052.14_Monitoring&Reporting\0004-SamplingAnalysisPlan\02_PRODUCTION\MXD\FIGURES\Rev0\013605214-0004-012.mxd PRINTED ON: 2018-08-17 AT: 1:24:26 PM



LEGEND

Locations_All

Type_, DownGrad

- Well screened in UPA Upper Sand included in the Proposed 2018 Monitoring Program
- Well screened in UPA Upper Sand not included in the Proposed 2018 Monitoring Program
- Well screened in UPA Lower Sand included in the Proposed 2018 Monitoring Program
- Well screened in UPA Lower Sand not included in the Proposed 2018 Monitoring Program
- Well screened across UPA Upper Sand and Lower Sand included in the Proposed 2018 Monitoring Program
- Well screened across UPA Upper Sand and Lower Sand not included in the Proposed 2018 Monitoring Program
- Modeled Feature in the Well P-6 area
- Transducer Study Location
- Area of Attainment - The source of contaminated groundwater detected in well BW-2 remains under evaluation
- Waste Management Area

REFERENCE(S)

1. BASE DATA FROM NEW CASTLE COUNTY DELAWARE, DEPARTMENT OF LAND USE, "EPARCEL VIEW MAP" WEB SITE GIS DATA DOWNLOAD. DATA ACQUIRED 01/18/2012.

0 600 1,200

1 " = 600 feet Feet

IF THIS MEASUREMENT DOES NOT MATCH WHAT IS SHOWN, THE SHEET SIZE HAS BEEN MODIFIED FROM: ANSI B

1 in

0

CLIENT
DELAWARE SAND & GRAVEL SUPERFUND SITE
NEW CASTLE COUNTY, DELAWARE

PROJECT
SAMPLING ANALYSIS PLAN

CONSULTANT
GOLDER

YYYY-MM-DD	2018-08-10
DESIGNED	MBS
PREPARED	AM
REVIEWED	BAR
APPROVED	TAM

TITLE
AQUIFER TESTING - TRANSDUCER LOCATIONS
ON-GOING TRANSDUCER STUDY

PROJECT NO.	CONTROL	REV.	FIGURE
013-6052.14	0004-012	0	A-6A

PZ-4-INTR
DDA-07-TZ
DDA-07-US
PZ-6N
P4D
C18D
GA-101
C1D
PZ-11-EXT
C2D
PZ-6S
C-30
DDA-08-US
C3D
DDA-08-TZ
C19D
B2D
BG-1
C4D
DGC-2S
C20D
B3D
MHW-1S
MHW-1M
MHW-1D
DDA-14-TZ
B4DR
DDA-16-TZ
DDA-16-US
C22S
DDA-15-TZ
DDA-15-US
DDA-05
DDA-05-TZ-EXTR
DDA-01
DDA-18-TZ
DDA-18-US
PROPOSED TZExS
5 UPCUTZ WELLS
10 GPM COMBINED
DDA-02
DDA-17
DDA-06
DDA-06-TZ-EXTR
DDA-10-US
PROPOSED WELL DDA-10/12-US-EXTR
10 GPM
DDA-11-US
DDA-11-LS
DDA-03
DDA-20-TZ
DDA-20-US
DDA-13-TZ
GATE
DDA-12-TZ
DDA-12-US
METAL FRAMED
TREATMENT
BLDG.
DGC-7S
DDA-09-TZ
DDA-04
PW-1(U)
EXISTING WELL PW-1(U)
REDUCED FLOW TO
15 GPM
DDA-19-TZ
DDA-19-US

LEGEND

- FENCELINE
- CENTERLINE OF SLURRY WALL AROUND DRUM DISPOSAL AREA
- RAILROAD
- C19D — DDA LOW-FLOW EXTRACTION SYSTEM (LFExS) WELL
- DGC-2S — MONITORING WELL SCREENED IN UPPER POTOMAC AQUIFER
- DDA-05 — UPPER POTOMAC CONFINING UNIT TRANSITION ZONE (UPCUTZ) MONITORING WELLS
- POTENTIAL UPCUTZ EXTRACTION WELLS (TZEXS)
- PROTENTIAL UPA EXTRACTION WELLS
- PROPOSED UPCUTZ EXTRACTION WELL
- PROPOSED MONITORING WELL
- PUMPING TEST WELL
- TRANSDUCER LOCATION

NOTE(S)

1. LOCATIONS OF WELLS AND BORINGS ARE BASED ON SURVEY BY TAYLOR WISEMAN & TAYLOR (REVISED 2012)

REFERENCE(S)

1. BASE MAP TAKEN FROM DIGITAL U.S.G.S. 7.5 MINUTE QUADRANGLES OF WILMINGTON SOUTH, DELAWARE/NEW JERSEY (DATED 1984), DELAWARE CITY, DELAWARE (DATED 1984), NEWARK EAST, DELAWARE (DATED 1984) AND SAINT GEORGES, DELAWARE (DATED 1984).

0 30 60
1" = 30' FEET

CLIENT
DELAWARE SAND & GRAVEL SUPERFUND SITE
NEW CASTLE COUNTY, DELAWARE

PROJECT
SAMPLING AND ANALYSIS PLAN

TITLE
AQUIFER TESTING - TRANSDUCER LOCATIONS - STEPS 1 AND 2 - EXISTING WELL PW-1(U) SHUT DOWN AND RE-START

CONSULTANT
GOLDER

YYYY-MM-DD	2018-08-10
DESIGNED	MBS
PREPARED	GLS
REVIEWED	BAR
APPROVED	TAM

PROJECT NO.	CONTROL	REV.	FIGURE
0136052.14	0004-006	0	A-6B

IF THIS MEASUREMENT DOES NOT MATCH WHAT IS SHOWN, THE SHEET SIZE HAS BEEN MODIFIED FROM: ANSI D

Path: \\nttaurefilcad01\Projects\Delaware\Sand&Gravel\DS&G_NewCastleDelaware\99_PROJECTS\0136052.14_Monitoring&Reporting\0004-Sampling\Analysis\Plan\02_PRODUCTION\DWG\ | File Name: 01360524-0004-006.dwg | Last Edited By: amorales Date: 2018-08-13 Time:11:33:34 AM | Printed By: AMorales Date: 2018-08-17 Time:11:35:12 AM

PZ-4-INTR
DDA-07-TZ
DDA-07-US
PZ-6N
P4D
GA-101
C1D
PZ-11-EXT
C18D
C2D
PZ-6S
C-30
DDA-08-US
C3D
DDA-08-TZ
C19D
B2D
BG-1
C4D
DGC-2S
C20D
B3D
MHW-1S
MHW-1M
MHW-1D
DDA-14-TZ
B4DR
DDA-16-TZ
C22S
DDA-15-TZ
DDA-16-US
DDA-15-US
DDA-05
DDA-05-TZ-EXTR
DDA-01
DDA-06
DDA-17
DDA-06-TZ-EXTR
DDA-18-TZ
DDA-18-US
PROPOSED TZExS
5 UPCUTZ WELLS
10 GPM COMBINED
DDA-02
DDA-10-US
DDA-11-US
DDA-11-LS
PROPOSED WELL DDA-10/12-US-EXTR
10 GPM
DDA-03
DDA-20-TZ
DDA-20-US
DDA-13-TZ
GATE
DDA-12-TZ
DDA-12-US
METAL FRAMED
TREATMENT
BLDG.
DGC-7S
DDA-09-TZ
DDA-04
PW-1(U)
EXISTING WELL PW-1(U)
REDUCED FLOW TO
15 GPM
DDA-19-TZ
DDA-19-US

LEGEND

- FENCELINE
- CENTERLINE OF SLURRY WALL AROUND DRUM DISPOSAL AREA
- RAILROAD
- C19D — DDA LOW-FLOW EXTRACTION SYSTEM (LFExS) WELL
- DGC-2S — MONITORING WELL SCREENED IN UPPER POTOMAC AQUIFER
- DDA-05 — UPPER POTOMAC CONFINING UNIT TRANSITION ZONE (UPCUTZ) MONITORING WELLS
- POTENTIAL UPCUTZ EXTRACTION WELLS (TZEXS)
- PROTENTIAL UPA EXTRACTION WELLS
- PROPOSED UPCUTZ EXTRACTION WELL
- PROPOSED MONITORING WELL
- PUMPING TEST WELL
- TRANSDUCER LOCATION

NOTE(S)

1. LOCATIONS OF WELLS AND BORINGS ARE BASED ON SURVEY BY TAYLOR WISEMAN & TAYLOR (REVISED 2012)

REFERENCE(S)

1. BASE MAP TAKEN FROM DIGITAL U.S.G.S. 7.5 MINUTE QUADRANGLES OF WILMINGTON SOUTH, DELAWARE/NEW JERSEY (DATED 1984), DELAWARE CITY, DELAWARE (DATED 1984), NEWARK EAST, DELAWARE (DATED 1984) AND SAINT GEORGES, DELAWARE (DATED 1984).

0 30 60
1" = 30' FEET

CLIENT
DELAWARE SAND & GRAVEL SUPERFUND SITE
NEW CASTLE COUNTY, DELAWARE

PROJECT
SAMPLING AND ANALYSIS PLAN

TITLE
AQUIFER TESTING - TRANSDUCER LOCATIONS - STEP 3
PROPOSED EXTRACTION WELL DDA-10/12-US-EXTR

CONSULTANT
GOLDER

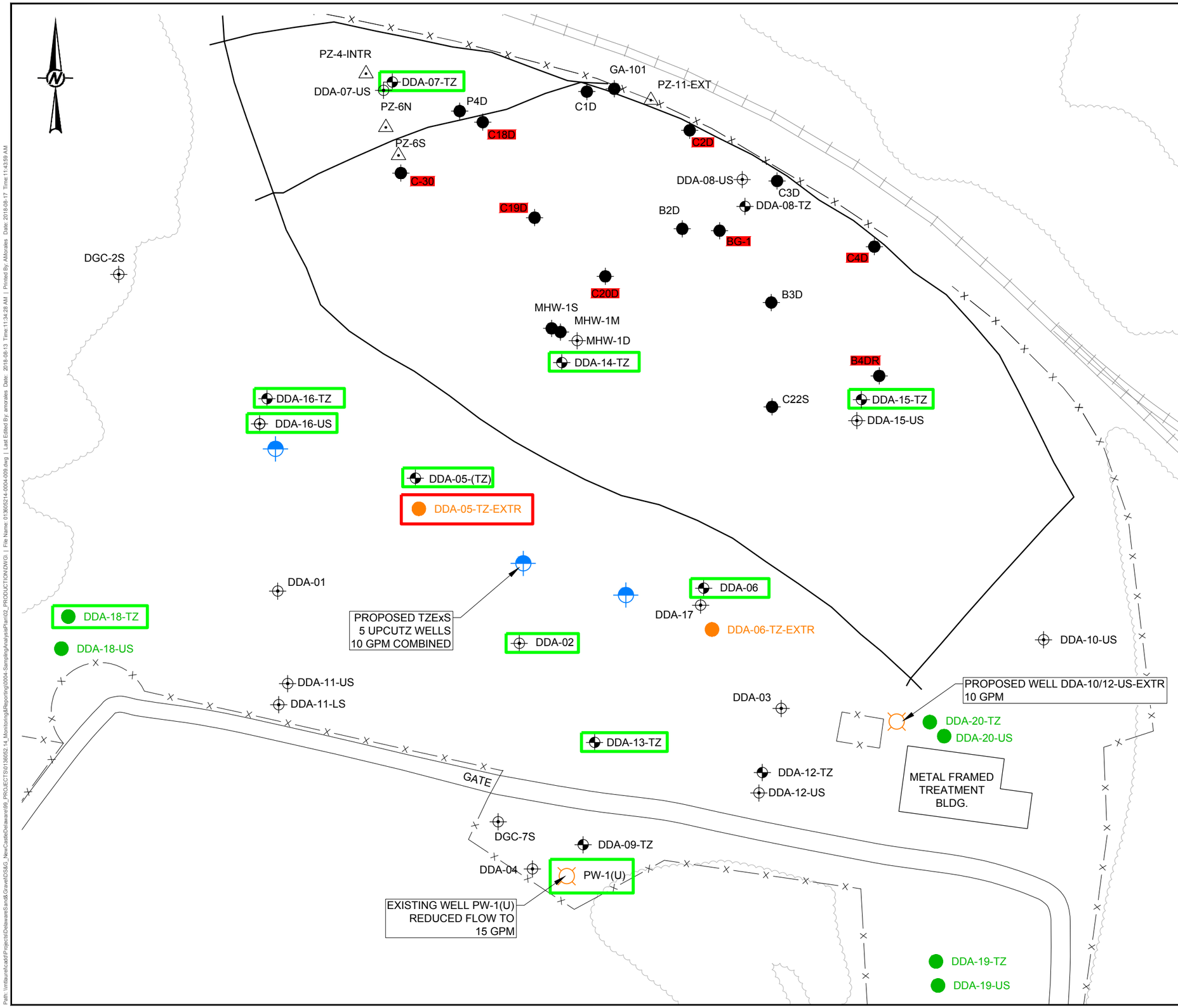
YYYY-MM-DD	2018-08-10
DESIGNED	MBS
PREPARED	GLS
REVIEWED	BAR
APPROVED	TAM

PROJECT NO.	CONTROL	REV.	FIGURE
0136052.14	0004-007	0	A-6C

IF THIS MEASUREMENT DOES NOT MATCH WHAT IS SHOWN, THE SHEET SIZE HAS BEEN MODIFIED FROM: ANSI D

0 1 in

Path: \\nttaurefilcad01\Projects\Delaware\Sand&Gravel\DS&G_NewCastleDelaware\99_PROJECTS\0136052.14_Monitoring&Reporting\0004-Sampling\Analysis\Plan\02_PRODUCTION\DWG\ | File Name: 0136052140004-007.dwg | Last Edited By: amorales Date: 2018-08-13 Time:11:34:02 AM | Printed By: AMorales Date: 2018-08-17 Time:11:44:13 AM



LEGEND

- FENCELINE
- CENTERLINE OF SLURRY WALL AROUND DRUM DISPOSAL AREA
- RAILROAD
- C19D — DDA LOW-FLOW EXTRACTION SYSTEM (LFExS) WELL
- DGC-2S — MONITORING WELL SCREENED IN UPPER POTOMAC AQUIFER
- DDA-05 — UPPER POTOMAC CONFINING UNIT TRANSITION ZONE (UPCUTZ) MONITORING WELLS
- POTENTIAL UPCUTZ EXTRACTION WELLS (TZEXS)
- PROTENTIAL UPA EXTRACTION WELLS
- PROPOSED UPCUTZ EXTRACTION WELL
- PROPOSED MONITORING WELL
- PUMPING TEST WELL
- TRANSDUCER LOCATION

NOTE(S)

1. LOCATIONS OF WELLS AND BORINGS ARE BASED ON SURVEY BY TAYLOR WISEMAN & TAYLOR (REVISED 2012)

REFERENCE(S)

1. BASE MAP TAKEN FROM DIGITAL U.S.G.S. 7.5 MINUTE QUADRANGLES OF WILMINGTON SOUTH, DELAWARE/NEW JERSEY (DATED 1984), DELAWARE CITY, DELAWARE (DATED 1984), NEWARK EAST, DELAWARE (DATED 1984) AND SAINT GEORGES, DELAWARE (DATED 1984).

0 30 60
1" = 30' FEET

CLIENT
DELAWARE SAND & GRAVEL SUPERFUND SITE
NEW CASTLE COUNTY, DELAWARE

PROJECT
SAMPLING AND ANALYSIS PLAN

TITLE
AQUIFER TESTING - TRANSDUCER LOCATIONS - STEP 4 - PROPOSED EXTRACTION WELL DDA-05-TZ-EXTR

CONSULTANT
GOLDER

YYYY-MM-DD	2018-08-10
DESIGNED	MBS
PREPARED	GLS
REVIEWED	BAR
APPROVED	TAM

PROJECT NO.	CONTROL	REV.	FIGURE
0136052.14	0004-009	0	A-6D

IF THIS MEASUREMENT DOES NOT MATCH WHAT IS SHOWN, THE SHEET SIZE HAS BEEN MODIFIED FROM: ANSI D

PZ-4-INTR
DDA-07-US
DDA-07-TZ
PZ-6N
P4D
C18D
GA-101
C1D
PZ-11-EXT
C2D
PZ-6S
C-30
C3D
DDA-08-US
DDA-08-TZ
C19D
B2D
BG-1
C4D
DGC-2S
C20D
B3D
MHW-1S
MHW-1M
MHW-1D
DDA-14-TZ
B4DR
DDA-16-TZ
DDA-16-US
C22S
DDA-15-TZ
DDA-15-US
DDA-05-(TZ)
DDA-05-TZ-EXTR
DDA-01
DDA-18-TZ
DDA-18-US
PROPOSED TZExS
5 UPCUTZ WELLS
10 GPM COMBINED
DDA-02
DDA-06
DDA-17
DDA-06-TZ-EXTR
DDA-10-US
PROPOSED WELL DDA-10/12-US-EXTR
10 GPM
DDA-11-US
DDA-11-LS
DDA-03
DDA-20-TZ
DDA-20-US
DDA-13-TZ
DDA-12-TZ
DDA-12-US
METAL FRAMED
TREATMENT
BLDG.
GATE
DGC-7S
DDA-09-TZ
DDA-04
PW-1(U)
EXISTING WELL PW-1(U)
REDUCED FLOW TO
15 GPM
DDA-19-TZ
DDA-19-US

LEGEND

- FENCELINE
- CENTERLINE OF SLURRY WALL AROUND DRUM DISPOSAL AREA
- RAILROAD
- C19D — DDA LOW-FLOW EXTRACTION SYSTEM (LFExS) WELL
- DGC-2S — MONITORING WELL SCREENED IN UPPER POTOMAC AQUIFER
- DDA-05 — UPPER POTOMAC CONFINING UNIT TRANSITION ZONE (UPCUTZ) MONITORING WELLS
- POTENTIAL UPCUTZ EXTRACTION WELLS (TZEXS)
- PROTENTIAL UPA EXTRACTION WELLS
- PROPOSED UPCUTZ EXTRACTION WELL
- PROPOSED MONITORING WELL
- PUMPING TEST WELL
- TRANSDUCER LOCATION

NOTE(S)

1. LOCATIONS OF WELLS AND BORINGS ARE BASED ON SURVEY BY TAYLOR WISEMAN & TAYLOR (REVISED 2012)

REFERENCE(S)

1. BASE MAP TAKEN FROM DIGITAL U.S.G.S. 7.5 MINUTE QUADRANGLES OF WILMINGTON SOUTH, DELAWARE/NEW JERSEY (DATED 1984), DELAWARE CITY, DELAWARE (DATED 1984), NEWARK EAST, DELAWARE (DATED 1984) AND SAINT GEORGES, DELAWARE (DATED 1984).

0 30 60
1" = 30' FEET

CLIENT
DELAWARE SAND & GRAVEL SUPERFUND SITE
NEW CASTLE COUNTY, DELAWARE

PROJECT
SAMPLING AND ANALYSIS PLAN

TITLE
AQUIFER TESTING - TRANSDUCER LOCATIONS - STEP 5 - PROPOSED EXTRACTION WELL DDA-06-TZ-EXTR

CONSULTANT
GOLDER

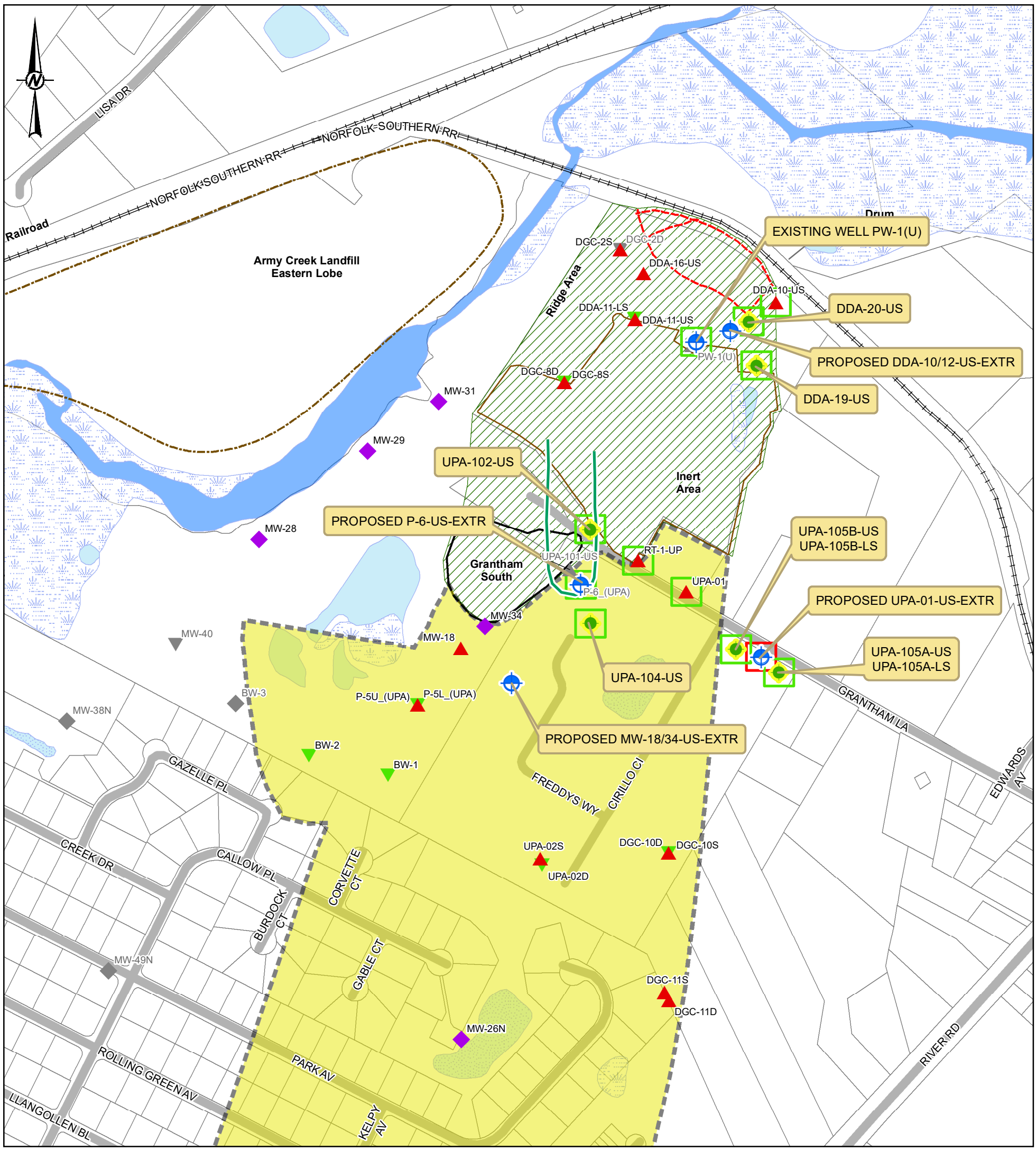
YYYY-MM-DD	2018-08-10
DESIGNED	MBS
PREPARED	AM
REVIEWED	BAR
APPROVED	TAM

PROJECT NO.	CONTROL	REV.	FIGURE
0136052.14	0004-016	0	A-6E

IF THIS MEASUREMENT DOES NOT MATCH WHAT IS SHOWN, THE SHEET SIZE HAS BEEN MODIFIED FROM: ANSI D

Path: \\nttaurefilcatd\Projects\Delaware\Sand&Gravel\DS&G_NewCastleDelaware\99_PROJECTS\0136052.14_Monitoring&Reporting\0004-Sampling\Analysis\Plan\02_PRODUCTION.DWG | File Name: 0136052140004-016.dwg | Last Edited By: amorales Date: 2018-08-13 Time:11:44:46 AM | Printed By: AMorales Date: 2018-08-17 Time:11:43:43 AM

PATH: W:\Projects\DelawareSand&Gravel\DS&G_NewCastleDelaware\99_PROJECTS\0136052.14_Monitoring&Reporting\0004-SamplingAnalysisPlan\02_PRODUCTION\MXD\FIGURES\Rev0\013605214-0004-013.mxd PRINTED ON: 2018-08-17 AT: 3:01:01 PM



LEGEND

- Proposed Extraction Well
- Proposed Assessment Location
- Well screened in UPA Upper Sand included in the Proposed 2018 Monitoring Program
- Well screened in UPA Upper Sand not included in the Proposed 2018 Monitoring Program
- Well screened in UPA Lower Sand included in the Proposed 2018 Monitoring Program
- Well screened in UPA Lower Sand not included in the Proposed 2018 Monitoring Program
- Well screened across UPA Upper Sand and Lower Sand included in the Proposed 2018 Monitoring Program
- Well screened across UPA Upper Sand and Lower Sand not included in the Proposed 2018 Monitoring Program
- Modeled Feature in the Well P-6 area
- Transducer Study Location
- Pumping Test Well
- Area of Attainment - The source of contaminated groundwater detected in well BW-2 remains under evaluation
- Waste Management Area

REFERENCE(S)

1. BASE DATA FROM NEW CASTLE COUNTY DELAWARE, DEPARTMENT OF LAND USE, "EPARCEL VIEW MAP" WEB SITE GIS DATA DOWNLOAD. DATA ACQUIRED 01/18/2012.

0 400 800

1 " = 400 feet Feet

IF THIS MEASUREMENT DOES NOT MATCH WHAT IS SHOWN, THE SHEET SIZE HAS BEEN MODIFIED FROM: ANSI B

1 in

0

CLIENT
DELAWARE SAND & GRAVEL SUPERFUND SITE
NEW CASTLE COUNTY, DELAWARE

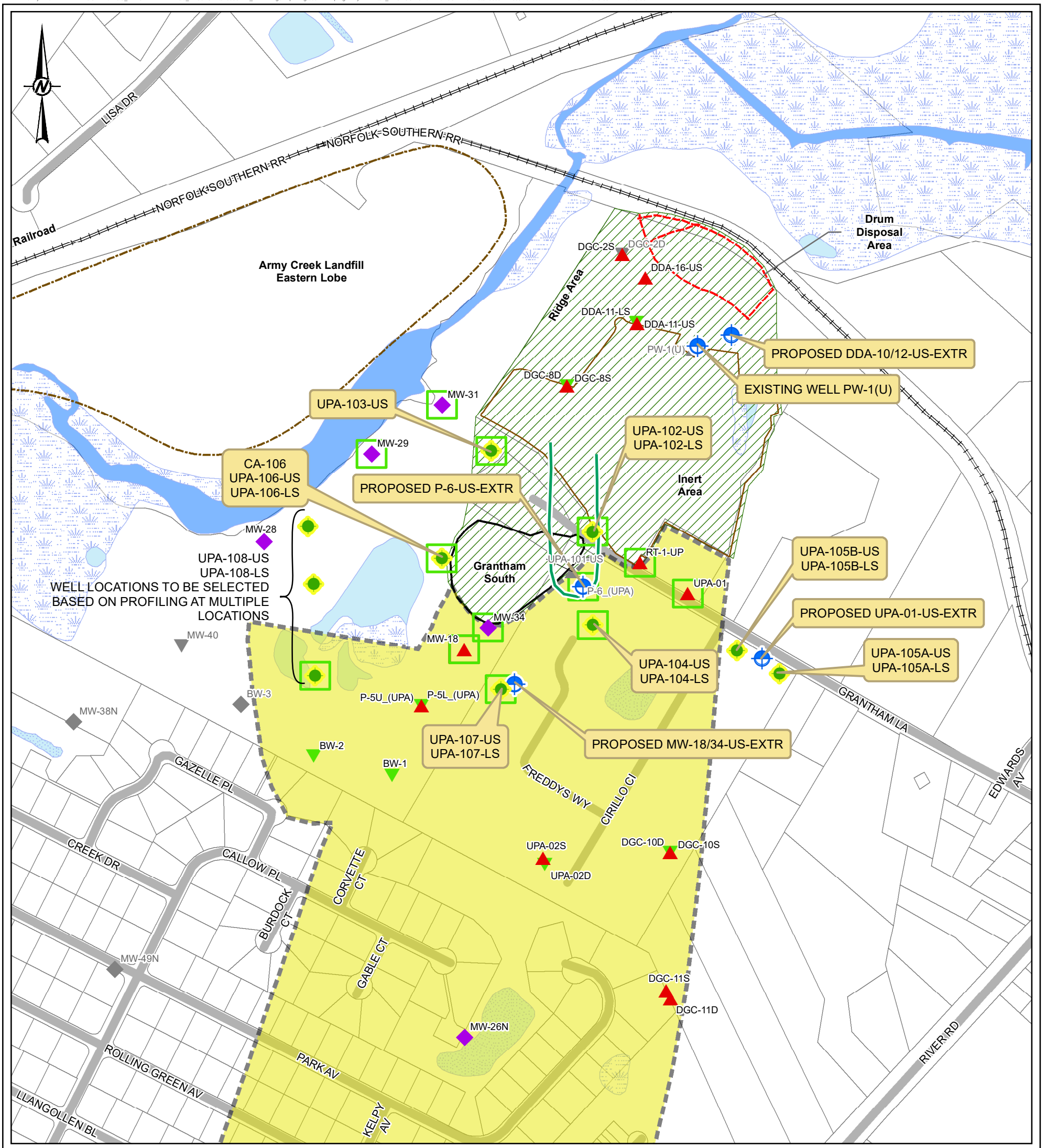
PROJECT
SAMPLING AND ANALYSIS PLAN

CONSULTANT
GOLDER

YYYY-MM-DD	2018-08-10
DESIGNED	MBS
PREPARED	AM
REVIEWED	BAR
APPROVED	TAM

TITLE
AQUIFER TESTING - TRANSDUCER LOCATIONS - STEP 6
PROPOSED EXTRACTION WELL UPA-01-US-EXTR

PROJECT NO.	CONTROL	REV.	FIGURE
013-6052.14	0004-013	0	A-6F



LEGEND

- Proposed Extraction Well
- Proposed Assessment Location
- Well screened in UPA Upper Sand included in the Proposed 2018 Monitoring Program
- Well screened in UPA Upper Sand not included in the Proposed 2018 Monitoring Program
- Well screened in UPA Lower Sand included in the Proposed 2018 Monitoring Program
- Well screened in UPA Lower Sand not included in the Proposed 2018 Monitoring Program
- Well screened across UPA Upper Sand and Lower Sand included in the Proposed 2018 Monitoring Program
- Well screened across UPA Upper Sand and Lower Sand not included in the Proposed 2018 Monitoring Program
- Modeled Feature in the Well P-6 area
- Transducer Study Location
- Pumping Test Well
- Area of Attainment - The source of contaminated groundwater detected in well BW-2 remains under evaluation
- Waste Management Area

REFERENCE(S)

1. BASE DATA FROM NEW CASTLE COUNTY DELAWARE, DEPARTMENT OF LAND USE, "EPARCEL VIEW MAP" WEB SITE GIS DATA DOWNLOAD. DATA ACQUIRED 01/18/2012.

0 400 800

1 " = 400 feet Feet

IF THIS MEASUREMENT DOES NOT MATCH WHAT IS SHOWN, THE SHEET SIZE HAS BEEN MODIFIED FROM: ANSI B

1 in

0

CLIENT
DELAWARE SAND & GRAVEL SUPERFUND SITE
NEW CASTLE COUNTY, DELAWARE

PROJECT
SAMPLING AND ANALYSIS PLAN

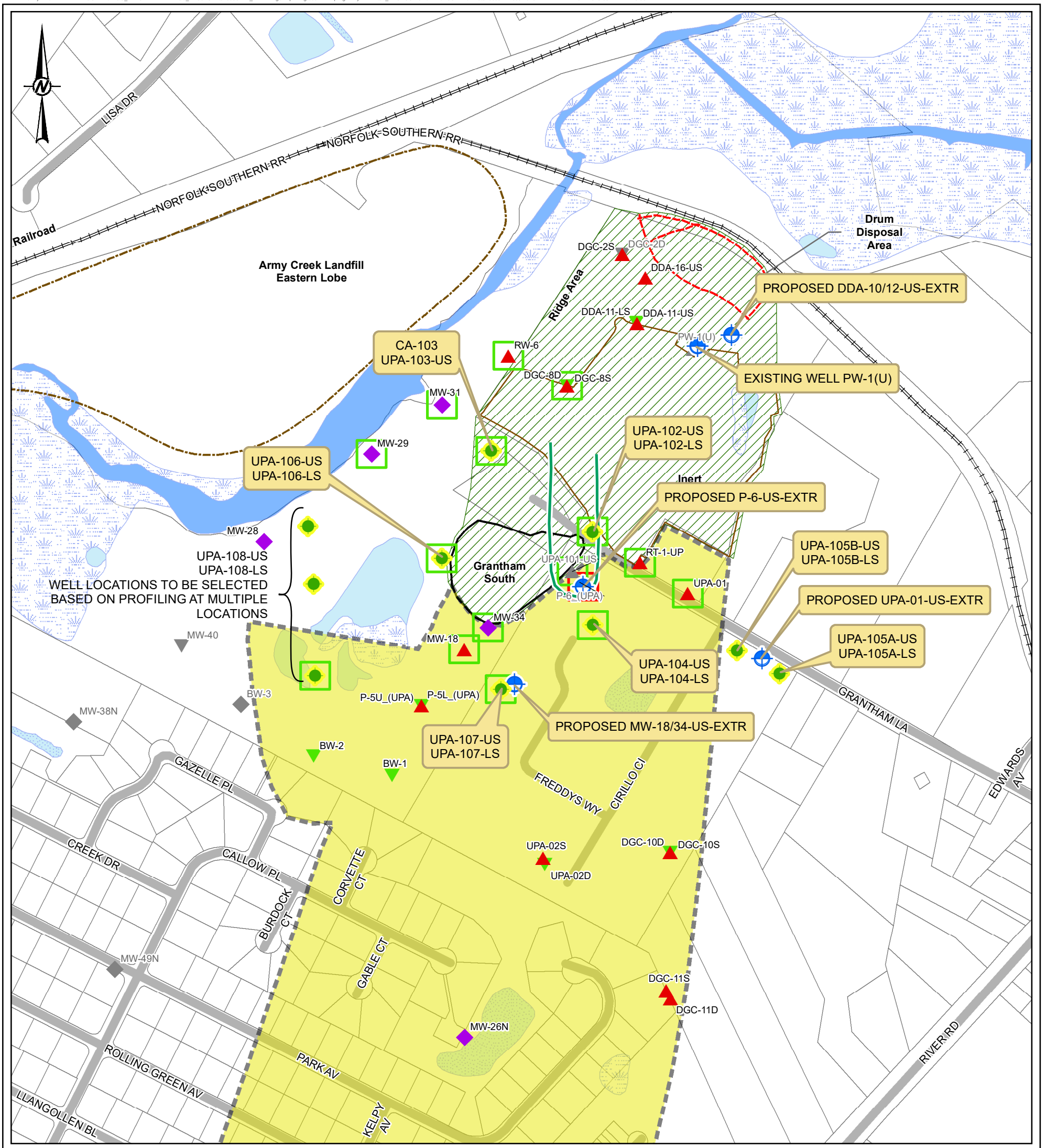
CONSULTANT
GOLDER

YYYY-MM-DD	2018-08-10
DESIGNED	MBS
PREPARED	AM
REVIEWED	BAR
APPROVED	TAM

TITLE
AQUIFER TESTING - TRANSDUCER LOCATIONS - STEP 7
PROPOSED EXTRACTION WELL MW-18/34-US-EXTR

PROJECT NO.	CONTROL	REV.	FIGURE
013-6052.14	0004-014	0	A-6G

PATH: W:\Projects\DelawareSand&Gravel\DS&G_NewCastleDelaware\99_PROJECTS\0136052.14_Monitoring&Reporting\0004-SamplingAnalysisPlan\02_PRODUCTION\MXD\FIGURES\Rev0\013605214-0004-015.mxd PRINTED ON: 2018-08-17 AT: 4:48:33 PM



LEGEND

- Proposed Extraction Well
- Proposed Assessment Location
- Well screened in UPA Upper Sand included in the Proposed 2018 Monitoring Program
- Well screened in UPA Upper Sand not included in the Proposed 2018 Monitoring Program
- Well screened in UPA Lower Sand included in the Proposed 2018 Monitoring Program
- Well screened in UPA Lower Sand not included in the Proposed 2018 Monitoring Program
- Well screened across UPA Upper Sand and Lower Sand included in the Proposed 2018 Monitoring Program
- Well screened across UPA Upper Sand and Lower Sand not included in the Proposed 2018 Monitoring Program
- Modeled Feature in the Well P-6 area
- Transducer Study Location
- Pumping Test Well
- Area of Attainment - The source of contaminated groundwater detected in well BW-2 remains under evaluation
- Waste Management Area

REFERENCE(S)

1. BASE DATA FROM NEW CASTLE COUNTY DELAWARE, DEPARTMENT OF LAND USE, "EPARCEL VIEW MAP" WEB SITE GIS DATA DOWNLOAD. DATA ACQUIRED 01/18/2012.

0 400 800

1 " = 400 feet Feet

CLIENT
DELAWARE SAND & GRAVEL SUPERFUND SITE
NEW CASTLE COUNTY, DELAWARE

PROJECT
SAMPLING AND ANALYSIS PLAN

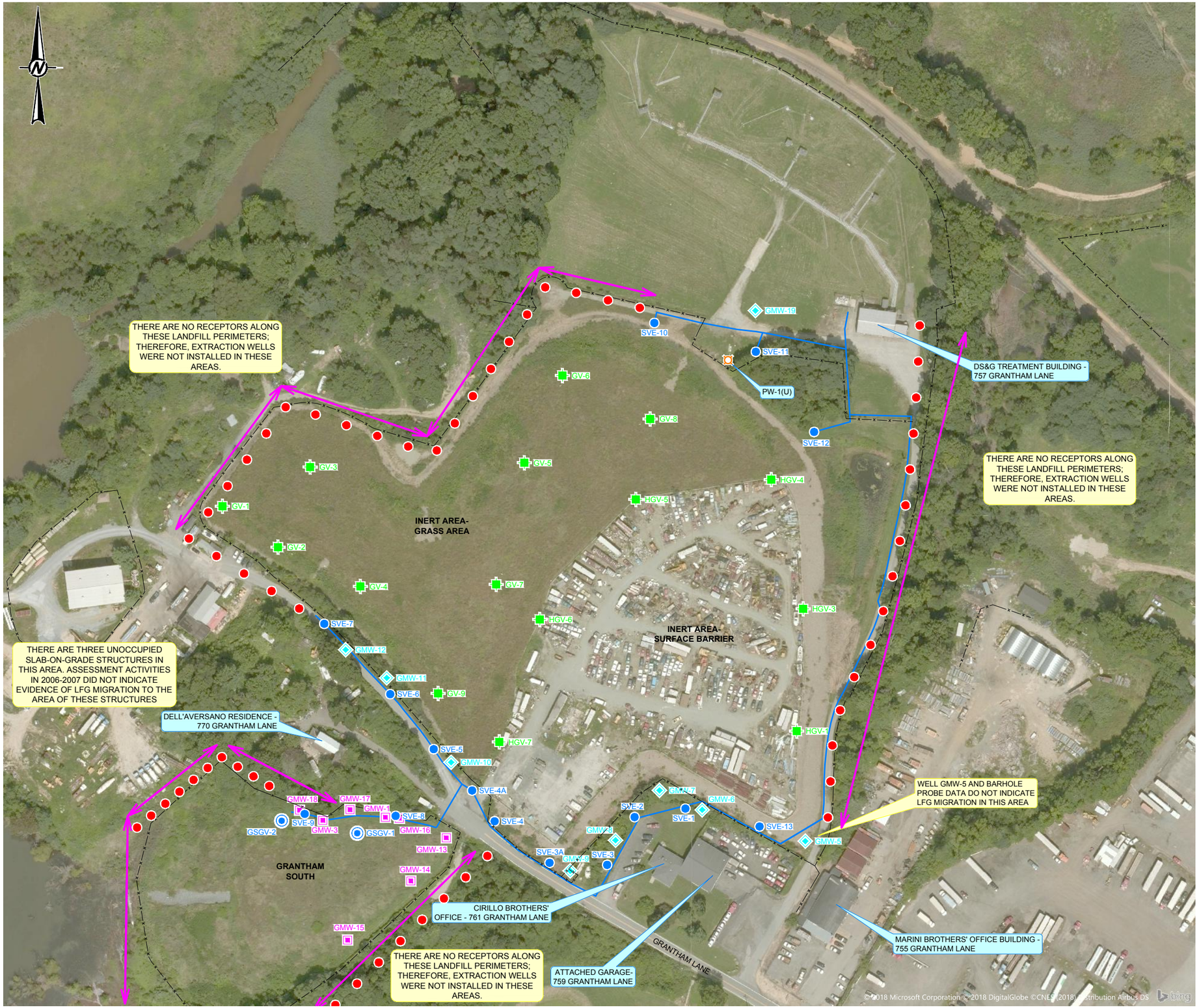
CONSULTANT
GOLDER

YYYY-MM-DD	2018-08-10
DESIGNED	MBS
PREPARED	AM
REVIEWED	BAR
APPROVED	TAM

TITLE
AQUIFER TESTING - TRANSDUCER LOCATIONS - STEP 8
EXISTING WELL P-6

PROJECT NO.	CONTROL	REV.	FIGURE
013-6052.14	0004-015	0	A-6H

IF THIS MEASUREMENT DOES NOT MATCH WHAT IS SHOWN, THE SHEET SIZE HAS BEEN MODIFIED FROM: ANSI B
1 in
0



LEGEND

- INERT AREA PERIMETER GAS MONITORING WELL (SEE REFERENCE 2)
- INERT AREA GAS VENT (SEE REFERENCE 2)
- GRANTHAM SOUTH PERIMETER GAS MONITORING WELL (SEE REFERENCE 2)
- EXISTING GRANTHAM SOUTH GAS VENT (SEE REFERENCE 2)
- CONVEYANCE PIPE ROUTE
- LANDFILL GAS EXTRACTION WELL
- PROPERTY LINE
- FENCE
- EXISTING PW-1(U) EXTRACTION WELL (SEE REFERENCE 4)
- LANDFILL PERIMETER ALONG WHICH THERE ARE NO RECEPTORS
- POTENTIAL BARHOLE LOCATIONS

REFERENCE(S)

1. BASE TOPOGRAPHY TAKEN FROM AUTOCAD FILE PREPARED BY TAYLOR WISEMAN & TAYLOR, WEST CHESTER, PA . CADD FILE WC-60044-4 DATED JULY 17, 1997.
2. MONITORING LOCATIONS SHOWN WERE TAKEN FROM PDF OF FIGURE ENTITLED "INERT AREA SITE BASE MAP," DATED MARCH 2014, BY ENVIRONMENTAL ALLIANCE, INC.
3. FENCE TAKEN FROM AUTOCAD FILE PREPARED BY TAYLOR WISEMAN & TAYLOR, WEST CHESTER, PA. CADD FILE WC-60044-4 DATED 07/17/97.
4. EXISTING PW-1(U) DISCHARGE LINE TAKEN FROM CADD DRAWING ENTITLED "POTENTIAL DISCHARGE PIPING LOCATIONS - ON PROPERTY," DATED 05-16-2016, BY GOLDER ASSOCIATES.

0 80 160
1" = 80' FEET

CLIENT
DELAWARE SAND & GRAVEL SUPERFUND SITE
NEW CASTLE COUNTY, DELAWARE

PROJECT
SAMPLING AND ANALYSIS PLAN

TITLE
BAREHOLE PROBE INVESTIGATION AREA

CONSULTANT
GOLDER

YYYY-MM-DD	2018-08-10
DESIGNED	MBS
PREPARED	GLS
REVIEWED	BAR
APPROVED	TAM

PROJECT NO.	CONTROL	REV.	FIGURE
0136052.14	0004-010	0	A-7

IF THIS MEASUREMENT DOES NOT MATCH WHAT IS SHOWN, THE SHEET SIZE HAS BEEN MODIFIED FROM: ANSI D

0 1 in

Path: \\ntlaurefcadd\Projects\Delaware\Sand&Gravel\DS&G_NewCastle\Delaware\99_PROJECTS\0136052.14_Monitoring&Reporting\0004-SamplingAnalysisPlan\02_PRODUCTION\DWG\ | File Name: 01360521 4-0004-010.dwg | Last Edited By: gsantell Date: 2018-08-09 Time: 1:02:07 PM | Printed By: AMorales Date: 2018-08-13 Time: 10:42:20 AM

ATTACHMENT A

Standard Operating Procedures (SOPs) for PFAS Monitoring Events

1.0 GENERAL APPLICABILITY

The purpose of this Standard Operating Procedure (SOP) is to describe the procedures that shall be used during implementation of this per- and polyfluoroalkyl substances (PFAS) sampling program.

Due to the extremely low method detection limits associated with PFAS analysis (i.e., nanograms per liter [ng/l]) and the many potential sources of trace levels of PFAS, field personnel shall employ the greatest caution by strictly following the protocols described herein. Frequent replacement of nitrile gloves and decontamination of non-dedicated sampling equipment in accordance with the appropriate procedures will reduce the potential for false detections of PFAS.

This SOP includes the following:

- Considerations regarding food packaging and food consumption during PFAS sampling programs
- Field gear and clothing restrictions
- Personal hygiene requirements
- Sample area access restrictions
- Field equipment decontamination

Some of the provisions of the PFAS sampling program requirements described herein may have resulted in modifications to standard health and safety procedures (e.g., use of insect repellant or sunscreen). The site-specific Health and Safety Plan (HASP; see Appendix B of the PDIWP) was prepared to address any potential conflicts between the requirements described herein and standard health and safety procedures.

2.0 RESPONSIBILITIES

The Field Team Leader and field personnel have the shared responsibility to oversee and ensure that the PFAS sampling program is performed in accordance with the program-specific protocols described in this SOP. The Field Team Leader shall ensure that on-site personnel, including subcontractors and third parties that may have direct access to the sampling area, understand and comply with this SOP. Field personnel shall be notified of these requirements a minimum of three days prior to the start of field work in order to have the time to appropriately comply with many of the food and clothing requirements prior to arriving at the site.

3.0 GENERAL FIELD METHODS

3.1 Food Consumption

Components of some food packages have been treated to resist wetting. Historically, this is achieved through the use of PFAS. Accordingly, field personnel shall avoid the use of paper bags and other paper packaging to transport food to the site, including pre-wrapped foods and snacks (e.g., chocolate bars, energy bars, granola bars, potato chips, etc.). Field personnel shall not bring any fast food to the site that uses any form of paper wrapping such as sandwiches or paper drinking cups. If possible, field personnel shall use hard plastic or stainless-steel food containers. Field personnel shall not use aluminum foil, wax paper, or coated textiles to transport food to the site.

The Teflon® coating on some frying pans contains fluorinated compounds and as such represents a potential source of PFAS. Field personnel shall not transport to or consume food at the site that has been prepared using a Teflon® coated cooking utensil.

Field personnel shall not consume food or beverages in the field vehicle or in the immediate vicinity of the sample location. Prior to consuming food or beverages, field personnel shall remove their nitrile gloves and coveralls and move to a location a minimum distance of 35 feet away from the sample location, preferably in the downwind direction. When finished eating or drinking, field personnel shall wash their hands, put their coveralls back on and put on a new pair of nitrile gloves prior to returning to the work area.

3.2 Field Gear and Clothing Restrictions

Because treatments to provide water resistant, water proof, or stain-resistant clothing include the use of PFAS, field personnel shall not wear any water resistant, water proof, stain-resistant treated clothing or Tyvek clothing during the field program. Permissible field clothing for PFAS sampling programs includes clothing made from natural fibers, preferably cotton. Clothing made of synthetic fibers shall be avoided (i.e., reflective vests).

Field clothing shall be laundered with a minimal amount of detergent and no fabric softener or scented products shall be used. Once field clothing has been washed appropriately, field clothing shall be washed a second time on a rinse-only cycle, using only water, prior to drying. Anti-static dryer sheets shall not be used when drying field clothing. Field clothing shall preferably be old cotton clothing that has been laundered many times, as new clothing may contain PFAS related treatments. Clothing containing Gore-Tex™ shall not be worn during the sampling program, as Gore-TexTM clothing contains a PFAS membrane.

Waterproof field books shall not be used; field notes shall be recorded on loose paper using aluminum clip boards. Plastic clip boards, self-sticking notes, binders or spiral hard cover notebooks shall not be used. Field notes shall be recorded in pen or pencil. Markers shall not be used.

Most safety footwear is constructed of leather and synthetic materials that have been treated to provide some degree of waterproofing and/or increased durability. Therefore, footwear materials represent a potential source of trace PFAS. Field personnel contact with safety footwear including donning footwear or tying laces shall not occur within 35-feet of the sampling area. If footwear must be adjusted, field personnel shall re-locate to an area a minimum of 35-feet from the sampling area, preferably in a downwind direction, and make the necessary adjustments. Nitrile gloves shall be worn when contacting footwear. The nitrile gloves worn while contacting footwear shall be removed and new nitrile gloves shall be put on prior to re-entering the sampling area.

Disposable nitrile gloves shall be worn at all times. A new pair of nitrile gloves shall be donned prior to the following activities at each sample location:

- Contact with laboratory-suppled sample containers or PFAS-free water containers
- Decontamination of sampling equipment
- Insertion of anything into the well (e.g., HDPE tubing, HydraSleeve, bailer, etc.)
- Insertion of silicon tubing into the peristaltic pump
- Completion of monitoring well purging
- Sample collection

- Handling of QA/QC samples including field blanks and equipment blanks
- After the handling of any non-dedicated sampling equipment or contact with non-decontaminated surfaces

Because field vehicle seats may have been treated with PFAS-containing products for stain resistance, the seats of field vehicles shall be covered with a well laundered cotton sheet or blanket for the duration of the field program in order to avoid direct contact between field personnel clothing and vehicle seat fabric. Measures taken to mitigate field personnel contact with field vehicle seat fabric shall not in any way interfere with the functionality or impede the use of vehicle safety belts.

3.3 Personal Hygiene

Field personnel shall not use shampoo, conditioner, body gel, cosmetic cream, or hand cream as part of their personal showering routine on the day of a sampling event, as these products may contain surfactants and represent a potential source of PFAS. Field personnel shall follow their normal hygiene routine the night before a sampling event and then rinse with water only the morning before a sampling event. The use of bar soap is acceptable; however, bar soap including moisturizers shall be avoided.

Field personnel shall not use moisturizers, cosmetics, dental floss, sunscreen, and/or insect repellent for the duration of the field program, either on-site or off-site, as these products may contain trace PFAS. Appropriate accommodation to address the prohibition of the use of these substances must be incorporated into a site-specific HASP.

3.4 Sample Area Access

Visitors, including contractors or site personnel, who are not following these general PFAS sampling program protocols shall not be allowed to approach within 35 feet of the sample area until PFAS sample collection activities are complete and the PFAS sample container has been sealed and placed in the sample cooler.

3.5 Field Equipment Decontamination

Use the procedures in this section to decontaminate all non-dedicated sampling equipment (e.g., submersible pumps, bladder pump components, tubing shears, etc.) used to collect samples:

- Rinse thoroughly with Citranox solution
- Rinse thoroughly with de-ionized (DI) water (certified PFAS free)
- Rinse with methanol (certified PFAS free)
- Rinse with DI water
- Allow to air dry

Submersible pumps used to sample PFAS, will be decontaminated at the monitoring well location to be sampled. Once the pump is decontaminated, it will be placed directly into the monitoring well and lowered to the sample interval to avoid accidental PFAS cross-contamination. Decontamination fluids used to clean equipment including Citranox, DI water, and methanol shall not be reused during field decontamination and shall be collected and discharged to the publicly-owned treatment works at the on-site treatment building.

1.0 GENERAL APPLICABILITY

The purpose of this Standard Operating Procedure (SOP) is to describe the procedures that shall be followed during monitoring well purging and the collection of groundwater samples for analysis of per- and polyfluoroalkyl substances (PFAS).

This SOP includes the following:

- Monitoring Well Groundwater Elevation Measurement
- Monitoring Well Purge
- Sample Container Considerations
- Groundwater Sample Collection Procedures
- Sample Shipping Requirements

With the exceptions provided in these SOPs, field personnel shall follow the monitoring well purge protocols included in the Pre-Design Investigation Work Plan (PDIWP) Sampling and Analysis Plan (SAP) dated August 2018. Sampling depths for the monitoring wells is included in this sampling program are included in the PDIWP SAP.

2.0 RESPONSIBILITIES

The Field Team Leader and field personnel have the shared responsibility to oversee and ensure that the monitoring well purge and PFAS groundwater sampling program is performed in accordance with the program-specific protocols described in this SOP. The Field Team Leader shall ensure that field personnel understand and comply with this SOP.

3.0 PURGING AND SAMPLING PROCEDURES

3.1 Water Level Measurement

Under normal conditions, the first step in conducting a groundwater sampling program is to collect a synoptic round of static water level measurements and monitoring well sounded depths. However, due to the extremely low detection limits for PFAS, collection of a synoptic round of groundwater elevation measurements shall only be conducted after the groundwater sampling program has been completed to help mitigate the possibility of cross-contamination.

Field personnel shall record a depth to water measurement in each well prior to initiating well purge procedures.

3.2 Monitoring Well Purge

Field personnel shall not use Teflon® or low-density polyethylene (LDPE) tubing or other equipment containing these materials for purging or sample collection. High-density polyethylene (HDPE) tubing is preferred. Field personnel shall not re-use materials between well sample locations. Following completion of monitoring well purge activities at a monitoring well location, field personnel shall place all disposable materials in heavy-duty (i.e., lawn waste) garbage bags for disposal. Field personnel shall wear nitrile gloves at all times.

Field personnel shall purge monitoring wells using a submersible pump and HDPE tubing. Field personnel shall inquire of the manufacturer and identify a submersible pump model whose construction does not include any

Teflon® components (e.g., check balls, O-rings, compression fittings, etc.). New HDPE tubing shall be used to purge groundwater at each bedrock well. Field personnel shall determine and cut the appropriate length of HDPE tubing to be used in each well using the previously measured arm span of the individual performing the monitoring well purge to avoid contact with any materials other than the well and submersible pump. Field personnel shall decontaminate non-dedicated components and sampling equipment (including pumps, tubing shears, etc.) in accordance with SOP-1 between well purge locations.

Purge water shall be collected and discharged to the publicly-owned treatment works at the on-site treatment building.

3.3 Sample Containers

Groundwater samples shall be collected in HDPE sample containers provided by the laboratory specifically for use in the collection samples for analysis of PFAS (i.e., HDPE without a Telfon® liner). Glass containers shall not be used due to the potential for loss of PFAS through adsorption.

Groundwater sample container lids shall remain on the sample container until immediately prior to sample collection and lids shall be resealed immediately following sample collection. Field personnel shall hold the sample container lid in their hand until the lid is replaced on the sample container. Field personnel shall not rinse groundwater sample container bottles during groundwater sample collection. Groundwater sample container labels shall be completed using a pen or a pencil after the lid has been re-secured on the sample container. Field personnel shall not use markers to complete sample container labels.

3.4 Sample Collection

With the exceptions provided in these SOPs, field personnel shall follow the groundwater sampling protocols included in the PDIWP SAP dated August 2018. Field personnel shall wash their hands and put on a new pair of nitrile gloves prior to sample collection. Once the nitrile gloves are put on, field personnel shall not handle papers, pens, clothes, etc. prior to the collection of groundwater samples. If field personnel need to take notes or handle anything other than the sample container prior to collecting the sample, the old nitrile gloves with which contact was made shall be removed and new nitrile gloves put on.

Field personnel shall hold the sample container in such a manner that the sample container does not come in direct contact with the HDPE tubing or pump equipment. The sampling container shall be filled completely. If field personnel observe suspended solids in the collected groundwater sample, a new sample shall be collected, if possible. If it is not possible to collect a sample with minimal suspended solids (i.e., no evidence of solids settling at the bottom of the sampling container), field personnel shall contact the project manager and, if the sample is submitted for analysis, indicate the presence of suspended solids as a note on the chain-of-custody.

Groundwater samples shall be placed directly into the laboratory-supplied HDPE containers. Once the groundwater sample container lid has been resealed, groundwater sample containers are to be placed into individual new Ziploc® storage bags. Following groundwater sample collection, groundwater sample containers enclosed within their Ziploc® storage bags shall be placed on ice in the laboratory-provided sample cooler. Field personnel shall minimize sample exposure to sunlight during sample handling and storage.

All sampling materials shall be treated as single use and disposed of following completion of groundwater sampling at each monitoring well location.

3.5 Sample Shipping

Groundwater sample containers shall be stored on ice and maintained at approximately 4 degrees Celsius (ºC) and transported by overnight courier to the laboratory. Field personnel shall only use new, fresh ice. Reusable chemical or gel ice packs shall not be used, as these may contain PFAS. Tracking numbers for all shipments shall be provided once the sample coolers have been shipped to ensure their timely delivery.

1.0 GENERAL APPLICABILITY

The purpose of this Standard Operating Procedure (SOP) is to describe the Quality Assurance / Quality Control (QA/QC) samples that shall be collected during a per- and polyfluoroalkyl substances (PFAS) sampling program.

This SOP includes protocols for the collection of the following QA/QC samples:

- Equipment/Rinsate Blanks
- Field Duplicates
- Field Blanks
- Trip Blanks
- Analytical QA/QC

2.0 RESPONSIBILITIES

The Field Team Leader and field personnel have the shared responsibility to oversee and ensure that the PFAS QA/QC sampling program is performed in accordance with the program-specific protocols described in this SOP. The Field Team Leader shall ensure that field personnel understand and comply with this SOP.

Field personnel shall inquire of the submersible pump manufacturer and identify a pump model whose construction does not include any Teflon® components (e.g., check balls, O-rings, compression fittings, etc.).

3.0 QA/QC PROTOCOLS

3.1 Equipment/Rinsate Blanks

Equipment/Rinsate blanks shall be collected at a rate of one per setup per event for non-dedicated sampling equipment (i.e., submersible pumps). Equipment/rinsate blanks shall be collected using laboratory-supplied PFAS-free water and shall be collected in laboratory-supplied high density polyethylene (HDPE) containers.

After decontamination of the submersible pump in accordance with the procedure described in SOP-1, equipment/rinsate blanks will be collected by pouring the laboratory-supplied PFAS-free water into a new and unused HDPE sample bottle and then pumping the PFAS-free water through new HDPE tubing and new silicon tubing with the submersible pump into the sample container. When the sample container is full, replace the sample container lid and re-seal. Equipment/rinsate blank container lids shall remain in the hand of field personnel until replaced on the sample container. Sample container labels shall be completed using a pen or pencil after the sample container lid has been resealed. Field personnel shall not use markers to complete sample container labels.

3.2 Field Duplicates

Field personnel shall collect one blind field duplicate for every 20 primary field samples collected. Field personnel shall collected field duplicates immediately after collection of the primary field samples. Field duplicates shall be collected in the laboratory-supplied PFAS-free HDPE sample containers. Field duplicate container lids shall remain in the hand of field personnel until replaced on the sample container. Sample container labels shall be completed as described above.

Field personnel shall collect groundwater field duplicates for analysis of PFAS using the following procedures:

- Field personnel shall stabilize groundwater parameters in accordance with the PDIWP SAP and SOP-2.
- Field personnel shall collect the primary sample in accordance with the PDIWP SAP and SOP-2.
- Following collection of the primary sample, change gloves and prepare to collect the field duplicate.
- Field duplicates shall be collected immediately following collection of the primary sample.
- Completely fill the laboratory-provided HDPE groundwater sample container.
- Replace and re-seal the lid on the groundwater sample containers and then complete the sample container label as described above.

3.3 Field Blanks

Field personnel shall submit of one field blank per day of sampling. Field blanks shall consist of PFAS-free water containerized in an HDPE sample container filled at the laboratory prior to beginning the field program. Field blank sample containers shall be opened during the collection of a sample and the laboratory-supplied PFAS-free water contained therein shall be poured directly into a laboratory-supplied HDPE sample container, then resealed. Field blank container lids shall remain in the hand of field personnel until replaced on the sample container. Sample container labels shall be completed as described above.

3.4 Trip Blanks

Field personnel shall submit one laboratory-supplied trip blank per day of sampling. Trip blanks shall consist of PFAS-free water containerized in an HDPE sample container filled at the laboratory prior to the beginning of the field program. Field personnel shall place one trip blank container in the sample cooler at the beginning of the day and the trip blank shall remain in the cooler for the duration of sampling activities conducted on that day. Trip blank containers shall be submitted to the laboratory with the daily field sample shipment.

3.5 Analytical QA/QC

Internal laboratory QA/QC shall consist of one laboratory blank and one matrix spike / matrix spike duplicate (MS/MSD) for every 20 primary field samples collected for analysis. Field personnel shall collected MS/MSDs immediately after collection of the primary field samples as described above for field duplicates.

As part of the internal QA/QC, relative percent difference (RPD) shall be calculated between samples and corresponding field or laboratory duplicates. The laboratory quality assurance portion of the laboratory certificates shall be reviewed to verify that all calculations/recoveries were within acceptable limits as established by the laboratory method.

3.6 Sample Shipping

QA/QC samples shall be maintained at a temperature between 0 and 4 ºC during shipping. Only new, fresh ice may be used in sample coolers. Field personnel shall not use reusable chemical or gel ice packs, as these may contain PFAS. Samples shall be shipped via courier service with priority overnight delivery. Tracking numbers for all shipments shall be provided once they have been sent out so to ensure their timely delivery.

ATTACHMENT B

Low-Flow Groundwater Purge/Sample Field Information Form

LOW FLOW GROUNDWATER PURGE/SAMPLE FIELD INFORMATION FORM

Site _____

Location: _____

Project Number: _____ Meter/Type/Serial #: _____

MONITORING WELL ID: _____ Meter Calibrated @: _____

Depth to Water Prior to Purging [ft-bmp]: _____ pH Calibration Check Measurment/Time*: _____

Well Casing Diameter [in]: _____ Sampling Date/Time: _____

Start Time (purging): _____ Sampler(s): _____

Purging Device: _____ Sampling Device: _____

Pump intake setting: _____ Sampling Purge Rate: _____

Well Screen Interval: _____ Sample Characteristics: _____

As-Built Construction Well Depth [ft-bmp]: _____ PID Measurement of Well Headspace (ppm): _____

Sounded Well Depth [ft-bmp]: _____ Analytical Parameters: _____

Weather Conditions:_____ Fe+2 result (field measurement):

Time [hh:mm]	**Temperature** [^{o}C]	**pH** [std]	**Specific Conductance** [S/m] or [mS/cm]	**Turbidity** [ntu]	**Dissolved Oxygen** [mg/l]	**Redox Potential** Note - Indicate if (+) or (-) [mV]	**Depth To Water** [ft-bmp]	**Volume Purged** [liters]	**Approximate Purge Rate** [ml/min]	**Observations** (PID readings, sample characteristics, equipment problems, etc.)

Comments: * If the pH check differs by more than ± 0.2 S.U from the temperature adjusted pH buffer value, then the instrument will need to be recalibrated.

Signature:

NJ Cert: 03027

ATTACHMENT C

Volume Average Groundwater Purge/Sample Field Information Form

WATER SAMPLE FIELD INFORMATION FORM

Site:

Location:

Project Number:

Sampling Team:

Sample Point ID: Date:

Time Start: Time Finish:

Depth to water before purging (ft-bmp)

Casing diameter (in) Meter/Type/Serial #: Horiba U-52 #

Purging device: Meter Calibrated @: cal check @

Pump intake setting:

Well screen interval:

As-built construction Well Depth (ft-bpm):

Sounded well depth (ft-bmp):

Casing volume (gal)

Volume purged (gal)

Depth to water after purging (ft-bmp)

Remarks: Volume-averaging (conventional) - purge 3 to 5 well volumes

PID Measurement of Well Headspace (ppm): Fe+2 result (field measurement in ppm):

Casing Volume Calculation

2"	4"	6"	8"
0.163 gal/ft	0.653 gal/ft	1.47 gal/ft	2.61 gal/ft

Well Volume = ((well depth) - (water level)) * (casing volume/ft)

FIELD MEASUREMENTS							Units	Calibration Notes
Time	1)	2)	3)	4)	5)			
Temp.	1)	2)	3)	4)	5)		**ºC**	
pH	1)	2)	3)	4)	5)		**std. units**	
Sp. Cond	1)	2)	3)	4)	5)		**mS/cm**	
Turbidity	1)	2)	3)	4)	5)		**ntu**	
DO	1)	2)	3)	4)	5)		**mg/L**	
ORP	1)	2)	3)	4)	5)		**mV**	
Water Level	1)	2)	3)	4)	5)		**ft-bmp**	
Volume	1)	2)	3)	4)	5)		**gal**	

Sample Collection Notes:

Weather conditions at time of sampling:

Analytical Parameters:

Sample characteristics:

Sample date / time: @ Sampling Device:

Sample sequence:

Signature: Company: **Golder Associates Inc.** Date:

ATTACHMENT D

Barhole Probe Advancement and Monitoring Form

Barhole Probe Monitoring Form

					Probe Measurements								
	Barhole ID	Location	Date	Time	% LEL	% CH_4	CO_2 (ppm)	H_2S (ppm)	CO (ppm)	% O_2	Depth of Probe (ft)	Purge Time (min)	Comments
1													
2													
3													
4													
5													
6													
7													
8													
9													
#													
#													
#													
#													
#													
#													

Meter Type and Calibration:

Notes:

ATTACHMENT E

TestAmerica

E-1

Laboratory Quality Manual

Quality Assurance Manual Cover Page

TestAmerica Edison
777 New Durham Road
Edison, NJ 08817
Phone No. 732-549-3900
Fax No. 732-549-3679
www.testamericainc.com

Copyright Information:
This documentation has been prepared by TestAmerica Laboratories, Inc. and its affiliates ("TestAmerica"), solely for their own use and the use of their customers in evaluating their qualifications and capabilities in connection with a particular project. The user of this document agrees by its acceptance to return it to TestAmerica upon request and not to reproduce, copy, lend, or otherwise disclose its contents, directly or indirectly, and not to use it for any purpose other than that for which it was specifically provided. The user also agrees not to give access to this document to any third parties including but not limited to consultants, unless such third parties specifically agree to these conditions.

THIS DOCUMENT CONTAINS VALUABLE CONFIDENTIAL AND PROPRIETARY INFORMATION. DISCLOSURE, USE OR REPRODUCTION OF THESE MATERIALS WITHOUT THE WRITTEN AUTHORIZATION OF TESTAMERICA IS STRICTLY PROHIBITED. THIS UNPUBLISHED WORK BY TESTAMERICA IS PROTECTED BY STATE AND FEDERAL LAW OF THE UNITED STATES. IF PUBLICATION OF THIS WORK SHOULD OCCUR THE FOLLOWING NOTICE SHALL APPLY:

©COPYRIGHT 2017 TESTAMERICA LABORATORIES, INC. ALL RIGHTS RESERVED.

Uncontrolled Copy

Facility Distribution No. ELECTRONIC	Distributed To: FACILITY INTRANET

Title Page:

Quality Assurance Manual
Approval Signatures

Laboratory Director – Mark Acierno	12/19/2017 Date
Quality Manager - Carl Armbruster	12/19/2017 Date
Technical Manager, Organics – Sylvanus Klusey	12/19/2017 Date.
Technical Manager, Inorganics – Donald Evans	12/19/2017 Date

Uncontrolled Copy

SECTION 2. TABLE OF CONTENTS

Sec. No.	Title	2009 TNI Standard Reference	ISO/IEC 17025:2005 (E) Reference	Page No.
-	QUALITY ASSURANCE MANUAL COVER PAGE	V1M2 Sec. 4.2.8.3		1
1.0	TITLE PAGE			2
2.0	TABLE OF CONTENTS	V1M2 Secs. 4.2.8.3-4.2.8.4		3
3.0	INTRODUCTION, SCOPE AND APPLICABILITY	V1M2 Sec. 4.2.8.4		13
3.1	Introduction And Compliance References	V1M2 Secs. 1.1; 1.2; 2.0; 3.2; 4.1.2; 4.2.4	4.1.2; 4.2.4	13
3.2	Terms And Definitions	V1M2 Secs. 3.0; 4.2.4	4.2.4	14
3.3	Scope / Fields Of Testing	V1M2 Secs. 1.2; 4.2.4	4.1.2; 4.2.4	14
3.4	Management Of The Manual	V1M2 Secs. 4.2.1; 4.2.7; 4.3.3.2; 4.3.3.3	4.2.1; 4.2.7; 4.3.3.2; 4.3.3.3	15
4.0	MANAGEMENT REQUIREMENTS	V1M2 Sec. 4		15
4.1	Overview	V1M2 Secs. 4.1.1, 4.1.3; 4.1.5	4.1.1; 4.1.3; 4.1.5; 4.2.6	15
4.2	Roles And Responsibilities	V1M2 Secs. 4.1.4; 4.1.5; 4.1.6; 4.2.1; 4.2.6; 5.2.4	4.1.3; 4.1.5; 4.1.6; 4.2.1; 4.2.6; 5.2.4	15
4.3	Deputies	V1M2 Secs. 4.1.5; 4.1.7.2; 4.2.7	4.1.5; 4.2.7	29
5.0	QUALITY SYSTEM			35
5.1	Quality Policy Statement	V1M2 Secs. 4.1.5; 4.2.2; 4.2.3; 4.2.8.3	4.1.5; 4.2.2; 4.2.3	35
5.2	Ethics And Data Integrity	V1M2 Secs. 4.1.5; 4.16; 4.2.2; 4.2.8.1; 5.2.7	4.1.5; 4.2.2	35
5.3	Quality System Documentation	V1M2 Secs. 4.1.5; 4.2.2; 4.2.5	4.2.2; 4.2.5	36
5.4	QA/QC Objectives For The Measurement Of Data	V1M2 Sec. 4.2.2	4.1.5; 4.2.2	37
5.5	Criteria For Quality Indicators			39
5.6	Statistical Quality Control			39
5.7	Quality System Metrics			40
6.0	DOCUMENT CONTROL	V1M2 Secs. 4.2.7; 4.3.1; 4.3.2.2 ; 4.3.3.3; 4.3.3.4	4.2.7; 4.3.1; 4.3.2.2; 4.3.3.3; 4.3.3.4	40
6.1	Overview			40
6.2	Document Approval And Issue	V1M2 Secs. 4.3.2; 4.3.2.1-4.3.2.3; 4.3.3.1	4.3.2.1; 4.3.2.2; 4.3.2.3; 4.3.3.1	40

Uncontrolled Copy

Sec. No.	Title	2009 TNI Standard Reference	ISO/IEC 17025:2005 (E) Reference	Page No.
6.3	Procedures For Document Control Policy	V1M2 Secs. 4.3.2.1–4.3.2.2; 4.3.3.1	4.3.2.1; 4.3.2.2; 4.3.3.1	41
6.4	Obsolete Documents	V1M2 Secs. 4.3.2.1–4.3.2.2	4.3.2.1; 4.3.2.2	41
7.0	SERVICE TO THE CLIENT	V1M2 Secs. 4.4.1 - 4.4.4	4.4.1; 4.4.2; 4.4.3; 4.4.4	41
7.1	Overview	V1M2 Secs. 4.4.5; 4.5.5; 5.7.1	4.4.5; 5.7.1	41
7.2	Review Sequence And Key Personnel	V1M2 Sec. 4.4.5	4.4.5	42
7.3	Documentation	V1M2 Sec. 5.7.1	5.7.1	43
7.4	Special Services	V1M2 Secs. 4.7.1-4.7.2	4.7.1; 4.7.2	45
7.5	Client Communication	V1M2 Secs. 4.7.1-4.7.2	4.7.1; 4.7.2	45
7.6	Reporting	V1M2 Secs. 4.7.1-4.7.2	4.7.1; 4.7.2	45
7.7	Client Surveys	V1M2 Secs. 4.7.1-4.7.2	4.7.1; 4.7.2	45
8.0	SUBCONTRACTING OF TESTS	V1M2 Secs. 4.4.3; 4.5.4	4.4.3; 4.5.4	46
8.1	Overview	V1M2 Secs. 4.5.1 - 4.5.3; 4.5.5; 5.3.1	4.5.1; 4.5.2; 4.5.3; 5.3.1	46
8.2	Qualifying And Monitoring Subcontractors	V1M2 Secs. 4.5.1; 4.5.2; 4.5.3; 4.5.5	4.5.1; 4.5.2; 4.5.3	46
8.3	Oversight And Reporting	V1M2 Sec. 4.5.5		47
8.4	Contingency Planning			49
9.0	PURCHASING SERVICES AND SUPPLIES	V1M2 Sec. 4.6.1	4.6.1	49
9.1	Overview	V1M2 Secs. 4.6.2; 4.6.3; 4.6.4	4.6.2; 4.6.3; 4.6.4	49
9.2	Glassware	V1M2 Sec. 5.5.13.1		49
9.3	Reagents, Standards & Supplies	V1M2 Secs. 4.6.2; 4.6.3; 4.6.4	4.6.2; 4.6.3; 4.6.4	49
9.4	Purchase Of Equipment / Instruments / Software			52
9.5	Services			53
9.6	Suppliers			53
10.0	COMPLAINTS	V1M2 Sec. 4.8	4.8	54
10.1	Overview			54
10.2	External Complaints			54
10.3	Internal Complaints			55
10.4	Management Review			55
11.0	CONTROL OF NON-CONFORMING WORK	V1M2 Secs. 4.9.1; 5.10.5	4.9.1; 5.10.5	55
11.1	Overview	V1M2 Secs. 4.9.1; 4.11.3; 4.11.5	4.9.1; 4.11.3; 4.11.5	55

Uncontrolled Copy

Sec. No.	Title	2009 TNI Standard Reference	ISO/IEC 17025:2005 (E) Reference	Page No.
11.2	Responsibilities And Authorities	V1M2 Secs. 4.9.1; 4.11.3; 4.11.5; 5.2.7	4.9.1; 4.11.3; 4.11.5	56
11.3	Evaluation Of Significance And Actions Taken	V1M2 Secs. 4.9.1; 4.11.3; 4.11.5	4.9.1; 4.11.3; 4.11.5	56
11.4	Prevention Of Nonconforming Work	V1M2 Secs. 4.9.4; 4.11.2	4.9.2; 4.11.2	57
11.5	Method Suspension / Restriction (Stop Work Procedures)	V1M2 Secs. 4.9.1; 4.9.2; 4.11.5	4.9.1; 4.9.2; 4.11.5	57
12.0	CORRECTIVE ACTION	V1M2 Sec. 4.11		58
12.1	Overview	V1M2 Secs. 4.9.2; 4.11.1; 4.11.2	4.9.2; 4.11.1; 4.11.2	58
12.2	General	V1M2 Sec. 4.11.2; 4.11.3	4.11.2; 4.11.3	58
12.3	Closed Loop Corrective Action Process	V1M2 Sec. 4.11.2; 4.11.3; 4.11.4; 4.11.6; 4.11.7; 4.12.2	4.11.2; 4.11.3; 4.11.4; 4.12.2	59
12.4	Technical Corrective Actions	V1M2 Sec. 4.11.6		61
12.5	Basic Corrections	V1M2 Secs. 4.11.1; 4.13.2.3	4.11.1; 4.13.2.3	62
13.0	PREVENTIVE ACTION / IMPROVEMENT	V1M2 Secs. 4.10; 4.12.1; 4.12.2	4.10; 4.12.1; 4.12.2	67
13.1	Overview	V1M2 Secs. 4.15.1; 4.15.2	4.15.1; 4.15.2	67
13.2	Management Of Change			68
14.0	CONTROL OF RECORDS	V1M2 Secs. 4.2.7; 4.13.1.1; 4.13.3	4.2.7; 4.13.1.1	69
14.1	Overview	V1M2 Secs. 4.13.1.1; 4.13.1.2; 4.13.1.3; 4.13.1.4; 4.13.2.1; 4.13.2.2; 4.13.2.3; 4.13.3	4.13.1.1; 4.13.1.2; 4.13.1.3; 4.13.1.4; 4.13.2.1; 4.13.2.2; 4.13.2.3	69
14.2	Technical And Analytical Records	V1M2 Sec. 4.13.2.2 - 4.13.2.3	4.13.2.2; 4.13.2.3	72
14.3	Laboratory Support Activities			73
14.4	Administrative Records			74
14.5	Records Management, Storage And Disposal	V1M2 Sec. 4.13.3		74
15.0	AUDITS			75
15.1	Internal Audits	V1M2 Sec. 4.2.8.1; 4.14; 4.14.1; 4.14.2 ; 4.14.3; 4.14.5; 5.9.1; 5.9.2	4.14.1; 4.14.2; 4.14.3; 5.9.1; 5.9.2	75
15.2	External Audits	V1M2 Secs.4.14.2; 4.14.3	4.14.2; 4.14.3; 4.14.4	77

Uncontrolled Copy

Sec. No.	Title	2009 TNI Standard Reference	ISO/IEC 17025:2005 (E) Reference	Page No.
15.3	Audit Findings	V1M2 Secs. 4.14.2; 4.14.3; 4.14.5		78
16.0	MANAGEMENT REVIEWS	V1M2 Sec. 4.1.6; 4.15; 4.15.1; 4.15.2	4.1.6; 4.15.1; 4.15.2	78
16.1	Quality Assurance Report			78
16.2	Annual Management Review	V1M2 Sec. 4.2.2; 4.15.3	4.2.2	78
16.3	Potential Integrity Related Managerial Reviews			79
17.0	PERSONNEL	V1M2 Secs. 5.2; 5.2.1	5.2.1	81
17.1	Overview	V1M2 Secs. 5.2.2; 5.2.3; 5.2.5	5.2.2; 5.2.3; 5.2.5	81
17.2	Education And Experience Requirements For Technical Personnel	V1M2 Secs. 5.2.1; 5.2.3; 5.2.4	5.2.1; 5.2.3; 5.2.4	81
17.3	Training	V1M2 Sec. 5.2.5	5.2.5	82
17.4	Data Integrity And Ethics Training Program	V1M2 Sec. 4.2.8.1; 5.2.7		83
18.0	ACCOMMODATIONS AND ENVIRONMENTAL CONDITIONS	V1M2 Sec. 5.3		84
18.1	Overview	V1M2 Secs. 5.3.1; 5.3.3; 5.3.4; 5.3.5	5.3.1; 5.3.3; 5.3.4; 5.3.5	84
18.2	Environment	V1M2 Secs. 5.3.1; 5.3.2; 5.3.3; 5.3.4; 5.3.5	5.3.1; 5.3.2; 5.3.3; 5.3.4; 5.3.5	85
18.3	Work Areas	V1M2 Secs. 5.3.3; 5.3.4; 5.3.5	5.3.3; 5.3.4; 5.3.5	85
18.4	Floor Plan			86
18.5	Building Security	V1M2 Sec. 5.3.4	5.3.4	86
19.0	TEST METHODS AND METHOD VALIDATION	V1M2 Sec. 5.4.1	5.4.1	86
19.1	Overview	V1M2 Sec. 5.4.1	5.4.1; 5.4.5.1	86
19.2	Standard Operating Procedures (Sops)	V1M2 Secs. 4.2.8.5; 4.3.3.1; 5.4.2	4.3.3.1; 5.4.2	87
19.3	Laboratory Methods Manual	V1M2 Sec. 4.2.8.5		87

Uncontrolled Copy

Sec. No.	Title	2009 TNI Standard Reference	ISO/IEC 17025:2005 (E) Reference	Page No.
19.4	Selection Of Methods	V1M2 Secs. 4.13.3; 5.4.1; 5.4.2; 5.4.3. V1M4 Secs. 1.4; 1.5.1; 1.6.1; 1.6.2; 1.6.2.1; 1.6.2.2	5.4.1; 5.4.2; 5.4.3; 5.4.4; 5.4.5.1; 5.4.5.2; 5.4.5.3	87
19.5	Laboratory Developed Methods And Non-Standard Methods	V1M2 Sec. 5.4.2. V1M4 Sec. 1.5.1	5.4.2; 5.4.4; 5.4.5.2; 5.4.5.3	91
19.6	Validation Of Methods	V1M2 Sec. 5.4.2. V1M4 Secs. 1.5.1; 1.5.2; 1.5.2.1; 1.5.2.2; 1.5.3	5.4.2; 5.4.4; 5.4.5.2; 5.4.5.3	91
19.7	Method Detection Limits (mdl) / Limits Of Detection (LOD)	V1M2 Sec. 5.9.3. V1M4 Secs. 1.5.2; 1.5.2.1; 1.5.2.2	5.4.5.3	92
19.8	Instrument Detection Limits (Idl)	V1M2 Sec. 5.9.3		93
19.9	Verification Of Detection And Reporting Limits	V1M2 Sec. 5.9.3. V1M4 Sec. 1.5.2.1		93
19.10	Retention Time Windows	V1M2 Sec. 5.9.3		93
19.11	Evaluation Of Selectivity	V1M2 Sec. 5.9.3. V1M4 Sec. 1.5.4; 1.7.3.6		93
19.12	Estimation Of Uncertainty Of Measurement	V1M2 Sec. 5.1.1; 5.1.2; 5.4.6	5.1.1; 5.1.2; 5.4.6.1; 5.4.6.2; 5.4.6.3	94
19.13	Sample Reanalysis Guidelines	V1M2 Sec 5.9.1	5.9.1	94
19.14	Control Of Data	V1M2 Secs. 5.4.7.1; 5.4.7.2; 5.9.1	5.4.7.1; 5.4.7.2; 5.9.1	95
20.0	EQUIPMENT and CALIBRATIONS	V1M2 Secs. 5.5.4; 5.5.5; 5.5.6	5.5.4; 5.5.5; 5.5.6; 5.6.1	100
20.1	Overview	V1M2 Secs. 5.5.1; 5.5.2; 5.5.3; 5.5.5; 5.5.10	5.5.1; 5.5.2; 5.5.3; 5.5.5; 5.5.10; 5.6.1	100
20.2	Preventive Maintenance	V1M2 Secs. 5.5.1; 5.5.3; 5.5.7; 5.5.9	5.5.1; 5.5.3; 5.5.7; 5.5.9; 5.6.1	101
20.3	Support Equipment	V1M2 Secs. 5.5.10; 5.5.11; 5.5.13.1	5.5.10; 5.5.11; 5.6.2.1.2; 5.6.2.2.1; 5.6.2.2.2	102
20.4	Instrument Calibrations	V1M2 Secs. 5.5.8; 5.5.10; 5.6.3.1. V1M4 Sec. 1.7.1.1; 1.7.2	5.5.8; 5.5.9; 5.5.10; 5.6.1; 5.6.2; 5.6.3.1	104
20.5	Tentatively Identified Compounds (TICS) – GC/MS Analysis			108
20.6	Gc/Ms Tuning			108

Uncontrolled Copy

Sec. No.	Title	2009 TNI Standard Reference	ISO/IEC 17025:2005 (E) Reference	Page No.
21.0	MEASUREMENT TRACEABILITY			134
21.1	Overview	V1M2 Sec. 5.6.3.1	5.6.2.1.2; 5.6.2.2.2; 5.6.3.1	134
21.2	NIST-Traceable Weights And Thermometers	V1M2 Secs. 5.5.13.1; 5.6.3.1; 5.6.3.2	5.6.3.1; 5.6.3.2	134
21.3	Reference Standards / Materials	V1M2 Secs. 5.6.3.1; 5.6.3.2; 5.6.3.3; 5.6.3.4; 5.6.4.1; 5.6.4.2; 5.9.1; 5.9.3	5.6.3.1; 5.6.3.2; 5.6.3.3; 5.6.3.4; 5.9.1	134
21.4	Documentation And Labeling Of Standards, Reagents, And Reference Materials	V1M2 Secs. 5.6.4.2; 5.9.3		135
22.0	SAMPLING			137
22.1	Overview	V1M2 Secs. 5.7.1; 5.7.3	5.7.1; 5.7.3	137
22.2	Sampling Containers			138
22.3	Definition Of Holding Time			138
22.4	Sampling Containers, Preservation Requirements, Holding Times			138
22.5	Sample Aliquots / Subsampling	V1M2 Sec. 5.7.1	5.7.1	138
23.0	HANDLING OF SAMPLES	V1M2 Sec. 5.8.1	5.8.1	139
23.1	Chain Of Custody (COC)	V1M2 Secs. 5.7.2; 5.7.4; 5.8.4; 5.8.7.5; 5.8.8; 5.9.1	5.7.2; 5.8.4; 5.9.1	139
23.22	Sample Receipt	V1M2 Secs. 5.8.1; 5.8.2; 5.8.3; 5.8.5; 5.8.7.3; 5.8.7.4; 5.8.7.5	5.8.2; 5.8.3	140
23.3	Sample Acceptance Policy	V1M2 Secs. 5.8.6; 5.8.7.2		141
23.44	Sample Storage	V1M2 Secs. 5.7.4; 5.8.4	5.8.4	142
23.5	Hazardous Samples And Foreign Soils			143
23.6	Sample Shipping	V1M2 Sec. 5.8.2	5.8.2	143
23.7	Sample Disposal			143
24.0	ASSURING THE QUALITY OF TEST RESULTS			151
24.1	Overview	V1M2 Secs. 5.9.2; 5.9.3	5.9.2	151
24.2	Controls	V1M2 Secs. 5.9.2; 5.9.3	5.9.2	151
24.3	Negative Controls	V1M2 Secs. 5.9.2; 5.9.3 V1M4 Secs. 1.7.3; 1.7.3.1; 1.7.4.1	5.9.2	151

Uncontrolled Copy

Sec. No.	Title	2009 TNI Standard Reference	ISO/IEC 17025:2005 (E) Reference	Page No.
24.4	Positive Controls	V1M2 Secs 5.9.2; 5.9.3. V1M4 Secs. 1.7.3; 1.7.3.2; 1.7.3.2.1; 1.7.3.2.2; 1.7.3.2.3	5.9.2	152
24.5	Sample Matrix Controls	V1M2 Secs. 5.9.2; 5.9.3. V1M4 Secs. 1.7.3 ; 1.7.3.3; 1.7.3.3.1; 1.7.3.3.2; 1.7.3.3.3	5.9.2	154
24.6	Acceptance Criteria (Control Limits)	V1M2 Sec. 5.9.3. V1M4 Secs. 1.7.4.2; 1.7.4.3		154
24.7	Additional Procedures To Assure Quality Control	V1M2 Sec. 5.9.3. V1M4 Sec. 1.7.3.4		156
25.0	REPORTING RESULTS			157
25.1	Overview	V1M2 Secs. 5.10.1; 5.10.2; 5.10.8	5.10.1; 5.10.2; 5.10.8	157
25.2	Test Reports	V1M2 Secs. 5.10.1; 5.10.2; 5.10.3.1; 5.10.3.2; 5.10.5; 5.10.6; 5.10.7; 5.10.8; 5.10.10; 5.10.11	5.10.1; 5.10.2; 5.10.3.1; 5.10.3.2; 5.10.5; 5.10.6; 5.10.7; 5.10.8	157
25.3	Reporting Level Or Report Type	V1M2 Secs. 5.10.1; 5.10.7; 5.10.8	5.10.1; 5.10.7; 5.10.8	159
25.4	Supplemental Information For Test	V1M2 Secs. 5.10.1; 5.10.3.1; 5.10.5	5.10.1; 5.10.3.1; 5.10.5	160
25.5	Environmental Testing Obtained From Subcontractors	V1M2 Secs. 4.5.5; 5.10.1; 5.10.6	5.10.1; 5.10.6	161
25.6	Client Confidentiality	V1M2 Secs. 4.1.5; 5.10.7	4.1.5; 5.10.7	161
25.7	Format Of Reports	V1M2 Sec. 5.10.8	5.10.8	161
25.8	Amendments To Test Reports	V1M2 Sec. 5.10.9	5.10.1; 5.10.9	162
25.9	Policies On Client Requests For Amendments	V1M2 Secs. 5.9.1; 5.10.9	5.9.1; 5.10.1; 5.10.5; 5.10.9	162

Uncontrolled Copy

LIST OF TABLES

Table No.	Title	2009 TNI Standard Reference	ISO/IEC 17025:2005 (E) Reference	Page No.
12-1	Example – General Corrective Action Procedures	V1M2 Sec. 4.11.6. V1M4 Sec. 1.7.4.1	4.11.2	64
14-1	Record Index		4.13.1.1	69
14-2	Example: Special Record Retention *Requirements*			71
15-1	Types Of Internal Audits And Frequency		4.14.1	75
20-1	Example: Instrumentation List		5.5.4; 5.5.5	109
20-2	Example: Schedule Of Routine Maintenance			131
24-1	Example – Negative Controls			151
24-3	Sample Matrix Control			154

LIST OF FIGURES

Figure No.	Title	2009 TNI Standard Reference	ISO/IEC 17025:2005(E) Reference	Page No.
4-1	Corporate And Laboratory Organization Charts	V1M2 Sec. 4.1.5	4.1.3; 4.1.5; 4.2.6	31
12-1	Example - Corrective Action Report			63
19-1	Example - Demonstration Of Capability Documentation			100
23-1	Example: Chain Of Custody (COC)			145
23-2	Example: Sample Acceptance Policy	V1M2 Sec. 5.8.6; 5.8.7.1. V1M4 Sec. 1.7.5		146
23-3	Example: Cooler Receipt Form		5.8.3	150

LIST OF APPENDICES

Appendix No.	Title	Page No.
1	Laboratory Floor Plan	163
2	Glossary/Acronyms	164
3	Laboratory Certifications, Accreditations, Validations	172

Uncontrolled Copy

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

SOP Reference	Title
ED-GEN-002	Document Control
ED-GEN-026	Evaluation of Analytical Accuracy and Precision Through The Use of Control Charts
ED-GEN-010	Calibration of Analytical Balances
ED-GEN-003	Control of Non-Conformances and Corrective Action
ED-GEN-024	Record Storage and Retention
ED-GEN-022	Training
ED-GEN-001	Data Management and Handling Procedures
ED-GEN-021	Data Review
ED-SPM-001	Login
ED-RP-001	Reports Production
ED-GEN-014	Thermometer Calibration

Uncontrolled Copy

SOP Reference	Title
ED-GEN-011	Calibration and Use of Lab Pipettes
ED-FLD-008, -009	Groundwater Sampling and Flow Monitoring
ED-FLD-014	Wastewater Sampling
ED-FLD-001 thru -010	Field Analytical Parameters
ED-GEN-007	Subsampling
ED-SPM-007	Disposal of Samples and Associated Laboratory Waste
ED-SPM-006	Procedure for Acceptance and Handling of Regulated Domestic and Foreign Soils

Uncontrolled Copy

SECTION 3. INTRODUCTION, SCOPE AND APPLICABILITY

3.1 Introduction and Compliance References

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

The QAM has been prepared to assure compliance with The NELAC Institute (TNI) Standard, dated 2009, Volume 1 Modules 2 and 4. 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 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.*
- 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) (DW labs only)*
- *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, 21st, and on-line Editions.
- Nuclear Regulatory Commission (NRC) Quality Assurance Requirements.
- Marine Protection, Research, and Sanctuaries Act (MPRSA).
- Toxic Substances Control Act (TSCA).

Uncontrolled Copy

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 TestAmerica Edison Work Instruction No. EDS-WI-009 (Analytical Capabilities). 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.

Uncontrolled Copy

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 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 SOP No. ED-GEN-002, Document Control).

SECTION 4. MANAGEMENT REQUIREMENTS

4.1 Overview

TestAmerica Edison is a local operating unit of TestAmerica Laboratories, Inc.. The organizational structure, responsibilities and authorities of the corporate staff of TestAmerica Laboratories, Inc. are presented in the CQMP. 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 Edison is presented in Figure 4-1.

4.2 Roles and Responsibilities

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

4.2.1 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 Edison 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

Uncontrolled Copy

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 Operation Officer (COO)

The COO reports directly to the President and CEO of TestAmerica. The COO~~VPO~~ oversees the operations of all TestAmerica laboratories and the EMLab P&K business unit. The VP's of Operations report directly to COO

4.2.4 Vice President of Operations

Each VP of Operations reports directly to the Executive 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.

Uncontrolled Copy

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

Uncontrolled Copy

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.

Uncontrolled Copy

- 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/Lead Technical Manager

TestAmerica Edison's Laboratory Director is responsible for the overall quality, safety, financial, technical, human resource and service performance of the whole laboratory and reports to their 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:

- Serves as lead Technical Manager for all fields of testing.
- Ensures that all analysts and supervisors have the appropriate education and training to properly carry out the duties assigned to them and ensures that this training has been documented.
- Ensures that personnel are free from any commercial, financial and other undue pressures which might adversely affect the quality of their work.
- Ensures TestAmerica's human resource policies are adhered to and maintained.
- Ensures that sufficient numbers of qualified personnel are employed to supervise and perform the work of the laboratory.
- Ensures that appropriate corrective actions are taken to address analyses identified as requiring such actions by internal and external performance or procedural audits. Procedures that do not meet the standards set forth in the QAM or laboratory SOPs may be temporarily suspended by the Laboratory Director.
- Monitors standards of performance in quality control and quality assurance.
- Monitors the validity of analyses performed and data generated in the lab to assure reliable data.
- Reviews and approves all SOPs prior to their implementation and ensures all approved SOPs are implemented and adhered to.
- Interfaces with Project Management and Customer Service to forecast receipts, provide quality analytical data to clients and meet on-time delivery dates.
- Ensures that the facility has appropriate Information Technology resources and that they are used effectively to support operational requirements.
- Actively participates in the process of sharing and adopting best practices within TestAmerica. Provides technical assistance to other TestAmerica laboratories as needed to improve productivity and customer service.

Uncontrolled Copy

- Ensures client specific reporting and quality control requirements are met.
- Captains the management team, consisting of the QA Manager, the Operations Manager, the Laboratory Client Services Manager, the Client Services Manager, the Service Center Manager, the Environmental, Health and Safety Manager and the Support Services 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 their Corporate Quality Director. 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 may be temporarily suspended during the investigation.

Uncontrolled Copy

- 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.
- 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. (where applicable)

4.2.17 Quality Assurance (QA) Specialist

The Quality Assurance (QA) Specialist is responsible for performing data audits, special audits, assisting with external and systems audits, overseeing the maintenance of QC records, certifications, Standard Operating Procedures (SOPs), training records, DOCs, arranging and managing PT samples. Additional responsibilities may include assisting with systematic problems within the laboratory, assisting in reviewing and/or writing of Quality Assurance Project Plans, and technical and QC specifications in contracts and other functions in support of the QA Manager's responsibilities as assigned.

- Assist QA Manager in conducting QA training courses, including ethics training.
- Performs data audits.
- Assist in performing special audits as deemed necessary by data audits, client inquiries, etc.
- Assisting in, conducting and responding to external audits conducted by clients and regulatory agencies.

Uncontrolled Copy

- Assisting in reviewing and/or writing of Quality Assurance Project Plans, and technical and QC specifications in contracts.
- Maintaining all necessary laboratory certifications.
- Arranging and managing PT samples.
- Reviewing laboratory SOPs. Writing SOPs as needed.
- Maintaining historical indices of all technical records including SOPs, QC records, laboratory data, etc.
- Ensuring maintenance of records archives.
- Assisting in and monitoring laboratory's method compliance.
- Ensuring maintenance of DOCs for all analysts.
- Ensuring maintenance of training records for all employees.
- Assisting in identification of systematic problems within laboratories.
- Recommends resolutions for ongoing or recurring nonconformance.
- Providing statistical feedback to Departments on error rates, and assisting in identifying systematic improvements to minimize errors.
- Assists in tracking of customer complaints, providing statistical feedback to the laboratory, and assisting in identifying improvements.
- Overseeing and reviewing MDL studies.
- Ensuring control charts are generated, oversees and approves setting of control limits.
- Assists in monitoring new regulations and communicating them to the laboratory.

4.2.18 Operations/Technical Manager(s)

The Organics and Inorganics Operations/Technical Managers report directly to the Laboratory Director (throughout this text the term 'Technical Manager' refers to these individuals).. He/she is accountable for all analyses and analysts under their experienced supervision 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

Uncontrolled Copy

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.

4.2.19 Department Manager(s)

The Department Managers manage and direct the analytical production sections of the laboratory and act as Technical Manager of their department. He/She reports directly to either the Organics or Inorganics Operations/Technical Manager (as applicable). He/She assists the Operations/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 Operations/Technical Manager and QA Manager and in compliance with regulatory requirements.
- Ensures that scheduled instrument maintenance is completed.
- Is responsible for efficient utilization of supplies.

Uncontrolled Copy

- 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.

4.2.20 Environmental, Health and Safety Manager

The Environmental, Health and Safety Manager reports directly to the VP of Quality, Environmental, Heath and Safety. The duties of this position consist of:

- Supervises the Environmental, Health and Safety/Facilities Team.
- Conduct ongoing, necessary safety training and conduct new employee safety orientation.
- Assist in developing and maintaining the Chemical Hygiene/Safety Manual.
- Administer dispersal of all Material Safety Data Sheet (MSDS) information.
- Perform regular chemical hygiene and housekeeping instruction.
- Give instruction on proper labeling and practice.
- Serve as chairman of the laboratory safety committee.
- Provide and train personnel on protective equipment.
- Oversee the inspection and maintenance of general safety equipment – fire extinguishers, safety showers, eyewash fountains, etc. and ensure prompt repairs as needed.
- Supervise and schedule fire drills and emergency evacuation drills.
- Determine what initial and subsequent exposure monitoring, if necessary to determine potential employee exposure to chemicals used in the laboratory.
- When determined necessary, conduct exposure monitoring assessments.
- Determine when a complaint of possible over-exposure is "reasonable" and should be referred for medical consultation.
- Assist in the internal and external coordination of the medical consultation/monitoring program conducted by TestAmerica's medical consultants.
- Staying current with the hazardous waste regulations.
- Continuing training on hazardous waste issues.
- Reviewing and updating annually the Hazardous Waste Contingency Plan in the Environmental Health & Safety Manual.
- Auditing the staff with regard to compliance with the Hazardous Waste Contingency Plan.
- Contacting the hazardous waste subcontractors for review of procedures and opportunities for minimization of waste.

Uncontrolled Copy

4.2.21 EH&S/Facilities Coordinator

The EH&S/Facilities Coordinator reports directly to the Laboratory Director with a dotted line to the Environmental, Health and Safety Manager. The duties of this position consist of:

- Monitors laboratory for unsafe conditions or acts to keep lab in compliance with the Chemical Hygiene Plan, EH&S Procedures, and company policies.
- Ensures the proper personal protective equipment is available and personnel are properly trained in its use.
- Assists the Environmental, Health and Safety Manager in the investigation of accidents, incidents, and near misses and identifies and eliminates root cause.
- Conducts monthly facility inspections for compliance with health, safety and environmental regulations and procedures. Completes and forwards monthly inspection report to safety committee and laboratory management for corrective actions.
- Conducts safety equipment checks to ensure proper working order and sufficient inventory.
- Plans and tracks completion of monthly general awareness training sessions and compliance training, including new employee EH&S orientation.
- Coordinates emergency response team to provide prompt medical attention and stabilize emergency situation. After emergency is over, assists in determining appropriate clean up procedures.
- Conducts the monthly EH&S committee meeting.
- Participates in monthly EH&S conference call.
- Reviews and maintains MSDS's for laboratory materials.
- Coordinates the management and disposal of laboratory wastes.
- Assists in the preparation and maintenance of the laboratory Integrated Contingency Plan.
- Monitors air quality in facility, including monitoring fume hoods for proper operation and ventilation.
- Maintains overall building facilities and equipment as well as administers prevention maintenance measures.
- Contacts outside contractors as necessary to repair/maintain items outside the realm of reasonable maintenance.
- Performs miscellaneous errands, buying parts for labs, janitorial supplies.
- Oversees storage facilities, files and outside storage.

4.2.22 Laboratory Analysts and Technicians

Laboratory analysts and technicians 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:

Uncontrolled Copy

- 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.
- Adhere to all environmental, health and safety protocols and attend safety meetings as required.
- Attend and participate in all staff meetings.

4.2.23 Sample Control Manager

The Sample Control Manager reports to the Inorganics Operation Manager. The responsibilities are outlined below:

- Direct the logging of incoming samples into the LIMS.
- Ensure the verification of data entry from login.
- Manages the preparation and shipment of bottle kits to clients.
- Oversees the responsibilities of all Sample Control Technicians.
- Supervises the storage and disposal of all samples.

4.2.24 Senior Client Services Director

The Senior Client Services Director reports to the Vice President of Operations and Client Services with a dotted line to the laboratory director and serves as the primary interface between the laboratory and the Sales and Marketing staff. Responsibilities include:

- Laboratory's primary client representative.
- Ensures client complaints are handled professionally, and resolved in a timely manner.
- Compiles and interprets receipts forecast to show near term business trends.

Uncontrolled Copy

- Manages a minimal list of projects/programs for key client accounts. (Note: sufficient time is needed to manage the PM group and the CSM must not be overwhelmed with project management.)
- Prepares proposals for new business opportunities.
- Compiles and interprets Bid Activity Report.
- Compiles and interprets receipts forecast to show near term business trends.
- Prepares proposals for new business opportunities.
- Provides general sales support to Account Executives for business development activities started in the field.
- Develops and maintains business materials and organized information resource files that include project descriptions, resumes, original proposals, boilerplates, and company qualifications materials.

4.2.25 Manager of Project Managers

The Manager of Project Managers reports to the Senior 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 functions of this position are outlined below:

- Technical training and growth of the Project Management team.
- Technical liaison for the Project Management team.
- Human resource management of the Project Management team.
- Responsible for ensuring that clients receive the proper sampling supplies, as appropriate.
- Accountable for response to client inquiries concerning sample status.
- Responsible for assistance to clients regarding the resolution of problems concerning COC.
- Ensuring that client specifications, when known, are met by communicating project and quality assurance requirements to the laboratory.
- Notifying the supervisors of incoming projects and sample delivery schedules.
- Accountable to clients for communicating sample progress in daily status meeting with agreed-upon due dates.
- Responsible for discussing with client any project-related problems, resolving service issues, and coordinating technical details with the laboratory staff.
- Responsible for staff familiarization with specific quotes, sample log-in review, and final report completeness.
- Monitor the status of all data package projects in-house to ensure timely and accurate delivery of reports.
- Inform clients of data package-related problems and resolve service issues.
- Coordinate requests for sample containers and other services (data packages).

Uncontrolled Copy

4.2.26 Project Manager (PM)

The Project Managers report directly to the Director of Project Management and serve as liaisons between the laboratory and its clients. The Project Manager's responsibilities include:

- Ensure client specifications are met by communicating project and quality assurance requirements to the laboratory.
- Notify laboratory personnel of incoming projects and sample delivery schedules.
- Monitor the status of all projects in-house to ensure timely delivery of reports.
- Inform clients of project-related problems, resolving service issues and coordinating technical issues with the laboratory staff.
- Accountable for response to client inquiries concerning sample status.
- Responsible for assistance to clients regarding the resolution of problems concerning COC.
- Ensuring that client specifications, when known, are met by communicating project and quality assurance requirements to the laboratory.
- Notifying the supervisors of incoming projects and sample delivery schedules.
- Coordinate client requests for sample containers and other services.
- Schedule sample pick-ups from client offices or project sites and notifying the laboratory staff of incoming samples.
- Coordinate subcontract work.
- Respond to client inquiries concerning sample status.
- Generates final laboratory reports and has signature authority for those reports (as designated and approved by the Laboratory Director).
- Performs final completeness review of data packages prior to release to client.

4.2.27 Project Management Assistant

The Project Management Assistant coordinates and monitors scheduling, timely completion and maintenance of project documentation files and completion of project set up and final report review, invoicing, and EDD's. Assists the Project Manager in servicing the client's needs. Specific responsibilities include:

- Reviews login confirmation reports for accuracy and corrects as needed.
- Generates diskettes for electronic data deliverables (EDD's) for electronic delivery to clients.
- Enters data that was subcontracted to other laboratories.
- Monitors report due dates for timely delivery.
- Assists Project Manager in changing compound lists, TAT, deliverables and other client specific requirements in the LIMs project and/or job database.

Uncontrolled Copy

- Invoices completed data packages and generates credit or debit invoices to ensure proper payment.

4.2.28 Service Center Manager

The Service Center Manager (SCM) manages the service center and acts as a liaison between the laboratory and the local client base. The SCM is in charge of maintaining the Service Center facility, managing service center couriers, samplers and other personnel, and working with sales to develop, maintain and grow the client base in the area.

- Local area primary client representative for service center location.
- May head project start up meetings to ensure project objectives are successfully met and hands off project detail to assigned Project Manager(s).
- Works with the Quality Assurance Manager and Account Executives (AE) to evaluate and establish project requirements for the service center area.
- Ensures client complaints are handled professionally, and resolved in a timely manner.
- Is in charge of scheduling service center couriers and samplers, preparing bottle orders for delivery, scheduling sample pick ups and shipping samples to the designated laboratory for analysis.
- May manage a minimal list of projects/programs for key client accounts.
- Maintains the facilities at the service center and is responsible for all EH&S policies of TestAmerica at the service center.
- Responsible for all company vehicles that operate out of the service center.
- Provides general sales support to AEs for business development activities started in the field.

4.3 Deputies

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

Key Personnel	Deputy
Mark Acierno Laboratory Director	In the event of absence the Laboratory Director's responsibilities are shared by the Laboratory Operations Manager, the Quality Assurance Manager and the Client Services Manager, as appropriate
Carl Armbruster Quality Assurance Manager	Emmylou Digiacomo Quality Assurance Specialist Mark Acierno Laboratory Director
Sylvanus Klusey Organic Technical Manager	Donald Evans Inorganic Technical Manager

Uncontrolled Copy

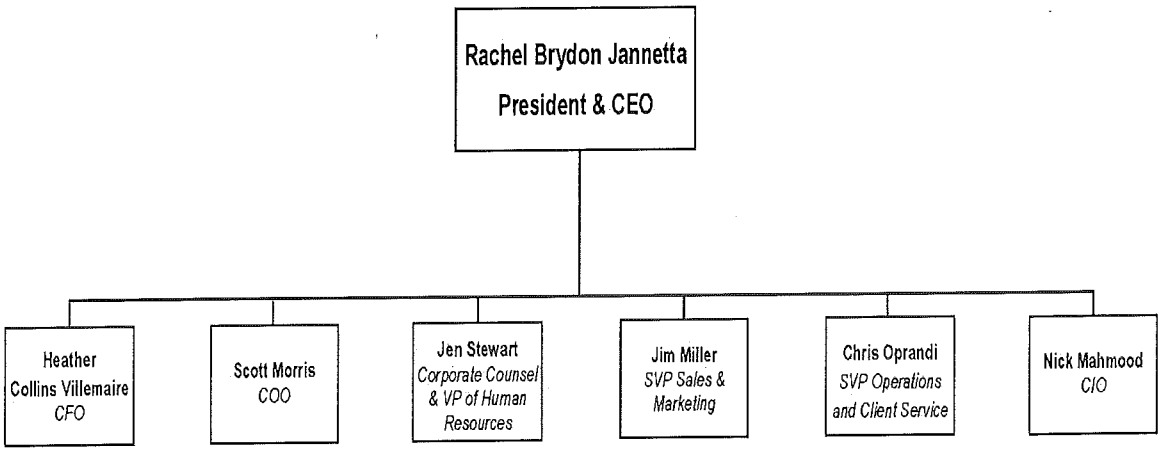
Key Personnel	Deputy
Donald Evans Inorganic Technical Manager	Sylvanus Klusey Organic Technical Manager
VOA, SVOA & Organic Prep Technical Managers	Sylvanus Klusey Organic Technical Manager
Wet Chemistry, Metals and Sample Control Managers	Donald Evans Inorganic Technical Manager
Dan Helfrich EH&S Manager	Edward Roche EH&S Coordinator
Mark Nemec Client Services Manager	Kristyn Tempe Project Manager
Kate Harrelson South Jersey/King of Prussia Service Center Manager	Alonzo Hall, Sample Control Tech. South Jersey Service Center Jeffery Keehn, Sample Control Tech. King of Prussia Service Center
John Kates Fields Services Manager	Stephen Schulze Thomas Lesinski Field Specialists – Edison Rick Toogood Dustin Miller Field Specialists – South Jersey

Uncontrolled Copy

Figure 4-1. Corporate and Laboratory Organization Charts

TestAmerica
THE LEADER IN ENVIRONMENTAL TESTING

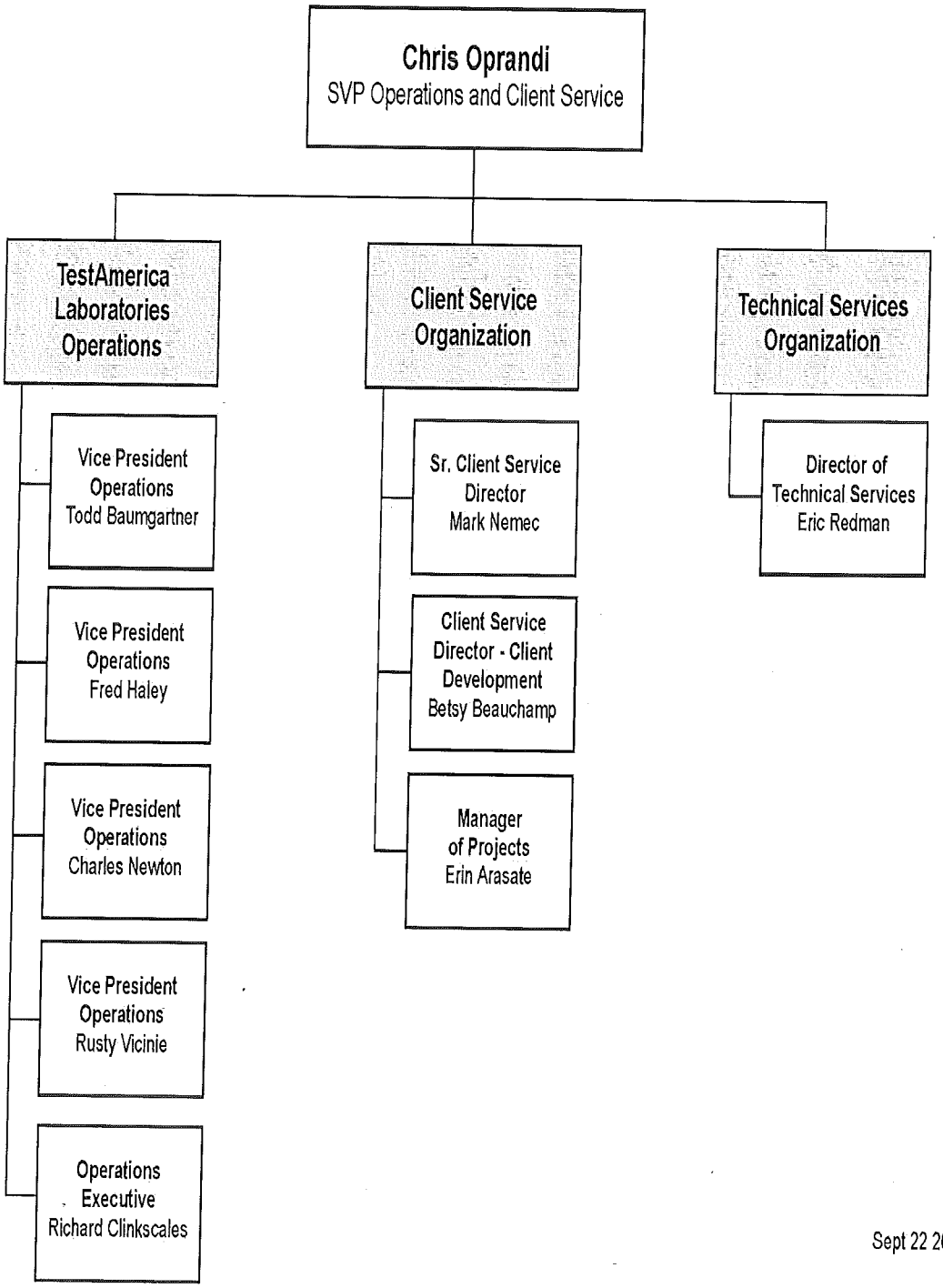
Executive Committee



February 24 2017

TestAmerica
THE LEADER IN ENVIRONMENTAL TESTING

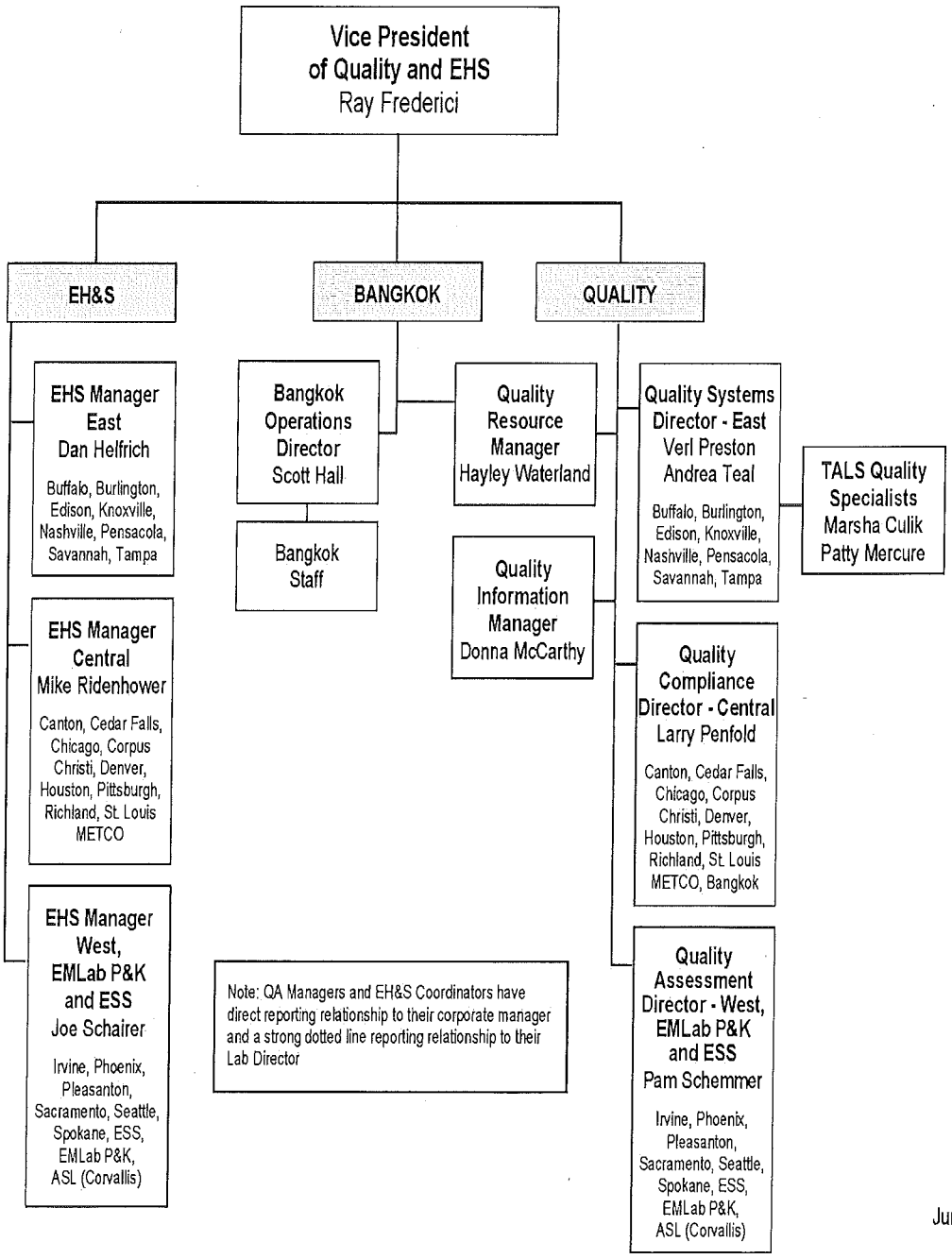
TestAmerica Laboratories Operations and Client Service



Sept 22 2017

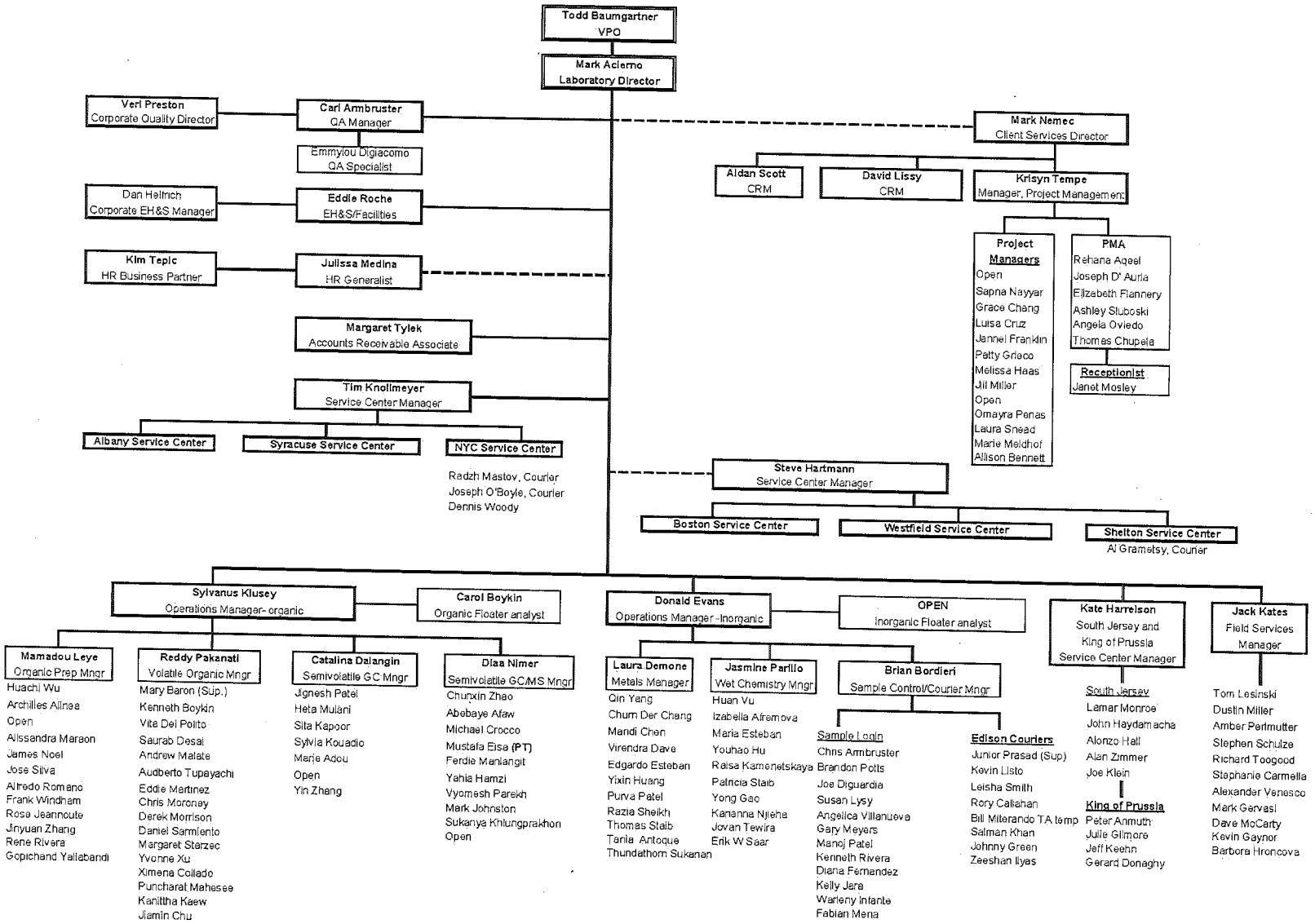
TestAmerica
THE LEADER IN ENVIRONMENTAL TESTING

Quality, EHS, Bangkok



June 5, 2017

TestAmerica Edison 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.
- 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.

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. CA-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.

Uncontrolled Copy

- 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.

Uncontrolled Copy

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.

Uncontrolled Copy

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..

Uncontrolled Copy

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 Limits within the Method Limit Group tables in TALS (the laboratory's LIMS) that summarize the precision and accuracy acceptability limits for performed analyses. This summary includes an effective date, is updated each time new limits are generated and are managed by the laboratory's QA department. Unless otherwise noted, limits within these tables are laboratory generated. Some acceptability limits are derived from US EPA methods when they are required. Where US EPA method limits are not required, the laboratory has developed limits from evaluation of data from similar matrices. Criteria for development of control limits is contained in Section 24.

5.6 Statistical Quality Control

Statistically-derived precision and accuracy limits are required by selected methods (such as SW-846) and programs [such as the Ohio Voluntary Action Plan (VAP)]. The laboratory routinely utilizes statistically-derived limits to evaluate method performance and determine when corrective action is appropriate. The analysts are instructed to use the current limits in the laboratory (dated and approved by the 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 Method Limit Group tables in TALS (LIMS). 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

The QA Manager generates QC charts using the TALS Control Chart program as detailed in SOP No. ED-GEN-026 (Evaluation of Analytical Accuracy and Precision Through The Use of Control Charts). In addition to their use in generating lab specific spike recovery limits and in the evaluation of MDL studies, these charts are used to determine if adjustments need to be made or for corrective actions to methods. All such findings are documented and kept on file in the QA Department.

Uncontrolled Copy

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. ED-GEN-002 (Document Control).

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

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

6.2 Document Approval and Issue

The pertinent elements of a document control system for each document include a unique document title and number, 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 (Technical) Manager submits an electronic draft to the QA Department

Uncontrolled Copy

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 and revised as appropriate. Changes to documents occur when a procedural change warrants.

6.3 Procedures for Document Control Policy

For changes to the QA Manual, refer to SOP No. ED-GEN-002 (Document Control) Uncontrolled copies must not be used within the laboratory. Previous revisions and back-up data are stored by the QA Department. Electronic copies are stored on the Public server in the QA folder for the applicable revision.

For changes to SOPs, refer to SOP No. CW-Q-S-002, Writing a Standard Operating Procedure SOP or list your labs SOP on this topic. 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 ED-GEN-002 (Document Control).

6.4 Obsolete Documents

All invalid or obsolete documents are removed, or otherwise prevented from unintended use. The laboratory has specific procedures as described above to accomplish this. In general, obsolete documents are collected from employees according to distribution lists and are marked obsolete on the cover or destroyed. At least one copy of the obsolete document is archived according to SOP No. ED-GEN-002 (Document Control).

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.

Uncontrolled Copy

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.

Uncontrolled Copy

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 (CRM) or CRM 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
- The Laboratory Client Services Manager
- The 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.

The Contracts Administrator maintains copies of all signed contracts. The applicable Project Manager maintains local copies of signed contracts.

7.3 Documentation

Appropriate records are maintained for every contract or work request. All stages of the contract review process are documented and include records of any significant changes. These records are maintained in the project file by the Project Manager and/or Key Account Executive. The contract will be distributed to and maintained by the appropriate sales/marketing personnel and the Account Executive. A copy of the contract and formal quote will be filed with the laboratory PM and the Laboratory Director.

Uncontrolled Copy

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. It is the PM's responsibility to ensure that project-specific technical and QC requirements are effectively evaluated and communicated to the laboratory personnel before and during the project. QA department involvement may be needed to assist in the evaluation of custom QC requirements.

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

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

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

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

Uncontrolled Copy

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 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.

Uncontrolled Copy

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 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 (e.g., USDA) or contracts require notification prior to placing such work.

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:

- 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

Uncontrolled Copy

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 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 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

Uncontrolled Copy

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 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.

Uncontrolled Copy

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 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 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

Uncontrolled Copy

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

If an item is not available from the on-site consignment, the analyst must provide the master item number (from the master item list that has been approved by the 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 Department (Technical) Manager or the Laboratory Operations Manager places the order.

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 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

Uncontrolled Copy

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' (refer to item 3 listed below).

- An expiration date **cannot** be extended if the dry chemical/solvent is discolored or appears otherwise physically degraded, the dry chemical/solvent must be discarded.
- Expiration dates can be extended if the dry chemical/solvent is found to be satisfactory based on acceptable performance of quality control samples (Continuing Calibration Verification (CCV), Blanks, Laboratory Control Sample (LCS), etc.).
- If the dry chemical/solvent is used for the preparation of standards, the expiration dates can be extended 6 months if the dry chemical/solvent is compared to an unexpired independent source in performing the method and the performance of the dry chemical/solvent is found to be satisfactory. The comparison must show that the dry chemical/solvent meets CCV limits. The comparison studies are maintained in the analytical Department.

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°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.

Uncontrolled Copy

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 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, 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, a unique identification name is assigned and provided to the QA Department for inclusion on the laboratory master 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.

Uncontrolled Copy

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 and/or the Laboratory Operations Managers.

Analytical balances are serviced and calibrated annually in accordance with TestAmerica Edison SOP ED-GEN-010 (Calibration of Analytical 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

Uncontrolled Copy

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 TestAmerica Edison SOP No. ED-GEN-003 (Control of Non-Conformances and Corrective Action).

10.2 External Complaints

An employee that receives a complaint initiates the complaint resolution process by first documenting the complaint according to TestAmerica Edison SOP No. ED-GEN-003 (Control of Non-Conformances and Corrective Action).

Uncontrolled Copy

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 Department (Technical) Manager for resolution. The

Uncontrolled Copy

manager may elect to discuss it with the Lab Director and/or QA Manager or have a representative contact the client to decide on a logical course of action. Once an approach is agreed upon, the analyst documents it using the laboratories corrective action system described in Section 12. This information can then be supplied to the client in the form of a footnote or a case narrative with the report.

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

Uncontrolled Copy

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. 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.

Uncontrolled Copy

Within 72 hours, the QA Manager will determine if compliance is now met and reports can be released, OR determine the plan of action to bring work into compliance, and release work. A team, with all principals involved (Laboratory Director, Laboratory Operations Managers, QA Manager, Department Technical 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 TestAmerica's Incident and Corrective Action Tracker (iCAT) (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.

Uncontrolled Copy

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

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 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 CAR is used for this documentation.

Uncontrolled Copy

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 CAR is entered into a the iCAT database for tracking purposes and a monthly summary of all corrective actions is printed out for review to aid in ensuring that the corrective actions have taken effect.
- TestAmerica laboratories began using the Incident/Corrective Action Tracker (iCAT) database developed by the company in 2015. (Previously, a local Excel 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 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.

Uncontrolled Copy

- Any out-of-control situations that are not addressed acceptably at the laboratory level may be reported to the Corporate Quality Director by the QA Manager, indicating the nature of the out-of-control situation and problems encountered in solving the situation.

12.3.5 Follow-up Audits

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

(Also refer to Section 15.1.4, Special Audits.)

12.4 Technical Corrective Actions

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

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

Table 12-1 provides some general guidelines for identifying the individual(s) responsible for assessing each QC type and initiating corrective action. The table also provides general guidance on how a data set should be treated if associated QC measurements are unacceptable. Specific procedures are included in Method SOPs, Work Instructions, 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 and appropriate corrective action (e.g., reanalysis) is taken and documented.

Uncontrolled Copy

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.

Uncontrolled Copy

Figure 12-1.
Example - Corrective Action Report

TestAmerica Edison - Quality Assurance

Corrective Action 8698

Incident 3098: WS0717 PT Failures
Incident Status: Closed
Response Due to Client: Sunday, Oct 8, 2017

Created By:	Carl Armbruster	Created On:	Friday, Sep 8, 2017
Laboratory Function:	Other Lab Functions	Date Follow-up Due:	
Corrective Action Type:	PT and/or Double Blind Failures	Date Follow-up Done:	
Department:	Metals	Response Due to QA:	Sunday, Oct 8, 2017
Finding Number:	1	Planned Closure Date:	Sunday, Oct 8, 2017
Is Repeat Finding?:	No	Date Closed:	Monday, Oct 16, 2017
Finding Reference:			
Assigned To/Lead:	Laura Demone		
Priority:	2 (High Importance)		
Follow-up Assigned To:			
C.A. Status:	Closed		

Issue Requiring Corrective Action:
Rec'd evaluation of 'Not Acceptable for Arsenic by EPA 200.8: Assigned value: 8.17 ug/l Reported result: 10.9 ug/l Acceptance limits: 6.72 - 10.6 ug/l. Rec'd evaluation of 'Not Acceptable for Iron by EPA 200.7: Assigned value: 304 ug/l Reported result: 218 ug/l Acceptance limits: 258 - 349 ug/l. Investigate and submit correction action plan along with completed PT corrective action checklist (attached to this iCAT item).

Investigation/Response:
Drinking water PT 460-137368 sample 12 (PT study WS0717) failed low for iron by EPA 200.7 and failed high for arsenic by EPA 200.8.

Root Cause:
PT sample results as well as instrument and batch QC samples were checked for any anomalies or issues that may have contributed to the failures. For EPA 200.7, the calibration curve and blanks look very good for iron, and QC recoveries are within the acceptable range. For EPA 200.8, the same is true, the arsenic curve and all blanks are very good and all QC recoveries are also within the acceptable range for this method. The only issue noticed in the evaluation of the 200.8 data is that the serial dilution result for arsenic went down with higher dilution. For example,. the serial dilution at 5x was 9.62 ug/L (this is high but within the acceptance limits for arsenic) and at 10x the result was 8.12 ug/L, which is essentially the assigned value of arsenic. Perhaps there was an interference (both copper and zinc needed dilution for this sample) that caused high bias for the arsenic that was removed by the dilution of the sample.

Corrective Action Plan:
As per an older corrective action response, when laboratory sample is logged in for both an ICP and an ICP-MS method, the final data is compared before reporting, and this of course is also true for PT samples. The iron result on the ICP-MS for EPA 200.8 was 301 ug/L but since iron was not logged in as a reportable analyte for this method, this data was not compared to the ICP result. The same is true for arsenic, this analyte was not reported via Method 200.7 so the data was not compared either, (the arsenic result via method 200.7 was 9.98 ug/L, which is high but within the acceptable range) Had these results been compared, the discrepancies would certainly have been noticed and investigated further. Going forward, all data will be compared between the two methods, even when only one method is actually needed for reporting purposes, and all QC data will be examined more carefully to check for possible interferences. In addition, part of the issue is simply that the metals PT samples are either started early but then not reviewed/ compared until close to the due date or they are not started until well after they are received. If there were more time to carefully review data and compare results, it is much more likely that any issues would be caught and resolved. With all future PT studies, metals samples will be digested, analyzed and reviewed within two weeks of login to TALS and if all data is acceptable, results will also be submitted in this time frame. Phenova sent a QC sample (Trace Metals QC Lot #9066-04) with the rapid response PT sample for drinking water by EPA 200.7 and 200.8 with a certified value of 933 ug/L for iron and acceptance limits of 790-1070 ug/L, and a certified value of 21.1 ug/L for arsenic and acceptance limits of 14.8-27.4 ug/L. This sample (460-140875-2) was digested and analyzed with the rapid response PT sample and we got an iron result of 1020 ug/L which is within the acceptable recovery range. We also ran this rapid response QC sample on the ICP-MS for method 200.8 arsenic and we got a result of 23.2 ug/L (this is on A468012, not identified since it is not logged in for this method in TALS) which is within the acceptable range. We also carefully checked QC recoveries on the ICP/ ICPMS and ran the samples on the ICPMS and compared the results from the two instruments as usual, so we feel confident with the rapid response results as reported. Going forward for drinking water by method 200.8, analysts have been reminded to matrix match the standards with the samples (eg, rinse water, internal standard, calibration standards all use 2% HNO3 to match non-digested drinking waters). According to the batch notes this was done in the initial run of the PT.

Corrective Action Documents (3)

iCAT3098_PT corrective action narrative checklist_WS0717_Fe_460-137368.pdf	Con4AanA4.LONG_DATE_STRING 6:58 AM
iCAT3098_PT corrective action narrative checklist_WS0717_As_460-137368.pdf	Con58AanA58.LONG_DATE_STRING 7:3 AM
iCAT3098_PT corrective action QC sample Iron_140875-2.pdf	Con29PanP29.LONG_DATE_STRING 12:32 PM

For QA Use Only

QA Comments	Follow Up
Enter a QA Comment	*Enter Follow Up*

Notifications

Location QA (notification required)

Name	Title
Carl Armbruster	Quality Assurance Manager
Emmylou DiGiacomo	Quality Assurance Specialist
Hayley Waterland	

Additional Notifications

Name	Title
Mark Aciemo	Laboratory Director
Laura Demone	Department Manager I
Donald Evans	Operations Manager

Table 12-1. Example – General Corrective Action Procedures

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

Uncontrolled Copy

QC Activity *(Individual Responsible for Initiation/Assessment)*	Acceptance Criteria	Recommended Corrective Action
Laboratory Control Sample (LCS) *(Analyst, Data Reviewer)*	- % Recovery within limits specified in TALS Method Limit Groups.	- Batch must be re-prepared and re-analyzed. This includes any allowable marginal exceedance. When not using marginal exceedances, the following exceptions apply: 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; 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. **Note:** If there is insufficient sample or the holding time cannot be met, contact client and report with flags.
Surrogates *(Analyst, Data Reviewer)*	- % Recovery within limits of method or within three standard deviations of the historical mean.	- Individual sample must be repeated. Place comment in LIMS. - Surrogate results outside criteria shall be reported with qualifiers.
Method Blank (MB) *(Analyst, Data Reviewer)*	< Reporting Limit [1]	- Reanalyze blank. - If still positive, determine source of contamination. If necessary, reprocess (i.e. digest or extract) entire sample batch. Report blank results. - 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 *(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 *(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.

Uncontrolled Copy

QC Activity *(Individual Responsible for Initiation/Assessment)*	Acceptance Criteria	Recommended Corrective Action
Reporting / Calculation Errors (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 or TestAmerica Edison's Corrective Action SOP (ED-GEN-003),
Client Complaints *(Project Managers, Lab Director/Manager, Sales and Marketing)*	-	- Corrective action is determined by the type of complaint. For example, a complaint regarding an incorrect address on a report will result in the report being corrected and then follow-up must be performed on the reasons the address was incorrect (e.g., database needs to be updated).
QA Monthly Report (Refer to Section 16 for an example) (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 (Safety Officer, Lab Director/Manager, *Technical Manager(s)*)	- Environmental Health and Safety (EHS) Manual.	- Non-conformance is investigated and corrected through CAR system.

Uncontrolled Copy

Note:

1. See specific method SOPs for blank acceptance criteria. 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 phthalate.. 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, 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 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:

Uncontrolled Copy

- 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 various tracking indicators, the potential risks inherent with a new event or change are identified and evaluated. The risks are minimized or eliminated through pre-planning and the development of preventive measures. The types of indicators monitored under this collective system include:

- SOP Tracking
  Current Revisions w/ Effective Dates
  Required Biennial Revisions w/ Due Date

- Proficiency Testing (PT) Sample Tracking
  Pass / Fail – most current 2 out of 3 studies.

- Instrument / Equipment List
  Current / Location

- Accreditations
  New / Expiring

- Method Capabilities
  Current Listing by program (e.g., Potable Water, Soils, etc.)

- Key Personnel
  Technical Managers, Department Supervisors, etc..

These items are maintained on TestAmerica's Intranet (Proposal Library) or on our internal database (TotalAccess) which uploads to our company internet site

Uncontrolled Copy

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, which is backed up as part of the regular laboratory backup. Records are of two types; either electronic or hard copy paper formats depending on whether the record is computer or hand generated (some records may be in both formats). Technical records are maintained by Laboratory Operations under the direction of the Laboratory Operations Managers.

Table 14-1. Record Index[1]

	Record Types [1]:	Retention Time:
Technical Records	- Raw Data - Logbooks[2] - Standards - Certificates - Analytical Records - MDLs/IDLs/DOCs - Lab Reports	5 Years from analytical report issue*
Official Documents	- Quality Assurance Manual (QAM) - Work Instructions - Policies - SOPs - Policy Memorandums - Manuals - Published Methods	Indefinitely
QA Records	- Certifications - Method and Software Validation / Verification Data	Indefinitely
QA Records	- Internal & External Audits/Responses - Corrective/Preventive Actions - Management Reviews -- Data Investigation	5 Years from archival* **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)

Controlled Copy

	Record Types [1]:	Retention Time:
Project Records	- Sample Receipt & COC Documents - Contracts and Amendments - Correspondence - QAPP - SAP - Telephone Logbooks - 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 & Marketing	6 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 which provides a suitable environment to prevent damage or deterioration and to prevent loss. All records shall be protected against fire, theft, loss, environmental deterioration, and vermin. In the case of electronic records, electronic or magnetic sources, storage media are protected from deterioration caused by magnetic fields and/or electronic deterioration.

Access to the data is limited to laboratory and company employees. Records 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.

Uncontrolled Copy

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) 10 years - Radiochemistry (project records)
Drinking Water Lead and Copper Rule	12 years (project records)
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 for more information. For additional details please refer to refer to TestAmerica Edison SOP No. ED-GEN-024 (Record Storage and Retention).

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. Reference TestAmerica Edison SOP No. ED-GEN-024 (Record Storage and Retention).

Uncontrolled Copy

- Instrument data is stored sequentially by instrument. A given day's analyses are maintained in the order of the analysis. Run logs are maintained for each instrument or method; a copy of each day's run log or instrument sequence is stored with the data to aid in 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.

- 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 or on a benchsheet.

Uncontrolled Copy

- 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, 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 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

Uncontrolled Copy

- results of data review, verification, and crosschecking procedures

14.3.1 Sample Handling Records

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

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

14.4 Administrative Records

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

14.5 Records Management, Storage and Disposal

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

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

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

The laboratory has a record management system (a.k.a., document control) for control of laboratory notebooks, instrument logbooks, standards logbooks, and records for data reduction, validation, storage and reporting. Laboratory notebooks are issued on a per analysis basis, and are numbered sequentially. All data are recorded sequentially within a series of sequential notebooks. Bench sheets are filed sequentially. Standards are maintained in the LIMS – no logbooks are used to record that data. Records are considered archived when 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

Uncontrolled Copy

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 QA Technical Audits	Joint responsibility: a) QA Manager or designee b) Technical Manager or Designee (Refer to CW-Q-S-003)	QA Technical Audits Frequency: 50% of methods annually

Uncontrolled Copy

Description	Performed by	Frequency
SOP Method Compliance	Joint responsibility: a) QA Manager or designee b) Technical Manager or Designee (Refer to CW-Q-S-003)	SOP Compliance Review Frequency: • Every 2 years
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 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

15.1.3 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 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

Uncontrolled Copy

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 Hazardous Waste.

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

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

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.

Uncontrolled Copy

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 Managers 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 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 VPs of Operations.

16.2 Annual Management Review

The senior lab management team (Laboratory Director, Technical Managers, 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

Uncontrolled Copy

QA personnel is be included in this meeting at the discretion of the Laboratory Director. The LIMS review consists of examining any audits, complaints or concerns that have been raised through the year that are related to the LIMS. The laboratory will summarize any critical findings that cannot be solved by the lab and report them to Corporate IT.

This management systems review (Corporate SOP No. CW-Q-S-004 and 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 Operation 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

Uncontrolled Copy

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.

Uncontrolled Copy

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).

Uncontrolled Copy

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

As a general rule for analytical staff:

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

Uncontrolled Copy

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.

Further details of the laboratory's training program are described in the Laboratory Training SOP (TestAmerica Edison SOP No. ED-GEN-022).

Uncontrolled Copy

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 42,000 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.

Uncontrolled Copy

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.

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.

Uncontrolled Copy

Adequate measures are taken to ensure good housekeeping in the laboratory and to ensure that any contamination does not adversely affect data quality. These measures include regular cleaning to control dirt and dust within the laboratory. Work areas are available to ensure an unencumbered work area. Work areas include:

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

18.4 Floor Plan

A floor plan can be found in Appendix 1.

18.5 Building Security

Building keys are distributed to employees as necessary.

Visitors to the laboratory sign in and out in a visitor's logbook. A visitor is defined as any person who visits the laboratory who is not an employee of the laboratory. In addition to signing into the laboratory, the Environmental, Health and Safety Manual contains requirements for visitors and vendors. There are specific safety forms that must be reviewed and signed. Visitors (with the exception of company employees) are escorted by laboratory personnel at all times, or the location of the visitor is noted in the visitor's logbook.

SECTION 19. TEST METHODS AND METHOD VALIDATION

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.

Uncontrolled Copy

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.
- 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

Uncontrolled Copy

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*
- *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/ 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*

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.

Uncontrolled Copy

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) (reference TestAmerica Edison Training SOP No. ED-GEN-022) 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 Department Manager (i.e., 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).

Uncontrolled Copy

- The laboratory's nominal or default reporting limit (RL) is equal to the quantitation limit (QL), must be at or above the lowest non-zero standard in the calibration curve and must be reliably determined. Project RLs are client specified reporting levels which may be higher than the QL. Results reported below the QL must be qualified as estimated values. Also see Section 19.6.1.3, Relationship of Limit of Detection (LOD) to Quantitation Limit (QL).
- The client request is documented and the lab informs the client of its procedure for working with unusual compounds. The final report must be footnoted: *Reporting Limit based on the low standard of the calibration curve.*

19.4.3 Initial Demonstration of Capability (IDOC) Procedures

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

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

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

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

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

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

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

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

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

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

Uncontrolled Copy

Methods on line prior to the effective date of this Section shall be updated to the procedures outlined above as new analysts perform their demonstration of capability. A copy of the new record will replace that which was used for documentation in the past. At a minimum, the precision and accuracy of four mid-level laboratory control samples must have been compared to the laboratory's quality control acceptance limits.

19.5 Laboratory Developed Methods and Non-Standard Methods

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

19.6 Validation of Methods

Validation is the confirmation by examination and the provision of objective evidence that the particular requirements for a specific intended use are fulfilled.
All non-standard methods, laboratory designed/developed methods, standard methods used outside of their scope, and major modifications to published methods must be validated to confirm they are fit for their intended use. The validation will be as extensive as necessary to meet the needs of the given application. The results are documented with the validation procedure used and contain a statement as to the fitness for use.

19.6.1 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

Uncontrolled Copy

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. 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. CA-Q-S-006 for details on the laboratory's MDL process.

Uncontrolled Copy

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. 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

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.

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.

Uncontrolled Copy

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.

Uncontrolled Copy

- Homogenous samples: If a reanalysis agrees with the original result to within the RPD limits for MS/MSD or Duplicate analyses, or within ± 1 reporting limit for samples ≤ 5x the reporting limit, the original analysis will be reported. At the client's request, both results may be reported on the same report but not on two separate reports.

- If the reanalysis does not agree (as defined above) with the original result, then the laboratory will investigate the discrepancy and reanalyze the sample a third time for confirmation if sufficient sample is available.

- Any potential charges related to reanalysis are discussed in the contract terms and conditions or discussed at the time of the request. The client will typically be charged for reanalysis unless it is determined that the lab was in error.

- 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.

19.14 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 the TestAmerica Corporate IT SOPs and in TestAmerica Edison SOPs No. ED-GEN-001 (Data Management and Handling Procedures) and ED-GEN-002 (Document Control). The laboratory is currently running the TALS LIMS which is a custom in-house developed LIMS system that has been highly customized to meet the needs of the laboratory. It is referred to as LIMS for the remainder of this section. The LIMS utilizes Microsoft SQL Server which is an industry standard relational database platform. It is referred to as Database for the remainder of this section.

19.14.1.1 Maintain the Database Integrity – Assurance that data is reliable and accurate through data verification (review) procedures, password-protecting access, anti-virus protection, data change requirements, as well as an internal LIMS permissions procedure.

- LIMS Database Integrity is achieved through data input validation, internal user controls, and data change requirements.
- Spreadsheets and other software developed in-house must be verified with documentation through hand calculations prior to use. Cells containing calculations must be lock-protected and controlled.
- Instrument hardware and software adjustments are safeguarded through maintenance logs, audit trails and controlled access.

19.14.1.2 Ensure Information Availability – Protection against loss of information or service is ensured through scheduled back-ups, stable file server network architecture,

Uncontrolled Copy

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 Technical Manager or alternate analyst prior to updating the data in LIMS. The spreadsheets, or any other type of applicable documents, are signed by both the analyst and alternate reviewer to confirm the accuracy of the manual entry(s).

Manual integration of peaks will be documented and reviewed and the raw data will be flagged in accordance with the TestAmerica Corporate SOP No. CA-Q-S-002, *Acceptable Manual Integration Practices*.

Analytical results are reduced to appropriate concentration units specified by the analytical method, taking into account factors such as dilution, sample weight or volume, etc. Blank correction will be applied only when required by the method or per manufacturer's indication; otherwise, it should not be performed. Calculations are independently verified by appropriate laboratory staff. Calculations and data reduction steps for various methods are summarized in the respective analytical SOPs or program requirements.

19.14.2.1 All raw data must be retained in the worklist folder, computer file (if appropriate), and/or runlog. All criteria pertinent to the method must be recorded. The documentation is recorded at the time observations or calculations are made and must be signed or initialed/dated (month/day/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.

Uncontrolled Copy

19.14.2.5 The laboratory strives to import data directly from instruments or calculation spreadsheets to ensure that the reported data are free from transcription and calculation errors. For those analyses with an instrumental output compatible with the LIMS, the raw results and dilution factors are transferred into LIMS electronically after reviewing the quantitation report, and removing unrequested or poor spectrally-matched compounds. The analyst prints a copy of what has been entered to check for errors. This printout and the instrument's printout of calibrations, concentrations, retention times, chromatograms, and mass spectra, if applicable, are retained with the data file. The data file is stored in a monthly folder on the instrument computer; periodically, this file is transferred to the server and, eventually, to a tape file.

19.14.3 Logbook / Worksheet Use Guidelines

Logbooks and worksheets are filled out 'real time' and have enough information on them to trace the events of the applicable analysis/task. (e.g. calibrations, standards, analyst, sample ID, date, time on short holding time tests, temperatures when applicable, calculations are traceable, etc.)

- Corrections are made following the procedures outlined in Section 12.
- Logbooks are controlled by the QA department. A record is maintained of all logbooks in the lab.
- Unused portions of pages must be "Z"'d out, signed and dated.
- Worksheets are created with the approval of the Department 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 SOPs (including but not limited to, TestAmerica Edison SOP Nos. ED-GEN-021: Data Review, ED-SPM-001:Login, and ED-RP-001:Reports Production) to ensure that reported data are free from calculation and transcription errors, that QC parameters have been reviewed and evaluated before data is reported. The general review concepts are discussed below, more specific information can be found in the SOPs

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

Uncontrolled Copy

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.

Uncontrolled Copy

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).

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.

Uncontrolled Copy

Figure 19-1. Example - Demonstration of Capability Documentation

DEMONSTRATION OF CAPABILITIY (DOC)

Laboratory Name:_____
Laboratory Address:_____
Method:_____ Matrix:_____
Date:_____ Analyst(s):_____
Source of Analyte(s):_____

Analytical Results

Analyst	Conc. (Units)	Rep 1	Rep 2	Rep 3	Rep 4	Avg. % Recovery	% RSD

% RSD = Percent relative standard deviation = standard deviation divided by average % Recovery

Raw data reference: _____

Certification Statement:

We, the undersigned, certify that:
1. The cited test method has met Demonstration of Capability requirements.
2. The test method was performed by the analyst(s) identified on this certification.
3. A copy of the test method and the laboratory-specific SOPs are available for all personnel on site.
4. The data associated with the method 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, and the associated information is well organized and available for review.
6.

_____
Analyst Signature Date

_____
Technical Manager Signature Date

_____
Quality Assurance Coordinator Signature Date

Uncontrolled Copy

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. The most current list of laboratory instrumentation can be found in TestAmerica Edison Work Instruction No. ED-WI-002 (Equipment Inventory).

Equipment is only operated by authorized and trained personnel. 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 Technical 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.)

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.

Uncontrolled Copy

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, 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.

20.3.2 pH, Conductivity, and Turbidity Meters

The pH meters used in the laboratory are accurate to $\pm$ 0.1 pH units, and have a scale readability of at least 0.05 pH units. The meters automatically compensate for the temperature, and are calibrated with at least two working range buffer solutions before each use.

Conductivity meters are also calibrated before each use with a known standard to demonstrate the meters do not exceed an error of 1% or one umhos/cm.

Uncontrolled Copy

Turbidity meters are also calibrated before each use. All of this information is documented in logs.

Consult pH and Conductivity, and Turbidity SOPs for further information.

20.3.3 Thermometers

All thermometers are 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.

IR thermometers, digital probes and thermocouples are calibrated quarterly. 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 digital NIST thermometer is recalibrated every five years (unless thermometer has been exposed to temperature extremes or apparent separation of internal liquid) by an approved outside service and the provided certificate of traceability is kept on file. The NIST thermometer(s) have increments of 1 degree (0.5 degree or less increments are required for drinking water microbiological laboratories), and have ranges applicable to method and certification requirements. The NIST traceable thermometer is used for no other purpose than to calibrate other thermometers.

All of this information is documented in logbooks. Monitoring method-specific temperatures, including incubators, heating blocks, water baths, and ovens, is documented in method-specific logbooks. More information on this subject can be found in the laboratory SOP No. ED-GEN-014 (Thermometer Calibration).

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 > 0°C and ≤ 6 °C.

Specific temperature settings/ranges for other refrigerators, ovens waterbaths, and incubators can be found in method specific SOPs.

All of this information is documented in Daily Temperature Logbooks and method-specific logbooks.

Uncontrolled Copy

20.3.5 Autopipettors, Dilutors, and Syringes

Mechanical volumetric dispensing devices including burettes (except Class A Glassware and Glass microliter syringes) are given unique identification numbers and the delivery volumes are verified gravimetrically, at a minimum, on a quarterly basis.

For those dispensers that are not used for analytical measurements, a label is / can be applied to the device stating that it is not calibrated. Any device not regularly verified cannot be used for any quantitative measurements. Refer to TestAmerica Edison SOP No. ED-GEN-011 (Calibration and Use of Lab Pipettes).

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 as needed based on manufacturer's recommendations.

20.4 Instrument Calibrations

Calibration of analytical instrumentation is essential to the production of quality data. Strict calibration procedures are followed for each method. These procedures are designed to determine and document the method detection limits, the working range of the analytical instrumentation and any fluctuations that may occur from day to day.

Sufficient raw data records are retained to allow an outside party to reconstruct all facets of the initial calibration. Records contain, but are not limited to, the following: calibration date, method, instrument, analyst(s) initials or signatures, analysis date, analytes, concentration, response, type of calibration (Avg RF, curve, or other calculations that may be used to reduce instrument responses to concentration.)

Sample results must be quantitated from the initial calibration and may not be quantitated from any continuing instrument calibration verification unless otherwise required by regulation, method or program.

If the initial calibration results are outside of the acceptance criteria, corrective action is performed and any affected samples are reanalyzed if possible. If the reanalysis is not possible, any data associated with an unacceptable initial calibration will be reported with appropriate data qualifiers (refer to Section 12).

Note: Instruments are calibrated initially and as needed after that and at least annually *(the annual requirement does not apply to Isotope dilution).*

Uncontrolled Copy

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. 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.

Uncontrolled Copy

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 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).

Uncontrolled Copy

If the results of a CCV are outside the established acceptance criteria and analysis of a second consecutive (and immediate) CCV fails to produce results within acceptance criteria, corrective action shall be performed. Once corrective actions have been completed & documented, the laboratory shall demonstrate acceptable instrument / method performance by analyzing two consecutive CCVs, or a new initial instrument calibration shall be performed.

Sample analyses and reporting of data may not occur or continue until the analytical system is calibrated or calibration verified. However, data associated with an unacceptable calibration verification may be fully useable under the following special conditions:

a). when the acceptance criteria for the CCV are exceeded high (i.e., high bias) and the associated samples within the batch are non-detects, then those non-detects may be reported with a footnote or case narrative explaining the high bias. Otherwise the samples affected by the unacceptable CCV shall be re-analyzed after a new calibration curve has been established, evaluated and accepted; or

b). when the acceptance criteria for the CCV are exceeded low (i.e., low bias), those sample results may be reported if they exceed a maximum regulatory limit/decision level. Otherwise the samples affected by the unacceptable CCV shall be re-analyzed after a new calibration curve has been established, evaluated and accepted.

Samples reported by the 2 conditions identified above will be appropriately flagged.

20.4.1.2 Verification of Linear and Non-Linear Calibrations

Calibration verification for calibrations involves the calculation of the percent drift or the percent difference of the instrument response between the initial calibration and each subsequent analysis of the verification standard. These calculations are available in the laboratory method SOPs. Verification standards are evaluated based on the % Difference from the average CF or RF of the initial calibration or based on % Drift or % Recovery if a linear or quadratic curve is used.

Regardless of whether a linear or non-linear calibration model is used, if initial verification criterion is not met, then no sample analyses may take place until the calibration has been verified or a new initial calibration is performed that meets the specifications listed in the method SOPs. If the calibration cannot be verified after the analysis of a single verification standard, then adjust the instrument operating conditions and/or perform instrument maintenance, and analyze another aliquot of the verification standard. If the calibration cannot be verified with the second standard, then a new initial calibration is performed.

- 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

Uncontrolled Copy

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.

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.

Uncontrolled Copy

Table 20-1. Example: Instrumentation List

TestAmerica
THE LEADER IN ENVIRONMENTAL TESTING

TestAmerica Edison Instrument List

Instrument Type (Name)	Manufacturer	Model Number	Serial Number	Year Put into Service	Condition When Received
Metals Department					
ICP (ICP5)	Thermo Jarrell Ash	ICAP 6500 DUO View	IC5D20121709	2012	New
ICP (ICP4)	Thermo Jarrell Ash	ICAP 6500 DUO View	ICP-20073407	2009	New
ICP/MS (ICPMS1)	Agilent Technologies	7500CE	JP51201560	2006	New
Heat Exchanger	Agilent Technologies	3370	G57335		
Autosampler	Cetac	G3286A	US0808108A520		
ICP/MS (ICPMS2)	Agilent Technologies	7500CX	JP82802644	2010	New
Heat Exchanger	Agilent Technologies	3370	1C1160265	2006	Used
Autosampler	Cetac	ASX500	021134A520	2006	New
ICP/MS (ICPMS3)	Agilent Technologies	G8403A	JP16121357	2016	New
Heat Exchanger	PolyScience	G3292-80000	2U1570105	2016	New
Autosampler	Agilent Technologies	G8410A	AU16101128	2016	New
Mercury Analyzer 6	Leeman Labs	Hydra II	2008 112-00064-1	2014	New
Mercury Analyzer 7	Leeman Labs	Hydra II	US17159010	2017	New
Hot Block (Hot Block 2)	Environmental Express Limited	SC-154	2391CE1273	2004	New
Hot Block (Hot Block 3)	Environmental Express Limited	SC-150	4298CEC2048	2004	New
Hot Block (Hot Block 4)	Environmental Express Limited	SC-150	4507CEC2115	2006	New
Hot Block (Hot Block 5)	Environmental Express Limited	SC-150	466CEC2183	2006	New
Hot Block (Hot Block 7)	Environmental Express Limited	SC-150	2772CDC1378	2006	New
Hot Block (Hot Block 8)	Environmental Express Limited	SC154	1423CEC1154		
Hot Block (Hot Block 9)	Environmental Express Limited	SC-150	1917CEC1155		

TestAmerica
THE LEADER IN ENVIRONMENTAL TESTING

Instrument Type (Name)	Manufacturer	Model Number	Serial Number	Year Put Into Service	Condition When Received
Hot Block (Hot Block 11) **OUT FOR REPAIR – 11/7/17**	Environmental Express Limited	DigiPrep	4102030316		
Hot Block (Hot Block 12)	Environmental Express Limited	SC-154	3098CEC1508		
Top Loading Balance (Balance #35)	Acculab	VIC-412	18255989	2005	New
Top Loading Balance (Balance #33)	Ohaus	AR5120	F0461200521139	2001	New
Semivolatile GC/MS Department					
GC/MS System (BNAMS2)					
Gas Chromatograph	Agilent Technologies	6890A	CN10447031	TBD	New
MS Detector	Agilent Technologies	5973	US44647039	TBD	New
Autosampler Tower	Agilent Technologies	7683B	US82410944	TBD	New
Autosampler Tray	Agilent Technologies	7683	US85002901	TBD	New
GC/MS System (BNAMS4)					
Gas Chromatograph	Hewlett Packard	5890 Series II	3108A34490	1986	New
MS Detector	Hewlett Packard	5971A	3114A02077	1986	New
Autosampler Tower	Hewlett Packard	7673A	3429A33228	1986	New
Autosampler Tray	Hewlett Packard	7673	3624A42191	1986	New
Controller	Hewlett Packard		2803A11211	1986	New
GC/MS System (BNAMS5)					
Gas Chromatograph	Agilent Technologies	7890A	CN10726100	2005	New
MS Detector	Agilent Technologies	5975C	US35120328	2005	New
Autosampler Tower	Agilent Technologies	7673B	CN72441261	2005	New
Autosampler Tray	Agilent Technologies	7673	CN43830668	2005	New
Controller	Agilent Technologies		CN40427800	2005	New

Company Confidential & Proprietary

TestAmerica
THE LEADER IN ENVIRONMENTAL TESTING

Instrument Type (Name)	Manufacturer	Model Number	Serial Number	Year Put into Service	Condition When Received
GC/MS System (BNAMS9)					
Gas Chromatograph	Agilent Technologies	6890N	CN10349071	2004	New
MS Detector	Agilent Technologies	5973	US35120328	2004	New
Autosampler Tower	Agilent Technologies	7683	CN34433497	2004	New
Autosampler Tray	Agilent Technologies	7683	CN40327770	2004	New
Controller	Agilent Technologies		CN40427800	2004	New
GC/MS System (BNAMS11)					
Gas Chromatograph	Agilent Technologies	7890A	CN10727109	2007	New
MS Detector	Agilent Technologies	5975C	US71236621	2007	New
Autosampler Tower	Agilent Technologies	7683B	CN72441255	2007	New
Autosampler Tray	Agilent Technologies	7683	CN72544441	2007	New
Controller	Agilent Technologies			2007	New
GC/MS System (BNAMS12)					
Gas Chromatograph	Agilent Technologies	6890A	CN10531011	2012	New
MS Detector	Agilent Technologies	5975C	US52420834	2012	New
Autosampler Tower	Agilent Technologies	7683B	CN61732705	2012	New
Autosampler Tray	Agilent Technologies	7683	CN24322270	2012	New
Controller	Agilent Technologies				
GC/MS System (BNAMS13)					
Gas Chromatograph	Agilent Technologies	6890A	CN10529024	2012	New
MS Detector	Agilent Technologies	5975C	US10503712	2012	New
Autosampler Tower	Agilent Technologies	7683B	CN48320808	2012	New
Autosampler Tray	Agilent Technologies	7683	CN40327583	2012	New

Company Confidential & Proprietary

TestAmerica
THE LEADER IN ENVIRONMENTAL TESTING

Instrument Type (Name)	Manufacturer	Model Number	Serial Number	Year Put into Service	Condition When Received
Controller	Agilent Technologies			2012	New
GC/MS System (BNAMS14)					
Gas Chromatograph	Agilent Technologies	7890B	CN14393167	2012	New
MS Detector	Agilent Technologies	5975C	US83131020	2012	New
Autosampler Tower	Agilent Technologies	7683B	CN759A2046	2012	New
Autosampler Tray	Agilent Technologies	7683	CN72544431	2012	New
Controller	Agilent Technologies			2012	New
GC/MS System (BNAMS15)	Agilent Technologies				
Gas Chromatograph	Agilent Technologies	9000 Intuvo	CN17010006	2015	New
MS Detector	Agilent Technologies	5977A	US1433L427	2015	New
Autosampler Tower	Agilent Technologies	7963A	CN14410091	2015	New
Autosampler Tray	Agilent Technologies	7963	CN14360040	2015	New
GC/MS System (BNAMS16)					
Gas Chromatograph	Agilent Technologies	6890N	CN10402079	TBD	New
MS Detector	Agilent Technologies	5975	US55132182	TBD	New
Autosampler Tower	Agilent Technologies	7683	CN22025342	TBD	New
Autosampler Tray	Agilent Technologies	G2614A	CN404427800	TBD	New
GC/FID System (BNAGC8)					
Gas Chromatograph	Hewlett-Packard	5890	3121A35833	1986	New
Autosampler Tower	Hewlett-Packard	7673A	2704805765	1986	New
Autosampler Tray	Hewlett-Packard		3131A25914	1986	New
Controller	Hewlett-Packard		2921A03449	1986	New
Manifold	Western Enterprise	Innovator HBAC2-5-4	28452	2004	New

TestAmerica
THE LEADER IN ENVIRONMENTAL TESTING

Instrument Type (Name)	Manufacturer	Model Number	Serial Number	Year Put into Service	Condition When Received
Volatiles Department (GC and GC/MS)					
GC/MS System (VOAMS1)					
Gas Chromatograph	Agilent Technologies	6890N	CN10606023	2006	New
MS Detector	Agilent Technologies	5975	US60532504	2006	New
Autosampler	OI	4551A	D607458342	2006	New
Concentrator	OI	Eclipse 4660	D608466853	2006	New
Spiker	OI	SAM	E54175492	2006	New
GC/MS System (VOAMS2)					
Gas Chromatograph	Hewlett-Packard	7890A	CN10813013	2008	New
MS Detector	Hewlett-Packard	5975C	US80838709	2008	New
Autosampler	OI	4552 (Archon 5100)	14608	2008	New
Concentrator	OI	Eclipse 4660	K41746689OP	2008	New
GC/MS System (VOAMS3)					
Gas Chromatograph	Agilent Technologies	6890N	CN10406105	2004	New
MS Detector	Agilent Technologies	5973 Inert	US35120382	2004	New
Autosampler	EST	Centurion	CENT140051304	2004	New
Concentrator	EST	Encon	478021506	2004	New
Concentrator	EST	Encon	478021506	2004	New
GC/MS System (VOAMS4)					
Gas Chromatograph	Hewlett-Packard	7890A	CN10813014	2008	New
MS Detector	Hewlett-Packard	5975C	US80838712	2008	New
Autosampler	OI	4552	15264	2008	New
Concentrator	OI	2008	D809466076	2008	New
GC/MS System (VOAMS5)					
Gas Chromatograph	Agilent	6890	US10518019	TBD	New
MS Detector	Hewlett-Packard	5973	US44621422	TBD	New
Autosampler	OI	4552 (Archon 5100)	12206	1998	New
Concentrator	OI/EST	Encon	425042704	TBD	New
Concentrator	OI/EST	Encon	425042705	TBD	New

Company Confidential & Proprietary

TestAmerica
THE LEADER IN ENVIRONMENTAL TESTING

Instrument Type (Name)	Manufacturer	Model Number	Serial Number	Year Put into Service	Condition When Received
GC/MS System (VOAMS6)					
Gas Chromatograph	Agilent Technologies	6890N	CN10406076	2004	New
MS Detector	Agilent Technologies	5973 Inert	US35120322	2004	New
Autosampler	OI	4551A	D54645B461	2005	New
Concentrator	OI	4660	D548466579	2005	New
Spiker	OI	SAM	C425475656	2004	New
GC/MS System (VOAMS7)					
Gas Chromatograph	Agilent Technologies	6890N	CN10437064	2006	New
MS Detector	Agilent Technologies	5973 Inert	US43110514	2004	New
Autosampler	Teledyne Tekmar	Solatek	US08121007	2008	New
Concentrator	Teledyne Tekmar	Stratum	US08007007	2008	New
GC/MS System (VOAMS8)					
Gas Chromatograph	Hewlett-Packard	6890	US00028879	TBD	New
MS Detector	Hewlett-Packard	5973	US91411758	TBD	New
Autosampler	EST Archon	5100A	13097	TBD	New
Concentrator	EST Encon	4560	389092704E	TBD	New
GC/MS System (VOAMS9)					
Gas Chromatograph	Agilent	6890	CN10517107	TBD	New
MS Detector	Agilent	5973	US44610847	TBD	New
Autosampler	OI	4552	15266	2008	New
Concentrator	OI	4660	D548466579	TBD	New
GC/MS System (VOAMS10)					
Gas Chromatograph	Agilent	6890	CN10419047	TBD	New
MS Detector	Hewlett-Packard	5973	US10461695	TBD	New
Autosampler	OI	4100	D714410066	TBD	New
Concentrator	OI	4760	A714447065	TBD	New
GC/MS System (VOAMS11)					
Gas Chromatograph	Agilent Technologies	6890N	CN10324011	2003	New
MS Detector	Agilent Technologies	5973N	US30965664	2003	New

TestAmerica
THE LEADER IN ENVIRONMENTAL TESTING

Instrument Type (Name)	Manufacturer	Model Number	Serial Number	Year Put into Service	Condition When Received
Autosampler	EST Archon	5100A	13970	2003	New
Concentrator	EST	Encon Eclipse	A714447060	2017	New
GC/MS System (VOAMS12)					
Gas Chromatograph	Agilent Technologies	6890N	CN10439051	2005	New
MS Detector	Agilent Technologies	5973 inert	US43110519	2004	New
Autosampler	EST	5100A	14448	2005	New
Concentrator	EST	Encon	430051605	2005	New
Turbo Pump Upgrade	Agilent Technologies	Performance	56115832	2005	New
GC/MS System (VOAMS13)					
Gas Chromatograph	Agilent Technologies	6890N	CN10439052	2005	New
MS Detector	Agilent Technologies	5973 inert	US43110517	2004	New
Autosampler	EST	5100A	14449	2005	New
Concentrator	EST	Encon	431051605	2005	New
Turbo Pump Upgrade	Agilent Technologies	Performance	56069171	2005	New
GC/MS System (VOAMS14)					
Gas Chromatograph	Agilent Technologies	7890B	17293078	2017	New
MS Detector	Agilent Technologies	5977B	US1731M027	2017	New
Autosampler					
Concentrator					
GC/MS System (VOAMS15)					
Gas Chromatograph	Agilent Technologies	7890B	CN17293101	2017	New
MS Detector	Agilent Technologies	5977B	US1731M011	2017	New
Autosampler					
Concentrator					
GC/FID System (VOAGC1)					
Gas Chromatograph	Agilent Technologies	6890N	US10610006	2006	New
Autosampler	OI	4552	14608	2006	New
Concentrator	OI	4660	D607466340	2006	New
Autosampler	OI	4551A	D60745B342	2006	New

Company Confidential & Proprietary

TestAmerica
THE LEADER IN ENVIRONMENTAL TESTING

Instrument Type (Name)	Manufacturer	Model Number	Serial Number	Year Put into Service	Condition When Received
Concentrator	OI	4660	D607466341	2006	New
Spiker	OI	SAM	E610475713	2006	New
GC/FID System (VOAGC3)					
Gas Chromatograph	Hewlett-Packard	5890 II	3310A49242	1996	New
Autosampler	Dynatech Archon	5100	11780-795	1996	New
Concentrator	OI	4560	J437460274	1996	New
GC/FID System (VOAGC4)					
Gas Chromatograph	Hewlett-Packard	6890	00031073	TBD	New
Headspace Autosampler	Teledyne Tekmar	HT3	US17187006	TBD	New
Headspace Autosampler	Teledyne Tekmar	HT3	US1715201	TBD	New
GC/FID System (VOASCREEN5/6)					
Gas Chromatograph	Hewlett-Packard	5890 II	2921A23492	1993	New
Autosampler	Tekmar	7050	US04156005	2004	New
Headspace Sampler	Tekmar	7000	US04156003	2004	New
Autosampler	Tekmar	7050	US04148014	2004	New
Headspace Sampler	Tekmar	7000	US04163001	2004	New
Top Loading Balance (Balance #22)	Mettler	PB1501	2115517886	1997	New
Precision Balance (Balance #50)	Ohaus	Explorer Pro 413	1125573353	2006	New
Top Loading Balance (Balance #32)	Denver Instruments	P602	126008	2009	New
Drying Oven	Fisher Isotemp Oven	13-246-516G	502N0045	2005	New
Drying Oven	Baxter	DX-1	199012	2000	New
H-Nu PID	H-Nu Systems	PI101	801023	1989	New
Fume Hood	Air Science	PurAir15	P41007	2004	New
GC Semivolatile Department					
Analytical Balance (Balance #30)	A&D	HR200C	12315880		
Ion Chromatograph (IC-B)	Metrohm Peak, Inc.			2005	New
Pump 1	Metrohm Peak, Inc.	818	04187	2005	New
Pump 2	Metrohm Peak, Inc.	818	04197	2005	New

TestAmerica
THE LEADER IN ENVIRONMENTAL TESTING

Instrument Type (Name)	Manufacturer	Model Number	Serial Number	Year Put into Service	Condition When Received
Conductivity Detector	Metrohm Peak, Inc.	819	03195	2005	New
Injector and Oven	Metrohm Peak, Inc.	820	04147	2005	New
2-channel interface	Metrohm Peak, Inc.	830	04184	2005	New
Liquid Handling 1	Metrohm Peak, Inc.	833	04154	2005	New
Liquid Handling 12	Metrohm Peak, Inc.	833	04118	2005	New
Autosample	Metrohm Peak, Inc.	838	03198	2005	New
Ion Chromatograph (IC-A)	Metrohm Peak, Inc.			2010	New
Pump 1	Metrohm Peak, Inc.	818	SS4818011006190	2010	New
Pump 2	Metrohm Peak, Inc.	818	SS1818011003192	2010	New
UV-VIS Detector	Metrohm Peak, Inc.	1010 (Bischoff)	SS1153001010101	2010	New
Conductivity Detector	Metrohm Peak, Inc.	819	03181	2005	New
IC Interface	Metrohm Peak, Inc.	830	SS1830002003180	2010	New
Separation Center	Metrohm Peak, Inc.	820	SS1820023003168	2010	New
Sample Processor	Metrohm Peak, Inc.	838	SS1838001009171	2010	New
Filter Pump 1	Emerson		N SA55-NXGTB 4142		New
Filter Pump 2	Emerson	SA55JXgtd-4144	G8ECX	2002	New
Ion Chromatograph (IC-1)	Metrohm USA, Inc.	Compact IC Flex 930.2460		2017	New
Compact Flex Oven/Ses/Deg	Metrohm USA, Inc.	29302460	00103716	2017	New
Conductivity Detector	Metrohm USA, Inc.	28509010			
Dosino	Metrohm USA, Inc.	28000010	00099636		
MSM-A Rotor	Metrohm USA, Inc.	28509010	00102177		
Metrohm Data Management System	Metrohm USA, Inc.	66059321	JB5FKH2		

Company Confidential & Proprietary

TestAmerica
THE LEADER IN ENVIRONMENTAL TESTING

Instrument Type (Name)	Manufacturer	Model Number	Serial Number	Year Put into Service	Condition When Received
ProfIC Sample Processor Pump	Metrohm USA, Inc.	28580020	000091657		
Ion Chromatograph (IC-2)	Metrohm USA, Inc.	Compact IC Flex 930.2460		2017	New
Compact Flex Oven/Ses/Deg	Metrohm USA, Inc.	29302460	00103714	2017	New
Conductivity Detector	Metrohm USA, Inc.	28509010			
Dosino	Metrohm USA, Inc.	28000010	00099712		
Dosino Invial Dilution	Metrohm USA, Inc.	28000010	00099731		
MSM-A Rotor	Metrohm USA, Inc.	28509010	00102177		
Metrohm Data Management System	Metrohm USA, Inc.	66059321	JB5DKH2		
ProfIC Sample Processor Pump	Metrohm USA, Inc.	28580020	000098576		
GC/FID System (BNAGC1)					
Gas Chromatograph	Agilent Technologies	6890N	US10248079		
Autosampler	Agilent Technologies	G2913A	**CHECK**		
Autosampler	Agilent Technologies	G2913A	US95110886 (Compco)		
Autosampler Tray	Agilent Technologies	G2914A	CN82949935		
GC/FID System (BNAGC2)					
Gas Chromatograph	Agilent Technologies	6890N	US00005410		
Autosampler	Agilent Technologies	G2913A	US00210996		
Autosampler	Agilent Technologies	G2914A	**CHECK**		
Autosampler Tray	Agilent Technologies	G2914A	CN21420543		
GC/FID System (BNAGC3)					
Gas Chromatograph	Agilent Technologies	6890N	US10202132		

Company Confidential & Proprietary

TestAmerica
THE LEADER IN ENVIRONMENTAL TESTING

Instrument Type (Name)	Manufacturer	Model Number	Serial Number	Year Put into Service	Condition When Received
Autosampler	Agilent Technologies	G2913A	CN22025340		
Autosampler	Agilent Technologies	G2914A	US00210996		
Autosampler Tray	Agilent Technologies	G2914A	CN43830663		
GC/FID System (BNAGC4)					
Gas Chromatograph	Agilent Technologies	6890N	US10610005	2006	New
Autosampler	Agilent Technologies	G2913A	CN24428046	2006	New
Autosampler	Agilent Technologies	G2913A	CN43820799	2006	New
Autosampler Tray	Agilent Technologies	G2914A	CN43930714	2006	New
GC/ECD System (PESTGC1)					
Gas Chromatograph	Hewlett-Packard	5890A	2612A07669	1992	New
Autosampler	Hewlett-Packard	G1513A	US72202218	1992	New
Autosampler Tray	Hewlett-Packard	18596C	3401A34202	1992	New
Controller	Hewlett-Packard	G1512A	CN00005085	1992	New
GC/ECD System (PESTGC3)					
Gas Chromatograph	Hewlett-Packard	5890A	3223A42873	1992	New
Autosampler	Hewlett-Packard	18593B	3216A38325	1992	New
Autosampler Tray	Hewlett-Packard	18596B	3120A27740	1992	New
Controller	Hewlett-Packard	18594B	3049A23890	1992	New
GC/ECD System (PESTGC4)					
Gas Chromatograph	Hewlett-Packard	5890A Series II	US00024529	1997	New
Autosampler	Hewlett-Packard	18593B	**Check?**	1997	New
Autosampler Tray	Hewlett-Packard	7673	US13807350	1997	New

Company Confidential & Proprietary

TestAmerica
THE LEADER IN ENVIRONMENTAL TESTING

Instrument Type (Name)	Manufacturer	Model Number	Serial Number	Year Put into Service	Condition When Received
Controller	Hewlett-Packard	18594B	3227A29129	1997	New
GC/ECD System (PESTGC5)					
Gas Chromatograph	Agilent Technologies	6890N	US10226033	2002	New
Autosampler	Agilent Technologies	G2613A	US83301937	2002	New
Autosampler Tray	Agilent Technologies	G2614A	CN61739524	2002	New
GC/ECD System (PESTGC6)					
Gas Chromatograph	Hewlett-Packard	5890A	2950A26642	1998	New
Autosampler	Hewlett-Packard	18593B	US83301937	1998	New
Autosampler Tray	Hewlett-Packard	18596B	2942A20025	1998	New
Controller	Hewlett-Packard	G1512A	CN00004777	1998	New
GC/ECD System (PESTGC7)					
Gas Chromatograph	Hewlett-Packard	5890A	3029A29927	1998	New
Autosampler	Hewlett-Packard	18593A	3120A28315	1998	New
Autosampler Tray	Hewlett-Packard	18596B	3346A33861	1998	New
Controller	Hewlett-Packard	18594A	626059	1998	New
GC/ECD System (PESTGC8)					
Gas Chromatograph	Agilent Technologies	6890	US00004463	2000	New
Autosampler	Agilent Technologies	G1513A	**Check**	2000	New
Autosampler Tray	Agilent Technologies	18596M	3334A32973	2000	New
Controller	Agilent Technologies	G1512A	3631A05939	2000	New
GC/ECD System (PESTGC9)					
Gas Chromatograph	Agilent Technologies	6890	US00043694	2001	New
Autosampler	Agilent Technologies	G1513A	**Check**	2001	New

Company Confidential & Proprietary

TestAmerica
THE LEADER IN ENVIRONMENTAL TESTING

Instrument Type (Name)	Manufacturer	Model Number	Serial Number	Year Put into Service	Condition When Received
Autosampler Tray	Agilent Technologies	18596B	3228A29094	2001	New
Controller	Agilent Technologies	G1512A	CN00004150	2001	New
GC/ECD System (PESTGC11)					
Gas Chromatograph	Agilent Technologies	6890	US00008746	2003	New
Autosampler	Agilent Technologies	18593B	3120A28308	2003	New
Autosampler Tray	Agilent Technologies	18596C	3249A30680	2003	New
Controller	Agilent Technologies	G2512A	US72202100	2003	New
Wet Chemistry Department					
UV/VIS Spectrophotometer	HACH	DR2800	1205122	2007	New
UV/VIS Spectrophotometer	HACH	DR2800	1204684	2007	New
UV/VIS Spectrophotometer	HACH	DR2800	1120442	2007	New
Turbidimeter	HACH	HACH 2100N	12050C028810	2006	New
Ion Selective Meter	Orion	720A	006825	1994	New
Ion Selective Meter	Orion	720A+	092904	2007	New
pH Meter	Orion	A211	X16125	2017	New
pH Meter	Orion	A211	X39284	2017	New
pH Meter	Orion	A211	X37844	2017	New
pH Meter	Orion	A211	26256	2017	New
pH Meter	Orion	A211	Y28592	2016	New
Oven	VWR	1320	0402001	2001	New
Oven	VWR	1300U	VWR	2001	New
Oven	VWR	1305U	VWR	2001	New
Oven	Fisher	230G	Fisher	1997	New
Muffle Furnace	Fisher	550-14	901N002	2002	New
Drying Oven	VWR	1320	VWR	2001	New
Analytical Balance (Balance #27)	A&D	HR-200C	12315883	2005	New
Analytical Balance (Balance #29)	A&D	HR-200C	12315872	2005	New
Micro Balance (Balance #100)	Mettler	MX-5	122423439	2006	New
Top Loading Balance (Balance #13)	Sartorius	LC-421	50709085		
Top Loading Balance (Balance #200)	Denver Instruments	P-602	095010		
Analytical Balance (Balance #900)	Ohaus	GA-110	2220		
Top Loading Balance (Balance #25)	Sartorius		3403003		

Company Confidential & Proprietary

TestAmerica
THE LEADER IN ENVIRONMENTAL TESTING

Instrument Type (Name)	Manufacturer	Model Number	Serial Number	Year Put into Service	Condition When Received
Water Bath	Precision	50	9302-112	1995	New
Water Bath	Precision	50	9305-024	1995	New
COD Reactor	Hach	DRB 200	960800014651		
COD Reactor	Hach	DRB 200	13060C0213	2007	New
COD Reactor	Hach	DRB 200	1202323	2007	New
COD Reactor	Hach	DRB 200	1209887	2015	New
Flow Injection Autoanalyzer	Lachat	Quickem 8000	A83000	1997	New
Flow Injection Autoanalyzer	Lachat	8000 Series	8300-1658	2000	New
Total Organic Carbon (TOC) Analyzer	Shimadzu	TOC 5000	31242909	1997	New
Autosampler	Shimadzu	ASI 5000	31816800	1997	New
Solid Sample Module	Shimadzu	SSM-5000A	31303115	1997	New
Total Organic Carbon (TOC) Soil Analyzer #2	Thermo Electron Corp.	Flash EA 1112 Series	20034945	2004	New
Printer	Epson	LQ570	41NE28676	1997	New
Total Organic Carbon (TOC) Analyzer	Shimadzu	TOC_VCSH	H51104335164	2006	New
Autosampler	Shimadzu	ASI-V	H52104301656SA	2006	New
Solid Sample Module	Shimadzu	SSM-500A	H52504300040NK	2006	New
BOD Meter	YSI	5000	97S0534AE	1998	New
BOD Meter1 Rinse Pump Interface Control Box Automax 390 autosampler Software Seed pump Dilution pump Computer Monitor		PBM-BOD/AM39010 PC-1000-475 PC-1085-00 PB-10030 PC-1000-1074 PB-10600 PC-1000-408 PC-1000-443	 MT-1F7-414 MT-1G7-249 MT-1H7-200 MT-1H7-100 MT-1K6-559 MT-1G7-230 MXL7112917 CNK72411GS	2017	New
Incubator	GCA Precision Scientific			1998	New
Hot Plate	Corning		103N0071	2001	New
Hot Plate	Fischer Scientific	PC-400	370301092774	2007	New
Hot Plate	Corning	PC-420	390502148495	2007	New
Hot Plate	Fischer Scientific	PC-620	220897070707	2007	New
Conductivity Meter	Fischer Scientific	Accumetab 30	81209007	2002	New
Vortex Mixer	Thermolyne	M63215	632000855604	2002	New
Dishwasher	Miele Professional	G7783CD	208479	2003	New
Easy-Dist Distillation	Westco		1095	2003	New

Company Confidential & Proprietary

TestAmerica
THE LEADER IN ENVIRONMENTAL TESTING

Instrument Type (Name)	Manufacturer	Model Number	Serial Number	Year Put into Service	Condition When Received
Easy	Westco		J097	2003	New
Easy	Westco		1063	2007	New
Easy	Westco		1110	2007	New
Automated Discrete Analyzer #1	Konelab	20	S2019177	2003	New
Automated Discrete Analyzer #2	Konelab	20	2519236	2003	New
Computer	Dell		246175	2003	New
PC-Titration Plus Autotitrator	Mantech Assoc., Inc			2004	New
Interface		PC-1000-102/4	MS-0H4-373		
Titra-Rinse 1		PC-1000-408	MS-0G4-198		
Titra-Rinse 2		PC-1000-408	MS-0G4-200		
Buret Module 1		PC-1104-00	MS-0H4-627		
Buret Module 2		PC-1104-00	MS-0H4-625		
Titration Module		PC-1300-475	MS-0B5-657		
Redox Meter	VWR	8005	001149	1997	New
TCLP Extraction Laboratory					
Peristaltic Pump	PC-Titrate	PC-1000-40	MS-OF3-568	2004	New
TCLP Extraction Apparatus/Timer included	Assoc. Design and Mfg. Co.	3740-4 BRE	1320	2006	New
TCLP Extraction1 Apparatus/Timer included	Assoc. Design and Mfg. Co.	3740-12 BRE	1352	1997	New
TCLP Extraction2 Apparatus/Timer included	Assoc. Design and Mfg. Co.	3740-12 BRE	1053	1997	New
TCLP Extraction3 Apparatus/Timer included	Assoc. Design and Mfg. Co.	3740-12 BRE	1249	1997	New
TCLP Extraction4 Apparatus/Timer included	Environmental Express Ltd	LE 1002	3384-12-473	2005	New
TCLP Extraction5 Apparatus/Timer included	Environmental Express Ltd	LE 1002	3384-12-472	2005	New
TCLP Extraction6 Apparatus/Timer included	Assoc. Design and Mfg. Co.	3740-12 BREII	2125	2006	New
TCLP Extraction7 Apparatus/Timer included	Assoc. Design and Mfg. Co.	3740-12 BREII	2126	2006	New
Sample Control Department					
Top Loading Balance (Balance #104)	Denver Instruments	P602	126006		
Oven #1	Fisher	Isotemp 637G	410B01117	2005	New
Oven #2	Fisher	Isotemp 637G	505N0063	2005	New
Oven #3	Fisher	6921			
DI Water System (LOGIN)	Millipore		07348-C	1990	New

Company Confidential & Proprietary

TestAmerica
THE LEADER IN ENVIRONMENTAL TESTING

Instrument Type (Name)	Manufacturer	Model Number	Serial Number	Year Put into Service	Condition When Received
Organic Prep Department					
Analytical Balance (Balance #28)	A&D	HR200C	12315879		
Analytical Balance (Balance #A1)	Denver Instruments	P214	1050008		
Top Loading Balance (Balance #60)	Denver Instruments	P602	115003		
DI Water System	Barnstead	D11911	1191020210415	1995	New
Microwave Extraction System	MARS Xpress 230/60	907501	MD5095	2012	Inter-company transfer
Microwave Extraction System	MARS Xpress 230/60	907501	MD1952	2009	
Microwave Extraction System	MARS Xpress 230/60	907501	MD4965	2014	
Sonicator (Controller)	Sonic & Material, Inc.	VC750	58783	2012	Inter - company transfer
Sonicator Horn	Sonic & Material, Inc.	CV335	33107206	2012	Inter-company transfer
N-Evap #1	Organomation	112	52792	2004	New
N-Evap #2	Organomation	112	1694	1990	New
N-Evap #3	Organomation	112	59284	2014	New
N-Evap #4	Organomation	112	10203	2014	New
Water Bath #1	Fischer	15-491	605021280	2005	New
Water Bath #2	Fischer	15-491	204272	2007	New
Large Muffle Furnace	Wilt Industries	210	041213	2001	New
Dishwasher #1	Miele Professional	G7783CD	53081646	2003	New
Dishwasher #2	LabConco	4540031	130576299 E	2003	New
Vacuum Pump #1	Emerson Electric MLD	5KH36KN90HX	UNL231171	1990	New
Vortex Mixer	Scientific Industries	6560	2-318564	1995	New
Electric Mixer	Barnstead/Thermlyne		125404091646	1995	New
Mini Stirring Hotplate	VWR	220	33918-604	1995	New
Separatory Funnel Rotator 1	AP & R Machine & Tool		222307	2003	New
Separatory Funnel Rotator 2	AP & R Machine & Tool		222306	2003	New
Separatory Funnel Rotator 3	AP & R Machine & Tool		222305	2003	New

Company Confidential & Proprietary

TestAmerica
THE LEADER IN ENVIRONMENTAL TESTING

Instrument Type (Name)	Manufacturer	Model Number	Serial Number	Year Put into Service	Condition When Received
Separatory Funnel Rotator 4	AP & R Machine & Tool		222304	2003	New
Separatory Funnel Rotator 5	AP & R Machine & Tool		222303	2003	New
Separatory Funnel Rotator 6	AP & R Machine & Tool		222302	2003	New
Centriguge #1	Sigma	2-5	78646	2001	New
Field Services Department					
pH meter E-036	HACH	Sension1-5170060	10120C210304		New
pH meter E-019	HACH	Sension1-5170060	412000029333	2006	New
pH meter E-035	HACH	Sension1-5170060	08040C310395		
pH meter M-016	Hach	Sension1	050400022762	2005	New
pH meter M-039	Hach	Sension1	08040C410063	2006	New
pH meter M-031	Hach	HQ40D			
pH meter	Hach	Sension1	050600C10445	2005	New
pH meter E-036	HACH	5170060	0C260049		New
pH meter M-042	HACH	5170060	08110C111808		New
pH meter M-010	HACH	5170060	40300004162		New
pH meter E-021	ThermoOrion	230A	017900		New
pH meter E-033	ThermoOrion	230A	017788		New
pH/COND M-059	HACH	HQ40Dmulti	130800092563		New
pH+EC Sensor EP-091	HACH	5059	230		New
pH+ORP+EC Sensor MP-162	HACH	5048	151		New
DO meter M-056	HACH	Sension+DO6	401123		New
DO meter M-058	HACH	Sension+DO6	326101		New
DO meter M-032	HACH	Sension6-5185060	05070C360249	2006	New
DO meter E-037	HACH	Sension6-5185060	10120C260049		
DO meter M-027	Hach	Sension6	050500C60066	2005	New
DO meter M-007	Hach	Sension6	020900001321		New
DO meter	Hach	Sension6			New
DO meter M-015	Hach	Sension6	001200002352	2000	New
DO Meter E-037	HACH	5185000	0C260049		New
DO Meter	Hach	Sension +DO6	348155		New
DO Meter	Hach	Sension +DO6	403037		New
Conductivity Meter E-038	HACH	Sension5-5180060	10120C250073		New
Conductivity Meter M-028	HACH	Sension5-5180060	050500C50288	2005	New

Company Confidential & Proprietary

TestAmerica
THE LEADER IN ENVIRONMENTAL TESTING

Instrument Type (Name)	Manufacturer	Model Number	Serial Number	Year Put into Service	Condition When Received
Conductivity Meter E-006	HACH	Sension5-5180060	501000002708		New
Conductivity Meter M-018	HACH	Sension5	050300013668		New
Conductivity Meter M-0055	HACH	HQ40D	12080078247		New
Conductivity Meter	HACH	Sension5			New
Conductivity Meter M-014	HACH	Sension5	030500006215		New
Conductivity Meter E-038	HACH	5180000	0C250073		New
Conductivity Meter E-027	HACH	5180060	050500C50193		New
ORP Meter E-039	HACH	Sension1-5170060	10120C210070		New
ORP Meter M-060	HACH	HQ40Dmulti	130100083125		New
ORP Meter E-028	HACH	Sension1-5170060	40800010007		New
ORP Meter E-009	HACH	Sension1-5170060	402000003831		New
ORP Meter M-017	HACH	Sension1	05040002039		New
ORP Meter M-041	HACH	Sension1	08040C410042		New
ORP Meter	HACH	Sension1			New
ORP Meter M-006	HACH	Sension1	04030004238		New
ORP Meter E-039	HACH	5170000	0C210070		New
ORP Meter M-069	HACH	HQ40D	140300101090		New
Turbidity Meter E-056	Lamotte	2020we	584-0311		New
Turbidity Meter M-056	Lamotte	2020we	3458-2313		New
Turbidity Meter M-053	Lamotte	2020we	2404-3012		New
Turbidity Meter E-058	Lamotte	2020we	371-4610		New
Turbidity Meter M-047	Lamotte	2020we	567-0111		New
Turbidity Meter M-048	Lamotte	2020we	639-0411		New
Turbidity Meter	Lamotte	2020we			New
Turbidity Meter M-049	Lamotte	2020we	546-0111		New
Turbidity Meter M-066	Lamotte	2020we	4225-1014		New
Turbidity Meter M-054	Lamotte	2020we	2385-0312		New
Turbidity Meter E-059	Lamotte	2020we	3462-2313		New
Turbidity Meter E-055	Lamotte	2020we	180-3710		New
Turbidity Meter M-050	Lamotte	2020we	640-0411		New
Turbidity Meter EP-057	Lamotte	2020we	369-4610		New
Multi Meter (pH/Cond/ORP) M-064	Hach	HQ40D	131200097322		New
Multi Meter (pH/Cond/ORP) M-062	Hach	HQ40D	131100096885		New
Multi Meter (pH/Cond/ORP) M-068	Hach	HQ40D	140300101093		New
Chlorine Meter CL-007	HACH	Pocket Colorimeter II	040200011290	2006	New
Chlorine Meter CL-002	HACH	Pocket Colorimeter	020100174404	2006	New

Company Confidential & Proprietary

TestAmerica
THE LEADER IN ENVIRONMENTAL TESTING

Instrument Type (Name)	Manufacturer	Model Number	Serial Number	Year Put into Service	Condition When Received
Chlorine Meter CL-003	HACH	Pocket Colorimeter II	040200011345	2006	New
Chlorine Meter CL-004	HACH	Pocket Colorimeter	961200102549	2006	New
Chlorine Meter CL-006	HACH	Pocket Colorimeter	030400034505		New
Chlorine Meter CL-005	HACH	Pocket Colorimeter	020100174252		New
Chlorine Meter CL-008	HACH	Colorimeter 1200	4796-4900		New
PID	RAE	Min:RAE2000	110-010940		New
PID	RAE	Min:RAE2000	110-010984		New
Peristaltic Pump	Solonist	410	003270		New
Peristaltic Pump	Solonist	410	002642		
Peristaltic Pump	Solonist	410	002634		New
Peristaltic Pump	ISCO	ISCO 150	202F01835		New
Peristaltic Pump	ISCO	ISCO 150	195K01032		New
Peristaltic Pump	ISCO	ISCO 150	N/A		New
Peristaltic Pump	Sigma	N/A	22151		New
Peristaltic Pump	GeoPump	9001280	603003533		New
Composite sampler	ISCO	3700	204G00978		New
Composite sampler	ISCO	3700	210S00481		New
Composite sampler	ISCO	3700	211D00260		New
Composite sampler	ISCO	3700	212L00333		New
Grundfos	Grundfos	MP1/1A106003 P1	10460002		New
Grundfos	Grundfos	MP1/1A106003 P1	11030045		New
Grundfos	Grundfos	MP1/1A106003 P1	7460012		New
Grundfos	Grundfos	MP1/1A106003	98040466		New
Grundfos	Grundfos	MP1/1A106003	51418349		New
Sub Pump	Proactive	N/A	1371		New
Monsoon Control	Proactive	N/A	NA		New
Compressor	QED	3020	18323		New
Compressor	Coleman	CT5090412	D02812339		New
Compressor	Honda/Cambell	N/A	091593L689231VT69 7203AJ		New
Control Pack	QED	MP15	1278		New
Control Pack	QED	MP15	1299		New
Control Pack	QED	MP15	1079		New
Control Pack	QED	MP15	1528		New
Control Pack	QED	MP15	1280		New
Control Box	Grundfos	96765947	H100520040		New
Control Box	Grundfos	81128527	H0303130012		New
Control Box	Grundfos	91126028	H0412210115		New

Company Confidential & Proprietary

TestAmerica
THE LEADER IN ENVIRONMENTAL TESTING

Instrument Type (Name)	Manufacturer	Model Number	Serial Number	Year Put into Service	Condition When Received
Control Box	Grundfos	91126028	H0412210120		New
Control Box	Grundfos	SI-MP1115	151131		New
Control Box	Grundfos	SI-MP1115	203718		New
Bladder Pump	QED	MP-SPK-4P	12219		New
Bladder Pump	QED	MP-SPK-4P	10218		New
Bladder Pump	QED	MP-SPK-4P	12794		New
Bladder Pump	QED	MP-SPK-4P	12523		New
Bladder Pump	QED	MP-SPK-4P	11191		New
Bladder Pump	QED	MP-SPK-4P	11192		New
Bladder Pump	QED	MP-SPK-4P	11991		New
Bladder Pump	QED	MP-SPK-4P	11697		New
Bladder Pump	QED	MP-SPK-4P	11714		New
Bladder Pump	QED	MP-SPK-4P	12751		New
Bladder Pump	QED	MP-SPK-4P	11512		New
Bladder Pump	GeoTech	Model D 0.675	18		New
Bladder Pump	GeoTech	Model D 0.675	93		New
Generator	Honda	EU Inverter 2000	EAAS-2126666		New
Generator	Honda	EM 1800	E2CK-1011585		New
Generator	Honda	EM 3000	E2GL-1002925		New
Metal Detector	Fisher	FX-3	10123978		New
Marshaulk	N/A	N/A	N/A		New
Water Level Meter	Solonist	101	236477		New
Water Level Meter	Solonist	101	236505		New
Water Level Meter	Solonist	101	200769		New
Water Level Meter	Solonist	101	58356		New
Water Level Meter	Solonist	N/A	N/A		New
Interphase	Solonist	122	58167		New
Interphase	N/A	N/A	N/A		New
PID	RAE	MiniRae 2000	110-010953		New
PID	RAE	PGM-7615	103958		New
PID	RAE	MiniRae 3000	592-906947		New
YSI Meter	YSI Inc.	556MPS	06F1362 AC		New
Bladder Pump	QED	MP-SPK-4P	12870		New
Bladder Pump	QED	MP-SPK-4P	12175		New
Bladder Pump	QED	MP-SPK-4P	10996		New
Bladder Pump	QED	MP-SPK-4P	12023		New
Bladder Pump	QED	MP-SPK-4P	12752		New
Bladder Pump	QED	MP-SPK-4P	10997		New
Bladder Pump	QED	MP-SPK-4P	10200		New
Bladder Pump	QED	MP-SPK-4P	12087		New
Bladder Pump	QED	MP-SPK-4P	10649		New
Bladder Pump	QED	MP-SPK-4P	12568		New
Bladder Pump	QED	MP-SPK-4P	12654		New
Bladder Pump	Solinst	NA	NA		New
Bladder Pump	Solinst	NA	NA		New

Company Confidential & Proprietary

TestAmerica
THE LEADER IN ENVIRONMENTAL TESTING

Instrument Type (Name)	Manufacturer	Model Number	Serial Number	Year Put into Service	Condition When Received
Submersible Pump	Proactive	Monsoon XL	24730		New
Monsoon Control Box	Proactive	LCD Controller	H10867		New
Grundfos Pump	Grundfos	MP1/1A106003	044917629		New
Grundfos Pump	Grundfos	MP1/1A106003	06029591		New
Grundfos Pump	Grundfos	MP1/1A106003	051418361		New
Grundfos Pump	Grundfos	A 1A106003PI	10030064		New
Grundfos Pump	Grundfos	A 1A106003PI	07460012		New
Grundfos Pump	Grundfos	A 1A106003PI	11150009		New
Control Box	Grundfos	BMI/MP1-115V	9517		New
Control Box	Grundfos	SI/MP1-115/230V	203831		New
Control Box	Grundfos	IN2039B11	H1006080040		New
Control Box	Grundfos	IN0853A43	H0303130012		New
Control Box	Grundfos	IN0903A44	H0412210120		New
Control Pack	QED	MP-15	1181		New
Control Pack	QED	MP-15	1537		New
Control Pack	QED	MP-15	1615		New
Control Pack	QED	MP-15	1297		New
Control Pack	QED	MP-15	1060		New
Control Pack	QED	MP-15	1538		New
Control Pack	QED	MP-15	1180		New
Control Pack	QED	MP-15	1300		New
Peristaltic Pump	Solinst	410	002562		New
Peristaltic Pump	Solinst	410	002071		New
Peristaltic Pump	Solinst	410	001979		New
Peristaltic Pump	Solinst				New
Peristaltic Pump	GeoTech	900-1280	J02003143		New
Interface Meter	Solinst	122	59307		New
Interface Meter	Solinst	122	122 008699-1		New
Water Level Meter	Solinst	101	50333		New
Water Level Meter	Solinst	101	200766		New
Water Level Meter	Solinst	101	50331		New
Water Level Meter	Solinst	101	236317		New
Water Level Meter	Solinst	101	35312		New
Water Level Meter	Solinst	101	222654		New
Composite sampler	ISCO	3700 Sampler	212L00327		New
Composite sampler	ISCO	3700 Sampler	205C01380		New
Composite sampler	ISCO	3700 Sampler	205C01376		New
Composite sampler	ISCO	2700 Sampler	04939-054		New
Composite sampler	ISCO	2910 Sampler	197J00505		New
Composite sampler	ISCO	2700 Sampler	06522-094		New
Metal Detector	Chicago Steel Tape	Magna-Trak 102	102066024		New
Metal Detector	Chicago Steel Tape	Magna-Trak 102			New
Fresh Water Pump	NorthStar	10633	2252957		New

Company Confidential & Proprietary

TestAmerica
THE LEADER IN ENVIRONMENTAL TESTING

Instrument Type (Name)	Manufacturer	Model Number	Serial Number	Year Put into Service	Condition When Received
Generator	Honda	EB 3000C	EZGP-1145763		New
Generator	Honda	EB 3000C	EZGP-1151238		New
Generator	Honda	EM 3000C	EZGL-1002930		New
Compressor	Industrial Air	CTA5090412.GNE	K13110207A		New
Power Inverter	N Power	NA	120270929		New
PID	PE Photovac	2020	DQ-GD-302		New
Control Box	Grundfos	SI/MP1-115/230V	9909		New
Generator	Troy Bilt	01924	1010694921		New
Composite sampler	ISCO	2710 Sampler	05248-001		New
Mini Interface Meter	Water Mark	H01L/SM01L	01-1096		New
Interface Meter	Testwell Inc.	NA	NA		New
Water Level Meter	Fisher	M-Scope	NA		New
Water Level Meter	Fisher	M-Scope	NA		New
Varistaltic Pump	Manostat	72-310-000	M97005467		New
Utility Pump	TEEL	2P110A	1962		New
Utility Pump	TEEL	2P110B	3021		New
Utility Pump	TEEL	2P110B	0034		New
Utility Pump	TEEL	2P110B	0036		New

Company Confidential & Proprietary

Table 20-2. Example: Schedule of Routine Maintenance

Table 20-2. Example: Schedule of Routine Maintenance		
Instrument	**Procedure**	**Frequency**
Leeman Mercury Analyzer	Check tubing for wear	Daily
	Fill rinse tank with 10% HCl	Daily
	Change dryer tube	As needed
	Fill reductant bottle with 10% Stannous Chloride	Daily
ICP	Check pump tubing	Daily
	Check liquid argon supply	Daily
	Check fluid level in waste container	Daily
	Check filters	Weekly
	Clean or replace filters	As required
	Check torch	Daily
	Check sample spray chamber for debris	Monthly
	Clean and align nebulizer	Monthly
	Check entrance slit for debris	Monthly
	Change printer ribbon	As required
	Replace pump tubing	As required
ICP MS	Change pump tubing	Weekly or As required
	Clean torch	Weekly or As required
	Check / clean nebulizer	Weekly or As required
	Clean cones	Weekly or As required
	Check air filters	Weekly or As required
	Check multiplier voltages & do cross calibration	Weekly or As required
	Replace sample uptake tubing	Weekly or As required
	Check rotary pump oil	Weekly or As required
	Check oil mist filters	Monthly
	Check chiller water level	Monthly
UV-Vis Spectrophotometer	Clean ambient flow cell	As required
	Precision check/alignment of flow cell	As required
	Wavelength verification check	Semi-annually
Auto Analyzers	Clean sampler	Daily
	Check all tubing	Daily
	Clean inside of colorimeter	Daily
	Clean pump well and pump rollers	Quarterly
	Clean wash fluid receptacle	Weekly
	Oil rollers/chains/side rails	Weekly
	Clean optics and cells	Quarterly

Uncontrolled Copy

Table 20-2. Example: Schedule of Routine Maintenance

Instrument	Procedure	Frequency
Gas Chromatograph/Mass Spectrometer (GC/MS)	Ion gauge tube degassing	As required
	Pump oil-level check	Monthly
	Pump oil changing	Annually
	Analyzer bake-out	As required
	Analyzer cleaning	As required
	Resolution adjustment	As required
	COMPUTER SYSTEM AND PRINTER:	As required
	Air filter cleaning	As required
	Change data system air filter	As required
	Printer head carriage lubrication	As required
	Paper sprocket cleaning	As required
	Drive belt lubrication	
Gas Chromatograph	Compare standard response to previous day or since last initial calibration	Daily
	Check carrier gas flow rate in column	Daily via use of known compound retention
	Check temp. of detector, inlet, column oven	Daily
	Septum replacement	As required
	Glass wool replacement	As required
	Check system for gas leaks with SNOOP	W/cylinder change as required
	Check for loose/frayed wires and insulation	Monthly
	Bake injector/column	As Required
	Change/remove sections of guard column	As Required
	Replace connectors/liners	As Required
	Change/replace column(s)	As Required
Electron Capture Detector (ECD)	Detector wipe test (Ni-63)	Semi-annually
	Detector cleaning	As required
Flame Ionization Detector (FID)	Detector cleaning	As required
Photoionization Detector (PID)	Change O-rings	As required
	Clean lamp window	As required
HPLC	Change guard columns	As required
	Change lamps	As required
	Change pump seals	Semi-annually or as required
	Replace tubing	As required
	Change fuses in power supply	As required
	Filter all samples	Daily
	Change autosampler rotor/stator	As required
Balances	Class "S" traceable weight check	Daily, when used
	Clean pan and check if level	Daily
	Field service	At least Annually
Conductivity Meter	0.01 M KCl calibration	Daily
	Conductivity cell cleaning	As required
Turbidimeter	Check light bulb	Daily, when used
Deionized/Distilled Water	Daily conductivity check	Daily
	Check deionizer light	Daily
	Monitor for VOA's	Daily
	System cleaning	As required
	Replace cartridge & large mixed bed resins	As required

Uncontrolled Copy

Table 20-2. Example: Schedule of Routine Maintenance

Instrument	Procedure	Frequency
Drying Ovens	Temperature monitoring Temperature adjustments	Daily As required
Refrigerators/ Freezers	Temperature monitoring Temperature adjustment Defrosting/cleaning	Daily As required As required
Vacuum Pumps/ Air Compressor	Drained Belts checked Lubricated	Weekly Monthly Semi-annually
pH/Specific Ion Meter	Calibration/check slope Clean electrode	Daily As required
BOD Incubator	Temperature monitoring Coil and incubator cleaning	Daily Monthly
Centrifuge	Check brushes and bearings	Every 6 months or as needed
Water baths	Temperature monitoring Water replaced	Daily Monthly or as needed

Uncontrolled Copy

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 Laboratory Accreditation Cooperation) or APLAC (Asia–Pacific Laboratory Accreditation Cooperation). A calibration certificate and scope of accreditation is kept on file at the laboratory.

21.3 Reference Standards / Materials

Reference standards/materials, where commercially available, are traceable to certified reference materials. Commercially prepared 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

Uncontrolled Copy

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 maintained applicable analytical Departments. Records must be kept of the date of receipt and date of expiration of standards, reagents and reference materials. In addition, records of preparation of laboratory standards, reagents, and reference materials must be retained, stored appropriately, and be readily available for use and inspection. For detailed information on documentation and labeling, please refer to method specific SOPs.

Commercial materials purchased for preparation of calibration solutions, spike solutions, etc.., are usually accompanied with an assay certificate or the purity is noted on the label. If the assay purity is 96% or better, the weight provided by the vendor may be used without correction. If the assay purity is less than 96% a correction will be made to concentrations applied to solutions prepared from the stock commercial material. 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.

Uncontrolled Copy

21.4.1 All standards, reagents, and reference materials must be labeled in an unambiguous manner. Standards are logged into the laboratory's LIMS system, and are assigned a unique identification number. The following information is typically recorded in the electronic database within the LIMS.

- Standard ID
- Description of Standard
- Department
- Preparer's name
- Final volume and number of vials prepared
- Solvent type and lot number
- Preparation Date
- Expiration Date
- Standard source type (stock or daughter)
- Standard type (spike, surrogate, other)
- Parent standard ID (if applicable)
- Parent Standard Analyte Concentration (if applicable)
- Parent Standard Amount used (if applicable)
- Component Analytes
- Final concentration of each analyte
- Comment box (text field)

Records are maintained electronically for standard and reference material preparation. These records show the traceability to purchased stocks or neat compounds. These records also include method of preparation, date of preparation, expiration date and preparer's name or initials. Preparation procedures are provided in the Method SOPs.

21.4.2 All standards, reagents, and reference materials must be clearly labeled with a minimum of the following information:

- Expiration Date (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 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 by the facility Environmental Health and Safety Coordinator.

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)

Uncontrolled Copy

- 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 provides sampling services. Sampling procedures are described in the following SOPs as applicable::

- Groundwater Sampling (TestAmerica Edison SOP #s ED-FLD-008 and ED-FLD-009)
- Wastewater Sampling (TestAmerica Edison SOP # ED-FLD-014)
- Potable Sampling
- Waste Sampling
- Soil and Sediment Sampling
- Flow Monitoring (TestAmerica Edison SOP #s ED-FLD-008 and ED-FLD-009)
- Field Parameter Analysis (TestAmerica Edison SOPs ED-FLD-001 thru ED-FLD-007, ED-FLD-010)
- Cleaning and Decontamination of Field Equipment (see individual SOPs listed above and TestAmerica Edison SOP# ED-GEN-013)

Uncontrolled Copy

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 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 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.

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.

Uncontrolled Copy

Analysts should handle each sample as if it is potentially dangerous. At a minimum, safety glasses, gloves, and lab coats must be worn when preparing aliquots for analysis.

Guidelines on taking sample aliquots & subsampling are located SOP No. ED-GEN-007 (Subsampling).

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

Uncontrolled Copy

- 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

The laboratory may, upon special request, adhere to legal/evidentiary chain of custody requirements. If TestAmerica agrees to such procedures the samples are identified for legal/evidentiary purposes on the COC, login will complete the custody seal retain the shipping record with the COC, and initiate an internal COC for laboratory use by analysts and a sample disposal record.

23.2 Sample Receipt

Samples are received at the laboratory by designated sample receiving personnel and a unique laboratory project identification number is assigned. Each sample container shall be assigned a unique sample identification number that is cross-referenced to the client identification number such that traceability of test samples is unambiguous and documented. Each sample container is affixed with a durable sample identification label. Sample acceptance, receipt, tracking and storage procedures are summarized in the following sections.

23.2.1 Laboratory Receipt

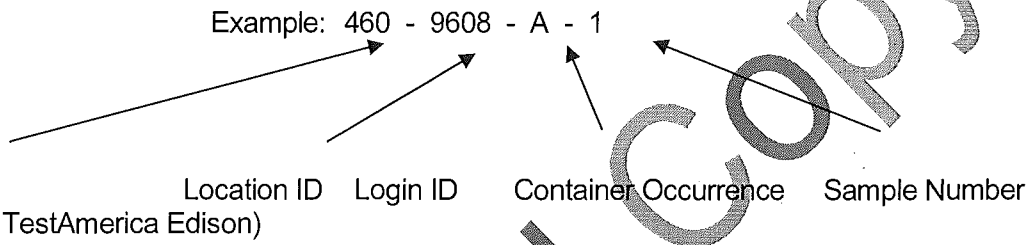
When samples arrive at the laboratory, sample receiving personnel inspect the coolers and samples. The integrity of each sample must be determined by comparing sample labels or tags with the COC and by visual checks of the container for possible damage. Any non-conformance, irregularity, or compromised sample receipt must be documented on a via the Sample Receipt application within TALS (the laboratory LIMS) 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.

Uncontrolled Copy

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 Edison Laboratory (Location 460). 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: 460 - 9608 - A - 1 - **A** ←—— **Secondary Container Occurrence**

Example: 460-9608-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);

Uncontrolled Copy

- 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.

Once sample acceptance is verified, the samples are logged into the LIMS according SOP No. ED-SPM-001.

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. Sample containers designated for metals only analysis are stored un-refrigerated. In addition, samples to be analyzed for volatile organic parameters are stored in separate refrigerators designated for volatile organic parameters only. Samples are never to be stored with reagents, standards or materials that may create contamination.

To ensure the integrity of the samples during storage, refrigerator blanks are maintained in the volatile sample refrigerators and analyzed every 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 30 days after delivery of the final report to the client, which meets or exceeds most sample holding times. After 30 days the samples are disposed of or, upon client request moved to a sample archive area where they are stored for an additional time period agreed upon with the client or dictated by the applicable analytical program (ex. USEPA CLP).

Uncontrolled Copy

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, hazardous and foreign soil samples are stored in an isolated area designated for hazardous waste only.

Procedures for the handling and storage of hazardous samples are addressed in the TestAmerica Corporate Safety Manual (TestAmercia Document No. CW-E-M-001) and in TestAmerica Edison SOP No. ED-SPM-001 (Sample Receipt, Login, Identification, and Storage).

Procedures for the acceptance and handling of USDA regulated domestic and foreign soils are detailed in TestAmerica SOP No. ED-SPM-006 (Procedure for Acceptance and Handling of Regulated Domestic and Foreign Soils).

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. 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

Uncontrolled Copy

may be disposed of in accordance with the laboratory's waste disposal procedures, TestAmerica Edison SOP No. ED-SPM-007 (Disposal of Samples and Associated Laboratory Waste). All procedures in the laboratory Environmental, Health and Safety Manual are followed during disposal. Samples are normally maintained in the laboratory no longer than 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).

Uncontrolled Copy

Figure 23-1. Example: Chain of Custody (COC)

CHAIN OF CUSTODY / ANALYSIS REQUEST

TestAmerica Edison
777 New Durham Road
Edison, New Jersey 08817
Phone: (732) 549-3900 Fax: (732) 549-3679

Page ___ of ___

Name (for report and Invoice)	Samplers Name (Printed)	Site/Project Identification
Company	P.O. #	State (Location of site): NJ: ☐ NY: ☐ Other:
		Regulatory Program:
Address	Analysis Turnaround Time Standard ☐	ANALYSIS REQUESTED (ENTER "X" BELOW TO INDICATE REQUEST)
City State	Rush Charges Authorized For: 2 Week ☐	
Phone Fax	1 Week ☐ Other ☐ _____	

Sample Identification	Date	Time	Matrix	No. of Cont.										LAB USE ONLY Project No: Job No: Sample Numbers
Preservation Used: 1 = ICE, 2 = HCl, 3 = H_2SO_4, 4 = HNO_3, 5 = NaOH				Soil:										
6 = Other _____, 7 = Other _____				Water:										

Special Instructions _____ Water Metals Filtered (Yes/No)? _____

Relinquished by	Company	Date / Time	Received by	Company
1)		/	1)	
Relinquished by	Company	Date / Time	Received by	Company
2)		/	2)	
Relinquished by	Company	Date / Time	Received by	Company
3)		/	3)	
Relinquished by	Company	Date / Time	Received by	Company
4)		/	4)	

Laboratory Certifications: New Jersey (12028), New York (11452), Pennsylvania (68-522), Connecticut (PH-0200), Rhode Island (132).

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.

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 collectors name
   - The matrix description
   - The container description
   - The total number of each type of container
   - Preservatives used
   - Analysis requested
   - Requested turnaround time (TAT)
   - Any special instructions
   - Purchase Order number or billing information (e.g. quote number) if available
   - The date and time that each person received or relinquished the sample(s), including their signed name.
   - The date and time of receipt must be recorded between the last person to relinquish the samples and the person who receives the samples in the lab, and they must be exactly the same.
   - Information must be legible

2) Samples must be properly labeled.
   - Use durable labels (labels provided by TestAmerica are preferred)
   - Include a unique identification number
   - Include sampling date and time & sampler ID
   - Include preservative used.
   - Use indelible ink
   - Information must be legible

3) Proper sample containers with adequate volume for the analysis and necessary QC are required for each analysis requested.

Uncontrolled Copy

4) Samples must be preserved according to the requirements of the requested analytical method.

Uncontrolled Copy

5) Most analytical methods require chilling samples to 4° C (other than water samples for metals analysis). For these methods, the criteria are met if the samples are chilled to below 6° C 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. **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).

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 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 (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.

Uncontrolled Copy

- 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 (< 48hr HT) sample must be received with at least 48 hrs (2 working days) remaining on the holding time for us to ensure analysis.

   - Analyses that are designated as "field" analyses (Odor, pH, Dissolved Oxygen, Disinfectant Residual; a.k.a. Residual Chlorine, and Redox Potential) should be analyzed ASAP by the field sampler prior to delivering to the lab (within 15 minutes). However, if the analyses are to be performed in the laboratory, TestAmerica will make every effort to analyze the samples within 24 hours from receipt of the samples in the testing laboratory. Samples for "field" analyses received after 4:00 pm on Friday or on the weekend will be analyzed no later than the next business day after receipt (i.e., Monday, unless Monday is a holiday). Samples will remain refrigerated and sealed until the time of analysis. The actual times of all "field" sample analyses are noted 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, do not seal very well and are prone to intrusion from the water which results from melted ice.

   - Water samples are best package when wrapped with bubble-wrap or paper (newspaper, or paper towels) and then placed in plastic zip-lock bags.

   - Fill cooler void spaces with bubble wrap.

Uncontrolled Copy

Figure 23-3. Example: Cooler Receipt Form

TestAmerica Edison
Receipt Temperature and pH Log

Page ___ of ___

Job Number: _____

Number of Coolers: _____ IR Gun # _____

Cooler Temperatures

	RAW	CORRECTED		RAW	CORRECTED		RAW	CORRECTED
Cooler #1:	°C	°C	Cooler #4:	°C	°C	Cooler #7:	°C	°C
Cooler #2:	°C	°C	Cooler #5:	°C	°C	Cooler #8:	°C	°C
Cooler #3:	°C	°C	Cooler #6:	°C	°C	Cooler #9:	°C	°C

TALS Sample Number	Ammonia (pH<2)	COD (pH<2)	Nitrate Nitrite (pH<2)	Metals* (pH<2)	Hardness (pH<2)	Pest (pH 5-9)	EPH or QAM (pH<2)	Phenols (pH<2)	Sulfide (pH>9)	TKN (pH<2)	TOC (pH<2)	Total Cyanide (pH>12)	Total Phos (pH<2)	Other	Other

If pH adjustments are required record the information below:

Sample No(s). adjusted: _____

Preservative Name/Conc.: _____ Volume of Preservative used (ml): _____

Lot # of Preservative(s): _____ Expiration Date: _____

The appropriate Project Manager and Department Manager should be notified about the samples which were pH adjusted.

**Samples for Metal analysis which are out of compliance must be acidified at least 24 hours prior to analysis.*

EDS-WI-038, Rev 4, 06/09/2014

Initials: _____ Date: _____

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. 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)	are used to assess preparation and analysis for possible contamination during the preparation and processing steps.
	The specific frequency of use for method blanks during the analytical sequence is defined in the specific standard operating procedure for each analysis. Generally it is 1 for each batch of samples; not to exceed 20 environmental samples.
	The method blank is prepared from a clean matrix similar to that of the associated samples that is free from target analytes (e.g., Reagent water, Ottawa sand, glass beads, etc.) and is processed along with and under the same conditions as the associated samples. The method blank goes through all of the steps of the process (including as necessary: filtration, clean-ups, etc.).
	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.

Uncontrolled Copy

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) 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

Uncontrolled Copy

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.

Uncontrolled Copy

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.

Uncontrolled Copy

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 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 ≤ 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.

24.6.1.1 The QA Department generates and reviews Quality Control Limit Summaries using the TALS Control Chart module. These tables summarize the updated, proposed precision and accuracy acceptability limits for each applicable analysis performed at TestAmerica Edison. Once the QA Department is satisfied that the proposed limits are satisfactory the tables are forwarded to the applicable Department (Technical) Manager for final review. Once the proposed limits have been reviewed they entered into the appropriate TALS Method Limit Group database and approved for use (effectively replacing the existing limits in the database). The Quality Assurance Department maintains an archive of all limits used within the laboratory. Reference TestAmerica

Uncontrolled Copy

Edison SOP No. ED-GEN-026 (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.

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.

Uncontrolled Copy

SECTION 25. REPORTING RESULTS

25.1 Overview

The results of each test are reported accurately, clearly, unambiguously, and objectively in accordance with State and Federal regulations as well as client requirements. Analytical results are issued in a format that is intended to satisfy customer and laboratory accreditation requirements as well as provide the end user with the information needed to properly evaluate the results. Where there is conflict between client requests and laboratory ethics or regulatory requirements, the laboratory's ethical and legal requirements are paramount, and the laboratory will work with the client during project set up to develop an acceptable solution. Refer to Section 7.

A variety of report formats are available to meet specific needs.

In cases where a client asks for simplified reports, there must be a written request from the client. There still must be enough information that would show any analyses that were out of conformance (QC out of limits) and there should be a reference to a full report that is made available to the client. Review of reported data is included in Section 19.

25.2 Test Reports

Analytical results are reported in a format that is satisfactory to the client and meets all requirements of applicable accrediting authorities and agencies. A variety of report formats are available to meet specific needs. The report is printed on laboratory letterhead, reviewed, and signed by the appropriate project manager. At a minimum, the standard laboratory report shall contain the following information:

25.2.1 A report title (e.g. 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.

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

Uncontrolled Copy

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 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.

Uncontrolled Copy

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 a separate report on the official letterhead of the subcontractor. All TestAmerica subcontracting is clearly identified on the report as to which laboratory performed a specific analysis.

25.2.28 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 I is a report with the features described in Section 25.2 above.
- Level II (also called 'Results/QA) is a Level I report plus summary information, including results for the method blank reported to the laboratory MDL, percent recovery for laboratory control samples and matrix spike samples, and the RPD values for all MSD and sample duplicate analyses.
- NJDEP Reduced Deliverables Format which contains, at minimum, the elements listed in the current NJDEP Technical Requirements for Site Remediation, N.J.A.C. 7:26E.
- NJDEP Full Deliverables Format (Non-USEPA CLP Methods) which contains, at minimum, the elements listed in the current NJDEP Technical Requirements for Site Remediation, N.J.A.C. 7:26E.
- NJDEP Full Deliverables Format (USEPA CLP Methods) which contains, at minimum, the elements listed in the current NJDEP Technical Requirements for Site Remediation, N.J.A.C. 7:26E.
- NYSDEC ASP 'A' and 'B' Deliverables Format which contain, at minimum, the elements listed in the current *New York State Department of Environmental Conservation Analytical Services Protocol*.

In addition to the various levels of QC packaging, the laboratory also provides reports in diskette deliverable form. Initial reports may be provided to clients by facsimile. Procedures used to ensure client confidentiality are outlined in Section 25.6.

Uncontrolled Copy

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 Edison offers a variety of EDD formats including NJ Hazsite Deliverables, Excel, Dbase, GISKEY, and Text Files.

EDD specifications are submitted to the IT department by the PM for review and undergo the contract review process. Once the facility has committed to providing data in a specific electronic format, the coding of the format may need to be performed. This coding is documented and validated. The validation of the code is retained by the IT staff coding the EDD.

EDDs shall be subject to a review to ensure their accuracy and completeness. If EDD generation is automated, review may be reduced to periodic screening if the laboratory can demonstrate that it can routinely generate that EDD without errors. Any revisions to the EDD format must be reviewed until it is demonstrated that it can routinely be generated without errors. If the EDD can be reproduced accurately and if all subsequent EDDs can be produced error-free, each EDD does not necessarily require a review.

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

Uncontrolled Copy

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.

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, include 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.

Uncontrolled Copy

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 "Rev (n)" where 'n' is the revision number. The revised report will have the words "Revision (n)" on the report cover page beneath the report date. Additionally, a section entitled"Revised Report" will appear on the Case Narrative page. A brief explanation of the reasons for the re-issue will be included in this section.

When the report is re-issued, a notation of "report re-issue "is placed on the cover/signature page of the report *or at the top of the narrative page* with a brief explanation of reason for the re-issue and a reference back to the last final report generated. *For Example: Report was revised on 11/3/08 to include toluene in sample NQA1504 per client's request. This final report replaces the final report generated on 10/27/08 at 10:47am.*

25.9 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.

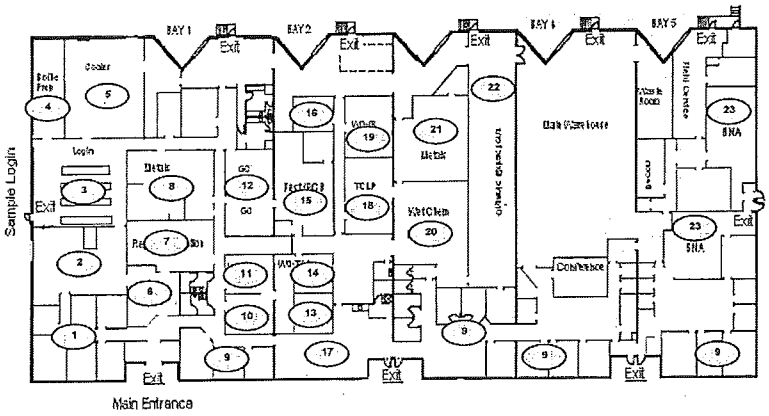
25.9.2 Multiple Reports

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.

Uncontrolled Copy

Appendix 1. Laboratory Floor Plan

Edison Floor Plan

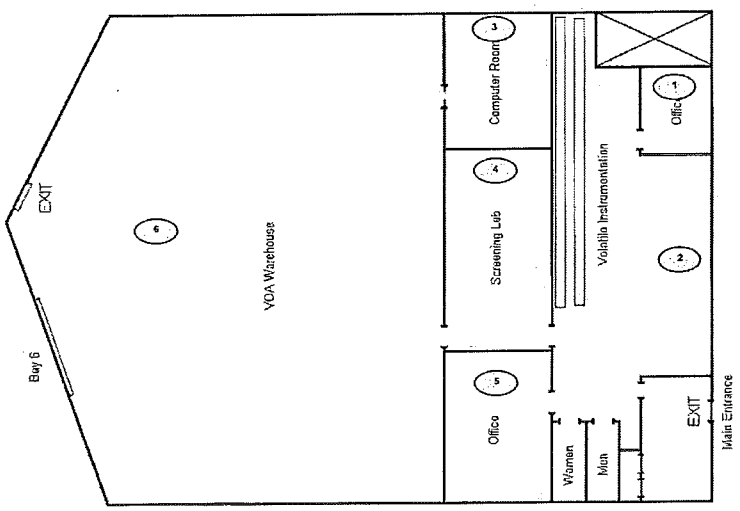


Key Areas *

1. PM-PMA/Admin.
2. Project Management
3. Sample Login
4. Bottle Prep
5. Sample Cooler
6. Reception
7. Report Production
8. Metals
9. Offices
10. Wet Chem-IC
11. Wet Chem - Lachat
12. GCs
13. DAI Instruments
14. Wet Chem-TOC
15. Pest/PCBs
16. Mercury Rm
17. Cafeteria
18. TCLP Extration
19. Wet-Chem-IR
20. Wet Chem Lab
21. Metals
22. Organic Extractions
23. BNA

TestAmerica
THE LEADER IN ENVIRONMENTAL TESTING

Edison Floor Plan



Key Areas *

1. Office
2. Volatile Organics
3. Computer Rm
4. Screening Rm
5. Office
6. Warehouse

TestAmerica
THE LEADER IN ENVIRONMENTAL TESTING

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)

Uncontrolled Copy

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)

Uncontrolled Copy

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.

Uncontrolled Copy

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 & 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

Uncontrolled Copy

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)

Uncontrolled Copy

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 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)

Uncontrolled Copy

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

Uncontrolled Copy

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

Uncontrolled Copy

Appendix 3. Laboratory Certifications, Accreditations, Validations

TestAmerica Edison 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 TestAmerica Edison laboratory has accreditation/certification/licensing with the following organizations:

TestAmerica
THE LEADER IN ENVIRONMENTAL TESTING

TestAmerica Certifications

Laboratory	Program	Authority	Identification	Expiration Date
TestAmerica Edison	Federal	USDA	NJCA-003-08	06/13/2020
TestAmerica Edison	NELAP	New Jersey	12028	06/30/2018
TestAmerica Edison	NELAP	New York	11452	04/01/2018
TestAmerica Edison	NELAP	Pennsylvania	68-00522	02/28/2018
TestAmerica Edison	State Program	Connecticut	PH-0200	09/30/2018
TestAmerica Edison	State Program	DE Haz. Subst. Cleanup Act (HSCA)	N/A	12/31/2017
TestAmerica Edison	State Program	Rhode Island	LAO00132	12/30/2017

Page 1 of 1

12/15/17

* 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
©2010, TestAmerica Laboratories, Inc. All rights reserved. TestAmerica & Design ™ are trademarks of TestAmerica Laboratories, Inc.

The certificates and accredited parameter lists are available for each State/Program at www.testamericainc.com under Analytical Services Search – Certifications.

E-2

Laboratory Standard Operating Procedures

Anions by Ion Chromatography using EPA Method 300.0, SW846 9056A and SM 4110 B

Once printed, this is considered an uncontrolled document.

Approvals (Signature/Date):

Catalina R. Dalangin	01/18/18	Mark. A. Acierno	01/18/18
Department Manager	Date	Laboratory Director	Date
Carl Armbruster	01/18/18	Dan Helfrich	01/18/18
Quality Assurance Manager	Date	Health & Safety Manager/Coordinator	Date

Copyright Information:
This documentation has been prepared by TestAmerica Laboratories, Inc. and its affiliates ("TestAmerica"), solely for their own use and the use of their customers in evaluating their qualifications and capabilities in connection with a particular project. The user of this document agrees by its acceptance to return it to TestAmerica upon request and not to reproduce, copy, lend, or otherwise disclose its contents, directly or indirectly, and not to use it for any other purpose other than that for which it was specifically provided. The user also agrees that where consultants or other outside parties are involved in the evaluation process, access to these documents shall not be given to said parties unless those parties also specifically agree to these conditions.

THIS DOCUMENT CONTAINS VALUABLE CONFIDENTIAL AND PROPRIETARY INFORMATION. DISCLOSURE, USE OR REPRODUCTION OF THESE MATERIALS WITHOUT THE WRITTEN AUTHORIZATION OF TESTAMERICA IS STRICTLY PROHIBITED. THIS UNPUBLISHED WORK BY TESTAMERICA IS PROTECTED BY STATE AND FEDERAL LAW OF THE UNITED STATES. IF PUBLICATION OF THIS WORK SHOULD OCCUR THE FOLLOWING NOTICE SHALL APPLY:

©COPYRIGHT 2018 TESTAMERICA LABORATORIES, INC. ALL RIGHTS RESERVED.

Facility Distribution No. ELECTRONIC **Distributed To:** FACILITY INTRANET

1.0 Scope and Application

1.1 Analytes, Matrix(s), and Reporting Limits

This SOP describes the procedures used to analyze for anions in surface water, groundwater, wastewater, drinking waters and soils using the following methods: EPA Method 300.0, SW846 9056A and SM 4110 B**.**

Parameter	CAS Registry No.	Water Reporting Limits (mg/L)	Soil Reporting Limits (mg/kg) (soil sample prep is 1:10 ratio)
Bromide	24959-67-9	0.400	4.0
Chloride	16887-00-6	0.120	1.20
Fluoride	16984-48-8	0.080	0.80
Nitrate-N	14797-55-8	0.100	1.0
Nitrite-N	14797-65-0	0.120	1.20
o-Phosphate	14265-44-2	0.200	2.0
Sulfate	14808-79-8	0.600	6.0

Note: The most current MDLs and RLs for this method can be found in the active TestAmerica LIMS (TALS) Method Limit Group (MLG) database.

1.2 On occasion clients may request modifications to this SOP. These modifications are handled following the procedures outlined in Section 7 (*Review of Work Request*) and Section 19 (*Test Methods and Method Validation)* of TestAmerica Edison's Quality Assurance Manual (ED-QA-LQM).

2.0 Summary of Method

2.1 A filtered aqueous sample of 20 ul is injected into an ion chromatograph (IC) with the use of an automated sampler. For solid materials, samples are extracted at 1:10 time ratio prior to analysis. The sample merges with an eluent stream and is pumped through the system. The ion exchanger separates the anions of interest. Ions are separated based on their affinity for the exchange sites of the resin. The separated anions are converted to their acid forms via electrolytic suppression, and then measured using an electrical conductivity cell. Anions are identified based on their retention times compared to known standards. Quantitation is accomplished by measuring the peak height or area and comparing it to a calibration curve generated from known standards.

3.0 Definitions

For a complete list of definitions refer to Appendix 2 in the most current revision of TestAmerica Edison's Quality Assurance Manual (ED-QA-LQM).

4.0 Interference

4.1 Interference can be caused by substances with retention times that are similar to and overlap those of the anion of interest. Anions of high concentrations can interfere with the peak resolution of an adjacent anion. Diluting the sample may minimize overlap.

4.2 Method interference may be caused by contaminants in the reagent water, reagents, glassware and other sample processing apparatus that lead to discrete artifacts or an elevated baseline in the ion chromatograms.

4.3 All samples must be pre-filtered through a 0.45um filter before injection. If particles contaminate the guard or analytical columns, follow the manufacturer's suggestions for cleaning, or simply replace the column.

4.4 The water dip or negative peak that elutes near and can interfere with the Fluoride peak can usually be eliminated by the addition of the equivalent of 1ml of concentrated eluent to 100ml of each standard and sample.

4.5 Acetate, formate and other low molecular weight organic acids elute early and can interfere with Fluoride during the chromatographic run. Therefore, this method is not recommended for leachates of solid samples where acetate is used.

5.0 Safety

Employees must abide by the policies and procedures in the Corporate Safety Manual and this document.

This procedure may involve hazardous material, operations and equipment. This SOP does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of the method to follow appropriate safety, waste disposal and health practices under the assumption that all samples and reagents are potentially hazardous. Safety glasses, gloves, lab coats and closed-toe, nonabsorbent shoes are a minimum.

5.1 Specific Safety Concerns or Requirements

Exercise caution when using syringes with attached filter assemblies. Application of excessive force has, upon occasion, caused a filter disc to burst during the process.

5.2 Primary Materials Used

The following is a list of the materials used in this method, which have a serious or significant hazard rating. **Note: This list does not include all materials used in the method. The table contains a summary of the primary hazards listed in the MSDS for each of the materials listed in the table.** A complete list of materials used in the method can be found in the reagents and materials section. Employees must review the

information in the MSDS for each material before using it for the first time or when there are major changes to the MSDS.

Material (1)	Hazards	Exposure Limit (2)	Signs and symptoms of exposure
Sulfuric Acid	Corrosive Oxidizer Dehydrator Poison Carcinogen	1 Mg/M3-TWA	Inhalation produces damaging effects on the mucous membranes and upper respiratory tract. Symptoms may include irritation of the nose and throat, and labored breathing. Symptoms of redness, pain, and severe burn can occur. Contact can cause blurred vision, redness, pain and severe tissue burns. Can cause blindness.
1 – Always add acid to water to prevent violent reactions.			
2 – Exposure limit refers to the OSHA regulatory exposure limit.			

6.0 Equipment and Supplies

6.1 Instrumentation

6.1.1 Ion chromatograph complete with all required accessories:

6.1.1.1 Anion separator column capable of resolving bromide, chloride, fluoride, nitrate, sulfate, nitrite and o-phosphate (as P). Metrosep A Supp 5 150/4.0 Cat # 6.1006.520; Cat# 10014.0; Cat# 6.1006.500

6.1.1.2 Guard column to protect the separator column from fouling by particles. Metrosep RP 2 Guard Cat # 6.1011.030; Cat# 6.1006.510

6.1.1.3 Pump able to deliver 1.2 ml/min of constant flow rate.

6.1.1.4 Data collection and analysis system.

6.1.1.5 Automated sampler.

6.1.1.6 Reagent Fee Controller

6.1.1.7 Column Temperature Stabilizer

6.1.1.8 Carbonate Removal Device (4mm)

6.1.1.9 ASRS-Ultra II (4mm)

6.1.1.10 Conductivity detector with temperature control and separate working and reference electrodes

6.2 Supplies

6.2.1. Various laboratory glassware such as Class A graduated cylinders, syringes, volumetric flasks and pipettes.

6.2.2. 10 ml syringes and 0.45 um syringe filters for colored samples

6.2.3. Analytical balance, capable of weighing to the nearest 0.0001g.

6.2.4. Filter caps for clean samples purchased from Dionex

6.2.5. 10 ml sample vials purchased from a outside vendor

6.2.6. 4 oz. glass container for soil extraction

7.0 Reagents and Standards

7.1 Sample bottles: Glass or polyethylene bottles of sufficient volume to allow replicate analyses of anions of interest.

7.2 Reagent water: Distilled or deionized water free or ELGA water from metals, of the anions of interest. Water should contain particles no larger than 0.20 microns.

7.3 Ottawa sand will be used for all soil QC samples to represent soil matrix.

7.4 Eluent solution: 0.32 M Na2CO3/.10M NaHCO3 (Metrohm Cat # REAIC1102) pour all contents in tube (10 ml) directly into a 1L flask. Rinse the tube with several aliquots of reagent water adding to flask. Bring flask to volume and mix well. Final conc 3.20mM Na2CO3/1.00mM NaHCO3 suggested by manufacturer (Metrohm).

7.5 Regeneration Solution: For instrument ID IC-A and IC-B prepare by diluting 2.8 ml of concentrated sulfuric acid to 4L with reagent water. For instrument ID IC-1 and IC-2 prepare the solution by adding 28 ml of conc. H2SO4 to 1L with reagent water.

7.6 Multi-Element Ion Chromatography (IC) Standards (Stock Solution) purchased from Inorganic Ventures: Catalog No. 300-Cal-A. Contains:

• Bromide	100	ug/ml
• Chloride	30	ug/ml
• Fluoride	20	ug/ml
• Nitrate-N	25	ug/ml
• Nitrite-N	30	ug/ml
• O-Phosphate as P	50	ug/ml
• Sulfate	150	ug/ml

Note: A separate lot number of this standard solution (IV Catalog No. 300-Cal-A) is used for preparation of the Laboratory Control Sample (LCS), Matrix Spike/Matrix Spike Duplicate (MS/MSD) and Initial Calibration Verification (ICV).

7.7 **Standards Preparation**: Prepare the calibration standards for a 7-point curve by measuring the following volumes of the Multi-Element IC standard described in

Section 7.6 into separate 50 ml Class A volumetric flasks. Bring to the final volume of 50 ml with analyte-free reagent water.

	Level 1	Level 2	Level 3	Level 4	Level 5	Level 6	Level 7
Volume of the Multi-Element IC Standard (Cat. No. 300-Cal-A)	0.0	0.20 ml	0.50 ml	2.5 ml	5.0 ml	7.5 ml	10 ml
Final Volume with Reagent Water	50 ml	50 ml	50 ml	50 ml	50 ml	50 ml	50 ml

The final concentration for each anion in the 7 calibration levels is summarized in the table below:

Calibration Standards Preparation Instructions (mg/L)

Anion	Level 1	Level 2	Level 3	Level 4	Level 5	Level 6	Level 7
Fluoride	0.0	0.08	0.2	1.0	2.0	3.0	4.0
Chloride	0.0	0.12	0.3	1.5	3	4.5	6
Nitrite-N	0.0	0.12	0.3	1.5	3	4.5	6
Bromide	0.0	0.4	1.0	5.0	10	15	20
Nitrate-N	0.0	0.1	0.25	1.25	2.5	3.75	5
O-Phosphate	0.0	0.2	0.5	2.5	5	7.5	10
Sulfate	0.0	0.6	1.5	7.5	15	22.5	30

The Initial Calibration Verification (ICV) and Continuing Calibration Verification (CCV) is prepared by adding 2.5 ml of Multi-Element IC standard describe in Section 7.6 into 50.0 ml Class A volumetric flasks. Bring to the final volume of 50ml with analyte-free reagent water. Note: ICV is prepared from a source different from the lot used for the Initial Calibration Range. The ICV is analyzed and evaluated as described in Section 9.2.1. Working standards must be prepared fresh daily.

Note: Prepare the CCV used for instrument IC-2 in 200 ml volumetric flask. Add 10 ml of Multi-Element IC standard describe in Section 7.6 into 200 ml Class A volumetric flasks.

8.0 Sample Collection, Preservation, Shipment and Storage

Listed below are the holding times and the references that include preservation requirements.

Matrix	Sample Container	Min. Sample Size	Preservation	Holding Time[1]	Reference
Waters	Plastic	50 mL	None Cool 4 ± 2°C	28 Days	40 CFR Part 136.3 SM 4110B, SW846 9056A

Soils	Plastic	20g	None Cool 4 ± 2°C	28 Days	N/A

[1] The holding time for Nitrate-N, Nitrite-N and O-Phosphate as P is 48 Hours.

9.0 Quality Control

9.1 Sample QC - The following quality control samples are prepared with each batch of samples. When running a batch with multiple methods (300.0 4110B and 9056A), use the more stringent limits.

Quality Controls	Frequency	Control Limit
Method Blank (MB)	1 in 20 or fewer samples	< ½ RL
Laboratory Control Sample (LCS)[1]	1 in 20 or fewer samples	**300.0:** ± 10% **9056A**: ± 20% **4110B**: Statistical Limits [3]
Matrix Spike (MS)[2]	**300.0**: 1 in 10 or fewer samples **9056A & 4110B:** 1 in 20 or fewer samples	**300.0:** ± 10% **9056A**: ± 20% **4110B**: Statistical Limits [3]
Matrix Duplicate (MD) [2]	1 in 20 or fewer samples	RPD: 15% and 50% for low range samples

[1] LCS Duplicate (LCD) is performed only when insufficient sample is available for the MS/MSD or when requested by the client/project/contract.
[2] The sample selection for MS and MD is random, unless specifically requested by a client.
[3] Statistical control limits are updated annually into LIMS.

9.1.1 Method Blank (MB): To determine freedom from contamination; prepare a MB at the beginning of the analytical procedure. The blank consists of 10 ml reagent water that is treated the same way as the samples and standards. The blanks must be free of the analytes of concern and must be less one-half the reporting limit.

- Reanalyze all samples associated with an unacceptable method blank unless the detected concentrations in the samples are < ½ RL or detected concentrations in the blank are < 10X amount in associated sample.

9.1.2 Laboratory Control Sample (LCS): Prepare and analyze an LCS near the beginning of each analytical run at the concentration near the midpoint of the curve. The recovery of the LCS must be within 90-110% of the true value. If the

LCS recovery is unacceptable re-analysis is required. Any samples associated with an unacceptable LCS must be reanalyzed with a passing LCS. For final concentration see Sec 9.1.2.2 for LCS soil and Sec 10.2.1.2 for LCS water.

9.1.2.1 For water samples: The LCS (and when required) the LCSD is prepared by adding 500 ul of Multi-Element IC Standard described in Section 7.6 into 9.5 ml of analyte-free reagent water. A lot separate from the lot used in preparation of the ICAL must be used.

9.1.2.2 For soil samples: The LCS (and when required) the LCSD is prepared as follows: Weigh 10 grams of Ottawa sand into a 4 oz. glass container. Using class A graduated cylinder add 90 ml of analyte-free reagent water. Pipet 5 ml of Multi-Element IC Standard as describe in Section 7.5. (A lot separate from the lot used in preparation of the ICAL must be used). Mix for 10 minutes using a magnetic stirring device. Filter the resulting slurry before injecting using a 0.45u membrane type filter.

Analyte	Final conc. (mg/Kg)
Bromide	50
Chloride	15
Fluoride	10
Nitrate-N	12.5
Nitrite-N	15
O-Phosphate-P	25
Sulfate	75

9.1.3. Laboratory Control Sample Duplicate (LCSD): prepare and analyze one LCSD per batch. Refer to Sec 9.1.2 for preparation instructions and acceptance limits. The RPD is ≤15 % for all samples concentrations near or at the midrange of calibration. RPD is ≤ 50 % for concentration near the low range of the calibration curve

9.1.4. Matrix Duplicate (MD): a sample duplicate is analyzed for each matrix type daily or one per batch. The RPD is ≤ 15 % for all samples concentrations near or at the midrange of calibration. RPD is ≤ 50 % for concentration near the low range of the calibration curve.

9.1.4 Matrix Spike (MS): a matrix spike is to be prepared and analyzed after every 10 or fewer samples. Deviations may occur due to specific client, state, or protocol requirements. The method control limits for Method 300.0 is 90-110% and Method 9056A is 80-120%. If the % recovery is outside of the method limits and all other QC data (i.e. LCS/LCSD) is within limits, a matrix effect is suspected. For final concentration see Sec 9.1.2.2 for MS soil and Sec 10.2.1.2 for MS water.

9.1.4.1 For water samples The MS and MSD are prepared by adding 500 ul of Multi-Element IC Standard described in Section 7.6 into 9.0 ml of

the sample being spiked. A lot separate from the lot used in preparation of the ICAL must be used.

9.1.4.2 For soil samples: Weigh 10 grams of the sample into a 4 oz glass container. Using class A graduated cylinder add 90 ml of analyte-free reagent water. Add 5 ml of Multi-Element IC Standard as describe in Section 7.6 (A lot separate from the lot used in preparation of the ICAL must be used). Mix for 10 minutes using a magnetic stirring device. Filter the resulting slurry before injecting using a 0.45um membrane type filter.

9.2 Instrument QC

9.2.1. **Initial Calibration Verification (ICV):** analyzed an ICV immediately after the calibration curve to verify the accuracy of the curve The ICV is prepared from a source or lot different from the ICAL, see Sec 7.8 for the standard preparation instruction. The results must be within 90-110% of the true value.

9.2.2. **Initial Calibration Blank (ICB):** The ICB is prepared using 10 mls of deionized water. The ICB must be free of the analytes of concern at levels less than one-half the reporting limit (RL).

9.2.3. **Continuing Calibration Verification (CCV):** Analyze CCV after every 10 samples and at the end of the run using two standard concentrations, Level 4 and Level 7 standards in the Initial Calibration (See Sec 7.7). Alternate analysis of these standards after each 10 samples.

CCV1: (Level 4)
CCB1
10 samples
CCV 2: (Level 7)
CCB2
10 samples
CCV3 (Level 4)
CCB3 ……

9.2.3.1. The recovery must be within 90-110%. If the CCV fails it may be reanalyzed.

- If 2nd analysis is acceptable, analytical sequence can continue, however the previous 10 samples must be reanalyzed.
- If 2nd analysis is unacceptable, analyze a new ICAL.

9.2.4. **Continuing Calibration Blank (CCB)** - The CCB is analyzed after every 10 samples and at the end of the run. Use 10 mls of reagent water for the CCB; it must be free of target analytes at levels less than one-half the reporting limit (RL). CCB must not exceed the concentration specified below:

Fluoride	0.04 ug/ml
Chloride	0.06 ug/ml
Nitrite-N	0.06 ug/ml

Bromide	0.2 ug/ml
Nitrate-N	0.05 ug/ml
O-Phosphate	0.1 ug/ml
Sulfate	0.3 ug/ml

9.2.5. **Lower Limit of Quantitation (LLOQ)**: LLOQ is the lowest point concentration in the calibration curve and should be analyzed daily. Prepare the standard the same way as the Level 2 standard in the Initial calibration, see Sec 7.7. The LLOQ recoveries must be within 50 % of the true value to verify the data reporting limit.

9.2.6. **LCR (Linear Calibration Range)** – The LCR must be determined initially and verified every six months or whenever a significant change in instrument response is observed or expected. The initial demonstration of linearity must use sufficient standards to insure that the resulting curve is linear. The verification of linearity must use a minimum of a blank and three standards. If any verification data exceeds the initial values by +/- 10 %, linearity must be established. If any portion of the range is shown to be nonlinear, sufficient standards must be used to clearly define the nonlinear portion.

10.0 Procedure

10.1 System Equilibrium:

10.1.1 Set up the ion chromatograph as specified in the manufacturer's instructions.

10.1.2 Turn on and prime the pump.

10.1.3 Adjust the eluent flow rate to 1.0 ±0.1 mL/min. for all instruments equipped with the RFC.

10.1.4 Allow the system to come to equilibrium (15-20 minutes). A stable baseline indicates system equilibrium.

10.2 Calibration

10.2.1 Prepare a standard calibration curve that consists of a blank and at least seven different concentrations for each anion to be measured. Plot instrument's peak area responses against corresponding concentration values. The correlation coefficient (R value) must be $\geq$0.995 or a new calibration is prepared.

10.2.1.1 Verify the calibration curve daily, or whenever the detector setting, eluent or regenerant is changed. If the verification fails, a new calibration curve is prepared.

10.2.1.2 Calibration Acceptance Summary:

Step	*Standards in mg/L*	*Type*	*Control Limit*
ICAL	7 standards for each analyte at the concentrations listed below	Linear	R value ≥ .995
	Bromide: 0.0, 0.4, 1.0, 5.0, 10, 15, 20	Linear	R value ≥ .995
	Chloride: 0.0, 0.12, 0.3, 1.5, 3.0, 4.5, 6.0	Linear	R value ≥ .995
	Fluoride: 0.0, 0.08, 0.2, 1.0, 2.0, 3.0, 4.0	Linear	R value ≥ .995
	Nitrate as N: 0.0, 0.1, 0.25, 1.25, 2.5, 3.75, 5	Linear	R value ≥ .995
	Nitrite as N: 0.0, 0.12, 0.3, 1. 5, 3, 4.5, 6	Linear	R value ≥ .995
	O-Phosphate as P: 0.0, 0.2, 0.5, 2.5, 5, 7.5, 10	Linear	R value ≥ .995
	Sulfate: 0.0, 0.6, 1.5, 7.5, 15, 22.5, 30	Linear	R value ≥ .995
ICV	Prepared from a standard source or lot separate from that of ICAL. Analyzed after successful ICAL at concentrations listed below		90-110%
	Bromide at 5.0mg/L		90-110%
	Chloride at 1.5 mg/L		90-110%
	Fluoride at 1.0 mg/L		90-110%
	Nitrate-N at 1.25 mg/L		90-110%
	Nitrite-N at 1.5 mg/L		90-110%
	O-Phosphate-P at 2.5 mg/L		90-110%
	Sulfate at 7.5 mg/L		90-110%
CCV	Prepared from a source similar to the ICAL and at the same concentrations as the ICV	Linear	90-110%
ICB/ CCB	10mls of reagent water		All targets < ½ RL

10.2.1.3 SM4110B requires each calibration point to be back calculated and the true concentration should agree within +/- 50 % for the first calibration standard and within +/- 10 % for the rest of the standards.

10.2.1.4 A Non-Conformance Memo (NCM) must be completed in TALS for any of the following conditions:

- Holding time exceedance
- QC (MB, LCS, MS, MD) outside of specifications.
- Dilutions resulting in RLs raised above client requirements.
- Other anomalies as observed during the prep/analysis of the samples.
- Insufficient sample volume and reanalysis.

10.3 Sample analysis:

10.3.1 Cloudy aqueous samples must be filtered through a pre-washed 0.45um pore diameter membrane filter. If the sample is clean no filtration is required.

10.3.2 **Soil Extraction**: For soil samples the following extraction should be used. Add 10 grams of soil sample to 100 ml of deionized water (10 times the weight of dry solid material). This slurry is mixed for 10 minutes using a magnetic stirring device. Filter the resulting slurry using a 0.45µm membrane filter. Once filtered this sample is ready to be loaded onto the autosampler.

10.3.3 **ASTM extraction:** Soil samples which require ASTM extraction should be extracted utilizing the ASTM D3987 procedure described in SOP# ED-ORP-041 (ASTM LEACHATE, Procedure for the Extraction of Solid Waste with Water).

10.3.4 Fill autosampler vials with the filtered samples to the fill line marked on the vial body (approximately 10 mL). Place vial cap into vial.

10.3.5 Place the filled vial into the sampler cassette and fully insert the cap using the insertion tool.

10.3.6 Place the filled cassettes into the automated sampler and start the run.

10.3.7 If the peak area responses exceed the working calibration range, then dilute the sample appropriately with reagent water. (Note: chrome data results are in-column data, dilution factor not included). TALS will calculate the dilution factor to the raw data results prior to data reporting.

10.3.8 Retention time (migration time) is the expected time or migration time in minutes for the component. The retention time of each component is compared to the expected retention time and the component table is updated (retentions are always in the order F, Cl, NO2, Br, NO3, o-PO4 and SO4). The retention time for the analytes must be closely monitored on each day of the analysis and throughout the lifetime of the analytical column. The retention time windows must be within +/-0.20 minutes of the expected retention time or a new initial calibration is prepared.

11.0 Calculations / Data Reduction

11.1 Using TALS and the instrument software packages, prepare a linear calibration curve for each analyte by plotting instrument response against standard concentration. Compute sample concentration by comparing sample response with the standard curve. The response factor produced will be represented by a r^2 number of 0.995 or greater.

11.2 The calibration curve will calculate the analyte concentrations from the peak area. This area is determined from automatic peak integration by the instrument.

11.3 Manual peak integration may be performed if required to improve the instrument integration. If a manual integration is performed, chromatograms carefully detailing the peak before and after integration are stored in TALS and the instrument software. All manual integrations will be dated and initialed by the second level reviewer, on the data review checklist during secondary review.

11.4 The analyst corrects the results for dilution factors:
Xf = Xj * Dilution Factor

Where:
Xf = Final sample concentration
Xj = calculated concentration of sample at instrument

11.5 Report only those values that are within the calibration range. Samples with responses exceeding the highest standard must be diluted and reanalyzed.

11.6 For solid samples, the result is expressed as mg/kg on a dry weight basis.
To convert the mg/l result obtained from the calibration curve to
mg/kg use the following equation:

mg/kg (wet) = [mg/l X final vol. of leached sample] / grams sample used

mg/kg (dry) = mg/kg (wet) / decimal dry weight

NOTE: All dry weight corrections are made in LIMS at the time the final report is prepared.

11.7 Accuracy:

$$\text{ICV / CCV, LCS \% Recovery} = \frac{\text{observed concentration}}{\text{known concentration}} \times 100$$

$$\text{MS \% Recovery} = \frac{\text{(spiked sample) - (unspiked sample)}}{\text{spiked concentration}} \times 100$$

11.8 Precision (RPD):

$$\text{Matrix Duplicate (MD)} = \frac{|\text{orig. sample value - dup. sample value}|}{[(\text{orig. sample value} + \text{dup. sample value})/2]} \times 100$$

12.0 Method Performance

12.1. Method Detection Limit Study (MDL)

The method detection limit (MDL) is the lowest concentration that can be detected for a given analytical method and sample matrix with 99% confidence that the analyte is present. The MDL is determined according to the laboratory's MDL procedure in Section 19 (Test Methods and Method Validation) of TestAmerica Edison's Quality Assurance Manual (ED-QA-LQM). MDLs reflect a calculated (statistical) value determined under ideal laboratory conditions in a clean matrix, and may not be achievable in all environmental matrices. The laboratory maintains MDL studies for analyses performed; these are verified at least annually unless method requirements require a greater frequency. Method EPA 300.0 MDL studies are performed every 6 months as required by the method.

12.2. Demonstration of Capabilities

For DOC procedure refer to Section 19 in the most current revision of TestAmerica Edison's Quality Assurance Manual (ED-QA-LQM).

12.3. Training Requirements

Refer to TestAmerica SOP No. ED-GEN-022, *Training*, for the laboratory's training program.

13.0 Pollution Control

13.1. Pollution prevention encompasses any technique that reduces or eliminates the quantity or toxicity of waste at the point of generation. Numerous opportunities for pollution prevention exist in laboratory operation. The USEPA has established a prevention hierarchy of environmental management techniques that places pollution prevention as the management option of first choice. Whenever feasible, laboratory personnel should use pollution prevention techniques to address their waste generation. When wastes cannot be feasibly reduced at the source, the agency recommends recycling as the next best option.

13.2. The quantity of chemical purchased should be based on expected usage during its shelf life and disposal cost of unused material. Actual reagent preparation volumes should reflect anticipated usage.

14.0 Waste Management

14.1 Waste management practices are conducted consistent with all applicable rules and regulations. Excess reagents, samples and method process wastes are disposed of in an accepted manner. Waste description rules and land disposal restrictions are followed. Waste disposal procedures are incorporated by reference to TestAmerica SOP No. ED-SPM-008 (Laboratory Waste Disposal Procedures).

14.2 The following waste streams are produced when this method is carried out:

- Aqueous waste is neutralized in the neutralization tank located in the waste room.
- Solid wastes consist of contaminated plastic materials such as IC syringes, filters, caps and vials utilized for sample preparation. All solid wastes are disposed in the recycling containers located throughout the lab

15.0 References / Cross-References

15.1 Environmental Monitoring Systems Laboratory, Office of Research and Development, U.S. EPA, Cincinnati, Ohio. Method 300.0, “Determination of Inorganic Anions by Ion Chromatography”, Revision 2.1, August 1993.

15.2 Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, U.S. Environmental Protection Agency, Office of Solid Waste and Emergency Response. Method 9056A, “Determination of Inorganic Anions by Ion Chromatography,” SW-846, Revision 1, 2007.

15.3 Standard Methods for the Examination of Water and Wastewater , 22nd Edition, American Public Health Association, Washington, DC 2011, Editorial Revision 2011, SM 4110 B-11.

15.4 IC Net 2.3 Operational Tutorials manual by Metrohm USA Inc and IC Net 2.3 (6.6034.033) software guide

15.5 TestAmerica Edison Document No. ED-QA-LQM, *Laboratory Quality Manual*, most current revision.

15.6 TestAmerica Edison SOP No. ED-GEN-022, *Training,* most current revision.

15.7 TestAmerica Edison SOP No. ED-SPM-008, *Laboratory Waste Disposal Procedures*, current revision.

15.8 TestAmerica Corporate Environmental Health and Safety Manual, Document No. CW-E-M-001, most current revision.

16.0 Method Modifications:

Item	Method #	Modification
7.4	300.0; 9056A; 4110B	The eluent concentration (3.20mM Na2CO3/1.00mM NaHCO3) being used in this SOP is suggested by the instrument manufacturer (Metrohm).

17.0 Attachments

Attachment 1: Analytical Run Sequence
Attachment 2: Eluent solution (Metrohm Cat # REAIC1102)

18.0 Revision History

- **Rev 6, dated 18 Jan 2018**
  - Sec 2: Revised the amount of sample injected into the IC instrument to 20 ul.
  - Sec 6.1.1.1 & 6.1.1.2: Added catalog part# used for the anion separator column and guard column.
  - Sec 6.2.6: Added 4 oz. glass container to list of supplies.
  - Sec 7.5: Added the preparation procedure for the regeneration solution – instrument IC-1 and IC-2.
  - Sec 7.7: Revised the final volume to 50 ml and the spiking amount to add for the ICAL and ICV/CCV standards. Added note and prep instructions for the CCV solution used for instrument IC-2.
  - Sec 9.1.2.2: Revised the initial/final volume and spiking amount added for the LCS soil.
  - Sec 9.1.4.2: Revised the initial/final volume and spiking amount added for the soil Matrix Spike.
  - Sec 10.2.1.3: Clarified the % recovery criteria for the lowest ICAL standard for Method 4110B.
  - Sec 10.3.2: Revised the amount of sample used when extracting soil samples from 1 gram to 10 grams; revised the amount of DI water added to 100ml.

- **Rev 5, dated 16 Mar 2016**
  - Section 12.1 revised to reflect the requirement that Method EPA 300.0 MDL studies be performed every 6 months as required by the method.

- **Rev 4, dated 08 Oct 2015**
  - Sec 7.7: Added following statement: “Working standards must be prepared fresh daily.”

- **Rev 3, dated 19 August 2014**
  - Sec 2.1: Clarified the amount of sample and standard injected into the ion chromatography.

- **Rev 2, dated 12 November 2013**

- Revised entire procedure to include proper method requirements, Quality Control, control limits preparation instructions and procedures.
- Updated all references to SW846 9056 and SM 4110B to the updated SW846 Method 9056A and 4110B-11.
- Sec. 6.1.1.1 & 6.1.1.2: Added separator and guard column information: Metrosep A Supp 5 150/4.0 Cat # 6.1006.520; Metrosep RP 2 Guard Cat # 6.1011.030.
- Sec. 6.1.1.10: Added Conductivity detector to the list of equipment.
- Sec. 7.4: Revised Eluant solution preparation instructions.
- Sec. 7.5: Added to clarify the preparation instructions of the regeneration solution; subsequent sections adjusted accordingly.
- Sec. 7.7 (previously Sec 7.6): Changed 5-point curve to 7-point curve. Table revised.
- Sec. 7.8 (previously Sec 7.7): Revised preparation instruction for ICV.
- Sec 16: Added method modification.
- Sec 17: Replaced Attachment 1 with Edison's run log. Added Attachment 2.

- **Rev 1 dated 10 January 2013**
  - Section 7.6, Table titled 'Calibration Standards Prep Instructions': revised the concentration of Nitrate in level 5 from 25 mg/L to 5.0 mg/L.

- **Rev 0 New 01/08/2013**

Attachment 1:
Example Analytical Run Sequence

TestAmerica Laboratories
Worklist Run Log Report

Worklist Name: 11/26/2013 ical
Instrument: IC B
Batch Directory: \\EDICHROM\ChromData\ICB\20131126-7176.b
Anaylysis Type: Semi VOA
Inj Volume: 10.00

Worklist Num: 7176
Method: anions-icb
Creator: Boykin, Carol B
Inj Vol Units: ul

Lab ID	Worklist ID	Sample Type	Cal Lvl	Inj Date/Time	File Name	Vial	Dil Factor	Client ID	Fract	pH	Std Lot	~~Comments~~ STA Lot
ccb	460-0007176-001	CCB		26-Nov-2013 00:21:00	XB260021.d		1.0		sv		OK	
ICB	460-0007176-002	ICB		26-Nov-2013 00:42:00	XB260042.d		1.0		sv		OK	
IC 1	460-0007176-003	IC	1	26-Nov-2013 01:02:00	XB260102.d		1.0		sv		OK	SGAnionsL1_00010
ic 2	460-0007176-004	IC	2	26-Nov-2013 01:23:00	XB260123.d		1.0		sv		OK	SGAnionsL2-00010
ic 3	460-0007176-005	IC	3	26-Nov-2013 01:44:00	XB260144.d		1.0		sv		OK	SGAnionsL3-00010
ic 4	460-0007176-006	IC	4	26-Nov-2013 02:05:00	XB260205.d		1.0		sv		OK	SGAnionsL4_00010
ic 5	460-0007176-007	IC	5	26-Nov-2013 02:25:00	XB260225.d		1.0		sv		OK	SGAnionsL5_00010
ic 6	460-0007176-008	IC	6	26-Nov-2013 02:46:00	XB260246.d		1.0		sv		OK	SGAnionsL6-00010
ic 7	460-0007176-009	IC	7	26-Nov-2013 03:07:00	XB260307.d		1.0		sv		OK	SGAnionsL7-00010
icv	460-0007176-010	ICV		26-Nov-2013 03:27:00	xb260327.d		1.0		sv		OK	SGAnionsICV-00010
ccv	460-0007176-011	CCV		26-Nov-2013 03:48:00	xb260348.d		1.0		sv		OK	SGAnionsL4_00010
ccb	460-0007176-012	CCB		26-Nov-2013 04:09:00	xb260409.d		1.0		sv		OK	
MB 460-194370/1-A	460-0007176-013	MB		26-Nov-2013 04:30:00	xb260430.d		1.0		sv		OK	
LCS 460-194370/2-A	460-0007176-014	LCS		26-Nov-2013 04:50:00	xb260450.d		1.0		sv		OK	
LCSD 460-194370/3-A	460-0007176-015	LCSD		26-Nov-2013 05:11:00	xb260511.d		1.0		sv		OK	
460-65403-A-1-B	460-0007176-016	Client		26-Nov-2013 05:53:00	xb260553.d		1.0	Anions	sv		OK	
460-65403-A-1-B	460-0007176-017	Client		26-Nov-2013 05:57:00	XB260557.d		20.0	Anions	sv		OK	
460-65403-A-1-B	460-0007176-018	Client		26-Nov-2013 07:38:00	XB260738.d		5.0	Anions	sv		OK	
460-65403-A-1-B	460-0007176-019	Client		26-Nov-2013 08:20:00	XB260820.d		2.0	Anions	sv		OK	
ccv	460-0007176-020	CCV		26-Nov-2013 09:01:00	XB260901.d		1.0		sv		OK	SGAnionsL4_00010
CCB	460-0007176-021	CCB		26-Nov-2013 09:22:00	XB260922.d		1.0		sv		OK	
mb	460-0007176-022	MB		26-Nov-2013 09:43:00	XB260943.d		1.0		sv		OK	
ccv anions LLOQ	460-0007176-023	CCV		26-Nov-2013 10:03:00	XB261003.d		1.0		sv		OK	
ccv anions LLOQ	460-0007176-024	CCV		26-Nov-2013 10:24:00	XB261024.d		1.0		sv		OK	
ccv anions LCR1	460-0007176-025	CCV		26-Nov-2013 10:45:00	XB261045.d		1.0		sv		OK	SGAnionsLCR_00003
ccv anions LCR2	460-0007176-026	CCV		26-Nov-2013 11:26:00	XB261126.d		1.0		sv		OK	SGAnionsLCR_00004
ccv anions LCR3	460-0007176-027	CCV		26-Nov-2013 12:08:00	XB261208.d		1.0		sv		OK	SGAnionsLCR_00005

Eluent_00005 + Suppressor regenerant Solution_00005

Worklist Run Log: 11/25/2013 ical

Lab ID	Worklist ID	Sample Type	Cal Lvl	Inj Date/Time	File Name	Vial	Dil Factor	Client ID	Fract	pH	Std Lot	Comments
CCV	460-0007176-028	CCV		26-Nov-2013 13:10:00	XB261310.d		1.0		sv		OK	SGAnionsL4_00010
CCB	460-0007176-029	CCB		26-Nov-2013 13:31:00	XB261331.d		1.0		sv		OK	

Attachment 2:

Eluent Solution (Metrohm Cat # REAIC1102)

Metrohm USA Inc.

Metrohm USA, Inc.
6555 Pelican Creek Circle
Riverview, FL 33578

Product Name: A Supp 5 Eluent Concentrate

Part Number: REAIC1102
Lot Number: 072413

Expiration Date: 07/15
Solvent: 18 megohm Water

Compound Number	Analyte	Gravimetric Concentration	Final Concentration (1:100 Dilution)
W-1800H-30	Sodium Carbonate	0.32 M	3.20 mM
W-1385H-12	Sodium Bicarbonate	0.10 M	1.00 mM

TRACEABILITY: This solution was manufactured and certified in our ISO 9001 registered facility. The neat source material was weighed on an electronic balance calibrated by an ISO 17025 metrology firm. The balances are calibrated daily at multiple levels using NIST traceable masses. Final solution is prepared in Class A volumetric glassware. Sodium content analytically traceable to NIST SRM 919a.

STORAGE & HANDLING: Can be stored at room temperature. Do not freeze.

INSTRUCTIONS: Hold PFA tube vertically. Snip the top off the tube with a pair of sharp scissors. Pour contents directly into a 1000 mL flask. The tube will release all 10 mL of the concentrate. Rinse the tube with several small aliquots of reagent water adding to flask. Bring flask to volume and mix well.

Approved By: Cynthia K. Loftus, Quality Manager

07/29/13
Date

Metrohm
International Headquarters

IR 9/20/2013

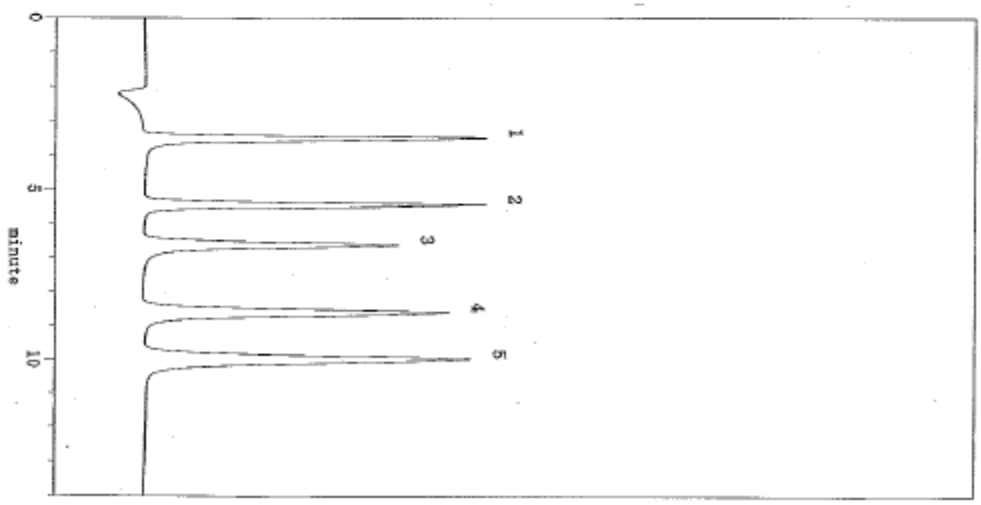
CERTIFICATE OF ANALYSIS

Column : METROSEP A Supp 5 150/4.0
Column size : 150mmL×4.0mmID
Cat No. : 6.1006.520
Gel lot number : 130226

(Analysis condition)
Eluent : 3.2mM Na2CO3+1.0mM NaHCO3
Flow rate : 0.7 ml/min
Detector(ID) : Suppressed CD (J002)
Temperature : Ambient (ca. 25℃)
Sample size : 20 μl
Sample : 1. F(-) (2ppm) 2. Cl(-) (3ppm)
: 3. NO2(-) (5ppm) 4. Br(-) (10ppm)
: 5. NO3(-) (10ppm)
Pressure : 7.4 MPa

Column number : 7304645

No.	Rt (minute)	Plate Count	Fas
5	9.957	8900	1.61



Title: Semivolatile Organic Compounds by Gas Chromatography/Mass Spectrometery (GC/MS), SW846 Method 8270D

Once printed, this is considered an uncontrolled document

Approvals (Signature/Date):

Sylvanus Klusey Organics Operations Manager	6/8/2018 Date	Dan Helfrich Health & Safety Manager	6/8/2018 Date
Carl Armbruster Quality Assurance Manager	6/8/2018 Date	Mark Acierno Laboratory Director	6/8/2018 Date

Diaa Nimer — 6/8/2018 Date
SVOA GC/MS Manager

Copyright Information:
This documentation has been prepared by TestAmerica Laboratories, Inc. and its affiliates ("TestAmerica"), solely for their own use and the use of their customers in evaluating their qualifications and capabilities in connection with a particular project. The user of this document agrees by its acceptance to return it to TestAmerica upon request and not to reproduce, copy, lend, or otherwise disclose its contents, directly or indirectly, and not to use it for any other purpose other than that for which it was specifically provided. The user also agrees that where consultants or other outside parties are involved in the evaluation process, access to these documents shall not be given to said parties unless those parties also specifically agree to these conditions.

THIS DOCUMENT CONTAINS VALUABLE CONFIDENTIAL AND PROPRIETARY INFORMATION. DISCLOSURE, USE OR REPRODUCTION OF THESE MATERIALS WITHOUT THE WRITTEN AUTHORIZATION OF TESTAMERICA IS STRICTLY PROHIBITED. THIS UNPUBLISHED WORK BY TESTAMERICA IS PROTECTED BY STATE AND FEDERAL LAW OF THE UNITED STATES. IF PUBLICATION OF THIS WORK SHOULD OCCUR THE FOLLOWING NOTICE SHALL APPLY:

©COPYRIGHT 2018 TESTAMERICA LABORATORIES, INC. ALL RIGHTS RESERVED.

1.0 Scope and Application

1.1 Analytes, Matrix(s), and Reporting Limits

USEPA Method 8270D is an analytical method which employs the use of GC/MS to determine the concentration of semivolatile organic compounds in extracts prepared from many types of solid waste matrices, soils, and water samples. TestAmerica Edison has the capability to analyze and report the compounds listed in Table 1 via Method 8270D.

Table 1

Compound	CAS No.	Compound	CAS No.
1,1'-Biphenyl	92-52-4	Anthracene **(1)**	120-12-7
1,2,4,5-Tetrachlorobenzene	95-94-3	Atrazine	1912-24-9
1,2,4-Trichlorobenzene	120-82-1	Benzaldehyde	100-52-7
1,2-Dichlorobenzene	95-50-1	Benzidine	92-87-5
1,2-Diphenylhydrazine	122-66-7	Benzo[a]anthracene **(1)**	56-55-3
1,3-Dichlorobenzene	541-73-1	Benzo[a]pyrene **(1)**	50-32-8
1,3-Dimethylnaphthalene	575-41-7	Benzo[b]fluoranthene **(1)**	205-99-2
1,4-Dichlorobenzene	106-46-7	Benzo[g,h,i]perylene **(1)**	191-24-2
1,4-Dichlorobenzene-d4 **(ISTD)**	3855-82-1	Benzo[k]fluoranthene **(1)**	207-08-9
1,4-Dioxane **(1) (2)**	123-91-1	Benzoic acid	65-85-0
1-Methylnaphthalene	90-12-0	Benzyl alcohol	100-51-6
1-Naphthylamine	134-32-7	Bis(2-chloroethoxy)methane	111-91-1
2,2'-oxybis[1-chloropropane]	108-60-1	Bis(2-chloroethyl)ether **(1)**	111-44-4
2,3,4,6-Tetrachlorophenol	58-90-2	Bis(2-ethylhexyl) phthalate	117-81-7
2,3,7,8-TCDD	1746-01-6	Bisphenol-A	80-05-7
2,3-Dihydroindene	496-11-7	Butyl benzyl phthalate	85-68-7
2,3-Dimethylaniline	87-59-2	Caprolactam	105-60-2
2,4,5-Trichlorophenol	95-95-4	Carbamazepine	298-46-4
2,4,5-Trimethylaniline	137-17-7	Carbazole	86-74-8
2,4,6-Tribromophenol **(Surrogate)**	118-79-6	Chrysene **(1)**	218-01-9
2,4,6-Trichlorophenol	88-06-2	Chrysene-d12 **(ISTD)**	1719-03-5
2,4-Dichlorophenol	120-83-2	Coumarin	91-64-5
2,4-Dimethylphenol	105-67-9	Dibenz(a,h)anthracene **(1)**	53-70-3
2,4-Dinitrophenol	51-28-5	Dibenzofuran	132-64-9
2,4-Dinitrotoluene	121-14-2	Diethyl phthalate	84-66-2
2,4-Xylidine	95-68-1	Dimethyl phthalate	131-11-3
2,6-Dinitrotoluene	606-20-2	Di-n-butyl phthalate	84-74-2
2-Chloronaphthalene	91-58-7	Di-n-octyl phthalate	117-84-0
2-Chlorophenol	95-57-8	Fluoranthene **(1)**	206-44-0
2-Ethylaniline	578-54-1	Fluorene **(1)**	86-73-7
2-Fluorobiphenyl **(Surrogate)**	321-60-8	Hexachlorobenzene **(1)**	118-74-1
2-Fluorophenol **(Surrogate)**	367-12-4	Hexachlorobutadiene	87-68-3
2-Methylnaphthalene	91-57-6	Hexachlorocyclopentadiene	77-47-4
2-Methylphenol	95-48-7	Hexachloroethane	67-72-1
2-Naphthylamine	91-59-8	Indeno[1,2,3-cd]pyrene **(1)**	193-39-5
2-Nitroaniline	88-74-4	Isophorone	78-59-1
2-Nitrophenol	88-75-5	n,n'-Dimethylaniline	121-69-7
2-tertbutyl-4-methylphenol	2409-55-4	Naphthalene **(1)**	91-20-3
2-Toluidine	95-53-4	Naphthalene-d8 **(ISTD)**	1146-65-2
3 & 4 Methylphenol	15831-10-4	n-Decane	124-18-5

Table 1

Compound	CAS No.	Compound	CAS No.
3,3'-Dichlorobenzidine	91-94-1	Nitrobenzene	98-95-3
3,4-Dimethylaniline	95-64-7	Nitrobenzene-d5 **(Surrogate)**	4165-60-0
3,5-di-tert-butyl-4-hydroxytol	128-37-0	N-Nitrosodimethylamine **(1)**	62-75-9
3-Nitroaniline	99-09-2	N-Nitrosodi-n-propylamine	621-64-7
4,6-Dinitro-2-methylphenol **(1)**	534-52-1	N-Nitrosodiphenylamine	86-30-6
4-Bromophenyl phenyl ether	101-55-3	n-Octadecane	593-45-3
4-chloro-2-methylaniline	95-69-2	o-Toluidine-d9 **(Surrogate)**	194423-47-7
4-Chloro-3-methylphenol	59-50-7	Pentachloronitrobenzene	82-68-8
4-Chloroaniline	106-47-8	Pentachlorophenol **(1)**	87-86-5
4-Chloroaniline–d4 **(Surrogate)**	191656-33-4	Perylene-d12 **(ISTD)**	1520-96-3
4-Chlorophenyl phenyl ether	7005-72-3	Phenanthrene **(1)**	85-01-8
4-Methylphenol	106-44-5	Phenanthrene-d10 **(ISTD**)	1517-22-2
4-Nitroaniline	100-01-6	Phenol	108-95-2
4-Nitrophenol	100-02-7	Phenol-d5 **(Surrogate)**	4165-62-2
Acenaphthene **(1)**	83-32-9	Phenyl ether	101-84-8
Acenaphthene-d10 **(ISTD)**	15067-26-2	Pyrene **(1)**	129-00-0
Acenaphthylene **(1)**	208-96-8	Pyridine	110-86-1
Acetophenone	98-86-2	Terphenyl-d14 **(Surrogate)**	1718-51-0
Aniline	62-53-3	Total Cresols	STL00160
Aniline-d5 **(Surrogate)**	4165-61-1		

(1) Compound can be analyzed by full scan or Selected Ion Monitoring (SIM).
(2) Compound can also be analyzed by Isotope Dilution/SIM.

1.2 For a listing of method detection limits (MDLs) and Reporting Limits (RLs) please refer to the currently active Method 8270D Method Limit Groups in TALS (TestAmerica LIMS).

1.3 On occasion clients may request modifications to this SOP. These modifications are handled following the procedures outlined in Section 7 (*Review of Work)*, and Section 19 (*Test Methods and Method Validation)* in TestAmerica Edison's Quality Assurance Manual (TestAmerica Edison Document No. ED-QA-LQM).

2.0 Summary of Method

2.1 This method is used for the analysis of aqueous and solid matrices for semi-volatile base, neutral and acid organic compounds that are extracted from the sample matrix with an organic solvent.

2.2 An aliquot of sample containing surrogate spiking compounds is extracted with an organic solvent. The extract is concentrated on a steam bath to a suitable volume. Internal standards are added to the extract.

2.3 Sample extraction techniques are specified for each matrix in the following TestAmerica Edison SOPs:

- ED-ORP-002 (*Extraction of Semivolatile Organic Compounds in Water by Separatory Funnel, SW846 Method 3510C*);

- ED-ORP-043 (*SW846 Method 3580A - Waste Dilution Prep for Analysis of BNAs by SW846 Method 8270)*
- ED-ORP-0044 (*Microwave Extraction for Solids, SW846 Method 3546*);
- ED-ORP-006 (*Extraction of Semivolatile Compounds in Soil Using Medium Level Extraction Techniques, SW846 Method 3550B)*.

2.4 A small aliquot of the extract is injected into a gas chromatograph (GC) equipped with a capillary column. The GC is temperature programmed to separate the compounds which were recovered during the extraction step by boiling point. The effluent of the gas chromatograph is interfaced to a mass spectrometer (MS) which is used to detect the compounds eluting from the GC. The detected compounds are fragmented with an electron beam to produce a mass spectrum which is characteristic of the compound introduced into the MS. Identification of target analytes is accomplished by comparing their mass spectra with the electron ionization spectra of authentic standards. Quantitation is accomplished by comparing the response of a major ion (quantitation ion) relative to an internal standard established through a five-point calibration (six points for second order regression). Specific calibration and quality control steps are included in the method that must be performed and must meet the specifications of SW846 Method 8270D.

2.5 Standard procedure involves preparation of aqueous samples using a Reduced Volume Extraction (RVE) followed by analysis using a Large Volume Injection (LVI). Optionally, a full volume (1000 ml nominal) may be employed. The details of the extractions are outlined in the applicable prep SOPs while the analytical details for 8270D are presented in this SOP.

2.6 This method is also applicable to the analysis of samples by Selected Ion Monitoring (SIM) for the purpose of obtaining lower reporting limits for the following compounds:

Table 2 – SIM Analytes

SIM Analytes	CAS #
1,4-Dioxane	123-91-1
4,6-Dinitro-2-methylphenol	534-52-1
Acenaphthene	83-32-9
Acenaphthylene	208-96-8
Anthracene	120-12-7
Benzo[a]anthracene	56-55-3
Benzo[a]pyrene	50-32-8
Benzo[b]fluoranthene	205-99-2
Benzo[g,h,i]perylene	191-24-2
Benzo[k]fluoranthene	207-08-9
Bis(2-chloroethyl)ether	111-44-4
Chrysene	218-01-9

Table 2 – SIM Analytes	
SIM Analytes	**CAS #**
Dibenz(a,h)anthracene	53-70-3
Fluoranthene	206-44-0
Fluorene	86-73-7
Hexachlorobenzene	118-74-1
Indeno[1,2,3-cd]pyrene	193-39-5
Naphthalene	91-20-3
N-Nitrosodimethylamine	62-75-9
Pentachlorophenol	87-86-5
Phenanthrene	85-01-8
Pyrene	129-00-0

2.7 An isotope dilution selected ion monitoring (SIM) technique for the analysis of 1,4-dioxane in water at a reporting level of 0.4 ug/l is also described in this SOP. Using this technique 1,4-dioxane-d8 is added prior to sample extraction and is used as an internal standard to calculate the concentration of 1,4-dioxane present. Additionally, 1,4-dichorobenzene-d4 is added to the extract prior to analysis to monitor the recovery of 1,4-dioxane-d8.

3.0 Definitions

For a complete list of definitions refer to Appendix 2 in the most current revision of the Quality Assurance Manual (ED-QA-LQM).

4.0 Interferences

4.1 GC/MS data from all blanks, samples, and spikes must be evaluated for interferences. Analysts must take steps to determine the source of the interference and take corrective action to eliminate the problem.

4.1.1 Contamination by carryover can occur whenever high-concentration and low-concentration samples are sequentially analyzed. To reduce carryover, the sample syringe is automatically rinsed with solvent between sample injections. Whenever an unusually concentrated sample is encountered, it should be followed by the analysis of a solvent blank to check for cross-contamination. Alternately, verify that the sample analyzed after the high concentration sample does not show any carryover through inspection of chromatogram and target results.

4.1.2 Contaminants from the extraction process, detected in the method blank should be evaluated to determine the impact on the analysis. Interferences from any target analyte must not be present in the method blank above the reporting limit for that

compound. If these types of interferences occur, corrective action is required. The source should be identified and corrective action initiated to eliminate the interference from the extraction process. Affected samples must be re-extracted and re-analyzed.

4.1.3 The analyst must take precautions to make sure that contaminants do not enter the analytical system. These precautions include systematic procedures designed to eliminate interferences.

4.2 Some compounds analyzed by this method are unstable or sensitive. Benzidine, for example, is easily oxidized during extraction. Hexachlorocyclopentadiene breaks down photochemically and can decompose from high temperatures, particularly in the injection port of the GC. 1,2-Diphenylhydrazine is unstable even at room temperature and readily converts to azobenzene. Phenols are sensitive to active sites and can give a low response or exhibit poor chromatography by tailing. Therefore, it is important the GC is maintained in the best possible condition. See Section 10.1 for proper daily maintenance.

5.0 Safety

Employees must abide by the policies and procedures in the Corporate Environmental Health and Safety Manual (CW-E-M-001) and this document. This procedure may involve hazardous material, operations and equipment. This SOP does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of the method to follow appropriate safety, waste disposal and health practices under the assumption that all samples and reagents are potentially hazardous. Safety glasses, gloves, lab coats and closed-toe, nonabsorbent shoes are a minimum.

5.1. Specific Safety Concerns or Requirements

The gas chromatograph and mass spectrometer contain zones that have elevated temperatures. The analyst needs to be aware of the locations of those zones, and must cool them to room temperature prior to working on them.

The mass spectrometer is under deep vacuum. The mass spectrometer must be brought to atmospheric pressure prior to working on the source.

There are areas of high voltage in both the gas chromatograph and the mass spectrometer. Depending on the type of work involved, either turn the power to the instrument off, or disconnect it from its source of power.

5.2. Primary Materials Used

The following is a list of the materials used in this method, which have a serious or significant hazard rating. **Note: This list does not include all materials used in the method. The table contains a summary of the primary hazards listed in the MSDS for each of the materials listed in the table.** A complete list of materials used in the method can be found in the reagents and materials section.

Employees must review the information in the MSDS for each material before using it for the first time or when there are major changes to the MSDS.

Material (1)	Hazards	Exposure Limit (2)	Signs and symptoms of exposure
Methanol	Flammable Poison Irritant	200 ppm-TWA	A slight irritant to the mucous membranes. Toxic effects exerted upon nervous system, particularly the optic nerve. Symptoms of overexposure may include headache, drowsiness and dizziness. Methyl alcohol is a defatting agent and may cause skin to become dry and cracked. Skin absorption can occur; symptoms may parallel inhalation exposure. Irritant to the eyes.
Methylene Chloride	Carcinogen Irritant	25 ppm-TWA 125 ppm-STEL	Causes irritation to respiratory tract. Has a strong narcotic effect with symptoms of mental confusion, light-headedness, fatigue, nausea, vomiting and headache. Causes irritation, redness and pain to the skin and eyes. Prolonged contact can cause burns. Liquid degreases the skin. May be absorbed through skin.
Toluene	Flammable Poison Irritant	200 ppm-TWA 300 ppm-Ceiling	Inhalation may cause irritation of the upper respiratory tract. Symptoms of overexposure may include fatigue, confusion, headache, dizziness and drowsiness. Peculiar skin sensations (e. g. pins and needles) or numbness may be produced. Causes severe eye and skin irritation with redness and pain. May be absorbed through the skin.
Dimethyl-dichloro-silane	Flammable	none	Can be corrosive to the respiratory tract causing severe irritation and tissue damage. Harmful if absorbed through the skin. May cause severe irritation and systemic damage. Severely irritating to the skin and eyes. Harmful if swallowed. Can cause abdominal discomfort, nausea, vomiting, diarrhea, and irritation to the mouth, throat and stomach.
1 – Always add acid to water to prevent violent reactions.			
2 – Exposure limit refers to the OSHA regulatory exposure limit.			

6.0 Equipment and Supplies

6.1 Gas chromatograph/mass spectrometer system

6.1.1 Gas chromatograph: An Agilent/HP 5890/6890/7890 (or equivalent) houses the capillary column. The GC provides a splitless injection port and allows the column to be directly coupled to the mass spectrometer. The oven is temperature programmable to meet the requirements of the method. An HP 7673/7683 autosampler (or

equivalent) with a 10 ul syringe provides automatic injection of sample extracts while the instrument is unattended.

6.1.2 Analytical Column: 30m x 0.25mm ID, 0.25 um film thickness, Restek Rxi-5Sil MS, Catalog #13623 Zebron ZB-Semivolatiles, Catalog # 7HG-G027-11.

6.1.3 Mass spectrometer: Agilent (HP) 5972, 5973, 5975 or 5977A Mass Selective Detector (MSD) Capable of scanning from 35 to 500 amu every 1 sec or less, using 70 volts electron energy in the electron ionization mode. The mass spectrometer must be capable of producing a mass spectrum for 50 ng of decafluorotriphenylphosphine (DFTPP) which meets the criteria in Section 9.2.1 when 2 ul of the 25 ug/ml GC/MS tuning standard is injected through the GC.

6.1.4 GC/MS interface: Any GC-to-MS interface may be used that gives acceptable calibration points at 50 ng per injection for each compound of interest and achieves acceptable tuning performance criteria.

6.1.5 Data system: The data system is interfaced to the mass spectrometer and accommodates continuous acquisition and storage of GC/MS data throughout the duration of the chromatographic program. The data system consists of a Hewlett-Packard Chemstation equipped with Mustang software used for instrument control and data acquisition. This, in turn, is interfaced to TestAmerica's Chrom software for data processing. Data from sample extract analysis can be accessed in real-time, while sample data reports and library searches can be performed on data files from previously run samples. The software is also capable of searching any GC/MS data file for ions of a specific mass whose abundances can be plotted versus time or scan number which allows integration of abundances for any extracted ion between specified times or scan-number limits. Library searches utilize a NIST 02.1 Mass Spectral Library.

6.2 Bottles, glass with polytetrafluoroethylene (PTFE)-lined screw caps or crimp tops.

6.3 Injection port liners, splitless

6.4 Injection port septa

6.5 Injection port graphite seals

6.6 Pre-silanized glass wool (Supelco 2-0411 or equivalent)

6.7 Syringes, Assorted sizes 10ul - 1000ul; gas-tight

6.8 Bottles, 10 and 5ml amber screw cap with Teflon liner

6.9 Vials, 2ml amber screw cap with Teflon liner

6.10 Wheaton microvials 100ul (or equivalent)

6.11 Volumetric Flasks, Class A with ground glass stoppers (2ml - 100ml)

6.12 Analytical balance, ASP Model SP-180 (or equivalent), capable of accurately weighing to 0.0001 gr.

7.0 Reagents and Standards

7.1. Reagents:

7.1.1. Methylene Chloride: J.T.Baker Resi-Analyzed, used for Organic Residue Analysis (P/N 9266-V8 or equivalent).

7.1.2. Methanol: J.T.Baker Purge and Trap Grade (P/N 9077-02 or equivalent).

7.1.3. Toluene: J.T.Baker Resi-Analyzed, for Organic Residue Analysis (P/N 9460-03 or equivalent).

7.1.4. Sylon-CT: Supelco (P/N 33065-U or equivalent). Sylon-CT is a highly reactive silanizing reagent consisting of 95% Toluene and 5% Dimethyldichlorosilane (DMDCS).

7.1.5. Each lot of solvent is screened for contaminants before being used for analysis as detailed in TestAmerica Corporate Quality SOP No. CA-Q-S-001 (*Solvent & Acid Lot Testing & Approval*) and TestAmerica Edison SOP No. ED-GEN-023 (*Bulk Solvent Testing and Approval*).

7.2. Standards:

7.2.1. **Calibration Standards (Full Scan Analysis)**: Stock analytical standard solutions are purchased mainly from Restek Corporation. Other standards are prepared in the laboratory as needed using neat compounds or prepared solutions purchased from SPEX CertiPrep, Chem Service, Accustandard, Supelco or other suppliers. Standards prep instructions are detailed for the following full scan analyte list options:

- Full Volume Aqueous Prep and Soils – Long Analyte List
- Full Volume Aqueous Prep and Soils – Short Analyte List
- Full Volume Aqueous Prep and Soils – Aromatic Amines
- Reduced Volume Aqueous Prep and Soils – Long Analyte List
- Reduced Volume Aqueous Prep and Soils – Short Analyte List
- Reduced Volume Aqueous Prep and Soils – Aromatic Amines

Secondary dilutions are either made from purchased stock solutions as listed below or from prepared solutions as listed in the following table:

NOTE: Second sources (from certified separate lots) are used for ICV standards.

Table 3 – Full Scan Stock Standards			
Target Analyte Standard Name	**Conc. (PPM)**	**Vendor**	**Catalog #**
1,2,3,4-TCDD	50	SPEX	SVO-TANJ-12
SPEX Super Mix (contains compounds listed in table below)	2000 *	SPEX	SVO-TANJ-16
8270 List 1/ Std #1 Megamix	Varied	Restek	567672
8270 List 1/ Std #7 N-Diphenylamine	2000	Restek	567676
8270 List 1/ Std #8	2000	Restek	568724
8270 Surrogate Standard	5000*	Restek	567685
8270 Internal Standard	2000	Restek	567684
8270 List 1/ Std#2 Amines	2000	Restek	567673
Custom Aromatic Amine Mix (see Table 5 below)	2000	Supelco	21892423
Custom Aromatic Amine Surrogate Standard (see Table 17A)	2000	Restek	569641
Bisphenol-A	1000	SPEX	S-509-MC

*SPEX Super Mix and 8270 Surrogate standard are diluted to 100ppm prior to the preparation of the 1.0ppm and 0.5ppm standards.

Table 4	
SPEX Super Mix SPEX Catalog No. SVO-TANJ-16	
Analyte	**Concentration (PPM)**
Pentachloronitrobenzene	2000
2 -tert-butyl-4-Methylphenol	2000
2,6-Di-tert-butyl-4-Methylphenol	2000
Coumarin	2000
Phenyl ether	2000
N,N'-Dimethylaniline	2000
N-Methylaniline	2000
Carbamazepine	2000
Benzonitrile	2000
1,3-Dimethylnaphthalene	2000

Table 5 Supelco Custom Aromatic Amine Mix Catalog No. 2168334	
Analyte	**Concentration (PPM)**
Aniline	2000
o-Toluidine	2000
2-Ethylaniline	2000
2,4-Dimethylaniline	2000
3,4-Dimethylaniline	2000
2,3-Dimethylaniline	2000
2,4,5-Trimethylaniline	2000
4-Chloro-o-Toluidine	2000
4-Chloroaniline	2000
2-Naphthylamine	2000

7.2.1.1. Individual calibration standards for full scan analysis are prepared in one of several ways depending upon the technique (full volume aqueous prep, soils prep, reduced volume prep with LVI) as well as the target analyte list (long list, short list, aromatic amines). The following tables detail the preparation of calibration standard solutions for each of these techniques and analyte lists. Prepare by combining the indicated volumes of each stock solution using volumetric flask. Dilute to the volume marker with methylene chloride.

Table 6 Full Volume Aqueous Prep and Soils – Long Analyte List Working Standards Preparation								
Solution Name	**120 PPM**	**80 PPM**	**50 PPM**	**20 PPM**	**10 PPM**	**5 PPM**	**1 PPM**	**0.5 PPM**
8270 List 1/ Std #1 Megamix	1200ul	800ul	500ul	200ul	100ul	50ul	10ul	5ul
8270 List 1/ Std #7	600ul	400ul	250ul	100ul	50ul	25ul	-	-
8270 List 1/ Std #8	600ul	400ul	250ul	100ul	50ul	25ul	-	-
SPEX Super Mix	600ul	400ul	250ul	100ul	50ul	25ul	100ul*	50ul*
1,2,3,4-TCDD	-	-	100ul	-	-	-	-	-
8270 Surrogate Standard	240ul	160ul	100ul	40ul	20ul	10ul	100ul*	50ul*
8270 Internal Standard	200ul	200ul	200ul	200ul	200ul	200ul	200ul	200ul
Bisphenol-A	600ul	400ul	250ul	100ul	50ul	25ul		
Final Volume (ml)	**10**	**10**	**10**	**10**	**10**	**10**	**10**	**10**

Note: The 1.0ppm and 0.5pmm standards (above) are prepared using the 100ug/ml standard for Spex Super Mix and 8270 Surrogate Standard.

Table 7 Full Volume Aqueous Prep and Soils – Short Analyte List Working Standards Preparation						
Solution Name	**120 PPM**	**80 PPM**	**50 PPM**	**20 PPM**	**10 PPM**	**5 PPM**
8270 Internal Standard	200ul	200ul	200ul	200ul	200ul	200ul
8270 List 1/ Std#8	600ul	400ul	250ul	100ul	50ul	25ul
Final Volume (ml)	**10**	**10**	**10**	**10**	**10**	**10**

Table 8 Full Volume Aqueous Prep and Soils - Aromatic Amines Working Standards Preparation						
Solution Name	**120 PPM**	**80 PPM**	**50 PPM**	**20 PPM**	**10 PPM**	**0.5 PPM**
8270 Internal Standard	200ul	200ul	200ul	200ul	200ul	200ul
Custom Aromatic Amine Mix	600ul	400ul	250ul	100ul	50ul	2.5ul
Custom Aromatic Amine Surrogate Std	600ul	400ul	250ul	100ul	50ul	2.5ul
Final Volume (ml)	**10**	**10**	**10**	**10**	**10**	**10**

Table 9 Reduced Volume Extraction/LVI – Long Analyte List Working Standards Preparation								
Solution Name	**24 PPM**	**16 PPM**	**10 PPM**	**4 PPM**	**2 PPM**	**1 PPM**	**0.2 PPM**	**0.1 PPM**
120 ppm Long Cal Std (see Table 6)	1.0 mL							
80 ppm Long Cal Std (see Table 6)		1.0 mL						
50 ppm Long Cal Std (see Table 6)			1.0 mL					
20 ppm Long Cal Std (see Table 6)				1.0 mL				
10 ppm Long Cal Std (see Table 6)					1.0 mL			
5.0 ppm Long Cal Std (see Table 6)						1.0 mL		
1.0 ppm Long Cal Std (see Table 6)							1.0 mL	
0.5 ppm Long Cal Std (see Table 6)								1.0 mL
Final Volume (ml)	**5**	**5**	**5**	**5**	**5**	**5**	**5**	**5**

Table 10 Reduced Volume Extraction/LVI – Short Analyte List Working Standards Preparation						
Solution Name	**24 PPM**	**16 PPM**	**10 PPM**	**4 PPM**	**2 PPM**	**1 PPM**
120 ppm Short Cal Std (see Table 7)	1.0 ml					
80 ppm Short Cal Std (see Table 7)		1.0 ml				
50 ppm Short Cal Std (see Table 7)			1.0 ml			
20 ppm Short Cal Std (see Table 7)				1.0 ml		

Table 10 Reduced Volume Extraction/LVI – Short Analyte List Working Standards Preparation						
Solution Name	**24 PPM**	**16 PPM**	**10 PPM**	**4 PPM**	**2 PPM**	**1 PPM**
10 ppm Short Cal Std (see Table 7)					1.0 ml	
5.0 ppm Short Cal Std (see Table 7)						1.0 ml
Final Volume (ml)	**5**	**5**	**5**	**5**	**5**	**5**

Table 11 Reduced Volume Extraction/LVI -Aromatic Amine Working Standards Preparation						
Solution Name	**24 PPM**	**16 PPM**	**10 PPM**	**4 PPM**	**2 PPM**	**0.1 PPM**
120 ppm Aromatic Amines Cal Std (see Table 8)	1.0 ml					
80 ppm Aromatic Amines Cal Std (see Table 8)		1.0 ml				
50 ppm Aromatic Amines Cal Std (see Table 8)			1.0 ml			
20 ppm Aromatic Amines Cal Std (see Table 8)				1.0 ml		
10 ppm Aromatic Amines Cal Std (see Table 8)					1.0 ml	
0.5 ppm Aromatic Amines Cal Std (see Table 8)						1.0 ml
Final Volume (ml)	**5**	**5**	**5**	**5**	**5**	**5**

7.2.1.2. **Initial Calibration Verification (full scan**): Second source ICVs for full scan analysis are prepared in one of several ways depending upon the technique (full volume aqueous prep, soils prep, reduced volume prep with LVI) as well as the target analyte list (long list, short list, aromatic amines). The following tables detail the preparation of ICVs for each of these techniques and analyte lists. Prepare by combining the indicated volumes of each stock solution using volumetric flask. Dilute to the volume marker with methylene chloride.

Table 12 8270/625 ICV -Long List Working Standards Preparation	
Solution Name	**25 PPM**
8270 List 1/ Std #1 Megamix (2nd Lot)	250ul
8270 List 1/ Std #7 (2nd Lot)	125ul
8270 List 1/ Std #8 (2nd Lot)	125ul
SPEX Super Mix (2nd Lot)	125ul
8270 Internal Standard	200ul
Bisphenol-A (2nd Lot)	125ul
Final Volume (ml)	**10**

Table 13 8270/625 ICV - Short List Working Standards Preparation	
Solution Name	**25 PPM**
8270 Internal Standard (2nd Lot)	200ul
8270 List 1/ Std#2 Amines (2nd Lot)	125ul
Final Volume (ml)	**10**

Table 14 Aromatic Amines ICV Working Standards Preparation	
Solution Name	**25 PPM**
8270 Internal Standard	200ul
Supelco Aromatic Amines 2nd Lot (Cat. No. 21467482)	125ul
Final Volume (ml)	**10**

Table 15 8270/625 ICV LVI - Long List Working Standards Preparation	
Solution Name	**5 PPM**
25PPM 8270/625 ICV (Long List) (see Table 12)	1.0 mL
Final Volume (ml)	**5**

Table 16 8270/625 ICV LVI -Short List Working Standards Preparation	
Solution Name	**5 PPM**
25PPM 8270/625 ICV (Short List) (see Table 13)	1.0 mL
Final Volume (ml)	**5**

7.2.1.3. **Surrogate Standards (Full Scan Analysis):** A 5000ppm Surrogate Standard is purchased from Restek for use in spiking blanks, samples and associated QC prior to extraction (reference the applicable sample prep SOPs for spiking instructions).

Table 17 Full Scan Surrogate Standards Solution Restek Catalog No. 567685	
Surrogate Standard Compounds	**Concentration (PPM)**
Nitrobenzene-d5	5000
p-Terphenyl-d14	5000
2,4,6-Tribromophenol	5000
Phenol-d5	5000
2-Fluorobiphenyl	5000
2-Fluorophenol	5000

7.2.1.3.1 Surrogate Standards (Aromatic Amine Analysis): A 2000 ppm Surrogate Standard is purchased from Restek (Cat. # 569641) for use in spiking blanks, samples and associated QC prior to extraction and analysis of samples for Aromatic Amines (reference the applicable prep SOPs for spiking instructions).

Table 17a Aromatic Amine Surrogate Standards Solutions Restek Catalog Nos. 569641	
Surrogate Standard Compounds	**Concentration (PPM)**
Aniline-d5	5000
o-Toluidine-d9	5000
4-Chloroaniline-d4	5000

7.2.1.4. Internal Standards (**Full Scan Analysis**): The Internal Standards Solution at 2000ppm is purchased from Restek (Catalog # 567684). The Internal Standard solution is stored in 10ml amber screw cap bottles with Teflon liners in the dark at 4°C. The Internal standard solution is used in preparing all analytical standards. Inject 20ul of this solution (2000ppm) per ml of sample extract prior to analysis resulting in a concentration of 40ppm (ug/ml) in the extract.

Table 18 Full Scan Internal Standards Solution Restek Catalog No. 567684	
Internal Standard Compounds	**Concentration (PPM)**
1,4-Dichlorobenzene-d4	2000
Phenanthrene-d10	2000
Naphthalene-d8	2000
Chrysene-d12	2000
Acenaphthene-d10	2000

Table 18 Full Scan Internal Standards Solution Restek Catalog No. 567684	
Internal Standard Compounds	**Concentration (PPM)**
Perylene-d12	2000

7.2.2. Calibration Standards (SIM analysis): The Edison lab currently analyzes only a select list of compounds by 8270D SIM (see Sections 1.0 and 2.0). Stock analytical SIM standard solutions are purchased mainly from Accustandard and Spex. Working standards are prepared from these solutions as listed in the tables in Section 7.2.2.1:

Table 19- Stock SIM Standards			
Standard Name	**Concentration**	**Vendor**	**Catalog #**
Pentachlorophenol	100ppm	Accustandard	App-9-176
n-Nitrosodimethylamine	100ppm	Accustandard	APP-9-149
Hexachlorobenzene	100ppm*	Accustandard	APP-9-112
PAH Mix	100ppm	Accustandard	M-610
Bis(2-chloroethyl)ether	100ppm*	Accustandard	App-9-027
4,6-Dinitro-2-methylphenol	100ppm	Accustandard	P-3845
1,4-Dioxane	1000ppm**	Accustandard	APP-9-096

*Hexachlorobenzene and Bis(2-chloroethyl)ether are diluted to 10ppm prior to SIM Standards prep

** 1,4-Dioxane is diluted (10x) to 100ppm prior to SIM Standards prep

NOTE: Second sources (from separate lots are used for ICV standards).

7.2.2.1 Individual calibration standards for SIM analysis are prepared in one of two ways depending upon the technique (full volume aqueous prep or reduced volume prep with LVI) as well as the target analyte list (long list, short list, aromatic amines). The following tables detail the preparation of calibration standard solutions for each of these techniques and analyte lists. Prepare by combining the indicated volumes of each stock solution using volumetric flask. Dilute to the volume marker with methylene chloride.

Table 20 Full Volume Aqueous Prep – SIM Working Standards Preparation						
	0.025 PPM	**0.05 PPM**	**0.1 PPM**	**0.5 PPM**	**1.0 PPM**	**5.0 PPM**
Pentachlorophenol	10uL	25uL	50uL	50uL	100uL	250uL
n-Nitrosodimethylamine	10uL	25uL	50uL	50uL	100uL	250uL
PAH mix	2.5uL	5uL	100uL	25uL	50uL	100uL

Table 20 Full Volume Aqueous Prep – SIM Working Standards Preparation						
	0.025 PPM	**0.05 PPM**	**0.1 PPM**	**0.5 PPM**	**1.0 PPM**	**5.0 PPM**
Hexachlorobenzene	10uL	25uL	100uL	500uL	1000uL	2500uL
Bis(2-chloroethyl)ether	10uL	25uL	100uL	500uL	1000uL	250uL*
4,6-dinitro-2-methylphenol	50ul	100ul	200ul	200ul	250ul	500ul
1,4-Dioxane	20ul	50ul	100ul	100ul	200ul	500ul
ISTD	200uL	200uL	200uL	100uL	100uL	100uL
Final Volume (ml)	**10**	**10**	**10**	**5**	**5**	**5**

*For Bis(2-chloroethyl)ether the 5.0 ppm level is prepared using the 100ppm standard.

Table 21 Reduced Volume Extraction/LVI – SIM Working Standards Preparation						
	0.005 PPM	**0.01 PPM**	**0.02 PPM**	**0.10 PPM**	**0.20 PPM**	**1.0 PPM**
0.025 PPM Std (see Table 20)	1.0 mL					
0.05 PPM Std (see Table 20)		1.0 mL				
0.1 PPM Std (see Table 20)			1.0 mL			
0.5 PPM Std (see Table 20)				1.0 mL		
1.0 PPM Std (see Table 20)					1.0 mL	
5.0 PPM Std (see Table 20)						1.0 mL
Final Volume (ml)	**5**	**5**	**5**	**5**	**5**	**5**

7.2.2.2 Initial Calibration Verification (SIM): A 0.1 ppm separate lot SIM ICV is prepared as detailed in Table 6 using the stock standards detailed in Section 7.2.1.4 (above)

Table 22 0.1ppm SIM ICV preparation	
Pentachlorophenol	25uL
n-Nitrosodimethylamine	25uL
PAH mix	5uL
Hexachlorobenzene	5uL
1,4-Dioxane	5ul
4,6-Dinitro-2-methylphenol	100ul
ISTD	**100uL**
Final Volume	**5 ml**

7.2.2.3 Internal Standard solution (SIM): A 50 ppm Internal Standard solution for SIM analysis is prepared by adding 125ul of the 2000ppm stock ISTD (see Section 7.2.1.4) and bringing to volume with Methylene Chloride in a 5ml volumetric flask.

7.2.2.3.1 For SIM analysis inject 20ul of this solution (50ppm) per ml of sample extract prior to analysis resulting in a concentration of 1ppm (ug/ml) in the extract.

.

7.2.3. Calibration Standards (Isotope Dilution SIM – 1,4-Dioxane): The Edison lab currently analyzes only for 1,4-dioxane by 8270D isotope dilution SIM (see Sections 1.0 and 2.0). Stock analytical isotope dilution SIM standard solutions are purchased mainly from Accustandard and Restek. Working standards are prepared from these solutions as listed in the tables below.

Table 23 -
Stock 1,4-Dioxane Isotope Dilution SIM Standards

Standard Name	Concentration	Vendor	Catalog #
1,4-Dioxane	1000ppm*	Accustandard	APP-9-096

* 1,4-Dioxane is diluted (10x) to 100ppm prior to SIM Standards prep

Table 24 -
Stock Labeled 1,4-Dioxane SIM Surrogate/Internal Standard (added at prep)

Standard Name	Concentration	Vendor	Catalog #
1,4-Dioxane-d8	2000ppm	Restek	A0120108

Table 25 -
Stock 1,4-Dioxane Isotope Dilution SIM Internal Standard (added to extract)

Standard Name	Concentration	Vendor	Catalog #
1,4-Dichlorobenzene-d4	2000ppm	Restek	A0121898

Table 26 -
Stock 1,4-Dioxane Isotope Dilution SIM Separate Source ICV

Standard Name	Concentration	Vendor	Catalog #
1,4-Dioxane	1000ppm	Absolute	70373

7.2.3.1 Individual calibration standards for 1,4-dioxane isotope dilution SIM analysis are prepared at the concentrations detailed in the following tables. Prepare by combining the appropriate volumes of each stock solution using volumetric flask. Dilute to the volume marker with methylene chloride.

Table 27 Reduced Volume Extraction/LVI – 1,4-Dioxane Isotope Dilution SIM ICAL Standard Concentrations (ug/ml)							
	Lev 1	**Lev 2**	**Lev 3**	**Lev 4**	**Lev 4**	**Lev 6**	**ICV***
1,4-Dioxane	10	2	0.8	0.4	0.1	0.04	0.2
1,4-Dioxane-d8	4	4	4	4	4	4	4
1,4-Dichlorobenzene-d4	0.2	0.2	0.2	0.2	0.2	0.2	0.2

***:** The ICV is prepared from the second source stock in Table 26.

7.2.4. GC/MS Instrument Performance Check (DFTPP): The DFTPP standard is prepared by is prepared at 25 ppm by adding 2.5ml of EPA 8270 GC/MS Tuning Solution II (Supelco Catalog # 47548-U) to a 100ml volumetric flask and bringing to volume with Methylene Chloride.

7.2.5. Information on prepared standard solutions must be recorded in a standards logbook or in the TALS Reagent Module. Information such as standard supplier, lot number, original concentration, a description of how the standard was made, are required along with the laboratory lot number, analyst's initials, date prepared, expiration date and verification signature. Standards must be remade every 6 months, or sooner, if the standards expire or begin to show signs of unacceptable degradation. Class "A" volumetric must be used at all times and syringes, preferably gas-tight syringes when available, should be checked for accuracy using an analytical balance. Class "A" pipettes should also be used if volumes permit.

7.2.6. Please refer to TestAmerica Edison SOP No. ED-GEN-008, Standard Operating Procedure for Preparation, Purity and storage of Reagents and Standards.

- Shelf Life of Standard: 6 months
- Storage Requirements: Stock standards are stored at 4°C and Working Standards stored at -10°C to -20°C.

8.0 Sample Collection, Preservation, Shipment and Storage

8.1 All samples must be stored at 4ºC (± 2ºC) upon receipt.

8.2 Sample Extract Storage. Samples extracts must be protected from light and refrigerated at 4ºC (± 2ºC) from time of extraction until analysis.

8.3 Sample Extract Holding Time. All sample extracts must be analyzed within 40 days of extraction.

Matrix	Sample Container	Min. Sample Size	Preservation	Holding Time	Reference
Waters	Amber glass, 1L	1000 ml or 250 ml (1)	Cool 4 ± 2°C	7 days to extraction; Analyze within 40 days of extraction	EPA Method SW846 8270D
Solids	Wide mouth glass, 8 or 16 oz.	50g	Cool 4 ± 2°C	14 days to extraction; Analyze within 40 days of extraction	EPA Method SW846 8270D

(1) : Reduced volume extaction (RVE) LVI option

9.0 Quality Control

9.1. **Sample QC** - The following quality control samples are prepared with each batch of samples.

Quality Controls	Frequency	Control Limit
Method Blank (MB)	1 in 20 or fewer samples	< Rpt. Limit
Laboratory Control Sample (LCS)[1]	1 in 20 or fewer samples	Statistical Limits [4]
Matrix Spike (MS)[2]	1 in 20 or fewer samples	Statistical Limits [4]
MS Duplicate (MSD)[2]	1 in 20 or fewer samples	Statistical Limits [4]
Surrogates	every sample[3]	Statistical Limits [4]
Internal Standards	Every sample	Response within -50% to +100% of CCV

[1] LCS Duplicate (LCD) is performed only when insufficient sample is available for the MS/MSD or when requested by the client/project/contract.
[2] The sample selection for MS/MSD are randomly selected, unless specifically requested by a client….predetermined by the extraction lab.
[3] Analytical and QC samples (MB, LCS, MS/MSD, Method Blank)
[4] Statistical control limits are updated annually and are updated into lab reporting software.

9.1.1. **Method blanks** are extracted with every sample batch on each day that samples are extracted. To be considered acceptable, the method blank must contain less than the reporting limit of all target compounds except for phthalates, which can be present at up to 5x the MDL. .

If method blanks are unacceptably contaminated with target compounds that are also present in field samples, all affected samples must be re-extracted and re-analyzed. Corrective action must be taken to identify and eliminate the contamination source. Demonstrate that acceptable blanks can be obtained before continuing with sample extraction and analysis. Method blanks must be analyzed on each instrument on which the associated samples are analyzed.

9.1.1.1. Surrogate recoveries for the method blank are compared to laboratory generated limits. If two or more surrogates for any one fraction (base-neutral or acid) are outside of recovery limits or if any one surrogate recovers at <10%, the sample must be re-extracted and re-analyzed to confirm matrix interference.. If any surrogate is still outside limits, all samples and QC samples associated with that method blank must be re-extracted (volume permitting).

9.1.2. Matrix Spike (MS)/Matrix Spike Duplicate (MSD): A matrix spike/matrix spike duplicate (MS/MSD) pair is extracted and analyzed with every 20 environmental samples of a specific matrix (defined as a sample batch). Full compound list spiking is employed for MS/MSDs and LCSs. These spikes are prepared and extracted concurrent with sample preparation. MS and MSD recoveries are calculated and compared to lab generated acceptance criteria. See the current active TALS 8270D Method Limit Group for QC limits. The MS/MSD spiking solution should the same as used for the calibration standards.

9.1.2.1 A Laboratory Control Sample Duplicate (LCSD) is extracted and analyzed only when insufficient client sample is available for preparation of an MS/MSD pair. The LCS/LSCD is evaluated in the same manner as the MS/MSD (see Section 9.1.2)

9.1.2.2 An LCS/LCSD may be substituted for the MS/MSD if insufficient sample volume is available.

9.1.3. Laboratory Control Sample (LCS)/Laboratory Control Sample Duplicate (LCSD): A Laboratory Control Sample (LCS) (aka blank spike) must be extracted and analyzed with each batch of 20 environmental samples. The LCS data is used to assess method performance if the MS/MSD recoveries fall outside of the lab generated limits (See the current active TALS 8270D Method Limit Group for QC limits). If the LCS recovery is within the current lab generated limits, the MS/MSD recoveries are attributed to matrix interference.

9.1.3.1 A Laboratory Control Sample Duplicate (LCSD) is extracted and analyzed only when insufficient client sample is available for preparation of an MS/MSD pair. The LCS/LSCD is evaluated in the same manner as the MS/MSD (see Section 9.1.2)

9.1.3.2 Spike recovery limits are lab generated and are updated annually. Certain state regulatory programs have defined recovery limits which, where applicable, are used for spike recovery evaluations. The TALS Method Limit Groups detail these regulatory program criteria.

9.1.4. Surrogate Standards: All full scan samples, blanks and QC samples are spiked with a six (6) component surrogate standard mix (see Section 7.2.1.3). The percent recovery of the surrogate standards is calculated and compared to lab generated limits (See the current active TALS 8270D Method Limit Group for QC limits). **Note:** Three (3) surrogates are used when analyzing for Aromatic Amines (see Section 7.2.1.3.1).

If any two or more surrogates for any one fraction (base-neutral or acid) are outside of recovery limits or if any one surrogate recovers at <10%, the sample must be re-extracted and re-analyzed to confirm matrix interference. If a surrogate is diluted to a concentration below that of the lowest calibration standard, no corrective action is necessary.

9.1.4.1 Surrogate recovery limits are lab generated and are updated annually. Certain state regulatory programs have defined recovery limits which, where applicable, are used for spike recovery evaluations. The TALS Method Limit Groups detail these regulatory program criteria.

9.1.5. Internal Standards: The response (area count) of each internal standard in the sample must be within -50 +100% of its corresponding internal standard in the CCV or, the ICAL midpoint for samples analyzed under the initial calibration range. Failure to meet these criteria is indicative of sample matrix effects. All samples failing these criteria must be reanalyzed to confirm matrix effects.

9.2. Instrument QC

9.2.1 GC/MS Instrument Performance Check (DFTPP): (**Note**: the DFTPP performance check applies only to full scan analyses and is not evaluated for SIM analysis). The GC/MS system is tuned using Perfluortributylamine (PFTBA) such that an injection of 50ng of Decafluorotriphenylphosphine (DFTPP) meet the abundance criteria listed in the table below. Prior to the analysis of any calibration standards or samples, the GC/MS system must meet all DFTPP key ion abundance criteria. This analysis will verify proper tuning of the system for a period of 12 hours post-injection. After 12 hours, the instrument performance must again be verified prior to the analysis of standards, QC or samples.

DFTPP Key Ions and Abundance Criteria	
Mass	**Ion Abundance Criteria**
51	30-60% of mass 198
68	<2% of mass 69
69	reference only
70	<2% of mass 69
127	40-60% of mass 198
197	<1% of mass 198
198	Base Peak, 100% relative abundance
199	5-9% of mass 198
275	10-30% of mass 198

DFTPP Key Ions and Abundance Criteria	
365	>1% of mass 198
441	present but less than mass 443
442	>40% of mass 198
443	17-23% of mass 442

9.2.1.1. Evaluate DFTPP using three scan averaging and background subtraction techniques. Select the scan at the peak apex, add +1 scan from the apex and -1 scans from the apex.

9.2.1.2. The mass spectrum of DFTPP may be background subtracted to eliminate column bleed or instrument background ions. Background subtract DFTPP by selecting a scan for subtraction ≤20 scans before the apex scan of DFTPP.

9.2.1.3. Check column performance using pentachlorophenol and the benzidine peaks (these compounds are included in the DFTPP solution). Benzidine & Pentachlorophenol should respond normally without significant peak tailing (Tailing Factor should be <2 measured at 10% peak height). If responses are poor and excessive peak tailing is present, corrective action for the GC/MS instrument may be required. Corrective actions may include:

9.2.1.3.1 Retune the GC/MS;
9.2.1.3.2 Clip the injector end of the GC column;
9.2.1.3.3 Replace the septum and injection port liner;
9.2.1.3.4 Change the injection port seal;
9.2.1.3.5 Replace the GC column;
9.2.1.3.6 Clean the injection port with MeCl2
9.2.1.3.7 Clean the MS ion source;
9.2.1.3.8 Place a service call.

9.2.1.4. The breakdown of 4, 4-DDT into 4,4-DDD and 4,4'DDE may also be used to assess GC column performance and injection port inertness. If so evaluated the breakdown must be <20%.

9.2.1.5. DFTPP parameter settings are stored in a tune file, which will be used in all subsequent analysis of standards and sample extracts.

9.2.2 Initial Calibration Range and Initial Calibration Verification

9.2.2.1. **Initial Calibration:** The initial calibration range consists of a minimum of five concentration levels of analytical standards (six for second order regression) prepared as described in Section

7.2. and analyzed once the DFTPP instrument performance check has met the criteria in Section 9.2.1. .

9.2.2.2. Initial Calibration Verification (ICV): An Initial Calibration Verification (ICV) standard is analyzed immediately after the Initial Calibration Range and before any samples are analyzed. The ICV is prepared as detailed in Section 7.2. The ICV must be from a source (or lot) separate from the standards used in the Initial Calibration Range.

9.2.3 Continuing Calibration Verification (CCV) and Low Level Continuing Calibration Verification (LLCCV): A mid-point Continuing Calibration Verification (CCV) must be analyzed every 12 hours after the DFTPP instrument performance check (when applicable).. The CCV is prepared as detailed in Section 7.2. (typically, 50 ug/ml for full volume aqueous and soils, 10 ug/ml for LV, 0.02 ug/ml for LVI SIM) and 0.2 for isotope dilution SIM). Additionally a Low Level Continuing Calibration Verification (LLCCV) is analyzed after the CCV for full scan analysis. The LLCCV is the same as the lowest calibration level analyzed with the initial calibration range (See Section 7.2).

9.2.4 Calibration Acceptance Summary

9.2.4.1. Retention Time Windows: Retention time windows must be established to compensate for minor shifts in absolute retention times as a result of sample loading and normal chromatographic variability. Obtain the retention time for all compounds from the analysis of the midpoint standard for the calibration curve. Establish the center of the retention time window by using the absolute retention time for each analyte, internal standard and surrogate from the calibration verification standard at the beginning of the analytical shift. For samples run during the same shift as an initial calibration, use the retention time of the mid-point standard of the initial calibration. For qualitative identification to be acceptable the retention time of the relative retention time (automatically calculated in Chrom) must be within 0.8 - 1.2 RRT units of its assigned internal standard. The relative retention times of each compound in the five calibration standards must agree within .06 relative retention time units.

9.2.4.2. Initial Calibration Range: Internal standard calibration is employed for this method. After the initial calibration range has been analyzed the relative response factor (RRF) for each target/surrogate compound at each concentration level is determined using the following equation.

$$RRF = \frac{A_x}{A_{is}} \times \frac{C_{is}}{C_x}$$

Where:
A_x = Area characteristic ion (see Table 31) for the compound
Ais = Area characteristic ion (see Table 31) of associated internal standard
Cis = Concentration of internal standard
Cx = Concentration of compound in standard

9.2.4.2.1. Determine the mean RRF for each compound. Minimum response factors must be met for each of the compounds listed in Table 28 (below). Any compound that fails the minimum response factor must be reported as estimated for detects and must have a demonstration of sensitivity in the analytical batch to report non-detects. To demonstrate adequate sensitivity for out of criterion compounds analyze the low level point of the calibration (LLCCV) in the analytical sequence. The criterion for the LLCCV is detection only but the standard qualitative identification criteria in the method must be met.

Table 28:
Minimum Response Factors

Compound	Minimum Response Factor
Benzaldehyde	0.010
Phenol	0.800
Bis(2-chloroethyl) ether	0.700
2-Chlorophenol	0.800
2-Methylphenol	0.700
2,2-Oxybis-(1-chloropropane)	0.010
Acetophenone	0.010
4-Methylphenol	0.600
N-Nitroso-di-n-propylamine	0.500
Hexachloroethane	0.300
Nitrobenzene	0.200
Isophorone	0.400
2-Nitrophenol	0.100
2,4-Dimethylphenol	0.200
Bis(2-chloroethoxy)methane	0.300
2,4-Dichlorophenol	0.200
Naphthalene	0.700
4-Chloroaniline	0.010
Hexachlorobutadiene	0.010
Caprolactam	0.010
4-chloro-3-methylphenol	0.200
2-Methylnaphthalene	0.400
Hexachlorocyclopentadiene	0.050
2,4,6-Trichlorophenol	0.200
2,4,5-Trichlorophenol	0.200

Table 28: Minimum Response Factors	
Compound	**Minimum Response Factor**
1,1'-Biphenyl	0.010
2-Chloronaphthalene	0.800
2-Nitroaniline	0.010
Dimethyl phthalene	0.010
2,6-Dinitrotoluene	0.200
Acenaphthylene	0.900
3-Nitroaniline	0.010
Acenaphthene	0.900
2,4-Dinitrophenol	0.010
4-Nitrophenol	0.010
Dibenzofuran	0.800
2,4-Dinitrotoluene	0.200
Diethyl phthalate	0.010
1,2,4,5-Tetrachlorobenzene	0.010
4-chlorophenyl-phenyl ether	0.400
Fluorene	0.900
4-Nitroanailine	0.010
4,6-Dinitro-2-methylphenol	0.010
4-Bromophenyl-phenyl ether	0.100
N-Nitrosodiphenylamine	0.010
Hexachlorobenzene	0.100
Atrazine	0.010
Pentachlorophenol	0.050
Phenanthrene	0.700
Anthracene	0.700
Carbazole	0.010
Di-n-butyl phthalene	0.010
Fluoranthene	0.600
Pyrene	0.600
Butyl benzyl phthalate	0.010
3,3'-Dichlorobenzidine	0.010
Benzo(a)anthracene	0.800
Chrysene	0.700
Bis-(2-ethylhexyl)phthalate	0.010
Di-n-octyl phthalate	0.010
Benzo(b)fluoranthene	0.700
Benzo(k)fluoranthene	0.700
Benzo(a)pyrene	0.700
Indeno(1,2,3-cd)pyrene	0.500
Dibenz(a,h)anthracene	0.400
Benzo(g,h,i)perylene	0.500
2,3,4,6-Tetrachlorophenol	0.010
Pentachloronitrobenzene	0.050

9.2.4.2.2. Calculate the Standard Deviation (SD) and Percent Relative Standard Deviation (% RSD) of the response factors for each compound:

$$\% \text{ RSD} = \frac{\text{Standard Deviation of RRFs}}{\text{Mean RRF}}$$

9.2.4.2.3. The % RSD of the RRF's must be ≤20% for each target analyte listed in Table 28. The % RSD of each target analytes must be ≤20% in order for the calibration range to be acceptable. If more than 10% of the compounds included with the initial calibration exceed the 20% RSD limit or do not meet the minimum correlation coefficient (0.99) for alternate fits (see below) then appropriate corrective maintenance action must be performed. If more than 10% of the compounds included with the initial calibration exceed the 20% RSD limit **AND** do not meet the minimum correlation coefficient (0.99) then recalibration is necessary.

9.2.4.2.4. If the above listed criteria is met, the system can be assumed to be linear and sample analysis may begin and the average RF from the initial calibration range is used to quantitate all samples.

9.2.4.2.4.1 Certain state regulatory programs have defined calibration acceptance limits which, where applicable, are used for calibration evaluations. The TALS ICAL Limit Groups detail these regulatory program criteria.

9.2.4.2.5. An alternative calibration technique may be employed for those any compounds exceeding the 20% RSD criteria:

9.2.4.2.5.1 Calculate the first order linear regression for any compound which did not meet the 20% criteria. First order linear regression calibration may be employed if alternative average response calibration procedures were not applicable. The r value (Correlation Coefficient) of the equation must be ≥0.99 for the calibration to be employed.

9.2.4.2.5.2 Second order regression calibration can be used for any compound that has an established history as a non-linear performer.

9.2.4.2.5.3 If second order regression calibration is used a minimum of six (6) calibration levels must be analyzed.

9.2.4.2.5.4 If second order regression calibration is used, the r^2 (Correlation Coefficient) value must be $\geq$ 0.99

9.2.4.2.5.5 Any compound that fails to meet the 20% RSD or or 0.99 correlation coefficient criteria must be flagged as estimated for detects (or must be noted in the narrative). If there are non-detects the compounds may be reported if there is adequate sensitivity to detect at the quantitation limit. To demonstrate adequate sensitivity analyze the low level point of the initial calibration in each analytical batch (LLCCV) The criteria for demonstrating adequate sensitivity is detection in the LLCCV using the standard qualitative identification criteria.

9.2.4.2.5.6. When calculating the calibration curve using the linear calibration model a minimum quantitation check on the viability of the lowest calibration point should be performed by re-fitting the response from the low concentration back into the curve. The recalculated concentration of the low calibration point should be within ±30% of the standard's concentration. This evaluation can be checked using the Initial Calibration %Drift Report in Chrom. Any detects for analytes calibrated using the linear model and failing this readback criterion must be flagged as estimated or detailed in the narrative.

9.2.4.3. Initial Calibration Verification (ICV):.Once the initial calibration has been analyzed and has met the above criteria, a second source Initial Calibration Verification (ICV) (as prepared in Section 7.2) must be analyzed and evaluated. The ICV must meet the criteria of 70-130% recovery for all compounds with the exception of the poor performing compounds listed in Attachment 1 which are allowed to be within 50-150% : An NCM must be initiated to denote any ICV non-conformances.

9.2.4.4. The ICV must meet the criteria of 70-130% recovery for all compounds however up to 10% of the compounds are allowed to exceed these criteria as long as their recoveries are within 65-135%. For the poor performers (see Attachment 1) the range is 50-150%. If the criterion is not met, a second ICV may be analyzed after corrective measures are taken. If a second ICV analysis fails to meet criteria proceed with corrective action and the analysis of a new initial calibration range. Flagging: If the ICV limits are outside of criteria (high) for an analyte and that analyte is undetected in the sample, no flagging or narration is

required. If the ICV limits are outside of criteria (low) for an analyte and that analyte is undetected in a sample, narrate the non-conformance in an NCM. When that out of spec analyte is detected in a sample, describe the issue in the narrative, or flag as estimated.

9.2.4.5. Continuing Calibration Verification (CCV): A CCV consisting of a standard at or near the midpoint of the Initial Calibration Range is analyzed every 12 hours of instrument operation or at the beginning of an analytical sequence to verify the initial calibration. The calibration verification consists of a DFTPP instrument performance check, and analysis of a calibration verification standard. **Note:** Certain state regulatory programs have defined calibration acceptance limits which, where applicable, are used for calibration evaluations. The TALS ICAL Limit Groups detail these regulatory program criteria.

9.2.4.5.1 Tune Verification: Follow the procedure for verifying the instrument tune described in section 9.2.1 using a 50 ng injection of DFTPP. If the tune cannot be verified, analysis must be stopped, corrective action taken and a return to "control" demonstrated before continuing with the calibration verification process.

9.2.4.5.2 Calibration Verification: Analyze the calibration verification standard immediately after a DFTPP that meets criteria. Use the mid point calibration standard (approximately 50ug/l). NOTE: The calibration standard contains internal standards; Dichlorobenzene d_4, Naphthalene d_8, Acenaphthene d_{10}, Phenanthrene d_{10}, Chrysene d_{12}, and Perylene d_{12} at 40ug/l (0.1ug/L for SIM). The calibration check standard must also include all the target analytes from the original calibration.

9.2.4.5.3 The RFs must meet the criteria for the compounds in Table 28. Any compound that fails the minimum response factor must be reported as estimated for detects and must have a demonstration of sensitivity to report non-detects. To demonstrate adequate sensitivity for out of criterion compounds analyze the low level point of the calibration (LLCCV) in the analytical sequence. The criterion for the LLCCV is detection only but the standard qualitative identification criteria in the method must be met

9.2.4.5.4 The percent difference (when using average response factor) or percent drift (when using linear regression) of

the compounds in Table 28 must be ≤20% for at least 80% of the total analyte list. If more than 20% of the compound list fail to 20% difference or drift criterion then appropriate corrective action must be taken prior to the analysis of the samples. Any individual compound that fails must be reported as estimated for detects and must have a demonstration of sensitivity to report non-detects. To demonstrate adequate sensitivity for out of criterion compounds analyze the low level point of the calibration (LLCCV) in the analytical sequence. The criterion for the LLCCV is detection only (%D criteria are not applied) but the standard qualitative identification criteria in the method must be met.

9.2.4.5.5 **CCV Poor Performers**: Refer to Attachment 1 for the identification of poor and/or erratic performing analytes. These analytes are allowed a %D >20% but must be <50 %D to be acceptable. If there are poor performers that exceed 50%D, the data may be reported provided results are noted as estimated. An NCM must be initiated to denote this situation.

9.2.4.5.6 The retention times of the internal standards from the calibration check must be within ±30 seconds of the internal standards from the mid point standard of the original calibration. If the retention time for any internal standard changes by more than 30 seconds from the latest daily (12 hour) calibration standard, the chromatographic system is inspected for malfunctions, and corrections made as required. If corrective action does not result in the retention time criteria being achieved, the system must be re-calibrated using four additional standards.

9.2.4.5.7 The response (area count) of each internal standard in the calibration verification standard must be within 50 - 100% of its corresponding internal standard in the mid-level calibration standard of the active calibration curve. If the EICP area for any internal standard changes by more than a factor of two (-50% +100%), the mass spectrometer system must be inspected for malfunction and corrections made as appropriate. When corrections are made, re-analysis of samples analyzed while the system was malfunctioning is required.

9.2.4.5.8 The relative retention times of each compound in the calibration verification standard must agree within .06 relative retention time units of its value in the initial calibration.

9.2.4.5.9 Use the average response factors from the original five-point calibration for quantitative analysis of target analytes identified in field samples.

9.2.4.5.10 Prepare a calibration summary or list indicating which compounds did not meet the 20% average percent difference criteria. Record this information in that run log.

9.2.4.6. Low Level Continuing Calibration Verification (LLCCV): An LLCCV consisting of the low level standard from the initial calibration range is analyzed every 12 hours of instrument operation after the CCV. The purpose and evaluation of the LLCCV is described in Section 9.2.4.4.4.

10.0 Procedure

10.1. Gas Chromatograph/Mass Spectrometer Operation

10.1.1. The sequence of events for GC/MS analysis involves many steps. First the injection system and column performance and calibration must be verified. Maintenance operations are performed as needed.

10.1.2. Preparation of the Injection Port Liner and Installation Procedure: Prior to the start of initial calibration and each daily analysis of sample extracts, a new liner for the injection port must be prepared. Once a liner has been used it is no longer inert and will cause serious chromatography problems with phenols and other compounds. When preparing the liner, proper laboratory protection must be worn and the liner must be prepared in a well-ventilated hood. When the procedure is completed all traces of toluene, Sylon-Ct and methanol will be removed immediately so that extraction solvents and preparation of sample extracts will not come into contact with these solvents and become contaminated.

10.1.2.1 Remove one liner from a 40ml VOA bottle containing other liners immersed in Sylon-Ct solution. Rinse off the liner with Toluene and wipe dry. Insert 1cm of pre-silanized glass wool partially into one end of the liner and trim neatly. Push the glass wool into the center of the liner so that it is 1 1/4" from the bottom. Do not use glass wool or solvents that are dirty (i.e. suspended particles) or use liners which are chipped on the ends, deformed or fractured. Inspect the glass wool for cleanliness after it has been inserted.

10.1.2.2 Using a Pasteur pipette flush out the interior of the liner containing the glass wool with Sylon-Ct. Rest the liner horizontally on a small beaker and allow the Sylon-Ct to re-deactivate the interior surfaces and the glass wool. There should be no air bubbles caught in the glass wool. After several minutes flush out the Sylon-Ct with toluene and finally with

methanol. Dry the outer surface of the liner and rest it on the injection port housing until the remaining methanol is boiled off

10.1.2.3 Insert the liner with the newly silanized glass wool plug into the injection port. Verify that the column extends up into the injection port and is perpendicular. Inspect the graphite seal and replace it if the edges are knife-shaped.

10.1.2.4 The septum is always replaced daily. Bake out the column at 300°C for 15 minutes after the vacuum in the analyzer has returned to normal.

10.1.2.5 Performance may enhanced by clipping a small portion of the column at the injection port end. Document this activity in the maintenance record.

10.1.3. Prior to calibration or sample analysis always verify that the analyzer is under sufficient vacuum and that the column has proper carrier gas flow.

10.1.4. Establish the following GC/MS operating conditions:

10.1.4.1 Full Scan Operating Mode

Full Scan Mode – Standard Injection Volume
Mass Range: 35 to 500amu
Scan Time: 1 sec/scan
Transfer Line Temperature: 300°C
Source Temperature: Preset by H.P. at 280°C
Scan start time: 1.0 minutes
Initial Column Temperature and Hold Time: 45°C for 0.5 minutes
Column Temperature Program: 20°C /min to 100°C 25°C/min to 270°C 10° C/min to 310°C
Final Column Temperature Hold: 310°C for 5 minutes
Carrier Gas: Ultra High Purity Grade Helium at 1.3ml/min
Injector Temperature: 275°C
Injector: Grob-type, pulse, splitless
Injection Volume: 1ul
Splitless Valve Time: 0.3 minutes

Full Scan Mode – Large Volume Injection (LVI)
Mass Range: 35 to 500amu
Scan Time: 1 sec/scan
Transfer Line Temperature: 300°C
Source Temperature: Preset by H.P. at 280°C
Scan start time: 1.0 minutes
Initial Column Temperature and Hold Time: 45°C for 0.5 minutes
Column Temperature Program: 20°C /min to 100°C 25°C/min to 270°C 10° C/min to 310°C
Final Column Temperature Hold: 310°C for 5 minutes
Carrier Gas: Ultra High Purity Grade Helium at 1.3ml/min
Injector Temperature: 275°C
Injector: Grob-type, pulse, splitless
Injection Volume: 5ul
Splitless Valve Time: 0.3 minutes

10.1.4.2 SIM Operating Mode

SIM Mode
Mass Range: 35 to 500amu
Scan Time: 1 sec/scan
Transfer Line Temperature: 300°C
Source Temperature: Preset by H.P. at 280°C
Scan start time: 1.5 minutes
Initial Column Temperature and Hold Time: 40°C for 0.5 minutes
Column Temperature Program: 20°C /min to 100°C 25°C/min to 270°C 10° C/min to 310°C
Final Column Temperature Hold: 310°C for 3 minutes
Carrier Gas: Ultra High Purity Grade Helium at 1.3ml/min
Injector Temperature: 275°C
Injector: Grob-type, pulse splitless
Injection Volume: 1ul
Splitless Valve Time: 0.3 minutes

10.1.4.3 Isotope Dilution Selected Ion Monitoring Mode :

SIM Parameters

Group 1
Plot 1 Ion: 74.0

Ions/Dwell in Group	(Mass	Dwell)	(Mass	Dwell)	(Mass	Dwell)
	42.0	50	43. 0	50	68.0	50
	74.0	50	128.0	50	129.0	50
	136.0	50	150.0	50	152.0	50
	93.0	50	66.0	50		
	58.0	50				
	88.0	50				

Group 2
Group Start Time: 6.00
Plot 1 Ion: 152.0

Ions/Dwell in Group	(Mass	Dwell)	(Mass	Dwell)	(Mass	Dwell)
	151.0	50	152.0	50	153.0	50
	154.0	50	162.0	50	164.0	50
	165.0	50	166.0	50		

Group 3
Group Start Time: 7.80
Plot 1 Ion: 188.0

Ions/Dwell in Group	(Mass	Dwell)	(Mass	Dwell)	(Mass	Dwell)
	94.0	50	101.0	50	142.0	50
	178.0	50	179.0	50	188.0	50
	202.0	50	264.0	50	266.0	50
	284.0	50				

Group 4
Group Start Time: 10.50
Plot 1 Ion: 228

Ions/Dwell in Group	(Mass	Dwell)	(Mass	Dwell)	(Mass	Dwell)
	120.0	50	228.0	50	229.0	50
	240.0	50				

Group 5
Group Start Time: 12.00
Plot 1 Ion: 252.0

Ions/Dwell in Group	(Mass	Dwell)	(Mass	Dwell)	(Mass	Dwell)
	138.0	50	139.0	50	252.0	50
	253.0	50	260.0	50	264.0	50
	267.0	50	276.0	50	278.0	50

Table 29: Target Compound - Primary and Monitoring Ions

Compound	1	2	3
1,4-Dioxane-d8	96	64	62
1,4-Dioxane	88	58	57
1,4-Dichlorobenzene-d4	152	150	

10.1.5. The above listed instrument conditions are used for all analytical standards for calibration and for all sample extracts analyzed by this method.

10.1.5.1 The column conditions, scan start time, and splitless valve time for analysis of DFTPP only are as follows are as follows:

Initial Column Temperature and Hold Time: 140°C for 0.5 minutes
Column Temperature Program: 140° to 320°C at 22°C/minute
Final Column Temperature Hold: 320C for 0.5 minutes
Scan Start Time: approx. 5 minutes
Splitless Valve Time: 0.3 minutes
Injection Volume: 2 ul

10.2. Analytical Sequence

10.2.1. Screening: All samples extracts must be screened by GC/FID using the identical chromatographic conditions described in section 9.2. Screening is used to determine the dilution factor of the sample (if any) prior to GC/MS analysis (for additional details see TestAmerica Edison SOP No. ED-GCS-001, *Preparation and Screening of Semivolatile Organic Extracts for GC/MS Analysis*, current revision).

10.2.1.1. Aqueous samples: Prior to extract screening, the extract is diluted to 2ml and split into two 1-ml aliquots:

- One 1-ml aliquot is internal standardized with 20ul of the 2000 ng/ul internal standard solution for full scan analysis and is analyzed by GC/FID for screening.
- The other aliquot is archived for SIM analysis which is internal standardized with 20ul of 50ppm SIM Internal Standard

10.2.1.2. Soil samples: Final volume is 1ml and extracts are internal standardized with 20ul of the 2000 ng/ul internal standard solution and analyzed by GC/FID for screening.

10.2.1.3. After screening analysis, the chromatogram is evaluated for high concentrations of organics. Determine dilutions by comparing the peak heights of compounds in the sample with the internal standard. The ratio of naturally present compounds to internal standards must be <5:1.

10.2.1.4. Dilutions are made based on the screening analysis and prior to GC/MS analysis. Dilutions are made in 1-ml vials using microsyringes. Calculate the dilution factor using the equation below:

$$DF = Ph / 5 \times Is$$

Where:

DF = Dilution Factor
Ph = Sample Peak Height
Is = Internal Standard Peak Height

When DF >1 but <2, combine 500ul of sample extract with 500ul methylene chloride in a 1 ml amber vial, add20 ul internal standard and crimp seal

Use **Table 30** to determine dilution and internal standard amount.

Table 30
Dilution Factor Calculations

DF Value	Volume of Sample (ul)	Volume of Methylene Chloride (ul)	Volume of ISTD (ul)
<1	1,000	None	None
>1, <2	500	500	10
>4, <5	200	800	16
>10, <20	100	900	36
>20	500*	500	10

*Prepare this dilution by serially diluting the >10, <20 dilution

10.2.2. Instrument Performance and Calibration Sequence

10.2.2.1. Once the GC/MS instrument has been setup and maintained as detailed in Section 10.1, the first operations to be performed are the performance checks and calibration standards.

10.2.2.2. Analyze the Instrument Performance Check Standard (DFTPP) as discussed in Section 9.2.1.

10.2.2.3. Initially and as required, analyze the Initial Calibration Range (minimum 5 points, six points for second order regression) as detailed in Sections 7.2.1 and 9.2.4.2. Evaluate the acceptability of the Initial Calibration Range as detailed in Section 9.2.4.2.

10.2.2.4. Immediately after the Initial Calibration Range only, analyze the Initial Calibration Verification (ICV) as detailed in Sections 7.2. and 9.2.4.3. Evaluate the acceptability of the ICV as detailed in Section 9.2.4.3.

10.2.2.5. Every 12 hours, reanalyze and evaluate the Instrument Performance Check Standard (DFTPP) followed by the Continuing Calibration Verification (CCV) and Low Level Continuing Calibration Verification (LLCCV) as detailed in Section 9.2.3, 9.2.4.4 and 9.2.4.5. Evaluate the acceptability of the CCV and LLCCV as detailed in Section 9.2.4.4

10.2.2.6. Client samples and QC samples are analyzed (as detailed in Section 10.2.3) after acceptable Instrument Performance and Calibration Checks and until the 12 hour clock expires. Repeat the sequence as required. The automation of GC/MS runs is accomplished via the “SEQUENCE” macro of the ChemStation.

10.2.3. Sample Analysis Sequence

10.2.3.1. Sample extracts are normally prepared on the same day as analysis. The GC/MS operator will prepare the extracts that will be run on his or her instrument. Volume adjustments to the extracts will be made at the discretion of the supervisor.

10.2.3.2. Prior to the start of sample analysis the GC/MS operator will generate a sequence program containing the list of the sample extracts to be analyzed, the position on the autosampler tray, and the proper acquisition and tune methods that are to be used. This sequence program contains all the necessary information on the samples to be analyzed and how the GC/MS system is to analyze them. The sample extracts are loaded onto the autosampler (ALS) tray. Their position is verified by checking them against the ALS number on the sequence. This batch analysis will be performed automatically over the 12-hour period.

10.2.3.3. The analytical run log is printed as a record of samples analyzed. The analyst will annotate the run log with any required information regarding anomalies or unusual events. The run log must be signed by the analyst and a reviewed and signed by a trained peer or manager

10.3. Data Processing

10.3.1. Prior to processing any standards or samples, target compound lists and sublists must be assembled. Chrom's auto-processing system queries TALS (LIMS) for each sample's processing parameters (including target compounds lists) and downloads the required processing methods from LIMS to analyze data. These lists are required for processing of all data files including calibration files. The data includes compound names, retention time data, quantitation ions, qualitative identification ions, and the assigned internal standard for qualitative and quantitative identification.

10.3.2. Key data is manually entered the first time a compound list is used for data processing. Processing data using a compound list automatically generates response factor data and updates retention information.

10.3.3. The characteristic ions for target compounds, surrogate compounds, and internal standards which can be determined using SW8270D are listed in Table 31.

10.4. Interpretation and Qualitative Identification: Qualitative identification of target compounds is based on retention time and mass spectral comparison with characteristic ions in the target compound list. The reference mass spectrum is taken from a standard of the target compound analyzed by this method. The characteristic ions are the three ions of greatest relative intensity or any ions over 30% relative intensity if less than three such ions occur in the reference spectrum. Compounds are identified as present when the following criteria are met:

10.4.1 Target Analytes: Qualitative identification of target compounds is based on retention time and mass spectral comparison with characteristic ions in the target compound list. The reference mass spectrum is taken from a standard of the target compound analyzed by this method. The characteristic ions are the three ions of greatest relative intensity or any ions over 30% relative intensity if less than three such ions occur in the reference spectrum. Compounds are identified as present when the following criteria are met:

10.4.1.1. Once the GC/MS instrument has been setup and maintained as detailed in Section 10.1, the first operations to be performed are the performance checks and calibration standards.

10.4.1.2. The intensities of the characteristic ions of a compound maximize in the same scan or within one scan of each other.

10.4.1.3. The relative retention time (RRT) of the sample component is within ± 0.06 RRT units of the RRT of the standard component.

10.4.1.4. The most abundant ion in the standard target spectrum that equals 100% MUST also be present in the sample target spectrum.

10.4.1.5. All other ions that are greater than 10% in the standard target spectra should also be present in the sample.

10.4.1.6. The relative intensities of the characteristic ions agree within 30% of the relative intensities of these ions in the reference spectrum. (Example: For an ion with an abundance of 50% in the reference spectrum, the corresponding abundance in a sample spectrum can range between 20% and 80%).

10.4.1.7. If the compound does not meet all of the criteria listed above, but is deemed a match in the technical judgment of the mass spectral interpretation specialist, the compound will be positively identified and reported with documentation of the identification noted in the raw data record.

10.4.2 Non-Target Analytes: Upon client request a library search to identify non-target Tentatively Identified Compounds (TIC) is performed. The NIST/EPA/NIH mass spectral library is used to identify non-target compounds (not including internal standard and surrogate compounds) of greatest apparent concentration by a forward search of the library. The following guidelines are used by the analyst when making TIC identifications:

10.4.2.1. Relative intensities of major ions in the reference spectrum (ions greater than 10% of the most abundant ion) should be present in the sample spectrum.

10.4.2.2. The relative intensities of the major ions should agree within ±20%. (Example: For an ion with an abundance of 50% in the standard spectrum, the corresponding sample ion abundance must be between 30 and 70%).

10.4.2.3. Molecular ions present in the reference spectrum should be present in the sample spectrum.

10.4.2.4. Ions present in the sample spectrum but not in the reference spectrum should be reviewed for possible background contamination or presence of co-eluting compounds.

10.4.2.5. Ions present in the reference spectrum but not in the sample spectrum should be reviewed for possible subtraction from the sample spectrum because of background contamination or co-eluting peaks. Data system library reduction programs can sometimes create these discrepancies.

10.4.2.6. If, in the technical judgement of the mass spectral interpretation specialist, no tentative identification can be

made, the compound will be reported as 'Unknown'. If the compound can be further classified the analyst may do so (i.e, 'Unknown hydrocarbon', 'Unknown acid' , etc.).

10.5. Data Reporting

10.5.1. Final Report. The Chom data system automatically produces a data report consisting of hardcopy reports corresponding to specific data reporting requirements, which is uploaded to the TALS LIMS System for the report production group.

10.5.1.1. Total Ion Chromatogram. Full length chromatogram depicting the full length of the GC/MS acquisition.

10.5.1.2. Spectra of all detected target compounds. A page for each detected target compound spectra with a standard reference spectrum for comparison.

10.5.1.3. The calculations of the concentrations of each target compound in the sample, reported in units of ppb, ug/kg or ug/l.

10.5.1.4. Data summaries for each method blank indicating which samples were extracted with the indicated blank.

10.5.1.5. A copy of the initial calibration range together with the calibration verification report, and tune report.

10.5.1.6. Quality Control (QC) data report for each batch including surrogate recoveries, internal standard area summaries, LCS, MS/MSD and RPD summaries.

10.6. The low-level calibration standard establishes the reporting limit. All reported data must be at a concentration at or above the low concentration standard. Any quantitative values below the report limit must be qualified as estimated.

.

11.0. Calculations/Data Reduction

11.1. **Target Compounds:** are quantitated using the internal standard method (see the formula in Section 11.3).

11.1.1. Identified target compounds are quantitated using the integrated abundance from the EICP of the primary characteristic ion. The internal standard used shall be the one nearest the retention time of the analyte).

11.1.2. The average response factor (RRF) from the initial calibration is used to calculate the target analyte concentration in client samples using the formula found in Section 11.3. See Section 9.2.4 for discussion of RRF.

11.1.3. Secondary ion quantitation is utilized only when there are sample interferences preventing use of the primary characteristic ion. If secondary ion quantitation is used an average relative response factor (RRF) must be calculated using that secondary ion.

11.2. Non-Target Compounds (Tentatively Identified Compounds): An estimated concentration for non-target (tentatively identified compounds) is calculated using the internal standard method (see formula in Section 11.3). For quantiation, the nearest eluting internal standard free of interferences is used. The procedure used for calculating the concentration of non-target compounds is the same as that used for target compounds (see Section 11.1) with the following revisions:

11.2.1. The total area count of the non-target compound is used for As (instead of the area of a characteristic ion).

11.2.2. The total area count of the chosen internal standard is used as Ais (instead of the area of a characteristic ion).

11.2.3. A RF on 1.0 is assumed.

11.2.4. The resulting concentration is qualified as estimated ('J') indicating the quantitative uncertainties of the reported concentration.

11.3. Internal Standard Calculation:

11.3.1. Aqueous Samples

$$\text{Concentration } (\mu g/L) = \frac{(As)(Cis)(D)}{(Ais)(RF)(Vs)\ (Vi)\ (1000)}$$

Where:

As	=	Area of the characteristic ion for the target analyte in the sample
Cis	=	Concentration of the internal standard (ug/L)
D	=	Dilution factor, if the sample or extract was diluted prior to analysis. If no dilution is performed, D = 1.
Vi	=	Volume of the extract injected (ul)
Ais	=	Area of the characteristic for the associated internal standard
RF	=	Average response factor from the initial calibration.
Vs	=	Volume of sample extracted (ml)

The 1000 in the denominator represents the number of ul in 1 ml.

11.3.2. Solid Samples

$$\text{Concentration (µg/KG)} = \frac{(As)(Cis)(D)(Vt)}{(Ais)(RF)(Ws)\ (Vi)\ (1000)}$$

Where:

As	=	Area of the characteristic ion for the target analyte in the sample
Cis	=	Concentration of the internal standard (ug/L)
D	=	Dilution factor, if the sample or extract was diluted prior to analysis. If no dilution is performed, D = 1.
Vi	=	Volume of the extract injected (ul)
Ais	=	Area of the characteristic for the associated internal standard
RF	=	Average response factor from the initial calibration.
Vt	=	Volume of concentrated extract (ul)
Ws	=	Weight of sample (g)

The 1000 in the denominator represents the number of ul in 1 ml.

11.4. Relative Response Factors

$$RRF = \frac{A_x}{A_{is}} \times \frac{C_{is}}{C_x}$$

Where:

A_x = Area characteristic ion for the compound (see Table 31)
Ais = Area characteristic ion of associated internal std (See Table 31)
Cis = Concentration of internal standard
Cx = Concentration of compound in standard

11.5. Percent Relative Standard Deviation (% RSD) : as discussed in Section 9.2.4.4 (Initial calibration):

$$\%\ RSD = \frac{\text{Standard Deviation of RRFs}}{\text{Mean RRF}}$$

11.6. Percent Difference (% D):as discussed in Section 9.2.4.4 (Continuing calibration):

$$\%\ D = \frac{RRF_c - \overline{RRF_i}}{\overline{RRF_i}} \times 100$$

Where: RRFc = RRF from continuing calibration

$\overline{RRF_i}$ = Mean RRF from current initial calibration

11.7. Percent Recovery (% R): Surrogates and Spikes

$$\text{Recovery (\%)} = \frac{\text{Concentration (or amount) found}}{\text{Concentration (or amount) added}} \times 100$$

11.8. Dry Weight Correction: All solid samples must be corrected for dry weight using the following formula for dry weight determination.

$$DW = \frac{Gd}{Gw} \times 100$$

Where:

DW	=	Percent % Dry Weight
Gd	=	Dry weight of selected sample aliquot
Gw	=	Wet weight of selected sample aliquot

Multiply the DW value times the wet weight of the sample extracted. NOTE: This calculation can also be performed automatically by the target system provided the DW value is available and entered into the system.

12.0. Method Performance

12.1. Method Detection Limit Study (MDL)

The method detection limit (MDL) is the lowest concentration that can be detected for a given analytical method and sample matrix with 99% confidence that the analyte is present. The MDL is determined according to the laboratory's MDL procedure in Section 19 (Test Methods and Method Validation) of TestAmerica Edison's Quality Assurance Manual (ED-QA-LQM). MDLs reflect a calculated (statistical) value determined under ideal laboratory conditions in a clean matrix, and may not be achievable in all environmental matrices. The laboratory maintains MDL studies for analyses performed; these are verified at least annually unless method requirements require a greater frequency.

12.2. Demonstration of Capabilities

For DOC procedure refer to Section 19 in the most current revision of TestAmerica Edison's Quality Assurance Manual (ED-QA-LQM).

12.3. Training Requirements

Refer to TestAmerica SOP No. ED-GEN-022, (*Training*), for the laboratory's training program.

13.0. Pollution Control

13.1 It is TestAmerica's policy to evaluate each method and look for opportunities to minimize waste generated (i.e., examine recycling options, ordering chemicals based on quantity needed, preparation of reagents based on anticipated usage and reagent stability). Employees must abide by the policies in Section 13 of the Corporate Environmental Health and Safety Manual (CW-E-M-001) for "Waste Management and Pollution Prevention."

14.0. Waste Management

14.1 Waste management practices are conducted consistent with all applicable rules and regulations. Excess reagents, samples and method process wastes are disposed of in an accepted manner. Waste description rules and land disposal restrictions are followed. Waste disposal procedures are incorporated by reference to TestAmerica Edison SOPs Nos. ED-SPM-007 (*Disposal of Samples and Associated Laboratory Waste, current revision)* and ED-SPM-008 (*Laboratory Waste Disposal Procedures, current revision).* The following waste streams are produced when this method is carried out:

- Auto sampler vials and expired standards: These vials are collected in satellite accumulation within the instrument laboratory. The vials are then placed into a 55 steel open top drum in the waste room. When the drums are full, the drum will be collected by the waste vendor for disposal. This waste is treated for incineration.

  Teris Profile Number: 50016652
  Onyx Profile WIP Number: 282493

- Mixed Solvent Waste: Mixed solvent waste is collected in a small beaker inside the bench top hood. This waste is then transferred into the satellite accumulation container in the Organic Prep. Lab. on a daily basis. This material is transferred into 5 gallon solvent cans as satellite accumulation. These cans are emptied every 24 hours into a steel drum in the waste room. This drum is kept in the walk in hood until it is full. The full drum is then removed from the hood and placed on secondary containment in the waste room.

  Teris Profile Number: 50016624
  Onyx Profile WIP Number: 545240

14.1. Pollution Prevention

14.2.1. Pollution prevention encompasses any technique that reduces or eliminates the quantity or toxicity of waste at the point of generation. Numerous opportunities for pollution prevention exist in laboratory operation. The USEPA has established a prevention hierarchy of environmental management techniques that places

pollution prevention as the management option of first choice. Whenever feasible, laboratory personnel should use pollution prevention techniques to address their waste generation. When wastes cannot be feasibly reduced at the source, the agency recommends recycling as the next best option.

14.2.2. The quantity of chemical purchased should be based on expected usage during its shelf life and disposal cost of unused material. Actual reagent preparation volumes should reflect anticipated usage and reagent stability.

15.0. References / Cross-References

15.1. United States Environmental Protection Agency, "Method SW8270D, Semivolatile Organic Compounds by Gas Chromatography/Mass Spectrometry", Test Methods for Evaluating Solid Wastes, SW846 Third Edition, Volume 1B: Laboratory Manual, Physical/Chemical Methods, Revision 4, February 2007.

15.2. United States Environmental Protection Agency, "Method SW8000C: Determinative Chromatographic Separations", Test Methods for Evaluating Solid Wastes, SW846, Laboratory Manual, Physical/Chemical Methods, Revision 3, March 2003.

15.3. TestAmerica Edison Document No. ED-QA-LQM, *Laboratory Quality Manual*, current revision.

15.4. TestAmerica Edison SOP No. ED-ORP-002, *SW846 Method 3510C-Extraction of Semi-Volatile Organic Compounds in Water by Separatory Funnel,* current revision.

15.5. TestAmerica Edison SOP No. ED-ORP-043, *SW846 Method 3580A - Waste Dilution Prep for Analysis of BNAs by SW846 Method 8270,* current revision.

15.6. TestAmerica Edison SOP No. ED-ORP-044, *Procedure for the Microwave Extraction of Solids, SW3546, current revision.*

15.7. TestAmerica Edison SOP No. ED-ORP-006, SW846 *Method 3550B- Extraction of Semi-Volatile Organic Compounds in Soil Using Medium--level Extraction Technique,* current revision.

15.8. TestAmerica Document No. CW-E-M-001, Corporate Environmental Health and *Safety Manual,* current revision.

15.9. TestAmerica Corporate Quality SOP No. CA-Q-S-001, *Solvent & Acid Lot Testing & Approval*, current revision.

15.10. TestAmerica Edison SOP No. ED-GEN-023 (*Bulk Solvent Testing and Approval*), current revision.

15.11. TestAmerica Edison SOP No. ED-GCS-001, *Preparation and Screening of Semivolatile Organic Extracts for GC/MS Analysis*, current revision.

15.12. TestAmerica Edison Work Instruction Document No. EDS-WI-012, *Client Complaint/Corrective Action Form,* current revision.

15.13. TestAmerica Edison SOP No. ED-GEN-003, *Standard Operating Procedure for Control of Non-Conformances and Corrective Action,* current revision.

15.14. TestAmerica Edison SOP No. ED-ORP-001, *Extraction of Semivolatile Organic Compounds in Water, EPA Method 625,* current revision.

15.15. TestAmerica Edison SOP No. ED-GEN-022, *Training,* current revision.

15.16. TestAmerica Corporate Quality Memorandum, CA-Q-QM-002, *GC/MS Tuning Policy*, current revision.

16.0. Method Modifications:

N/A

17.0. Attachments

Attachment 1 Poor Performing Analytes

18.0. Revision History

- Revision 7, date 06/08/2018
    - Section 2.3: revised to clarify that RVE/LVI is lab standard procedure.
    - Section 9.1.3: removed statement regarding allowance for up to five analytes to recover outside of lab acceptance limits in LCS/LCSD.
    - Section 9.2.4.3: Replace table 'ICV Poor Performers (50-150% Recovery) with expanded list of 'Poor Performing Analytes' in Attachment 1.
    - Added Section 9.2.4.4.5: CCV Poor Performers
    - Corrected number in section 9.2.4.5
    - Added Attachment 1 – Poor Performing Analytes
- Revision 6, date 01/12/2018:
    - Section 7.2.5 included to specify reagent and standard storage conditions.
    - Revised Section 9.1.3 to clarify requirements for specific LCS/LCSD evaluation criteria regarding the # of out of criteria analytes.
    - Revised Section 9.2.4.3 to add 2,4-Dimethylphenol as a poor performing analyte, increased the range for the poor performers to 50-150 and also expanded the guidelines for flagging the ICV outliers.
- Revision 5, dated 09/29/2017:
    - Revised Section 9.1.1 to clarify requirements for surrogate recovery in method blanks.

- Revision 4, dated 08/21/2017:
    - Updated throughout to add a procedure for the analysis of 1,4-dioxane by isotope dilution selected ion monitoring (SIM)
    - Added tables for isotope dilution SIM standards. Renumbered all tables as necessary.
    - Section 7.2.1: added a list of full scan calibration list options.
    - Table 3: Renamed 'Full Scan Stock Standards'.
    - Section 9.2.1: noted that DFTTP applies only to full scan analysis.
    - Section 9.2.3: updated CCV concentrations
    - Added reference to GC/MS Tuning Policy in Section 15.16.

- Revision 3, dated 01/07/2016:
    - Tables 1 and 2: added SIM as option for 1,4-Dioxane.
    - Section 2.3: removed SW3541 (Soxtherm) as option for soils prep (lab has discontinued use of this method). Also removed SW3541 SOP reference from Section 15.0.
    - Tables 19 and 20: added source and prep instructions for 1,4-Dioxane SIM standard. Updated source and prep instructions for 4,6-Dinitro-2-methylphenol.
    - Table 22: added prep instructions for 1,4-Dioxane and 4,6-Dinitro-2-methylphenol SIM ICV standard.
    - Corrected the information in the 'DFTPP Key Ions and Abundance Criteria' table in Section 9.2.1 to match the info found in SW846 8270C.
    - Section 10.1.4.2: updated "SIM Parameters" to included ion masses/dwell times for 1,4-Dioxane.

- Revision 2, dated 01/28/2015:
    - Extensively reformatted the SOP. Placed tables that had been in rear of document into the body of the text. Renumbered tables as applicable and fixed text references to tables.
    - Section 1.1, Table 1: Revised table to include all current analytes. Also footnoted those compounds which are currently analyzed by SIM.
    - Section 2.3: added options for extraction of solids by SW846 3456 (Microwave Extraction) and by SW3580A (Waste Dilution) and added SOP references. Deleted reference to SOP ED-ORP-005 (SW3550B – Low Level); Updated Section 15 (References).
    - Section 2.5: added text detailing the RVE/LVI options.
    - Section 2.6: added table which includes all analytes routinely analyzed by SIM.
    - Section 6: updated to include newer GC, MS and autosampler models currently in use.
    - Section 6.1.3: added Zebron ZB column as an option.
    - Section 7.2: extensively revised standards information to reflect switch to Restek standards.
    - Table 3:Added Custom Aromatic Amine Surrogate Standard and revised Table 8 to include initial calibration prep instructions for the Aromatic Amine surrogates.
    - Throughout document: removed references to Target and replaced with Chrom.
    - Section 7.2.1: Added reference to section 10.2.1.2 for LVI.
    - Added Section 7.2.1.3.1 and Table 17A both of which discuss use of Aromatic Amine surrogates.
    - Section 7.2.1.2: Added reference to Tables 9,10 and 11 (ICV Preparation)
    - Section 8.0: Added Sample container and minimum sample size (250 ml) for

Reduced volume extraction.
- Sections 9.1.2, 9.1.3, 9.1.4 and 9.2.4: added statement that certain state regulatory programs have defined recovery limits which, where applicable, are used for spike and calibration evaluations.
- Section 9.1.2: Deleted sentence "A minimum of 16 spiked analytes are reported to in client reports (the full list is reported at least once during each 2 year period because we employ full spiking list.
- Section 9.1.4: Added note regarding use of Aromatic Amine Surrogates.
- Section 9.2.2.2: Added reference to ICV Preparation tables in Section 7.2.
- Section 9.2.3: added more specific info as to the concentration of the CCVs for all techniques.
- Section 9.2.4.2.1: Changed to reflect that each analyte should meet minimum RF's, not the average across the calibration. Added LLCCV requirement.
- Section 10.3.1: added explanation of Chrom's interaction with TALS. Removed references to Target.
- Section 9.2.4.2.5.5: Added: (or can be noted in the narrative)
- Section 9.2.4.2.5.6: Revised last sentence to read: "This evaluation can be checked using the Initial Calibration %Drift Report in Chrom."
- Section 9.2.4.3: Removed 65-135% criteria and added "poor performing" analyte list and associated criteria of 60-140%.
- Section 9.2.4.4.3: Added LLCCV criterion for RFs
- Section 9.2.4.4.4: Added LLCCV criterion for %D
- Section 10.1.4: Updated GC/MS operating conditions for full scan, SIM and DFTPP.
- Section 10.1.4.1: added a table detailing operating conditions for LVI option.
- Table 2: Added 2-ethylaniline, 2,4-dimethylaniline, 3,4-dimethylaniline, 2,3-dimethylaniline, 2,4,5-trimethylaniline and 4-chloro-o-toluidine to Working Standards preparation information.
- Table 25: updated to include all current analytis/surrogates/internal standards and associated ions.
- Throughout document: updated LQM section references as appropriate as some have changed with the latest LQM revision.

- Revision 1, dated 11/07/2011

  - Section 1.1, Table 1: Added Pentachloronitrobenzene and associated CAS# to the analyte list.
  - Section 7.2.1: Added Pentachloronitrobenzene standard information.
  - Table 2: Added Pentachloronitrobenzene to Working Standards preparation information.
  - Table 4: Added Pentachloronitrobenzene and associated minimum RF.
  - Table 8: Added Pentachloronitrobenzene and associated ions.

- Revision 0, dated 02/22/2011: NEW

Table 31 Characteristic Ions Of Semi-Volatile Organic Compounds		
Compound	**Primary Ion**	**Secondary Ion(s)**
1,1'-Biphenyl	154	153,76
1,2,4,5-Tetrachlorobenzene	216	214, 179
1,2,4-Trichlorobenzene	180	182, 145
1,2-Dichlorobenzene	146	148, 111
1,2-Diphenylhydrazine	77	105, 182
1,3-Dichlorobenzene	146	148, 111
1,3-Dimethylnaphthalene	156	141, 115
1,4-Dichlorobenzene	146	148, 111
1,4-Dichlorobenzene d4 **(ISTD)**	152	150, 115
1,4-Dioxane	88	58, 43
1-Methylnaphthalene	142	141, 115
1-Naphthylamine	143	115, 116
2,2'-oxybis[1-chloropropane]	45	77, 121
2,3,4,6-Tetrachlorophenol	232	131, 230
2,3,7,8-TCDD (screen)	320	322, 324
2,3-Dihydroindene		
2,3-Dimethylaniline	106	129
2,4,5-Trichlorophenol	196	198, 200
2,4,5-Trimethylaniline	102	55, 56
2,4,6-Tribromophenol **(Surrogate)**	330	132, 141
2,4,6-Trichlorophenol	196	198, 200
2,4-Dichlorophenol	162	164, 98
2,4-Xylidine	121	120, 106
2,4-Dimethylphenol	122	107, 121
2,4-Dinitrophenol	184	63, 154
2,4-Dinitrotoluene	165	63, 89
2,6-Dinitrotoluene	165	63, 89
2-Chloronaphthalene	162	127, 164
2-Chlorophenol	128	64, 130
2-Ethylaniline	106	122,104
2-Fluorobiphenyl **(Surrogate)**	172	171
2-Fluorophenol **(Surrogate)**	112	64
2-Methylnaphthalene	142	141
2-Methylphenol	108	107
2-Naphthylamine	143	115, 116
2-Nitroaniline	65	108, 138
2-Nitrophenol	139	109, 65
2-tert-butyl-4-Methylphenol	149	121, 91
2-Toluidine	107	106, 77
3,3'-Dichlorobenzidine	252	254, 126
3,4-Dimethylaniline	106	129, 127
3,5-Di-tert-butyl-4-Hydroxytol	205	220, 145
3-Nitroaniline	138	108, 65
4,6-Dinitro-2-methylphenol	198	51, 105
4-Bromophenyl phenyl ether	248	250, 141
4-chloro-2-methylaniline	106	144, 142

Table 31 Characteristic Ions Of Semi-Volatile Organic Compounds		
Compound	**Primary Ion**	**Secondary Ion(s)**
4-Chloro-3-methylphenol	107	144, 142
4-Chloroaniline	127	129
4-Chloroaniline-d4 **(Surrogate)**	131	133
4-Chlorophenyl phenyl ether	204	206, 141
4-Methylphenol	108	107
4-Nitroaniline	138	108, 65
4-Nitrophenol	139	109, 65
Acenaphthene	154	153, 152
Acenaphthene d10 **(ISTD)**	164	162, 160
Acenaphthylene	152	151, 153
Acetophenone	105	77, 51
Aniline	93	66
Aniline-d5 **(Surrogate**)	98	71,42
Anthracene	178	176, 179
Atrazine	200	173,215
Benzaldehyde	77	105,106
Benzidine	184	92, 185
Benzo(a)anthracene	228	229, 226
Benzo(a)pyrene	252	253, 125
Benzo(b)fluoranthene	252	253, 125
Benzo(g,h,i)perylene	276	138, 277
Benzo(k)fluoranthene	252	253, 125
Benzoic Acid	122	105, 77
Benzyl Alcohol	108	79, 77
Bis(2-chloroethoxy)methane	93	95, 123
Bis(2-chloroethyl)ether	93	63, 95
Bis(2-ethylhexyl)phthalate	149	167, 279
Bisphenol-A	213	228, 119
Butyl benzyl phthalate	149	91, 206
Caprolactam	113	55,56
Carbamazepine	193	236, 135
Carbazole	167	166, 139
Chrysene	228	226, 229
Chrysene d12 **(ISTD)**	240	120, 136
Coumarin	146	118, 63
Dibenz(a,h)anthracene	278	139, 279
Dibenzofuran	168	139
Diethylphthalate	149	177, 150
Dimethylphthalate	163	194, 164
Di-n-butylphthalate	149	150, 104
Di-n-octylphthalate	149	167, 43
Fluoranthene	202	101, 203
Fluorene	166	165, 167
Hexachlorobenzene	284	142, 249
Hexachlorobutadiene	225	223, 227
Hexachlorocyclopentadiene	237	235, 272

Table 31 Characteristic Ions Of Semi-Volatile Organic Compounds		
Compound	**Primary Ion**	**Secondary Ion(s)**
Hexachloroethane	117	201, 199
Indeno(1,2,3-cd)pyrene	276	138, 227
Isophorone	82	95,138
Kepone	272	237, 355
N,N-Dimethylaniline	120	122, 104
Naphthalene	128	129, 127
Naphthalene d8 **(ISTD)**	136	68
n-decane	43	57
Nitrobenzene	77	123, 65
Nitrobenzene-d5 **(Surrogate)**	82	128, 54
N-Nitrosodimethylamine	42	74, 44
N-Nitroso-di-n-propylamine	170	42,101,130
N-Nitrosodiphenylamine	169	168, 167
n-Octadecane	57	43, 85
o-Toluidine-d9 **(Surrogate)**	114	112, 42
Pentachloronitrobenzene	237	214,295
Pentachlorophenol	266	264, 268
Perylene d12 **(ISTD)**	264	260, 265
Phenanthrene	178	179, 176
Phenanthrene d10 **(ISTD)**	188	94, 80
Phenol	94	65, 66
Phenol-d5 **(Surrogate)**	99	42, 71
Phenyl ether	170	77, 115
Pyrene	202	200, 203
Pyridine	79	52, 51
Terphenyl-d14 **(Surrogate)**	244	122, 212

Attachment 1
Poor Performing Compounds

1,2,4,5-Tetrachlorobenzene
1,4-Dioxane
1-Naphthylamine
2,3,4,6-Tetrachlorophenol
2,4-Dimethylphenol
2,4-Dinitrophenol
2-Chloroaniline
2-Naphthylamine
3&4-Methylphenol
3'3-Dichlorobenzidine
4,6-Dinitro-2-methyl- phenol
4-Chloroaniline
4-Nitrophenol
Aniline
Atrazine
Benzaldehyde
Benzidine
Benzoic Acid
Benzyl Alcohol
Biphenyl
Caprolactam
Diphenylamine
Hexachlorocyclopentadiene
Hexachloroethane
n-Decane
n-Nitrosodimethylamine
o,o,o-Triethylphosphorothioate
o-Toluidine
Pentachloronitrobenzene
Pentachlorophenol
Phenol
Pyridine

These analytes are exempt from the ICV and CCV criteria as detailed in this SOP

Title: Purge and Trap for Aqueous Samples, SW846 Method 5030B and 5030C

Once printed, this is considered an uncontrolled document

Approvals (Signature/Date):

Sylvanus Klusey VOA Department Manager	06/26/13 Date	Kene' Kasperek Health & Safety Manager / Coordinator	06/26/13 Date
Carl Armbruster Quality Assurance Manager	06/26/13 Date	Ann Gladwell Laboratory Director	06/26/13 Date

Copyright Information:

This documentation has been prepared by TestAmerica Laboratories, Inc. and its affiliates ("TestAmerica"), solely for their own use and the use of their customers in evaluating their qualifications and capabilities in connection with a particular project. The user of this document agrees by its acceptance to return it to TestAmerica upon request and not to reproduce, copy, lend, or otherwise disclose its contents, directly or indirectly, and not to use it for any other purpose other than that for which it was specifically provided. The user also agrees that where consultants or other outside parties are involved in the evaluation process, access to these documents shall not be given to said parties unless those parties also specifically agree to these conditions.

THIS DOCUMENT CONTAINS VALUABLE CONFIDENTIAL AND PROPRIETARY INFORMATION. DISCLOSURE, USE OR REPRODUCTION OF THESE MATERIALS WITHOUT THE WRITTEN AUTHORIZATION OF TESTAMERICA IS STRICTLY PROHIBITED. THIS UNPUBLISHED WORK BY TESTAMERICA IS PROTECTED BY STATE AND FEDERAL LAW OF THE UNITED STATES. IF PUBLICATION OF THIS WORK SHOULD OCCUR THE FOLLOWING NOTICE SHALL APPLY:

©COPYRIGHT 2013 TESTAMERICA LABORATORIES, INC. ALL RIGHTS RESERVED.

1.0 Scope and Application

1.1 Analytes, Matrix(s), and Reporting Limits

1.1.1 This method describes purge and trap techniques for the analysis of volatile organic compounds in aqueous samples by SW846 Method 5030B and 5030C and high concentration soil sample extracts prepared using SW846 Methods 5035 or 5035A.

1.1.2 SW846 Methods 5030B and 5030C can be used for most volatile compounds that have boiling points below 200° C and are insoluble or slightly soluble in water. Water-soluble compounds can be analyzed for by this method; however, quantitation limits will be higher due to poor purging efficiency.

1.1.3 This sample introduction step is to be followed by determinative methods such as SW-846 Method 8015 for GC analysis or SW-846 8260 for GC/MS analysis.

1.1.4 On occasion clients may request modifications to this SOP. These modifications are handled following the procedures outlined in Sections 7 (*Review of Work Request*) and 19 (*Test Methods and Method Validation*) of TestAmerica Edison's Quality Assurance Manual (ED-QA-LQM).

2.0 Summary of Method

2.1 An aliquot of the aqueous sample is purged with helium (or nitrogen, where applicable) as in a closed sparging vessel. For medium or high level samples, an aliquot of the extract prepared by SW846 Methods 5035 or 5035A is combined with organic free reagent water and then purged. The volatile compounds are transferred from the aqueous phase to the vapor phase. The vapor is swept through a sorbent column where the volatiles are trapped. After purging is complete, the sorbent trap is heated and backflushed with helium (or nitrogen, where applicable) to desorb the volatiles onto a gas chromatographic column.

3.0 Definitions

3.1 For a complete list of definitions refer to Appendix 2 in the most current revision of TestAmerica Edison's Quality Assurance Manual (ED-QA-LQM).

4.0 Interferences

4.1 This method and the determinative methods that follow are susceptible to contamination from a number of sources. Potential sources of contamination include organic solvents used in other laboratory procedures, impurities in the purge gas, improper cleaning of syringes or purge vessels, and carryover from high level samples. Samples can be contaminated by the diffusion of volatile organics through the septum

during shipment or storage. Steps have been taken to ensure that these potential problems are eliminated from the laboratory.

4.2 The volatile laboratory is housed in a separate building, away from the organic extraction area where large quantities of organic solvents are used. No organic solvents are used or stored in the volatile laboratory.

4.3 The helium (or nitrogen, where applicable) used as purge gas passes through a solvent trap prior to its inlet into the purge and trap units.

4.4 A trip blank prepared from organic-free reagent water is carried through the sampling, storage, and analysis of each group of samples to check for such contamination.

4.5 Individual samples are each handled with a unique syringe that has been baked in a drying oven at 105°C to ensure the absence of volatile compounds.

4.6 Purge vessels are removed from the autosampler units after each use, rinsed, baked, returned to the units and pre-purged before the next use.

4.7 Carryover can occur anytime a high level sample is analyzed. Screening procedures are employed to ensure that a sample is analyzed at an appropriate dilution to minimize potential carryover. When a high level sample is analyzed, it is followed by the analysis of a reagent water blank. If another sample was analyzed after the high level sample, this sample is inspected carefully for signs of carryover. If this sample does not contain any of the compounds found in the high level sample, the system can be considered contamination free.

4.8 The analytical system is checked daily with the analysis of a method blank. This blank must meet all quality control criteria for the method before sample analysis may take place.

5.0 Safety

Employees must abide by the policies and procedures in the Corporate Environmental Health and Safety Manual (CW-E-M-001) and this document. This procedure may involve hazardous material, operations and equipment. This SOP does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of the method to follow appropriate safety, waste disposal and health practices under the assumption that all samples and reagents are potentially hazardous. Safety glasses, gloves, lab coats and closed-toe, nonabsorbent shoes are a minimum.

Any questions pertaining to safety issues or procedures should be brought to the department manager or Edison Safety Officer.

5.1 Specific Safety Concerns or Requirements

- Latex, nitrile and vinyl gloves all provide adequate protection against the methanol used in this method.

- Purge vessels on purge-and-trap instruments can be pressurized by the time analysis is completed. Vent the pressure prior to removal of these vessels to prevent the contents from spraying out.

5.2 Primary Materials Used

The following is a list of the materials used in this method, which have a serious or significant hazard rating. **Note: This list does not include all materials used in the method. The table contains a summary of the primary hazards listed in the MSDS for each of the materials listed in the table.** A complete list of materials used in the method can be found in the reagents and materials section. Employees must review the information in the MSDS for each material before using it for the first time or when there are major changes to the MSDS.

Material (1)	Hazards	Exposure Limit (2)	Signs and symptoms of exposure
Hydrochloric Acid	Corrosive Poison	5 ppm-Ceiling	Inhalation of vapors can cause coughing, choking, inflammation of the nose, throat, and upper respiratory tract, and in severe cases, pulmonary edema, circulatory failure, and death. Can cause redness, pain, and severe skin burns. Vapors are irritating and may cause damage to the eyes. Contact may cause severe burns and permanent eye damage.
Methanol (MeOH)	Flammable Poison Irritant	200 ppm-TWA	A slight irritant to the mucous membranes. Toxic effects exerted upon nervous system, particularly the optic nerve. Symptoms of overexposure may include headache, drowsiness and dizziness. Methyl alcohol is a defatting agent and may cause skin to become dry and cracked. Skin absorption can occur; symptoms may parallel inhalation exposure. Irritant to the eyes.
1 – Always add acid to water to prevent violent reactions.			
2 – Exposure limit refers to the OSHA regulatory exposure limit.			

6.0 Equipment and Supplies

6.1 Instrumentation

Purge and trap unit that consists of three parts: the sample purger, the trap, and the concentrator.

6.1.1 Purge and trap units from several different manufacturers are used, depending upon the sample matrix and preparatory technique required.

6.1.2 Purge and trap units used include the the OI 4451 autosampler/4560 concentrator, the Archon 5100A automatic sampler/OI 4560 concentrator, OI 4551A autosampler/4660 Concentrator, Centurion

Autosampler/Encon, Archon/Encon, Solatek autosampler/Stratum, Aquatek 70/Tekmar 3000 concentrator and Archon/Evolution.

6.1.3 The purge chambers of each unit are designed to accept a 5mL sample with a water column at least 3cm deep. The headspace above the water has a volume less than 15 mL. The purge gas is introduced no more than 5 mm from the base of the water column. The purge gas passes through the water column as finely divided bubbles, each with a diameter of less than 3mm at the origin.

6.1.4 Different sorbent traps are used, depending on the determinative method that follows the purge and trap procedure. Alternate traps may be used provided the adsorption and desorption characteristics are equivalent to those of the trap recommended by the method.

6.1.5 The VOCARB 3000 (Supelco) is used in the Encon, Tekmar 3000 and Evolution. This trap is 25cm long with an inside diameter of 0.105 inches. The trap is packed with 10.0cm Carbopack B, 6.0 cm Carboxin 1000, and 1cm Carboxin 1001.

6.1.6 The OI Analytical purge trap #10 is used in the OI 4560 and 4660. This trap is 25cm long and has an inside diameter of 0.105 inches. The trap is packed to contain the following adsorbents: Tenax/silica gel/ carbon molecular sieve.

6.1.7 The concentrator of each unit is capable of rapidly heating the trap to 260°C and holding at that temperature for the duration of the desorb time.

6.1.8 Gas chromatograph. HP 5890/Agilent 6890N/7890 equipped with temperature programming capability.

6.1.9 GC column:

- 75M long x 0.53mm ID, J&W DB-624 capillary column with 3um film thickness or similar phase.
- 20M long x 0.18 ID, DB-624 or Rtx-VMS with 1 um film thickness.
- 30M long x 0.25 mm ID x 1.4 um film thickness Rtx-624.

6.1.10 Injection port liners. HP 18740-80200 or equivalent.

6.2 Supplies

- Microsyringes. 10 uL to 1000 uL.
- Syringes. 5mL and 30mL gas-tight.
- Volumetric flasks. Class “A” glassware, 10mL, 50mL, and 100 mL.

- Vials. 2mL amber glass with screw cap with Teflon-faced septa.
- VOA vials. 40ml glass with PTFE-faced septa

7.0 Reagents and Standards

7.1 Organic free reagent water. Distilled water purchased from Poland Spring.

7.2 Methanol: Purge and trap grade, purchased from JT Baker. (Cat # 9077-02)

7.3 Standard preparation and use is described in detail in each of the determinative methods.

8.0 Sample Collection, Preservation, Shipment and Storage

8.1 Aqueous samples are collected in the appropriate containers and preserved according to the determinative method requirements.

8.2 All aqueous samples are protected from light and stored at 4°C in an area free of organic solvents. No standards or solvents are stored in the sample refrigerators or in the surrounding area.

8.3 All aqueous samples are analyzed within 14 days of collection.

8.4 The collection, preservation and hold times for soil samples prepared via SW846 Method 5035 or 5035A are addressed in the corresponding TestAmerica Edison SOPs (see references in Section 15).

9.0 Quality Control

9.1 Specific quality control procedures are outlined in each of the determinative methods that follow this purge and trap technique of sample introduction. Standard quality assurance practices are used with all methods.

9.2 An initial demonstration of accuracy and precision is performed for each determinative method. This demonstration is done for each sample introduction technique.

9.3 Reagent water blanks are analyzed to ensure that reagents and/or sample dilutions are free of interferences.

9.4 Method blanks are prepared and analyzed in the same manner as samples, and are carried through the entire analytical procedure to show that each system is in control.

9.5 Each analysis batch contains spike and duplicate data as outlined in the method, as well as a laboratory control sample (blank spike) to show that replicate data can be generated and appropriate concentrations of target analytes recovered.

10.0 Procedure

10.1 This section provides guidance for the analysis of aqueous samples and/or methanol extracts prepared by SW846 Method 5035 by purge and trap techniques. Determinative methods SW846 Methods 8015 or 8260 will follow this purge and trap procedure

10.2 Instrument operating parameters are set at the beginning of a method of analysis and remain constant throughout the calibration and sample analysis steps. See each determinative method to verify specific instrument operating parameters.

Table 1: Example Instrument Operating Parameters

Purge and trap unit	
Purge Time	11 minutes
Dry Purge	1 Minutes
Purge Gas	Helium (or nitrogen, where applicable)
Purge Flow	40-45 ml/min
Purge Temp	Water: Ambient, Solids: 40^{0}C
Trapping Temp	Ambient, <30^{0}C
Desorb Time	1 Minute
Desorb Temp	VOCARB: 260^{0}C
Gas chromatograph	
Injector	180^{0}C
Carrier Gas	Helium
Carrier Flow	6 ml/min
Oven Program	35 - 250^{0}C with 2 ramps
Run Time	26 Minutes
Mass Spectrometer	
Electron Energy	70 volts (nominal)
Mass range	35-260 AMU
Scan time	0.9 sec./scan
Source Temp	200^{0}C
Separator Temp	180^{0}C

10.3 Screen all samples according to TestAmerica Edison SOP No. ED-GCV-001, (*Screening for Volatile Organics, Static Headspace with GC FID,, SW846 Method 5021* **,** current revision). Analysts are furnished with approximate dilution factors prior to analysis.

10.4 Allow all samples and standards solutions to warm to ambient temperature before use.

10.5 Prior to performing any purge and trap analysis, prepare each system in the following manner:

10.5.1 Condition concentrators by baking the trap at their bake temperatures for 5-12 minutes. Also condition the GC column by baking at 200°C.

10.5.2 Access the FLUSH menu on the Archon Autosampler. Rinse the syringe barrel with two 6mL portions of rinse water. Turn off the helium switch, and fill the water reservoir with reagent water. Visually inspect the standard vial and fill with the appropriate internal standard/surrogate mix. Turn the helium switch back on. Prime the standard loop by accessing the SYSTEM/MAINTENANCE/STANDARD CONTROL menu. Empty the waste reservoir.

10.5.3 Prepare other referenced autosamplers by filling internal standard reservoirs with the appropriate IS/SURR, empty the waste reservoir and fill the water rinse reservoir.

10.6 Prior to using this sample introduction technique, the GC or GC/MS system must be tuned and calibrated as required by each of the determinative methods (reference the applicable TestAmerica Edison SOP; see references in Section 15). All method requirements for tune, calibration, and blank analysis must be met prior to sample analysis.

10.7 Aqueous samples

10.7.1 Load the sample vials directly into the autosampler tray. The appropriate internal standard and/or surrogate solution will be added to the sample by the autosampler unit immediately before a sample is purged.

10.7.2 The process of using autosamplers also destroys the validity of the sample by puncturing the septum. Any re-analysis should be done using second vial of sample. If only one vial exists, do not use this autosampler.

10.8 Medium or high level sample

10.8.1 Prepare samples by adding the appropriate amount of sample to a 50mL volumetric flask containing reagent water.

10.8.2 Add the appropriate amount of sample to the flask. Dilute to volume with reagent water, cap, and invert three times. Pour the diluted sample into a 5mL syringe or a 40mL VOA vial.

10.8.3 If a dilution is required, reduce the volume of sample added to the

10.8.4 Load sample onto the autosampler.

10.8.5 Purge the sample for 11 minutes, with one minute dedicated to removing moisture from the purge and trap system (dry purge). Times may vary among different determinative methods.

10.8.6 After purging is complete, desorb the sample onto the GC column by rapidly heating the trap to 260°C and backflushing it with helium (or nitrogen, where applicable). (Trap temperatures will vary based on type used)

10.8.7 Begin the GC temperature program and data acquisition.

10.8.8 Re-condition the trap by baking at 260°C for 8-12 minutes.

10.8.9 Cool the trap to (<31°C). The trap is now ready for the next sample.

10.8.10 Process data using Chrom software, and evaluate as per each determinative method.

10.9 Technical acceptance criteria for sample analysis.

10.9.1 The samples must be analyzed on a GC or GC/MS system meeting the initial calibration, continuing calibration and blank technical acceptance criteria of the determinative methods (reference the applicable TestAmerica Edison SOP; see references in Section 15).

10.9.2 The sample must be analyzed within the required holding time.

10.9.3 The sample must have an associated method blank meeting the blank technical acceptance criteria.

10.9.4 The percent recovery of each of the system monitoring compounds in the sample must be within the acceptance windows.

10.9.5 After analyzing a sample that exceeds the initial calibration range the analyst must either analyze an instrument blank (using the same purge inlet if using an auto sampler) which must meet technical acceptance criteria for blank analysis or monitor the sample analyzed immediately after the contaminated sample for all compounds that were in the contaminated sample that exceeded the calibration range.

10.10 Corrective Action for Sample Analysis

10.10.1 Samples must meet technical acceptance criteria of the determinative methods (reference the applicable TestAmerica Edison SOP; see references in Section 15) before reporting data.

10.10.2 Corrective action must be completed prior to sample analysis if analysis failed to meet instrument performance checks, initial calibration, continuing calibration and method blanks.

10.10.3 Corrective action for system monitoring compounds and internal standard compounds that fail to meet acceptance criteria must be completed prior to sample analysis.

10.11 If any of the system monitoring compounds and internal standard compounds fail to meet acceptance criteria of the determinative methods (reference the applicable TestAmerica Edison SOP; see references in Section 15):

10.11.1 Check all calculations, instrument logs, the system monitoring compound and internal standard compound spiking solutions and the instrument operation. If the calculations were incorrect, correct calculations and verify that the system monitoring compound recoveries and internal standard compound responses meet acceptance criteria

10.11.2 Check the preparation of the internal standards and system monitoring compounds for concentration and expiration.

10.11.3 Verify that the instrument is operating correctly.

10.12 Data that fails to meet minimum acceptance criteria will be annotated (flagged) with qualifiers and/or appropriate narrative comments defining the nature of the outage. If applicable, a *Non-Conformance Memo* (NCM) (TestAmerica Edison Work Instruction No. EDS-WI-012) will be initiated in order to provide for investigation and follow-up. For further corrective actions and contingencies for handling out-of-control or unacceptable data see *Standard Operating Procedure for Control of Non-Conformances and Corrective Action* (TestAmerica Edison SOP No. EDS-GEN-003).

11.0 Calculations / Data Reduction

11.1 For specific calculations refer to the applicable determinative methods (reference the applicable TestAmerica Edison SOP; see references in Section 15

12.0 Method Performance

12.1 Method Detection Limit Study (MDL)

The method detection limit (MDL) is the lowest concentration that can be detected for a given analytical method and sample matrix with 99% confidence that the analyte is present. The MDL is determined according to the laboratory's MDL procedure in Section 19 (Test Methods and Method Validation) of TestAmerica Edison's Quality Assurance Manual (ED-QA-LQM). MDLs reflect a calculated (statistical) value determined under ideal laboratory conditions in a clean matrix, and may not be achievable in all environmental matrices. The laboratory maintains MDL studies for analyses performed; these are verified at least annually unless method requirements require a greater frequency.

12.2 Demonstration of Capabilities

For DOC procedure refer to Section 19 in the most current revision of TestAmerica Edison's Quality Assurance Manual (ED-QA-LQM).

12.3 Training Requirements

Refer to TestAmerica SOP No. ED-GEN-022, Training, for the laboratory's training program.

13.0 Pollution Control

13.1 Pollution prevention encompasses any technique that reduces or eliminates the quantity or toxicity of waste at the point of generation. Numerous opportunities for pollution prevention exist in laboratory operation. The USEPA has established a prevention hierarchy of environmental management techniques that places pollution prevention as the management option of first choice. Whenever feasible, laboratory personnel should use pollution prevention techniques to address their waste generation. When wastes cannot be feasibly reduced at the source, the agency recommends recycling as the next best option.

13.2 The quantity of chemical purchased should be based on expected usage during its shelf life and disposal cost of unused material. Actual reagent preparation volumes should reflect anticipated usage

14.0 Waste Management

14.1 The U.S. Environmental Protection Agency requires that laboratory waste management practices conducted be consistent with all applicable rules and regulations. All waste will be disposed of in accordance with Federal, State and Local regulations. Where reasonably feasible, technological changes have been implemented to minimize the potential for pollution of the environment. Employees will abide by this method and the policies in section 13 of the Corporate Environmental Health and Safety Manual (CW-E-M-001) for "Waste Management and Pollution Prevention."

14.2 The following waste streams are generated as a result of this analysis:

- Laboratory Generated Aqueous Waste (aqueous VOA vials – used and unused). This waste may have a pH of less than 2.0. These vials are collected in satellite accumulation. The vials are then transferred to the waste room. These vials are passed through a vial crusher and the liquid portion is separated from the solid portion. The solid is dumped into the municipal garbage. The liquid is pumped into the neutralization system where it is neutralized to a pH of 6 to 9 with sodium bicarbonate (Seidler Chemical SC-0219-25). When neutralization is complete, the material is transferred to the municipal sewer system.

- Expired Standards – The vials are collected in a 1 gallon polyethylene bucket. These vials are then transferred to an open top 55 gallon steel or polyethylene

waste drum. These drums are transported to a waste facility for proper disposal.

- Soil Retain Samples - These samples if not flagged in the system for any hazardous constituents are transferred to poly-lined cubic yard boxes. These boxes when full are sent to stabilization or incineration. These materials are sent out as hazardous for lead and chromium
  Teris Profile Number (incineration): 50016710
  Onyx Profile Number: (stabilization) 402535

- Methanol Preserved Samples - Methanol preserved sample vials are collected in satellite accumulation and then transferred to a 55 gallon open top steel waste drum in the waste room. This drum is then removed by a waste vendor for incineration.

  Teris Profile Number: 50016652
  Onyx Profile Number: 282493

- Returned Methanol Preservative - Methanol preserved vials are collected in satellite accumulation and then transferred to 55 gallon open top steel waste drums in the waste room. These waste drums are then removed by a waste vendor for incineration.

  Teris Profile Number: 50016652
  Onyx Profile Number: 282493

15.0 References / Cross-References

15.1 United States Environmental Protection Agency, *Method 5030B: Purge and Trap for Aqueous Samples*, Test Methods for Evaluating Solid Wastes, SW846 Third Edition, Volume 1B: Laboratory Manual, Physical/Chemical Methods, Revision 2 December 1996.

15.2 United States Environmental Protection Agency, *Method 5035: Closed System Purge and Trap and Extraction for Volatile Organics in Soil and Waste Samples*, Test Methods for Evaluating Solid Wastes, SW846 Third Edition, Volume 1B: Laboratory Manual, Physical/Chemical Methods, Revision 0, December 1996

15.3 United States Environmental Protection Agency , *Method 5035A*: *Closed-System Purge-and-Trap and Extraction for Volatile Organics in Soil and Waste Samples*, Test Methods for Evaluating Solid Wastes, SW846 Third Edition, Volume 1B: Laboratory Manual, Physical/Chemical Methods, Revision 0, July 2002.

15.4 United States Environmental Protection Agency , *Method 8260B*: *Volatile Organic Compounds by Gas Chromatograpy/Mass Spectrometry*, Test Methods for Evaluating Solid Wastes, SW846 Third Edition, Volume 1B: Laboratory Manual, Physical/Chemical Methods, Revision 2, December 1996.

15.5 United States Environmental Protection Agency , *Method 8015B*: *Non-Halogenated Organics by Gas Chromatography*, Test Methods for Evaluating Solid

Wastes, SW846 Third Edition, Volume 1B: Laboratory Manual, Physical/Chemical Methods, Revision 2, December 1996.

15.6 TestAmerica Edison SOP No. ED-MSV-005, *Volatile Organic Compounds by GC/MS,SW846 Method 8260B,* current revision.

15.7 TestAmerica Edison SOP No. ED-GCV-006, *Gasoline Range Organics using GC-FID Method, SW846 Method 8015B,* current revision.

15.8 TestAmerica Edison SOP No. ED-MSV-002, *Closed System Purge and Trap and Extraction for Volatile Organics in Soil, SW846 Method 5035,* current revision

15.9 TestAmerica Edison SOP No. ED-GCV-001, *Screening for Volatile Organics, Static Headspace with GC FID, SW846 Method 5021* **,** current revision

15.10 TestAmerica Edison Document No. ED-QA-LQM, *Laboratory Quality Manual*, most current revision.

15.11 TestAmerica Edison SOP No. EDS-GEN-003, *Standard Operating Procedure for Control of Non-Conformances and Corrective Action* , most current revision.

15.12 TestAmerica Edison Work Instruction No. EDS-WI-012, *Non-Conformance Memo* (*NCM),* most current revision.

15.13 United States Environmental Protection Agency, Method 5030B, Test Methods for Evaluating Solid Wastes, SW846 Third Edition, Volume 1B: Laboratory Manual, Physical/Chemical Methods, Revision 2, December 1996.

15.14 United States Environmental Protection Agency, Method 5030C, Test Methods for Evaluating Solid Wastes, SW846 Third Edition, Volume 1B: Laboratory Manual, Physical/Chemical Methods, Revision 3, May 2003.

16.0 Method Modifications:

16.1 N/A

17.0 Attachments

17.1 N/A

18.0 Revision History

Revision 9, dated: 06/26/2013:

- Revised title of SOP to include reference to SW846 5030C.
- Section 1.1.1 and throughout document as appropriate: added reference to SW846 5030C.
- Section 1.1.1: inserted word ‘extracts’ in reference to analysis of high concentration and soil samples by SW846 5035/5035A.
- Section 10.8.10: replaced Target reference with Chrom (newly implemented chromatography data system)

- Section 15.3: corrected method reference to '5035A'.
- Section 15.14: added reference to SW8465030C

Revision 8, dated: 11/27/2012:

- Updated referenced Lab Quality Manual section and appendix numbers as appropriate.

Revision 7, dated: 10/02/2008

- Revised SOP format in accordance with TestAmerica Corporate Quality SOP No. CW-QS-002 (Rev 0) Writing a Standard Operating Procedure (SOP).
- Section 1.2 Added reference to Quality Assurance Manual for method modifications. Added references to related methods and SOPs for determinative methods.
- Section 2 (and throughout document): added option of purging with nitrogen gas as applicable.
- Section 3: revised to reference new location for definitions.
- Section 5: Revised to include most up to date corporate health and safety references and information.
- Section 6: Newer instrumentation and GC column types added. See sections 6.1.2 and 6.1.9. All references to Tekmar instruments 2016 and 2000 and procedures to use with it removed in all relevant sections.
- Section 11: Removed reference to Organic Calculation SOP.
- Section 12: updated and revised the MDL requirements to reflect text in the current revision of the TestAmerica Edison Laboratory Quality Manual (LQM).
- References: Expanded to include more specific SOP references
- Section 18: Added this Revision History section

Title: SW846 Method 8260C, Volatile Organic Compounds by Gas Chromatography/Mass Spectrometry (GC/MS)

Once printed, this is considered an uncontrolled document

Approvals (Signature/Date):

Reddy Pakanati VOA Department Manager	01/17/18 Date	Sylvanus Klusey Organics Operations Manager	01/17/18 Date
Carl Armbruster Quality Assurance Manager	01/17/18 Date	Mark Acierno Laboratory Director	01/17/18 Date

Dan Helfrich — 01/17/18 Date
Health & Safety Manager / Coordinator

Copyright Information:
This documentation has been prepared by TestAmerica Laboratories, Inc. and its affiliates ("TestAmerica"), solely for their own use and the use of their customers in evaluating their qualifications and capabilities in connection with a particular project. The user of this document agrees by its acceptance to return it to TestAmerica upon request and not to reproduce, copy, lend, or otherwise disclose its contents, directly or indirectly, and not to use it for any other purpose other than that for which it was specifically provided. The user also agrees that where consultants or other outside parties are involved in the evaluation process, access to these documents shall not be given to said parties unless those parties also specifically agree to these conditions.

THIS DOCUMENT CONTAINS VALUABLE CONFIDENTIAL AND PROPRIETARY INFORMATION. DISCLOSURE, USE OR REPRODUCTION OF THESE MATERIALS WITHOUT THE WRITTEN AUTHORIZATION OF TESTAMERICA IS STRICTLY PROHIBITED. THIS UNPUBLISHED WORK BY TESTAMERICA IS PROTECTED BY STATE AND FEDERAL LAW OF THE UNITED STATES. IF PUBLICATION OF THIS WORK SHOULD OCCUR THE FOLLOWING NOTICE SHALL APPLY:

©COPYRIGHT 2018 TESTAMERICA LABORATORIES, INC. ALL RIGHTS RESERVED.

Facility Distribution No. ELECTRONIC **Distributed To: FACILITY INTRANET**

1.0 Scope and Application

1.1 Analytes, Matrix(s), and Reporting Limits

1.1.1 USEPA SW846 Method 8260C is used for the determination of volatile organic compounds in a variety of aqueous and solid matrices by purge and trap gas chromatography (GC)/mass spectrometery (MS). The method is applicable to the compounds listed in Table 1 (below). Actual target compound lists are determined through regulatory or project specifications. Method performance criteria for each target analyte will be determined prior to sample analysis.

1.1.2 This SOP also describes the optional procedure for analyses of compounds using Selected Ion Monitoring (SIM). SIM analyses is specific to target compounds: 1,2-dibromoethane, 1,2-dibromo-3-chloropropane, 1,2,3-Trichloropropane and 1,4-Dioxane. Benzene and Chloroform if needed.

Table 1: Method Analytes

COMPOUND	CAS#	COMPOUND	CAS#
Acetone	67-64-1	Epichlorohydrin	106-89-8
Acetonitrile	75-05-8	Ethylbenzene	100-41-4
Acrolein (Propenal)	107-02-8	Ethyl methacrylate	97-63-2
Acrylonitrile	107-13-1	Fluorobenzene (IS)	462-06-6
Allyl alcohol	107-18-6	Hexachlorobutadiene	87-68-3
Benzene	71-43-2	2-Hexanone	591-78-6
Benzyl chloride	100-44-7	Iodomethane	74-88-4
Bromochloromethane	74-97-5	Isobutyl alcohol	78-83-1
Bromodichloromethane	75-27-4	Isopropylbenzene	98-82-8
4-Bromofluorobenzene (surr)	460-00-4	Ethyl Ether	60-29-7
Bromoform	75-25-2	Freon 113	76-13-1
Bromomethane	74-83-9	Methylene chloride	75-09-2
n-Butanol	71-36-3	Methyl methacrylate	80-62-6
2-Butanone (MEK)	78-93-3	4-Methyl-2-pentanone (MIBK)	108-10-1
t-Butyl alcohol	75-65-0	Naphthalene	91-20-3
Butyl Acrylate	141-32-2	Isoprene	78-79-5
Butyl Methacrylate	97-88-1	n-Butyl Acetate	123-86-4
Camphene	79-92-5	n-Propyl Acetate	109-60-4
Camphor	76-22-2	2-Octanol	4128-31-8
Carbon disulfide	75-15-0	1-Propanol	71-23-8
Carbon tetrachloride	56-23-5	2-Propanol(Isopropanol)	67-63-0
Chlorobenzene	108-90-7	n-Heptane	142-82-5
Chlorobenzene-d5 (IS)	3114-55-4	n-Hexane	110-54-3
Chlorodibromomethane	124-48-1	tert-Amyl methyl ether	994-05-8
Chloroethane	75-00-3	tert-Butyl ethyl ether	637-92-3

COMPOUND	CAS#	COMPOUND	CAS#
2-Chloroethyl vinyl ether	110-75-8	Styrene	100-42-5
Chloroform	67-66-3	1,1,1,2-Tetrachloroethane	630-20-6
Chloromethane	74-87-3	1,1,2,2-Tetrachloroethane	79-34-5
Dibromomethane	74-95-3	Tetrachloroethene	127-18-4
1,2-Dichlorobenzene	95-50-1	Toluene	108-88-3
1,3-Dichlorobenzene	541-73-1	Toluene-d8 (surr)	2037-26-5
1,4-Dichlorobenzene	106-46-7	Pentyl Acetate(Amyl Acetate)	628-63-7
1,4-Dichlorobenzene-d4 (IS)	3855-82-1	1,2,4-Trichlorobenzene	120-82-1
trans-1,4-Dichloro-2-butene	110-57-6	1,1,1 -Trichloroethane	71-55-6
Dichlorodifluoromethane	75-71-8	1,1,2-Trichloroethane	79-00-5
1,1-Dichloroethane	75-34-3	Trichloroethene	79-01-6
1,2-Dichloroethane	107-06-2	Trichlorofluoromethane	75-69-4
1,2-Dichloroethane-d4 (surr)	17060-07-0	1,2,3-Trichloropropane	96-18-4
1,1-Dichloroethene	75-35-4	Vinyl acetate	108-05-4
trans-1,2-Dichloroethene	156-60-5	Vinyl chloride	75-01-4
1,2-Dichloropropane	78-87-5	o-Xylene	95-47-6
cis-1,3-Dichloropropene	10061-01-5	m-Xylene	108-38-3
1,3-Dimethylnaphthalene	575-41-7	p-Xylene	106-42-3
Diethyl ether	60-29-7	Bromobenzene	108-86-1
1,4-Dioxane	123-91-1	n-Butylbenzene	104-51-8
Methyl acrylate	96-33-3	sec-Butylbenzene	135-98-8
Methyl-t-butyl ether	163-404-4	tert-Butylbenzene	98-06-6
Methyl Acetate	79-20-9	Methyl Cyclohexane	108-87-2
n-Propylbenzene	103-65-1	2-Octanone	111-13-7
1,2,3-Trichlorobenzene	87-61-6	4-Chlorotoluene	106-43-4
1,2,4-Trimethylbenzene	95-63-6	cis-1,2-Dichloroethene	156-59-2
1,3,5-Trimethylbenzene	108-67-8	1,3-Dichloropropane	142-28-9
Tetrahydrofuran	109-99-9	2,2-Dichloropropane	590-20-7
2-Methylnaphthalene	91-57-6	p-Isopropyltoluene	99-87-6
1-Methylnaphthalene	90-12-0	Isopropyl Acetate	108-21-4
1,1,2-Trichloro-1,2,2-Trifluoroethane	76-13-1	Ethyl Acetate	141-78-6
1-Propene	115-07-1	Ethanol	64-17-5
2-Chloropropane	75-29-6	Xylenes (total)	133-0207
1-Chloropropane	540-54-5	Isopropyl Ether (DIPE)	108-20-3
Dichlorofluoromethane	75-43-4	2-Ethyl-1-Hexanol	104-76-7
Methacrylonitrile	126-98-7	Propionitrile	107-12-0
2-Chloro-1,3-butadiene (chloroprene)	126-99-8	Ethyl methacrylate	97-63-2
Isopropyl Alcohol	67-63-0	2-Nitropropane	79-46-9
Cyclopentene	142-29-0	Indan	496-11-7
trans-1,3-Dichloropropene	10061-02-6	Freon 114	76-14-2
2,2,4-Trimethylpentane (Isooctane)	540-84-1	t-Amyl Alcohol	75-85-4
1-Chlorohexane	544-10-5	1,4-Difluorobenzene	540-36-3
1,2,4,5- Tetramethylbenzene	95-93-2	1,4-Diethylbenzene	105-05-5
4- EthylToluene	622-96-8	Butadiene	106-99-0
Chlorotrifluoroethylene	79-38-9	1,2-Dichloro-1,1,2-triflioroethane (Freon 123a)	354-23-4

1.1.3 Method 8260C can be used to quantitate most volatile organic compounds that have boiling points below 200°C, and that are insoluble or slightly soluble in water. Water-soluble compounds can be included in this method, but quantitation limits will be higher due to poor purging efficiency.

1.1.4 The standard reporting limit (RL) is established at or above the low-level standard in the calibration curve. For a complete list of method detection limits (MDLs) and RLs, please see reference the current TALS (LIMS) active Method Limit Group database.

1.1.5 On occasion clients may request modifications to this SOP. These modifications are handled following the procedures outlined in Sections 7 (*Review of Work Request*) and 20 (*Test Methods and Method Validation*) of TestAmerica Edison's Quality Assurance Manual (ED-QA-LQM).

2.0 Summary of Method

2.1 Method 8260C is used to determine volatile organic compounds in aqueous, non-aqueous and solid matrices. Sample preparation techniques vary, depending on the matrix and the level of contamination expected. Purge and trap techniques are used to introduce the sample to the GC/MS system. Refer to TestAmerica Edison SOP Nos. ED-MSV-001*, Purge and Trap for Aqueous Samples, SW846 Method 5030*, current revision and ED-MSV-002, *Closed-System Purge-and-Trap and Extraction for Volatile Organics in Soil and Waste Samples, SW846 Method 5035,* current revision.

2.2 All samples extracts are screened by GC/FID static headspace analysis to provide the analyst with appropriate initial dilution factors. For additional details see TestAmerica Edison SOP No. ED-GCV-001, *Screening for Volatile Organics, Static Headspace with GC FID, SW846 Method 5021*, current revision.

2.3 An aliquot of sample containing internal standard and surrogate spiking solution is purged with nitrogen in a closed sparging vessel. The volatile compounds are transferred from the aqueous phase to the vapor phase. The vapor is swept through a sorbent column where the volatiles are trapped. After purging is complete, the sorbent column is heated and backflushed with helium to desorb the volatiles onto a gas chromatograph column.

2.4 Analytes eluted from the capillary chromatograhy column are introduced into the mass spectrometer via a direct connection. Identification of target analytes is accomplished by comparing their mass spectra with the electron impact spectra of authentic standards. Quantitation is accomplished by comparing the response of a major (quantitation) ion relative to an internal standard using a minimum of a five-point calibration curve.

2.5 For aqueous VOA samples submitted for New Jersey Groundwater Quality Standard (NJ GWQS) evaluation, a full scan analysis is initially performed using the 8260 method. No further analysis by SIM is required if all of the following

compounds are present above the full scan RL: 1,2-dibromoethane, 1,2-dibromo-3-chloropropane, 1,2,3-Trichloropropane and 1,4-dioxane, chloroform, vinyl chloride and benzene. If any of these compounds are undetected in the undiluted, full scan analysis, the sample must be analyzed via 8260C SIM for those compounds.

2.6 To meet lower reporting limits of 0.5ug/L for most analytes, 2.5 ug/L for ketones and generally lower limits for other non-routine analytical compounds, spike at the appropriate levels using existing purging conditions. The corresponding TALS login method for low level aqueous analysis is 8260_LL. See Table 3b for initial calibration levels and spike amounts.

3.0 Definitions

3.1 For a complete list of definitions refer to Appendix 2 in the most current revision of TestAmerica Edison's Quality Assurance Manual (ED-QA-LQM).

4.0 Interferences

4.1 This method is susceptible to contamination from a number of sources, including organic solvents used in other laboratory procedures, impurities in the purge gas, improper cleaning of syringes or purge vessels, and carryover from high level samples. Samples can be contaminated by the diffusion of volatile organics through the septum during shipment or storage. Steps have been taken to ensure that these potential problems are eliminated from the laboratory.

4.2 The volatiles analytical laboratory is housed in a separate building, away from the organic extraction lab area where large quantities of organic solvents are used. No organic solvents are used or stored in the volatiles laboratory.

4.3 The nitrogen used as purge gas passes through a solvent trap prior to its inlet into the purge and trap units.

4.4 A trip blank prepared from organic-free reagent water is carried through the sampling, storage and analysis of each group of samples to check for such contamination.

4.5 Individual samples are each handled with a unique syringe that has been baked in a drying oven at 105°C to ensure the absence of volatile compounds.

4.6 Carryover can occur anytime a high level sample is analyzed. Screening procedures are employed to ensure that a sample is analyzed at an appropriate dilution to minimize potential carryover. When a high level sample is analyzed, it is followed by the analysis of a reagent water blank. If another sample was analyzed after the high level sample, this sample is inspected carefully for signs of carryover. If this sample does not contain any of the compounds found in the high level sample, the system can be considered contamination free.

4.7 The analytical system is checked daily with the analysis of a method blank. This blank must meet all quality control criteria for the method before sample analysis may take place.

5.0 Safety

Employees must abide by the policies and procedures in the Corporate Environmental Health and Safety Manual (CW-E-M-001) and this document. This procedure may involve hazardous material, operations and equipment. This SOP does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of the method to follow appropriate safety, waste disposal and health practices under the assumption that all samples and reagents are potentially hazardous. Safety glasses, gloves, lab coats and closed-toe, nonabsorbent shoes are a minimum.

Any questions pertaining to safety issues or procedures should be brought to the department manager or Edison Safety Officer.

5.1 Specific Safety Concerns or Requirements

5.1.1 Latex, nitrile and vinyl gloves all provide adequate protection against the methanol used in this method.

5.1.2 Purge vessels on purge-and-trap instruments can be pressurized by the time analysis is completed. Vent the pressure prior to removal of these vessels to prevent the contents from spraying out.

5.1.3 The gas chromatograph and mass spectrometer contain zones that have elevated temperatures. The analyst needs to be aware of the locations of those zones, and must cool them to room temperature prior to working on them.

5.1.4 The mass spectrometer is under deep vacuum. The mass spectrometer must be brought to atmospheric pressure prior to working on the source.

5.1.5 There are areas of high voltage in both the gas chromatograph and the mass spectrometer. Depending on the type of work involved, either turn the power to the instrument off, or disconnect it from its source of power.

5.2 Primary Materials Used

The following is a list of the materials used in this method, which have a serious or significant hazard rating. **Note: This list does not include all materials used in the method. The table contains a summary of the primary hazards listed in the MSDS for each of the materials listed in the table.** A complete list of materials used in the method can be found in the reagents and materials section. Employees must review the information in the MSDS for each material before using it for the first time or when there are major changes to the MSDS.

Material (1)	Hazards	Exposure Limit (2)	Signs and symptoms of exposure
Methanol (MeOH)	Flammable Poison Irritant	200 ppm-TWA	A slight irritant to the mucous membranes. Toxic effects exerted upon nervous system, particularly the optic nerve. Symptoms of overexposure may include headache, drowsiness and dizziness. Methyl alcohol is a defatting agent and may cause skin to become dry and cracked. Skin absorption can occur; symptoms may parallel inhalation exposure. Irritant to the eyes.
1 – Always add acid to water to prevent violent reactions.			
2 – Exposure limit refers to the OSHA regulatory exposure limit.			

6.0 Equipment and Supplies

6.1 Instrumentation

6.1.1 Purge and trap units from several different manufacturers are used, depending upon the sample matrix and preparatory technique required. A purge and trap unit consists of three parts: the sample purge unit, the trap, and the concentrator. Unit configurations currently in use are:

- OI Analytical 4551, 4100 Automatic Sampler/4660,4760 concentrator;
- Archon 5100A Automatic sampler/ OI Analytical 4660,4760 concentrator;
- EST Centurion Autosampler/ EST Encon concentrator;
- Archon Autosampler/EST Encon concentrator.
- Archon/EST Evolution

6.1.2 A VOCARB 3000 trap from Supelco is used in the Encon concentrator. The trap is 25cm long with an inside diameter of 0.105 inches. The trap is packed with 10.0cm Carbopack B, 6.0 cm Carboxin 1000, and 1cm Carboxin 1001.

6.1.3 An OI analytical purge trap #10 is used for the OI 4560,4660 and 4760 concentrator. The trap is 25cm long with an inside diameter of 0.105 inches. The trap is packed to contain the following absorbents: Tenax/silica gel/carbon molecular sieve.

6.1.4 Alternate traps may be used provided the adsorption and desorption characteristics are equivalent to those of the trap recommended by the method.

6.1.5 Both the Encon and OI concentrators are capable of rapidly heating the trap to 260°C and holding at that temperature for the duration of the desorb time.

6.1.6 Gas chromatograph: HP Agilent 6890/7890 equipped with temperature programming capability.

6.1.7 GC column: 30M long x 0.25mm ID, 1.4um film thickness, 20M x 0.18mm x 1um DB-624 and 20M long x 0.18 mm ID Restek Rtx-VMS capillary column with 1um film thickness or similar phase.

6.1.8 Mass Spectrometer (Agilent 5973/5975/5977): scanning from 35-260 amu every 0.9 seconds, utilizing 70 volts (nominal) electron energy in the electron ionization mode and producing a mass spectrum which meets all EPA performance criteria when 50 ng of 4-Bromofluorobenzene (BFB) is injected through the gas chromatograph inlet.

6.1.9 GC/MS Interface: transfer lines heated to 180°C .

6.1.10 Data system: HP Chemstation II for data acquisition and TestAmerica Chrom for data processing.

6.2 Supplies

- Microsyringes: 10 ul to 1000 ul.
- Syringes: 5 ml to 25 ml gas-tight.
- Injection port liners: HP 18740-80200 or equivalent
- Volumetric flasks: Class “A” glassware, 5 ml to 500 ml.
- VOA vials: 20-ml and 40-ml glass with PTFE – faced septum.
- Vials: 2-ml amber glass with screw cap with Teflon-faced septa.
- Top loading analytical balance.
- Spatula: Narrow, stainless steel.
- Stir bars: PTFE coated, small enough to spin freely inside a VOA vial.

7.0 Reagents and Standards

7.1 Reagents

7.1.1 Organic free reagent water: Distilled water purchased from Poland Spring or equivalent.

7.1.2 Methanol: Ultra Resi-Analyzed, purge and trap grade, purchased from JT Baker or equivalent. (Cat # 9077-02)

7.1.2.1 Each lot of methanol is screened for contaminants before being used for analysis as detailed in TestAmerica Corporate Quality SOP No. CA-Q-S-001 (*Solvent & Acid Lot Testing & Approval*) and TestAmerica Edison SOP No. ED-GEN-023 (*Bulk Solvent Testing and Approval*).

7.2 Standards

7.2.1 **Calibration Standards** Stock target compound analytical standard solutions are purchased mainly from Restek, Supelco, Inc, Absolute Standards and Spex although standards of similar quality from other suppliers may be substituted as required. Standards noted with an asterisk (*) are custom mixes made especially for TestAmerica Edison.

Target Analyte Standard Name	Concentration	Vendor	Catalog #
8260 List 1 / Std #3 Gases*	2500 ppm	Restek	569722
8260 List 1 / Std #3 Gases – (SS)*	2500 ppm	Restek	569722 sec
8260 List 1 / Std #1 MegaMix*	1250-62500 ppm	Restek	569720
8260 List 1 / Std #1 MegaMix (SS)*	1250-62500 ppm	Restek	569720 sec
8260 List 1 / Std #2 Ketones *	12500 ppm	Restek	569721
8260 List 1 / Std #2 Ketones * (SS)	12500 ppm	Restek	569721 sec
8260 List 1 / Std #5 Acrolein *	20,000 ppm	Restek	568720
8260 List 1 / Std #5 Acrolein (SS)	20,000 ppm	Restek	568720 sec
8260 List 1 /Std #4 2 CEVE *	2500 ppm	Restek	569723
8260 List 1 /Std #4 2 CEVE (SS) *	2500 ppm	Restek	569723 sec
8260 List 1 /Std #6 Vinyl Acetate *	5000 ppm	Restek	569724
8260 List 1 /Std #6 Vinyl Acetate (SS) *	5000 ppm	Restek	569724 sec
8260 List 2 / Std #1 Additions *	2500-62500ppm	Restek	568725
8260 List 2 / Std #1 Additions (SS) *	2500-62500 ppm	Restek	568725 sec
8260 List 3 / Std #1 Polar Additions *	2500-100000ppm	Restek	568728
8260 List 3 / Std #1 Polar Additions (SS) *	2500-100000 ppm	Restek	568728 sec
VOC Extra Standard 2015 *	2500-5000 ppm	Absolute	98593
VOC Extra Standard 2015 * (SS)	2500-5000 ppm	Absolute	98593
Epichlorohydrin	1000 ppm	Absolute	70377
Acrolein	5000 ppm	Restek	91980
Acrolein *	Neat	Sigma	110221
2-Freon Mix quote # 12258 *	2500ppm	Absolute	12258
2-Freon Mix quote # 12258 * (SS)	2500ppm	Absolute	12258
1,4-Dioxane	Neat	Sigma	360481
Epichlorohydrin	Neat	Sigma	45340
2-Chloroethylvinyl ether	Neat	Sigma	109983
1,4-Dioxane	1000 ppm	Absolute	70373
1,4-Dioxane	10000 ppm	Absolute	92785
Benzene	1000 ppm	Absolute	70025
Chloroform	1000 ppm	Absolute	70076

(1): The separate source for this material is not available as a distinct catalog number. Analyst must ensure that a separate lot of the material is selected and used as required.

An asterisk (*) indicates a custom standard mix.

7.2.1.1. Prepare stock solutions at volumes and concentrations indicated in Table 2 (Working Standards Preparation) by combining the indicated volumes of each stock solution into a volumetric flask corresponding to the total final volume. Dilute to the volume marker with methanol.

7.2.1.2. Prepare individual calibration standards as applicable per Section 9.2.2.1, Table 3, Initial Calibration Standards Preparation, Low Level Soil, Table 3a, Initial Calibration Standards Preparation (Low Level), Aqueous or Table 3B Initial Calibration Standards Preparation, Aqueous.

7.2.1.3. The 'Second Source' standards listed are used in the preparation of the Initial Calibration Verification (ICV) standard (see Tables 4 and 4a for ICV preparation instructions) and the Laboratory Control Standard (LCS) (see Section 9.1.3 and Tables 4 and 4a).

7.2.2 Surrogate Standards: Surrogate standard solutions are prepared from the stock solution (2500ppm)

Surrogate Standard Name	Concentration	Vendor	Catalog #
4-Bromofluorobenzene	2500ppm	Restek	567650
Toluene-d8			
1,2-Dichloroethane-d4			
Dibromofluoromethane			

7.2.2.1 A primary surrogate stock solution (2500 ppm each) is prepared from the neat standards as follows:

7.2.2.2 Secondary surrogate standard solutions are prepared at two (2) levels using the 2500 ppm primary stock solution as detailed in the table below:

Standard Name	Vendor	Catalog #	Volume added	Concentration of Stock Std.	Concentration of Standard	Total Volume Volume in MeOH/Total volume of MeOH
8260 Surrogate Mix: 4-Bromofluorobenzene Toluene-d8 1,2-Dichloroethane-d4 Dibromofluoromethane	Restek	567650	1ml	2500ppm	250ppm	10mL 9.0mL TV/M

Standard Name	Vendor	Catalog #	Volume added	Concentration of Stock Std.	Concentration of Standard	Total Volume Volume in MeOH/Total volume of MeOH
8260 Surrogate Mix: 4-Bromofluorobenzene Toluene-d8 1,2-Dichloroethane-d4 Dibromofluoromethane	Restek	567650	1ml	2500ppm	50ppm	50mL 9.0mL TV/M

7.2.2.3 Methanol/Surrogate solution (2.5ug/mL): For methanol sampling field kits. Prepared by adding 1mL of 2500 ug/ml primary surrogate stock solution (see Section 7.2.2.1) to 1 L purge and trap grade methanol.

7.2.3 **Internal Standards:** Internal Standards Solutions are purchased from Restek:

Standard Name	Concentration	Vendor	Catalog #
8260 Internal Standard Mix: *Chlorobenzene-d5 *1,4-Dichlorobenzene-d4 *Fluorobenzene *1,4-Dioxane-d8 *TBA-d9	250-5000ppm	Restek	567649

7.2.4 **Internal Standard/Surrogate Mix (125 ppm each):** A solution containing both Internal Standards and Surrogates at 125 ppm is prepared in a 10ml volumetric flask as detailed below using the 2500 ppm surrogate stock solution prepared in Section 7.2.2.1 and the 2500 ppm internal standard mix detailed in Section 7.2.3:

Standard Name	Concentration of Stock Std.	Volume added to final volume of 20ml MeOH	Final Concentration of Standard
8260 Internal Standard/Surrogate Mix (125 ppm) For Aquatek Autosampler	2500 ppm Surrogate Mix	1.0ml	125 ppm each component
	250 Internal Std Mix	10 ml	

7.2.5 **Internal Standard/Surrogate Mix (SIM) (2.5/50 ppm each):** A solution containing both Internal Standards and Surrogates at 25 ppm is prepared in a 10ml volumetric flask as detailed below using the 2500 ppm surrogate stock solution prepared in Section 7.2.2.1 and the 250 ppm internal standard mix detailed in Section 7.2.3:

Standard Name	Concentration of Stock Std.	Volume added to final volume of 10ml MeOH	Final Concentration of Standard
8260 Internal Standard/Surrogate Mix (25 ppm) (SIM)	2500 ppm Surrogate Mix	10ul	2.5/50 ppm each component
	250 Internal Std Mix (Restek)	10ul	
1,4-Dioxane-d8	10000 ppm	50ul	

7.2.6 **GC/MS Instrument Performance Check (BFB):** The instrument performance check solution consists of 4-Bromofluorobenzene in addition to the other three surrogates in methanol. Prepare the solution at **50ppm as specified in section 7.2.2.2.** Assign an expiration date of 6 months.

7.2.7 All standards preparation information must be logged into the TALS Reagent Module. All pertinent information must be entered: Date prepared, Lot #'s, Expiration dates, Solvents used, Lab Lot # (expiration date), Manufacturer and Verification signature. Additionally, all prepped standards are typically given a unique Lot# and all information pertaining to standard preparation is entered into the GC/MS VOA Standard Preparation Log Book. Information such as standard supplier, lot number, original concentration, a description of how the standard was made, are required along with the laboratory lot number, analyst's initials, date prepared, expiration date and verification signature. Class "A" volumetric must be used at all times and syringes, preferably gas-tight syringes when available, should be checked for accuracy using an analytical balance. Class "A" pipettes should also be used if volumes permit.

7.2.8 Please refer to TestAmerica Edison SOP No. ED-GEN-008, *Standard Operating Procedure for Preparation, Purity and Storage of Reagents and Standards*, current revision. For Method 8260C:

- Shelf Life of Standard: Gas standards are replaced weekly. Non-gas standards must be replaced monthly.
- Storage Requirements: Stock standards are stored at 4°C and working standards stored at -6°C to -20°C.

8.0 Sample Collection, Preservation, Shipment and Storage

Sample container, preservation techniques and holding times may vary and are dependent on sample matrix, method of choice, regulatory compliance, and/or specific contract or client requests. Listed below are the holding times and the references that include preservation requirements.

Matrix	Sample Container	Min. Sample Size	Preservation	Holding Time	Reference
Waters	Glass 40 ml vials	40 mLs	HCl, pH < 2; Cool 4 °C ± 2°C	14 Days / preserved 7 Days / unpreserved	SW846 Method 5030
Waters	Glass 40 ml vials	40mLs	TSP,pH>11 Cool 4 °C ± 2°C	14 Days / preserved	SW846 Method 5030
Soils (Low)	Encore or Terracore (40 ml vials)	5 grams in 5 mls DI H_2O	Frozen Stored -7°C to -20°C	14 Days	SW846 Method 5035
Soils (Med)	Encore or Terracore (40 ml vials)	5 grams in 10 mls MeOH	Cool 4 °C ± 2°C	14 Days	SW846 Method 5030
Soils (High)	Glass (Lab Prepared Kits)	10 grams in 25 mls MeOH	Cool 4 °C ± 2°C	14 Days	SW846 Method 5030

8.1 Storage blanks are prepared by filling 40 mL VOA vials with reagent water and placing one in each refrigerator. After one week, the storage blanks are removed and analyzed. Additional details can be found in TestAmerica Edison SOP No. ED-SPM-004, *Sample Storage & Handling Procedures for Mitigation of Sample and Laboratory Contamination,* current revision.

9.0 Quality Control

9.1 **Sample QC** - The following quality control samples are prepared with each batch of samples:

Quality Controls	Frequency	Control Limit
Method Blank (MB)	1 in 20 or fewer samples	< Rpt. Limit
Laboratory Control Sample (LCS)[1]	1 in 20 or fewer samples	Statistical Limits [4]
Matrix Spike (MS)[2]	1 in 20 or fewer samples	Statistical Limits [4]
MS Duplicate (MSD)[2]	1 in 20 or fewer samples	Statistical Limits [4]
Surrogates	every sample[3]	Statistical Limits [4]
Internal Standards	Every samples	Response within -50% to +100% of CCV

[1] LCS Duplicate (LCD) is performed only when insufficient sample is available for the MS/MSD or when requested by the client/project/contract.
[2] The sample selection for MS/MSD are randomly selected, unless specifically requested by a client….predetermined by the extraction lab.
[3] Analytical and QC samples (MB, LCS, MS/MSD)
[4] Statistical control limits are updated annually and are updated into LIMS.

9.1.1. **Method blanks** are analyzed every 12 hours immediately after successful calibration verification (ICV and CCV) and before any samples are analyzed during the 12 hour clock. Analyze the blank in the same manner as the associated samples.

9.1.1.1. Prepare an aqueous blank by filling a 40 mL vial with reagent water and placing it in the autosampler. The autosampler will add the internal standard and/or surrogate standard.

9.1.1.2. Prepare a medium or high level blank in a 50 mL volumetric flask by adding 1.0 mL of purge and trap grade methanol to reagent water and bringing up to volume with the reagent water. The appropriate volume of this mix is added to the purge vessel. The autosampler will automatically internal standard and/or surrogate standard.

9.1.1.3. Prepare a low- level soil blank in a 40 ml VOA vial by adding a magnetic stir bar and 5 ml of reagent water and placing the vial in the autosampler tray. An additional 5mL of reagent water plus 1uL of 250ppm Internal Standard/Surrogate Mix (see Section 7.2.4) will be added by the Archon prior to purging.

9.1.1.4. To be considered acceptable, the method blank must not have any target analytes above the reporting limit. If method blanks are unacceptably contaminated with target compounds that are also present in field samples, all affected samples must be re-extracted and re-analyzed. Corrective action must be taken to identify and eliminate the contamination source. Demonstrate that acceptable blanks can be obtained before continuing with sample extraction and analysis. Method blanks must be analyzed on each instrument on which the associated samples are analyzed.

9.1.1.5. Surrogate recoveries for the method blank must be within the laboratory generated limits. (Method 8260C requires the use of a minimum of three (3) surrogates. Since we are spiking with four (4) surrogates, either 1,2-Dichloroethane-d4 or dibromofluoromethane can be recovered outside of control limits without corrective action) .Internal standard area counts in the method blank must be within method specified limits. If any surrogate or internal standard is outside the limits, the method blank must re-analyzed.

9.1.2. Matrix Spike (MS)/Matrix Spike Duplicate (MSD): A matrix spike/matrix spike duplicate (MS/MSD) pair is extracted and analyzed with every 20 environmental samples of a specific matrix (defined as a sample batch which may contain up to 20 samples, and additional samples can be added to the batch for 14 days after the first sample was analyzed). Full compound list spiking is employed for MS/MSDs and LCSs. These spikes are prepared (as described in Section 9.1.2.1) concurrent with sample preparation. MS and MSD recoveries are calculated and compared to lab generated acceptance criteria which are updated annually. For acceptance limits, reference the current TALS (LIMS) active Method Limit Group database.

9.1.2.1. Prepare the MS/MSD as follows:

9.1.2.1.1 Low Level Soil: The low level soil MS/MSD is prepared as detailed in the following table. This is prepared in duplicate (one for the MS, the other for the MSD) in a 5 ml syringe filled with reagent water. Once prepped the solution is added to separate 40 ml vials each containing 5 gram aliquots of the sample to be spiked :

Standard Solution (Reference Table 2, Lab Names)	Concentration	Volume of Standard (ul)Added to 5.0 ml of Reagent Water	Final Concentration (ug/kg)
Gas Mix Li	50ppm	2	20
8260 combined	50ppm	2	20
Acrolein	500 ppm	3	300
Propenes	50ppm (varied)	2	20 (varied)
Freons	50 ppm	2	20

9.1.2.1.2 Aqueous Samples: The MS/MSD for aqueous samples is prepared as detailed in the following table. This is prepared in duplicate (one for MS, the other for MSD) in 50 ml volumetric flasks filled with an aliquot of sample to be spiked. Once prepped the solution is poured into a 40 ml VOA vial and loaded onto the purge and trap autosampler:

Standard Solution (Reference Table 2, Lab Names)	Concentration	Volume of Standard (ul) Added to 50 ml of Sample	Final Concentration (ug/L)
Gas Mix Li	50ppm	20	20
8260 combined	50ppm	20	20
Acrolein	500 ppm	4	40
Propenes	50ppm (varied)	20	20 (varied)
Freons	50	20	20

9.1.2.1.3 Medium & High Level Soils: The MS/MSD for medium/high level soils is prepared as detailed in the following table. This is prepared in duplicate (one for MS, the other for MSD) in 50 ml volumetric flasks filled with reagent water which has been previously spiked with the methanol sample extract. Once prepped the solution is poured into a 40 ml VOA vial, the and loaded onto the purge and trap autosampler:

Standard Solution (Reference Table 2, Lab Names)	Concentration	Volume of Standard (ul) Added to 50 ml of Reagent Water containing sample methanol extract	Final Concentration (ug/L)
Gas Mix Li	50ppm	20	20
8260 conbined	50ppm	20	20
Acrolein	500ppm	4	40
Propenes	50ppm (varied)	20	20 (varied)
Freons	50 ppm	20	20

9.1.2.1.4 **SIM:** The MS/MSD for SIM samples is prepared as detailed in the following table. This is prepared in duplicate (one for MS, the other for MSD) in 100 ml volumetric flasks filled with an aliquot of sample to be spiked. Once prepped, two separate 10ml solution is poured into 40 ml VOA vials, 2ul of SIM IS/S is then added to each vial and loaded onto the purge and trap autosampler:

Standard Solution	Concentration	Volume of Standard Added to 100 ml of Sample (ul)	Final Concentration (ug/L)
8260SIM Mix1	10ppm	0.5	0.05
1,4-Dioxane	50ppm (varied)	10	5
Benzene/Chloroform	10ppm	0.5	0.05

9.1.2.2. An Laboratory Control Sample (LCS) /Laboratory Control Sample Duplicate (LCSD) may be substituted for the MS/MSD if insufficient sample volume is available (see Section 9.1.3).

9.1.3. Laboratory Control Sample (LCS)/Laboratory Control Sample Duplicate (LCSD): A Laboratory Control Sample (LCS) (aka blank spike) must be prepared analyzed with each batch of 20 environmental samples. The LCS data is used to assess method performance if the MS/MSD recoveries fall outside of the lab generated limits (see For acceptance limits, reference the current TALS (LIMS) active Method Limit Group database). If the LCS recovery is within the current lab generated limits, the MS/MSD recoveries are attributed to matrix interference. If the LCS recovery results are outside the method specified, the LCS is reanalyzed. If, upon reanalysis, the LCS is it is still outside of limits the entire batch must be reanalyzed.

9.1.3.1 For LCS preparation instructions please refer to Section 9.1.2.1 for low level soil introduction technique (note: use reagent water only, no solid matrix is used when preparing the LCS) and Sections 9.1.2.1.2 and 9.1.2.1.3 as applicable for aqueous/medium or high level solids introduction (note: use reagent water only, no sample or sample extract is used when preparing the LCS).

9.1.3.2 The LCS for SIM samples is prepared as detailed in the following table. This is prepared in a 200 ml volumetric flasks filled with organic free reagent water. Once prepped, 10ml of the solution is poured into a 40 ml VOA vial and 2ul IS/SS added manually and loaded onto the purge and trap autosampler

Standard Solution	Concentration	Volume of Standard Added to 200 ml of Reagent Water (ul)	Final Concentration (ug/L)
8260 Mix1	10ppm	1	0.05
1,4-Dioxane	50ppm	20	5
Benzene/Chloroform	10ppm	1	0.05

9.1.3.3 A Laboratory Control Sample Duplicate (LCSD) is analyzed only when insufficient client sample is available for preparation of an MS/MSD pair. The LCS/LSCD is evaluated in the same manner as the MS/MSD (see Section 9.1.2)

9.1.4. Surrogate Standards: All samples, blanks and QC samples are spiked with a four (4) component surrogate standard mix (see Section 7.2.2). The percent recovery of the surrogate standards is calculated and compared to lab generated limits (For acceptance limits, reference the current TALS (LIMS) active Method Limit Group database).

9.1.4.1. Surrogate recovery limits are lab generated and are updated annually.

9.1.4.2. Surrogate recoveries are calculated for the blank, samples, and QC samples. Surrogate recovery is calculated as:

$$\frac{\text{Concentration found}}{\text{Concentration added}} \times 100 = \%\ \text{RECOVERY}$$

9.1.4.3. If the surrogate recoveries of any blank, sample, or QC sample fails to meet the current recovery criteria, the sample must be re-analyzed. If a surrogate is diluted to a concentration below that of the lowest calibration standard, no corrective action is

necessary. Method 8260C requires the use of a minimum of three (3) surrogates. As we spike with four (4) surrogates, one can be recovered outside of control limits without corrective action.

9.1.5. Internal Standards: All samples, blanks, standards and QC samples are spiked with a three (3) component internal standard mix (See Section 7.2.3). The response (area count) and retention time of each internal standard in all samples, standards, blanks and QC samples are monitored.

9.1.5.1. The internal standard responses must be within -50 +100% of its corresponding internal standard in the mid-level calibration standard or the active calibration curve. Failure to meet these criteria is indicative of sample matrix effects. All samples failing these criteria must be reanalyzed to confirm matrix effects.

9.1.5.2. Internal standard retention time is evaluated immediately after acquisition. The retention times of the internal standards must be within ±30 seconds of the internal standards from the mid point standard of the initial calibration or the calibration verification standard. Any blank, sample, or QC sample that fails to meet these criteria must be re-analyzed.

9.2 Instrument QC

9.2.1 GC/MS Instrument Performance Check (BFB): The GC/MS system is tuned using Perfluortributylamine (PFTBA) such that an injection or purging of 50ng of 4-Bromofluorobenzene (BFB) meets the abundance criteria listed in the table below. Prior to the analysis of any calibration standards or samples, the GC/MS system must meet all BFB key ion abundance criteria. This analysis will verify proper tuning of the system for a period of 12 hours post-injection. After 12 hours, the instrument performance must again be verified prior to the analysis of standards, QC or samples.

BFB Key Ions and Abundance Criteria	
Mass	**Ion Abundance Criteria**
50	15.0-40.0 percent of the base peak
75	30.0-60.0 percent of the base peak
95	Base peak, 100% relative abundance
96	5.0-9.0 percent of the base peak
173	Less than 2.0% of mass 174
174	Greater than 50% of the base peak
175	5.0-9.0 percent of mass 174
176	Greater than 95.0% but less than 101% of mass174
177	5.0-9.0 percent of mass 176

9.2.1.1. The BFB mass spectrum may be evaluated using one of the procedures listed below. The spectrum may be background

subtracted using a single peak no more than 20 scans before the peak apex. The BFB spectrum must meet the technical acceptance criteria listed in the table above:

- A single scan on the peak;
- An average of the peak;
- Use of three scan averaging and background subtraction techniques. Select the scan at the BFB peak apex, add +1 scan from the apex and -1 scans from the apex;

9.2.1.2. BFB parameter settings are stored in a tune file, which ill be used in all subsequent analysis of standards and samples.

9.2.2 Initial Calibration Range and Initial Calibration Verification

9.2.2.1. Initial Calibration: The initial calibration range consists of a five-point concentrations (six points for second order regression) of analytical standards prepared as described in Tables 3, 3A and 3B as applicable (attached). The initial calibration range must be analyzed only after the BFB instrument performance check has met the criteria in Section 9.2.1. A separate initial calibration range is analyzed for each sample introduction technique.

9.2.2.2. If analysis by the SIM technique is required, prepare calibration standards for, Vinyl Chloride, Chloroform, Benzene 1,2-dibromoethane, 1,2,3-Trichloropropane and 1,2-dibromo-3-chloropropane at concentrations of 0.02, 0.04, .05, 0.10, 0.20, 0.50, 1.0 and 2.0 ppb; 1,4-Dioxane at 0.4, 1, 5, 10, 20, 30, 40 and 50ppb. See Table 5 that summarizes the preparation information.

9.2.2.3. Initial Calibration Verification (ICV): An Initial Calibration Verification (ICV) standard is analyzed immediately after the Initial Calibration Range and before any samples are analyzed. The ICV is prepared as detailed in Section 7.2.1.3 and Tables 4 and 4a (full scan) and Table 6 (SIM) (attached). The ICV must be from a source separate from the standards used in the Initial Calibration Range.

9.2.3 Continuing Calibration Verification (CCV): A approximately mid-point (20ug/ml and 0.050/5ug/ml for SIM) Continuing Calibration Verification (CCV) must be analyzed every 12 hours after the BFB instrument performance check. The CCV is prepared as detailed in Section 7.2.1.1 and Table 3 (attached).

9.2.4 Calibration Acceptance Summary

9.2.4.1. Retention Time: The relative retention times of each compound in the five calibration standards must agree within 0.06 relative retention time units.

9.2.4.2. Initial Calibration Range: Internal standard calibration is employed for this method. After the initial calibration range has been analyzed as detailed in Section 10.3.3 the relative response factor (RRF) for each target/surrogate compound at each concentration level is determined using the following equation.

$$RRF = \frac{A_x}{A_{is}} \times \frac{C_{is}}{C_x}$$

Where:
A_x = Area characteristic ion for the compound (see attached Table 7)
Ais = Area characteristic ion of internal standard (see attached Table 7)
Cis = Concentration of internal standard
Cx = Concentration of compound in standard

9.2.4.2.1. Determine the mean RRF for each compound using the five or six RFs from the initial calibration range.

9.2.4.2.2. The average RFs of the target analytes listed in the table below must meet the indicated minimum RF criteria:

Minimum Relative Response Factor	
Common Target Analytes	**Minimum RF**
Dichlorodifluoromethane	0.100
Chloromethane	0.100
Vinyl Chloride	0.100
Bromomethane	0.100
Chloroethane	0.100
Trichlorofluoromethane	0.100
1,1-Dichloroethene	0.100
1,1,2-Trichloro-1,2,2-trifluoroethane	0.100
Acetone *	0.050
Carbon disulfide	0.100
Methyl Acetate *	0.005
Methylene chloride	0.100
trans-1,2-Dichloroethene	0.100
cis-1,2-Dichloroethene	0.100
Methyl tert-Butyl Ether	0.100
1,1-Dichloroethane	0.200
2-Butanone *	0.050
Chloroform	0.200
1,1,1-Trichloroethane	0.100
Cyclohexane	0.100
Carbon tetrachloride	0.100
Benzene	0.500
1,2-Dichloroethane	0.100
Trichloroethene	0.200
Methylcyclohexane	0.100

Minimum Relative Response Factor	
Common Target Analytes	**Minimum RF**
1,2-Dichloropropane	0.100
Bromodichloromethane	0.200
cis-1,3-Dichloropropene	0.200
trans-1,3-Dichloropropene	0.100
4-Methyl-2-pentanone *	0.050
Toluene	0.400
1,1,2-Trichloroethane	0.100
Tetrachloroethene	0.200
2-Hexanone*	0.050
Dibromochloromethane	0.100
1,2-Dibromoethane	0.100
Chlorobenzene	0.500
Ethylbenzene	0.100
meta-/para-Xylene	0.100
ortho-Xylene	0.300
Styrene	0.300
Bromoform	0.100
Isopropylbenzene	0.100
1,1,2,2-Tetrachloroethane	0.300
1,3-Dichlorobenzene	0.600
1,4-Dichlorobenzene	0.500
1,2-Dichlorobenzene	0.400
1,2-Dibromo-3-chloropropane	0.050
1,2,4-Trichlorobenzene	0.200

Note: Alternate ions chosen for the analytes in the table above may result in lower than recommended value

* These values are lower than method recommended values.

9.2.4.2.3. Any individual analyte that fails the minimum response factor above must have a demonstration of sensitivity in the analytical batch to report non-detects. The demonstration of sensitivity is analysis of a low level CCV (at or below the reporting limit). The criterion for a passing LLCCV is detection only, and a passing LLCCV allows non-detects to be reported without flagging. The low level CCV would normally be analyzed immediately after the mid-level CCV

9.2.4.2.4. Calculate the Standard Deviation (SD) and Percent Relative Standard Deviation (% RSD) of the response factors for each compound:

$$\% \text{ RSD} = \frac{\text{Standard Deviation of RRFs}}{\text{Mean RRF}}$$

The % RSD of the common target compounds listed above must be ≤20% in order for the calibration range to be acceptable. If more than 10% of the compounds exceed the 20%RSD limit and do not meet the minimum correlation coefficient (0.99) for alternative curve fits, appropriate instrument maintenance like source cleaning should be performed. Any compound that do not meet the 20% RSD or 0.99 correlation coefficient criteria must be flagged as estimated for detects.

9.2.4.2.5. For all compounds (including those analyzed by SIM): in order to assume linearity, the % RSD of the RRF's for each target analyte must be ≤20%.

9.2.4.2.6. If the above listed criteria is met, the system can be assumed to be linear, sample analysis may begin and the average RF from the initial calibration range may be used to quantitate all samples.

9.2.4.2.7. An alternative calibration technique may be employed for those any compounds exceeding the 20% RSD criteria:

9.2.4.2.6.1 **Linear regression**: Calculate the first order linear regression for any compound which did not meet the 20% RSD criteria. The r value (Correlation Coefficient) of the equation must be ≥0.99 for linear regression to be employed.

9.2.4.2.6.2 **Quadratic (or second order) regression**: may be used if the linear regression correlation coefficient exceeds criteria. Quadratic regression requires the use of a minimum six calibration points. If second order regression calibration is used, the r^2 (Correlation Coefficient) value must be ≥ 0.99

9.2.4.2.8. If neither of the alternative calibration techniques meets acceptance criteria i.e for more than 10% of the analytes fail both 20%RSD and 0.990 the calibration is not valid. Corrective action must be taken and the initial calibration range reanalyzed.

9.2.4.2.9. Non-detect results for any analyte that fails both 20%RSD and 0.990 correlation coefficient may be reported without flagging if (and only if) there has been a successful analysis of a LLCCV (CCV at the reporting limit) in the same analytical batch. The criterion for the LLCCV is detection only (%D criteria are not applied) but the standard qualitative criteria in the method must be met. Flagging of detected analytes results as estimated is discouraged when the 20%RSD and 0.990 criteria

fails. In general no more than one or two of the poorest performing analytes should fail both criteria.

9.2.4.2.10. Due to significant bias to the lower portion of a calibration curve using the linear regression fit model a quantitation check on the viability of the lowest calibration point should be performed by re-fitting the response from the low concentration calibration standard back into the curve as if it were an unknown sample (rename the lower point calibration file as a separate data file before re-processing). The results should be within ±30% of the standard's true concentration. This is not required for average RF or quadratic fits. Additionally forcing a linear regression through zero will meet the requirement of not re-fitting. Analytes which do not meet the minimum quantitation calibration re-fitting criteria should be considered 'out of control'. Report those target analyte outliers as estimated when the concentration is at or near the lowest calibration point and/or report to the next reporting level (i.e., the next higher calibration point for the analyte).

9.2.4.2.11. For additional detail refer to TestAmerica Edison Work Instruction No. EDS-WI-096, *8260C ICAL Procedure*, latest revision.

9.2.4.3. Initial Calibration Verification (ICV): Once the initial calibration has been analyzed and has met the above criteria, a second source Initial Calibration Verification (ICV) (as prepared in Section 9.2.2.2) must be analyzed and evaluated. The ICV must meet the criteria of 70-130% recovery for all compounds however up to 10% of the compounds are allowed to exceed this criteria as long as their recoveries are within 65-135%. For the poor performers the range is 50-150%. If the criterion is not met, a second ICV may be analyzed after corrective measures are taken. If a second ICV analysis fails to meet criteria proceed with corrective action and the analysis of a new initial calibration range. Flagging: If the ICV limits are outside of criteria (high) for an analyte and that analyte is undetected in the sample, no flagging or narration is required. If the ICV limits are outside of criteria (low) for an analyte and that analyte is undetected in a sample, narrate the non-conformance in an NCM. When that out of spec analyte is detected in a sample, describe the issue in the narrative, or flag as estimated.

9.2.4.4. Continuing Calibration Verification (CCV): A CCV consisting of a standard at or near the midpoint of the Initial Calibration Range is analyzed every 12 hours of instrument operation or at the beginning of an analytical sequence to verify the initial calibration. The calibration verification consists of a BFB

instrument performance check, and analysis of a calibration verification standard.

9.2.4.4.1 Tune Verification: Follow the procedure for verifying the instrument tune described in section 9.2.1 using a 50 ng injection of BFB. If the tune cannot be verified, analysis must be stopped, corrective action taken and a return to "control" demonstrated before continuing with the calibration verification process.

9.2.4.4.1.1 Calibration Verification: Analyze the calibration verification standard immediately after a BFB that meets criteria. Use the mid point calibration standard (20ug/L). **NOTE:** The same sample introduction technique employed for the initial six-point calibration must be used for the calibration verification.

9.2.4.4.1.2 Calculate response factors (RF) for each compound using the internal standard method.

9.2.4.4.1.3 The RFs must meet the minimum RF criteria listed in the table in Section 9.2.4.2.2.

9.2.4.4.1.4 Calculate the % Difference for each response factor in the calibration check standard vs. the response factors from the initial calibration.

9.2.4.4.1.5 If the percent difference/drift (%D) for the compounds listed in the table in Section 9.2.4.2.2 is ≤20%, the initial calibration is assumed to be valid. If the ≤20% D criteria is not met for more than 20% of the compounds in the initial calibration, corrective action/ investigation may be taken. After corrective action, another calibration verification standard may be injected. If the response for the analyte is still not ≤20%, a new initial calibration range must be generated.

9.2.4.4.1.6 For the poor performing compounds listed below that fail the 20%D or 50%D criteria adequate sensitivity may be demonstrated by including a low level standard (LLCCV) in the analytical batch.

Poor Performers	
Acetone	Acrolein
Carbon disulfide	1,4-Dioxane
2-Butanone	Cyclohexane
2-Hexanone	Methyl cyclohexane
4-Methyl-2-pentanone	Benzyl chloride
Chlorodibromomethane	Naphthalene
1,2-Dibromo-3-chloropropane	Cis-Dichloropropene
Bromomethane	Trans-Dichloropropene
Chloroethane	All Alcohols

When samples have non-detects for an analyte that fails the SOP criteria with low recovery a low level CCV must be analyzed in the batch as a demonstration of adequate sensitivity. The criterion for a passing LLCCV is detection only, and a passing LLCCV allows non-detects to be reported without flagging. Any sample detects for an analyte that fails the SOP criteria must be flagged as estimated, or detailed in the case narrative. In all cases every effort should be made to re-analyze on an instrument with a passing CCV.

9.2.4.4.1.7 Percent drift is used instead of percent difference in calibrations employing either the linear or second order regression modes.

9.2.4.4.1.8 For the compounds not listed in the table in Section 9.2.4.2.2: No one individual compound of interest may exceed 50%D. For SIM analysis the %D is 20%.

9.2.4.4.1.9 The retention times of the internal standards from the calibration check must be within ±30 seconds of the internal standards from the mid point standard of the original calibration. If the retention time for any internal standard changes by more than 30 seconds from the latest daily (12 hour) calibration standard, the chromatographic system is inspected for malfunctions, and corrections made as required. If corrective action does not result in the retention time criteria being achieved, the system must be re-calibrated using four additional standards.

9.2.4.4.1.10 Internal standard area response is also evaluated immediately after acquisition. The response (area count) of each internal standard in the calibration verification standard must be within 50% - 100% of its

corresponding internal standard in the mid-level calibration standard of the initial calibration curve. If the EICP area for any internal standard changes by more than a factor of two (-50% to +100%), the mass spectrometer system must be inspected for malfunction and corrections made as appropriate. When corrections are made, re-analysis of samples analyzed while the system was malfunctioning is required.

10.0 Procedure

10.1. Gas Chromatograph/Mass Spectrometer Operation

10.1.1. The instrument operating parameters are set as follows at the beginning of a method of analysis and remain constant throughout the entire analytical procedure

10.1.1.1 Full Scan Operating Mode

Purge and trap unit

Purge Time:	11 minutes
Dry Purge:	1 Minutes
Purge Gas:	Nitrogen
Purge Flow:	40-45 ml/min
Purge Temp:	Water: Ambient; Solids: 40°C
Trapping Temp:	Ambient, <30°C
Desorb Time:	1 Minute
Desorb Temp:	VOCARB: 260°C, #10: 190°C

Gas chromatograph

Injector:	180°C
Carrier Gas:	Helium
Carrier Flow:	6 ml/min, 6890: 0.8 ml/min
Oven Program:	40°C for 1 min, 8°C/min to 90°C, 20°C/ min to 250°C for 3 min; 6890: 40°C for 1 min, 8°C/min to 100°C, 24°C/min to 220°C for 2 min
Run Time:	15 - 20 Minutes

Mass Spectrometer

Electron Energy:	70 volts (nominal)
Mass range:	35-260 AMU
Scan time:	0.9 sec./scan
Source Temp:	200°C
Separator Temp:	180°C

10.1.1.2 SIM Operating Mode

Purge and trap unit

Purge Time:	11 minutes
Dry Purge:	1 Minutes
Purge Gas:	Nitrogen
Purge Flow:	40-45 ml/min
Purge Temp:	Water: Ambient; Solids: 40°C
Trapping Temp:	Ambient, <30°C
Desorb Time:	1 Minute
Desorb Temp:	VOCARB: 260°C, #10: 190°C

Gas chromatograph

Injector:	180°C
Carrier Gas:	Helium
Carrier Flow:	6 ml/min, 6890: 0.8 ml/min
Oven Program:	40°C for 1 min, 8°C/min to 90°C, 20°C/ min to 250°C for 3 min; 6890: 40°C for 1 min, 8°C/min to 100°C, 24°C/min to 220°C for 2 min
Run Time:	15 - 20 Minutes

Mass Spectrometer

Electron Energy:	70 volts (nominal)
Mass range:	35-260 AMU
Scan time:	0.9 sec./scan
Source Temp:	200°C
Separator Temp:	180°C

SIM Parameters:

Group 1
Plot 1 Ion: 51.0/96

Ions/Dwell in Group	(Mass	Dwell)	(Mass	Dwell)	(Mass	Dwell)
	51.0	100	58.0	100	65.0	100
	67.0	100	70.0	100	88.0	100
	96.0	100	78.0	100	83.0	100
	85.0	100	62.0	100	64.0	100

Group 2
Group Start Time: 6.20
Plot 1 Ion: 82/117

Ions/Dwell in Group	(Mass	Dwell)	(Mass	Dwell)	(Mass	Dwell)
	82.0	100	107.0	100	109.0	100
	117.0	100				

Group 3
Group Start Time: 8.50
Plot 1 Ion: 75/157

Ions/Dwell in Group	(Mass	Dwell)	(Mass	Dwell)	(Mass	Dwell)
	75.0	100	95.0	100	150.0	100
	152.0	100	152.0	100	157.0	100
	174.0	100				

10.2. Sample Preparation

10.2.1. Screening: All samples extracts must be screened by GC/FID static headspace analysis to provide the analyst with appropriate initial dilution factors. For additional details see TestAmerica Edison SOP No. ED-GCV-001, *Screening for Volatile Organics, Static Headspace with GC FID, SW846 Method 5021*, current revision.

10.2.2. Aqueous Samples: Unopened 40 mls vials with aqueous samples are placed in an Archon autosampler. 1 uL of Internal Standard/Surrogate Mix (see Section 7.2.4) is added by the Archon as the 5 mL of the sample passes through the sample loop.

10.2.3. Medium or high level soils: Medium or high level extracts that will be run on an Archon autosampler are prepared in 50mL volumetric flasks. The Archon can be set up to add 1uL of 250ppm Internal Standard/Surrogate separately (see Section 7.2.3 and 7.2.2.2) to each sample as the 5mL portion passes through the sample loop.

10.2.4. Low level soils: Low level soils must be run on an Archon autosampler. 1uL of 250ppm Internal Standard/Surrogate separately (see Section 7.2.3 and 7.2.2.2) and 5mL reagent water is added to each sample vial by the Archon immediately before the sample is purged.

10.2.5. SIM analysis: Aliquot 10ml of sample and manually add 2ul of 2.5/50ppm of internal standard/surrogate mix. Load to soil section of the autosampler for heated purge.

10.3. Instrument Performance and Calibration Sequence

10.3.1. Once the GC/MS instrument has been setup and maintained as detailed in Section 10.1, the first operations to be performed are the performance checks and calibration standards.

10.3.2. Analyze the Instrument Performance Check Standard (BFB) as discussed in Section 9.2.1.

10.3.3. A unique initial calibration is then prepared for each sample introduction technique.:

10.3.3.1 **40 ml VOA Vial (Aqueous/Medium-High Level Soils):** Prepare aqueous calibration standards at six concentration levels for each parameter by adding the volumes of working standards listed in Table 3 to a 50mL volumetric flask of reagent water. Pour the calibration standards into 40mL VOA vials and load into the autosampler tray. If the internal standard is to be added by the Archon/OI autosamplers the addition of internal standard into the 50ml volumetric flaks may be omitted.

10.3.3.2 **40 ml VOA Vial (Low Level Soils):** If the calibration is for low-level soils prepared according to Method 5035, the calibration standards must be prepared by adding the volumes of working standards listed in Table 3 into a 5 mL syringe filled with reagent water and pouring the prepared standards into 40 mL VOA vials containing a magnetic stir bar.

10.3.4. Purge the standard for 11 minutes.

10.3.5. After purging is complete, desorb the sample onto the GC column by rapidly heating the trap to 260°C for VOCARB, 190°C for #10 and backflushing it with helium.

10.3.6. Begin the GC temperature program and data acquisition.

10.3.7. Re-condition the trap by baking for 12 minutes at 260°C for VOCARB, 210°C for #10.

10.3.8. Cool the trap to (<31°C). The trap is now ready for the next sample.

10.3.9. Transfer data to network, and process using CHROM software.

10.4. Sample Analysis Sequence

10.4.1. Once the initial calibration has been verified by successful analysis of an ICV and Method Blank, analysis of samples may begin.

10.4.2. Samples must be analyzed under the same instrument conditions and using the same injection volume as the calibration standards.

10.4.3. Equilibrate all samples to room temperature prior to analysis.

10.4.4. If the sample concentration exceeds that of the range, the sample must be diluted and re-analyzed.

10.4.5. The analytical run log is printed as a record of samples analyzed. The analyst will annotate the run log with any required information regarding anomalies or unusual events. The run log must be signed by the analyst and a reviewed and signed by a trained peer or manager

10.5. Data Processing

10.5.1. Prior to processing any standards or samples, target compound lists and sublists must be assembled in the Chrom system. These lists are required for processing of all data files including calibration files. The data includes compound names, retention time data, quantitation ions, qualitative identification ions, and the assigned internal standard for qualitative and quantitative identification.

10.5.2. Key data is manually entered the first time a compound list is used for data processing. Processing data using a compound list automatically generates response factor data and updates retention information.

10.5.3. Data is transferred from the acquisition PC to the network for auto-processing with CHROM software.

10.5.4. Each data file is checked for correct information including sample number, job number, QA batch, dilution factor, initial volume, final volume, and % moisture.

10.5.5. The data processing service from Chrom queries LIMS for the sample processing parameters.

10.5.6. Each data file is processed using calibration factors from the most recent initial calibration, quantitation from the daily calibration verification standard is not permitted.

10.5.7. The characteristic ions for target compounds, surrogate compounds, and internal standards which can be determined using SW8260CB are listed in Table 7.

10.6. Interpretation and Qualitative Identification:

10.6.1 Target Analytes: Qualitative identification of target compounds is based on retention time and mass spectral comparison with characteristic ions in the target compound list. The reference mass spectrum is taken from a standard of the target compound analyzed by this method. The characteristic ions are the three ions of greatest relative intensity or any ions over 30% relative intensity if less than three such ions occur in the reference spectrum. Compounds are identified as present when the following criteria are met:

10.6.1.1. Once the GC/MS instrument has been setup and maintained as detailed in Section 10.1, the first operations to be performed are the performance checks and calibration standards.

10.6.1.2. The intensities of the characteristic ions of a compound maximize in the same scan or within one scan of each other.

10.6.1.3. The relative retention time (RRT) of the sample component is within ± 0.06 RRT units of the RRT of the standard component.

10.6.1.4. The most abundant ion in the standard target spectrum that equals 100% MUST also be present in the sample target spectrum.

10.6.1.5. All other ions that are greater than 10% in the standard target spectra should also be present in the sample.

10.6.1.6. The relative intensities of the characteristic ions agree within 30% of the relative intensities of these ions in the reference spectrum. (Example: For an ion with an abundance of 50% in the reference spectrum, the corresponding abundance in a sample spectrum can range between 20% and 80%).

10.6.1.7. Structural isomers that produce very similar mass spectra should be identified as individual isomers if they have sufficiently different GC retention times. Otherwise, structural isomers are identified as isomeric pairs.

10.6.1.8. If the compound does not meet all of the criteria listed above, but is deemed a match in the technical judgment of the mass spectral interpretation specialist, the compound will be positively identified and reported with documentation of the identification noted in the raw data record.

10.6.2 Non-Target Analytes: Upon client request a library search to identify non-target Tentatively Identified Compounds (TIC) is performed. The NIST/EPA/NIH mass spectral library is used to identify non-target compounds (not including internal standard and surrogate compounds) of greatest apparent concentration by a forward search of the library. The following guidelines are used by the analyst when making TIC identifications:

10.6.2.1 Relative intensities of major ions in the reference spectrum (ions greater than 10% of the most abundant ion) should be present in the sample spectrum.

10.6.2.2 The relative intensities of the major ions should agree within ±20%. (Example: For an ion with an abundance of 50% in the standard spectrum, the corresponding sample ion abundance must be between 30 and 70%).

10.6.2.3 Molecular ions present in the reference spectrum should be present in the sample spectrum.

10.6.2.4 Ions present in the sample spectrum but not in the reference spectrum should be reviewed for possible background contamination or presence of co-eluting compounds.

10.6.2.5 Ions present in the reference spectrum but not in the sample spectrum should be reviewed for possible subtraction from the sample spectrum because of background contamination or co-eluting peaks. Data system library reduction programs can sometimes create these discrepancies.

10.6.2.6 If, in the technical judgement of the mass spectral interpretation specialist, no tentative identification can be made, the compound will be reported as 'Unknown'. If the compound can be further classified the analyst may do so (i.e, 'Unknown hydrocarbon', 'Unknown acid' , etc..).

10.7. Data Reporting

10.7.1. Final Report. LIMS TALS system automatically produces a data report consisting of key, hardcopy reports corresponding to specific data reporting requirements.

10.7.1.1. Total Ion Chromatogram. Full length chromatogram depicting the full length of the GC/MS acquisition.

10.7.1.2. Spectra of all detected target compounds. A page for each detected target compound spectra with a standard reference spectrum for comparison.

10.7.1.3. The calculations of the concentrations of each target compound in the sample, reported in units of ppb, ug/kg or ug/l.

10.7.1.4. Data summaries for each method blank indicating which samples were extracted with the indicated blank.

10.7.1.5. A copy of the initial calibration range together with the calibration verification report, and tune report.

10.7.1.6. Quality Control (QC) data report for each batch including surrogate recoveries, internal standard area summaries, LCS, MS/MSD and RPD summaries.

11.0. Calculations / Data Reduction

11.1. Target Compounds: are quantitated using the internal standard method.

11.1.1. Identified target compounds are quantitated using the integrated abundance from the EICP of the primary characteristic ion. The internal standard used shall be the one nearest the retention time of the analyte).

11.1.2. The average response factor (RRF) from the initial calibration is used to calculate the target analyte concentration in client samples using the formula found in Section 11.3.. See Section 9.2.4.2 for discussion of RRF.

11.1.3. Secondary ion quantitation is utilized only when there are sample interferences preventing use of the primary characteristic ion. If secondary ion quantitation is used an average relative response factor (RRF) must be calculated using that secondary ion.

11.1.4. Aqueous Samples

$$\text{Concentration } (\mu g/L) = \frac{(As)(Cis)(D)}{(Ais)(RRF)(Vs)}$$

Where:

As	=	Area of the characteristic ion for the target analyte in the sample
Cis	=	Concentration of the internal standard (ug/L)
D	=	Dilution factor, if the sample or extract was diluted prior to analysis. If no dilution is performed, D = 1.
Ais	=	Area of the characteristic for the associated internal standard
RRF	=	Average relative response factor from the initial calibration.
Vs	=	Volume of sample purged (ml)

11.1.5. Low Level Solid Samples

$$\text{Concentration } (\mu g/Kg) \text{ (dry wt)} = \frac{(As)(Cis)}{(Ais)(RRF)(Ws)\ (DW)}$$

Where:

As	=	Area of the characteristic ion for the target analyte in the sample
Cis	=	Concentration of the internal standard (ug/L)

DW = Dry wt correction = $\frac{100 - \%\ \text{moisture}}{100}$

Ais = Area of the characteristic for the associated internal standard

RRF = Average relative response factor from the initial calibration.

Ws = Weight of sample purged (g)

11.1.6. Medium Level Solid Samples

$$\text{Concentration (µg/Kg) (dry wt)} = \frac{(As)(Cis)(Vt)(1000)(D)}{(Ais)(RRF)(Va)(Ws)(DW)}$$

Where:

As = Area of the characteristic ion for the target analyte in the sample

Cis = Concentration of the internal standard (ug/L)

D = Dilution factor, if the sample or extract was diluted prior to analysis. If no dilution is performed, D = 1

DW = Dry wt correction = $\frac{100 - \%\ \text{moisture}}{100}$

Ais = Area of the characteristic for the associated internal standard

RRF = Average relative response factor from the initial calibration.

Va = Volume of the aliquot of sample methanol extract added to reagent water for purging in ul

Vt = Total volume of methanol extract in milliliters

Ws = Weight of sample purged (g)

11.2. Non-Target Compounds (Tentatively Identified Compounds): An estimated concentration for non-target (tentatively identified compounds) is calculated using the internal standard method . For quantiation, the nearest eluting internal standard free of interferences is used. The procedure used for calculating the concentration of non-target compounds is the same as that used for target compounds (see Section 11.1) with the following revisions:

11.2.1. The total area count of the non-target compound is used for As (instead of the area of a characteristic ion).

11.2.2. The total area count of the chosen internal standard is used as Ais (instead of the area of a characteristic ion).

11.2.3. A RF on 1.0 is assumed.

11.2.4. The resulting concentration is qualified as estimated ('J') indicating the quantitative uncertainties of the reported concentration.

11.3. Relative Response Factors

$$RRF = \frac{A_x}{A_{is}} \times \frac{C_{is}}{C_x}$$

Where:

A_x = Area characteristic ion for the compound (see Table 7)
Ais = Area characteristic ion of associated internal standard (See Table 7)
Cis = Concentration of internal standard
Cx = Concentration of compound in standard

11.4. Percent Relative Standard Deviation (% RSD) : as discussed in Section 9.2.4.2. (Initial calibration):

$$\% \text{ RSD} = \frac{\text{Standard Deviation of RRFs}}{\text{Mean RRF}}$$

11.5. Percent Difference (% D):as discussed in Section 9.2.4.4 (Continuing calibration):

$$\% D = \frac{RRF_c - \overline{RRF_i}}{\overline{RRF_i}} \times 100$$

Where: RRFc = RRF from continuing calibration

$\overline{RRF_i}$ = Mean RRF from current initial calibration

11.6. Percent Recovery (% R): Surrogates and Spikes

$$\text{Recovery (\%)} = \frac{\text{Concentration (or amount) found}}{\text{Concentration (or amount) added}} \times 100$$

11.7. Dry Weight Correction: All solid samples must be corrected for dry weight using the following formula for dry weight determination.

$$DW = \frac{Gd}{Gw} \times 100$$

Where:

DW	=	Percent % Dry Weight
Gd	=	Dry weight of selected sample aliquot
Gw	=	Wet weight of selected sample aliquot

Multiply the DW value times the wet weight of the sample extracted. NOTE: This calculation can also be performed automatically by the target system provided the DW value is available and entered into the system.

11.8. Accuracy:

$$\text{ICV , CCV and LCS \% Recovery} = \frac{\text{observed concentration}}{\text{known concentration}} \times 100$$

$$\text{MS \% Recovery} = \frac{\text{(spiked sample) - (unspiked sample)}}{\text{spiked concentration}} \times 100$$

11.9. Precision (RPD):

$$\text{Matrix Duplicate (MD)} = \frac{|\text{orig. sample value - dup. sample value}|}{[(\text{orig. sample value + dup. sample value})/2]} \times 100$$

12.0 Method Performance

12.1. Method Detection Limit Study (MDL)

The method detection limit (MDL) is the lowest concentration that can be detected for a given analytical method and sample matrix with 99% confidence that the analyte is present. The MDL is determined according to the laboratory's MDL procedure in Section 20 (Test Methods and Method Validation) of TestAmerica Edison's Quality Assurance Manual (ED-QA-LQM). MDLs reflect a calculated (statistical) value determined under ideal laboratory conditions in a clean matrix, and may not be achievable in all environmental matrices. The laboratory maintains MDL studies for analyses performed; these are verified at least annually unless method requirements require a greater frequency.

12.2. Demonstration of Capabilities

For DOC procedure refer to Section 20 in the most current revision of TestAmerica Edison's Quality Assurance Manual (ED-QA-LQM).

12.3. Training Requirements

Refer to TestAmerica Edison SOP No. ED-GEN-022, *Training*, current revision for the laboratory's training program.

13.0 Pollution Control

13.1. It is TestAmerica's policy to evaluate each method and look for opportunities to minimize waste generated (i.e., examine recycling options, ordering chemicals based on quantity needed, preparation of reagents based on anticipated usage and reagent stability). Employees must abide by the policies in Section 13 of the Corporate Environmental Health and Safety Manual (CW-E-M-001) for "Waste Management and Pollution Prevention."

14.0 Waste Management

14.1. Waste management practices are conducted consistent with all applicable rules and regulations. Excess reagents, samples and method process wastes are disposed of in an accepted manner. Waste description rules and land disposal restrictions are followed. Waste disposal procedures are incorporated by reference to TestAmerica Edison SOP No. ED-SPM-008, *Laboratory Waste Disposal Practices*, current revision. The following waste streams are produced when this method is carried out.

- Laboratory Generated Aqueous Waste (aqueous VOA vials – used and unused). This waste may have a pH of less than 2.0. These vials are collected in satellite accumulation. The vials are then transferred to the waste room. These vials are passed through a vial crusher and the liquid portion is separated from the solid portion. The solid is dumped into the municipal garbage. The liquid is pumped into the neutralization system where it is neutralized to a pH of 6 to 9 with sodium bicarbonate (Seidler Chemical SC-0219-25). When neutralization is complete, the material is transferred to the municipal sewer system**.**

- Expired Standards – The vials are collected in a 1 gallon polyethylene bucket. These vials are then transferred to an open top 55 gallon steel or polyethylene waste drum. These drums are transported to a waste facility for proper disposal.

- Soil Retain Samples - These samples if not flagged in the system for any hazardous constituents are transferred to poly-lined cubic yard boxes. These boxes when full are sent to stabilization or incineration. These materials are sent out as hazardous for lead and chromium

  Teris Profile Number (incineration): 50016710
  Onyx Profile Number: (stabilization) 402535

- Methanol Preserved Samples/Returned Methanol Preservative - Methanol preserved sample vials are collected in satellite accumulation and then transferred to a 55 gallon open top steel waste drum in the waste room. This drum is then removed by a waste vendor for incineration.

  Teris Profile Number: 50016652
  Onyx Profile Number: 282493

15.0 References / Cross-References

15.1. United States Environmental Protection Agency, "Method SW8260C, Volatile Organic Compounds by Gas Chromatography/Mass Spectrometry (GC/MS)", Test Methods for Evaluating Solid Wastes, SW846, August 2006.

15.2 United States Environmental Protection Agency, "Method SW8000C: Determinative Chromatographic Separations", Test Methods for Evaluating Solid Wastes, SW846, Laboratory Manual, Physical/Chemical Methods, Revision 3, March 2003.

15.3 TestAmerica Edison Document No. ED-QA-LQM, *Laboratory Quality Manual*, most current revision.

15.4 TestAmerica Edison SOP Nos. ED-MSV-001*, Purge and Trap for Aqueous Samples, SW846 Method 5030*, current revision.

15.5 TestAmerica Edison ED-MSV-002, *Closed-System Purge-and-Trap and Extraction for Volatile Organics in Soil and Waste Samples, SW846 Method 5035,* current revision.

15.6 TestAmerica Edison SOP No. ED-GCV-001, *Screening for Volatile Organics, Static Headspace with GC FID, SW846 Method 5021*, current revision.

15.7 TestAmerica Corporate Quality SOP No. CA-Q-S-001, *Solvent & Acid Lot Testing & Approval*, current revision.

15.8 TestAmerica Edison SOP No. ED-GEN-023, *Bulk Solvent Testing and Approval*, current revision.

15.9 TestAmerica Edison SOP No. ED-GEN-008, *Standard Operating Procedure for Preparation, Purity and Storage of Reagents and Standards*, current revision

15.10 TestAmerica Edison SOP No. ED-SPM-004, *Sample Storage & Handling Procedures for Mitigation of Sample and Laboratory Contamination,* current revision

15.11 TestAmerica Edison Work Instruction No. EDS-WI-096, *8260C ICAL Procedure*, current revision.

15.12 TestAmerica Edison SOP No. ED-GCV-001, *Screening for Volatile Organics, Static Headspace with GC FID, SW846 Method 5021*, current revision

15.13 TestAmerica Edison SOP No. ED-GEN-022, *Training*, current revision.

15.14 TestAmerica Edison SOP No. ED-SPM-008, *Laboratory Waste Disposal Practices*, current revision

16.0 Method Modifications:

N/A

17.0 Attachments

N/A

18.0 Revision History

- Revision 6, dated 01/17/2018:
    - Revised Table 5 and 6: revised to add additional levels plus Benzene and chloroform and to updated concentration level of ICV to current level.
    - 7.2.1 and Table 2: Table in Section 7.2.1 and Table 2 updated to include complete list of standards currently in use as well as to update vendor catalog number for several items.
    - Section 7.2.5: SIM IS/SS mix corrected to reflect lower concentration of IS mix .
    - Section 9.1.2.1.4: SIM MS/MSD preparation revised.
    - Section 9.1.3.2: SIM LCS/LCSD preparation revised.
    - Section 9.2.2.2: additional SIM levels added.
    - Section 9.2.3: SIM CCV level concentration revised to reflect lower concentration.
    - Section 10.2.5: New SIM analysis preparation narrative added
    - Section: 6.1: New instrumentation added.
    - Section: Section: 9.1.4.3: Revised to have any one surrogate out without the need for corrective action. This corrects previous narrative of one surrogate out of two.
- Revision 5, dated 12/11//2015:
    - Revised Table 5: new concentration of low standard (1,4-dioxane only).
- Revision 4, dated 12/08//2014:
    - Section 9.2.4.2.2: Table revised to reflect minimum RF of 0.050 for following compounds: acetone, 2-butanone, 4-methyl-2-pentanone, 2-hexanone.
    - Section 9.2.4.3: added statement ‘for poor performers the range is 50-150%’.

- Revision 3, dated 11/10/2014:

    - Tables 1 and 7: added 1,2,4,5-Trimethylbenzene, 1,4-Diethylbenzene, Butadiene, 1,4-Difluorobenzene, 1-Chlorohexane, Freon 114, Freon 123a, Isooctane, 4-Ethyltoluene, t-Amyl Alcohol, Chlorofluoroethylene to list of target compounds and list of standard sources.

    - Section 2.5: added chloroform, vinyl chloride and benzene to the list of SIM analytes addressed in this section.

    - Section 2.6: revised the concentration of the low ketone standard to 2.5 ug/l.

    - 7.2.1 and Table 2: Table in Section 7.2.1 and Table 2 updated to include complete list of standards currently in use as well as to update vendor catalog number for several items. All standards prep tables revised to reflect current standard prep instructions.

    - Section 8. Preservation by TSP and holding time is added.

    - Section 9.1.2.1: updated source of standards used in various spiking solutions.

    - Section 9.1.3: LCS/MS/MSD. Preparation tables now indicate using calibration mix and not the second source mix.

    - Sections 9.1.4.3 and 9.1.1 : Revised to indicate that we are now spiking with 4 surrogates instead of the method required 3. One surrogate is now allowed to be out of limit criteria for either 1,2-Dichloroethane-d4 and Dibromofluoromethane.

    - Section 9.2.2: Chloroform, Vinyl Chloride and Benzene added as SIM compounds.

    - Section 9.2.4.2.3.1. A list of 'poor performing compounds' is added with a ICAL RSD criteria of 50%.

    - Section 9.2.4.3: now specifies that up to 10% of the compounds are allowed to exceed the 70-130% ICV recovery criteria as long as their recoveries are within 65-135%..

    - Section 9.2.4.4.1.6: Added the following to the first sentence: '…or 50%D for the poor performing compounds'.

    - Section 10.1.1.2: updated masses/dwell time for Group 1 under SIM Parameters.

    - Throughout document as appropriate: Replaced references to Target with references to CHROM

    - Added Section 10.5.5: "The data processing service from Chrom queries LIMS for the sample processing parameters."

- Revision 2, dated 11/04/2013:

    - Tables 1 and 7: added methyl acrylate, 1-methylnaphthalene and 2-methylnapthalene.

- Revision 1, dated 09/16/2011:

    - Tables 1 and 7: added cyclopentene, 2-chloro-1,3-butadiene, methacrylonitrile, propionitrile, ethyl methacrylate, 2-nitropropane, indan and isobutyl alcohol to list of target compounds and list of standards sources.

    - Section 7.2.1 and Table 2: Table in Section 7.2.1 and Table 2 updated to include complete list of standards currently in use as well as to update vendor catalog number for several items.

    - Table 3: Initial Calibration Standards Preparation: is now split into three tables to include aqueous low level analysis.

    - Table 5: added following footnote:

      Levels 1 and 2 respectively are prepared in 500ml and 100ml final volumes
      [1]This level is also used as the Continuing Calibration Verification.

- Revision 0, dated 02/15/2011: New

Table 2: Working Standards Preparation

Target Compound Standard Name	Lab Name	Vendor	Cat. #	Vol. Std. Added	Conc. of Stock Std.	Concentration of Standard	Final Vol/ Total vol of MeOH
Gas Mix Hi	Gas (Hi)	Restek	567645	5ml mL	2000 ppm	500 ppm	20mL 15mL TV/M
Gas Mix Li	Gas (Li)	Restek	567645	500 uL	2000 ppm	50 ppm	20mL 19.5mL TV/M
8260Mix 1	Mix 1 (Hi)	Restek	567641 567646 567642 568022	2.5ml 2.5 ml 2.5 ml 2.5 ml	2000 ppm	500 ppm	10ml
8260 combined	Mix 1 (Li)	Restek	567641 567646 567642 568022 567643 568018 568713 568722 568723	1.0ml 1.0ml 1.0ml 1.0ml 1.0ml 1.0ml 1.0ml 1.0ml 1.0ml	2000 ppm	50 ppm	40ml 31ml TV/M
Acrolein	AC	Restek	82402	1.0ml	20000 ppm	500 ppm	40ml 39ml TV/M
8260 Mix 2	Mix 2 (Hi)	Restek	567643 568722 568019-fl 568713-fl	2.5ml 2.5 ml 2.5 ml 2.5 ml	2000 ppm	500 ppm	10mL
8260 Mix 3	Mix 3 (Hi)	Restek	568723 568021-fl	2.5ml 2.5ml	2000 ppm	500 ppm	10ml 5ml TV/M
1,4-Dioxane	1,4-Dioxane	Supelco	360481	483.6ul	Neat	50000 ppm	10ml/9.52TVM
1,4-Dioxane	1,4-Dioxane	Supelco	NA	100ul	50000 ppm	500 ppm	10ml/9.90TVM
Propenes*	Propenes	Supelco	21240202	NA	1000/2000 ppm	NA	NA
Propenes*	Propenes	Supelco	21240202	1ml	1000/2000 ppm	50 ppm (varied)	20ml/ 19ml
Gas SS	Gas SS	Restek	567645.sec	1ml	2000ppm	50 ppm	40ml 39ml/TV/M
8260 Mix 1 SIM	8260 Mix 1 SIM	Supelco	5-02111	50 ul	2000ppm	10 ppm	10ml 9.95 TV/M
1,4-Dioxane SIM	1,4-Dioxane	Supelco	NA	100 ul	50000 ppm	500 ppm	10ml/9.90TVM

Table 2: Working Standards Preparation

Target Compound Standard Name	Lab Name	Vendor	Cat. #	Vol. Std. Added	Conc. of Stock Std.	Concentration of Standard	Final Vol/ Total vol of MeOH
8260 SS	8260 SS	Restek	567641.sec 567646.sec 567642.sec 568022- sl 567643.sec 568019- sl 568713- sl 568722.sec 568723.sec 568021- sl	1 ml 1 ml 1 ml 1 ml 1 ml 1 ml 1 ml 1 ml 1 ml 1 ml	2000 ppm	50 ppm	40 ml 30 ml TV/M
Acrolein SS	AC SS	Restek	568720.sec	1 ml	20000 ppm	500 ppm	40 ml 39 ml TV/M
Propenes SS	Propenes SS	Supelco		1 ml	1000/2000 ppm	50/100 ppm	40 ml 39 ml TV/M
8260Mix 1 SIM SS	SIM MIX1 SS	Supelco	5S-02111	50ul	2000 ppm	10 ppm	10ml 9.95 TV/M
Benzene/ Chloroform	Ben/chl	Absolute	70025/ 70076	100ul each	1000ppm	10ppm	10ml 9.90 TV/M
1,4-Dioxane (SS)	1,4-Dioxane	Absolute	70373	1ml	1000 ppm	500 ppm	2ml/1ml TV/M

Asterisk (*) indicates a custom standard mix.

Table 3: Initial Calibration Standards Preparation, Low Level Soil

Standard Solution	Final Volume Reagent Water (ml)	Volume of Standard Added to Reagent Water (ul)					
		1ppb *	5ppb*	20ppb[1]	50ppb	200ppb	500ppb
Gas Mix (50ppm)	5	-	-	2.0	5	-	-
	50	1.0	5.0			-	-
Gas Mix (500ppm)	5	-	-	-		2.0	5.0
		-	-	-			
Mix 1 (combined) (50ppm)	5	-	-	2.0	5	-	-
	50	1.0	5.0			-	-
Mix 1 (Hi) (500ppm)	5	-	-	-	-	2.0	5.0
		-	-	-	-	-	-
Freon Mix							
AC (500ppm)	5	-	-	3.0	4.0	5.0	6.0
	50	10	20			-	-
Mix 2 (Hi) (500ppm)	5	-	-	-	-	2.0	5.0
		-	-	-	-		
Mix 3 (500ppm)	5					2.0	5
Propenes (50ppm)	-	-	-	-	-	-	-
	50	10.0	20.0		-	-	-
Propenes (Hi)(500ppm)	5	-	-	2.0	5.0	20	50
	-	-	-	-	-	-	-

[1]**This level is also used as the Continuing Calibration Verification.**

Table 3a: Initial Calibration Standards Preparation, Aqueous (LOW LEVEL)

Standard Solution	Volume of Standard Added to Reagent Water (ul)						
	0.5ppb*	1ppb*	5ppb*	20ppb[1]	50ppb	200ppb	500ppb
Gas Mix (500ppm)	0.5	1	1	2	5	20	50
Mix 1 (Hi) (500ppm)	0.5	1	1	2	5	20	50
Mix 2 (Hi) (500ppm)	0.5	1	1	2	5	20	50
Mix 3 (varied)	0.5	1	1	2	5	20	50
AC (500ppm)	2	4	4	4	10	20	40
1,4-Dioxane (500ppm)	15	30	-	-	-	-	-
Freons mix	0.5	1	1	2	5	20	50
Propenes (1000/2000ppm)	0.5	0.5	0.5	1	2.5	10	25
Methanol Compensate	3000	2800	610	300	280	190	0
Final vol. (reagent water)	500ml	500 ml	100ml	50 ml	50ml	50ml	50ml

[1]This level is also used as the Continuing Calibration Verification.

Table 3b: Initial Calibration Standards Preparation, Aqueous

Standard Solution	Volume of Standard Added to Reagent Water (ul)					
	1.0ppb*	5ppb*	20ppb[1]	50ppb	200ppb	500ppb
Gas Mix (500ppm)	1	1	2	5	20	50
Mix 1 (Hi) (500ppm)	1	1	2	5	20	50
Mix 2 (Hi) (500ppm)	1	1	2	5	20	50
Mix 3 (varied)	1	1	2	5	20	50
AC (500ppm)	4	4	4	10	20	40
1,4-Dioxane (500ppm)	30	-	-	-	-	-
Freons Mix	1	1	2	5	20	50
Propenes (1000/2000ppm)	0.25	0.5	1	2.5	10	25
Methanol Compensate	2800	610	300	280	190	0
Final vol. (reagent water)	500 ml	100ml	50 ml	50ml	50ml	50ml

[1]This level is also used as the Continuing Calibration Verification.

Table 4 : ICV Standard Preparation, Low Level Soil

Standard Solution	Concentration	Volume of Standard Added to 5.0 ml of Reagent Water (ul)	Final Concentration (ug/L)
GAS SS (Separate lot)	50ppm	2	20
8260 SS (Separate lot)	50ppm (+varied)	2	20
AC SS (separate lot)	500ppm	3	300
Freon SS (Separate lot)	50ppm	2	20
Propenes SS(separate lot)	50ppm (varied)	2	20 (varied)

Table 4a: ICV Standard Preparation, Aqueous

Standard Solution	Concentration	Volume of Standard Added to 50 ml of Reagent Water (ul)	Final Concentration (ug/L)
GAS SS (Separate lot)	50ppm	20	20
8260 SS (Separate lot)	5000ppm (varied)	20	20
AC SS (separate lot)	500ppm	4	400
Freons SS (Separate lot)	50ppm	20	20
Propenes (second source)	50ppm (varied)	20	20 (varied)

Table 5: SIM Initial Calibration Standards Preparation

Standard Solutions	Volume Standard Solution Added to Reagent Water (Final Concentration)							
8260 Mix 1 SIM (10ppm)	1 ul (0.02 ppb)	2 ul (0.04 ppb)	1 ul (0.05 ppb)	1 ul (0.1 ppb)	1 ul (0.2 ppb)	2.5 ul (0.5 ppb)	5 ul (1 .0 ppb)	10 ul (2.0 ppb)
1,4-Dioxane (500ppm)	4 ul (0.4 ppb)	2 ul (1.0 ppb)	1 ul (5.0 ppb)	1 ul (10 ppb)	1 ul (20 ppb)	2.5 ul (30 ppb)	5 ul (40 ppb)	10 ul (50 ppb)
SIM (ben/chl) 10ppm	1 ul (0.02 ppb)	1 ul (0.02 ppb)	1 ul (0.05 ppb	1 ul (0.1 ppb)	1 ul (0.2 ppb)	2.5 ul (0.5 ppb)	5 ul (1.0 ppb)	10 ul (2.0 ppb)
Final Vol. (reagent water)	**500ml**	**500ml**	**200ml**	**100ml**	**50ml**	**50ml**	**50ml**	**50ml**

levels 1 and 2 are respectively prepared in 500ml and 100ml final volumes
[1]**This level is also used as the Continuing Calibration Verification.**

Table 6 : SIM ICV Standard Preparation

Standard Solution	Concentration	Volume of Standard Added to 200 ml of Reagent Water (ul)	Final Concentration (ug/L)
SIM MIX1 SS (Second source)	10ppm	1	0.05
1,4-Dioxane SS	50ppm	20	5

TABLE 7
Characteristic Ions of Volatile Organic Compounds

Parameter	Primary ion	Secondary ion
1,1,1-Trichloroethane	97	99,117,119
1,1,2,2-Tetrachloroethane	83	85,131,133,166
1,1,2-Trichloroethane	97	83,85,99,132,134
1,1-Dichloroethane	63	65,83,85,98,100
1,1-Dichloroethene	96	61,98
1,1-Dichloropropene	75	110. 77
1,2,3-Trichlorobenzene	180	182
1,2,3-Trichloropropane	110	75
1,2,4-Trichlorobenzene	180	182, 145
1,2,4-Trimethylbenzene	105	120
1,2-Dibromo-3-Chloropropane	75	155, 157
1,2-Dibromomethane	107	109
1,2-Dichloroethane	62	64,100,98
1,2-Dichloroethene	96	61,98
1,2-Dichloropropane	63	65,114
1,2-Dichlorotrifluoroethene	67	117
1,2-Difluorotetrachloroethene	101	103, 167
1,3,5-Trimethylbenzene	105	120
1,3-Dichlorobenzene	146	148, 111
1,4-Dichlorobenzene	146	148, 111
1,4-Dioxane	88	58
1-Chloropropane	63	78
1-Methylnaphthalene	142	141
1-Propene	41	42
2,2-Dichloropropane	77	97

TABLE 7 Characteristic Ions of Volatile Organic Compounds		
2,4,4-trimethyl-1-pentene	41	57, 97
2-Butanone	72	57
2-Chloroethyl vinyl ether	63	65, 106
2-Chloropropane	78	63
2-Chlorotoluene	91	126
2-Chloro-1,3-butadiene	88	53
2-Hexanone	43	58,100
2-Methylnaphthalene	142	141, 115
2-Nitropropane	39	42, 44
2-Octane	43	58
2-Octanol	45	55
4-Chlorotoluene	91	126
4-Methyl-2-Pentanone	43	58,100
Methacrylonitrile	67	41
Acetone	43	58
Acetonitrile	39	40, 41
Acrolein	56	55
Acrylonitrile	53	52
Allyl Alcohol	57	40, 39
Allyl Chloride	76	41
Amyl Acetate	43	70, 61
Benzene	78	--
Benzyl Chloride	91	126, 65
Bromobenzene	156	77, 158
Bromochloromethane	129	49, 130
Bromodichloromethane	83	85
Bromoform	173	171,175,
Bromomethane	94	96
Butyl Acetate	73	56, 43
Butyl Acrylate	73	56, 55
Butyl methacrylate	87	69
Camphene	93	121
Camphor	95	81
Carbon disulfide	76	78
Carbon tetrachloride	117	119,121
Chlorobenzene	112	114
Chloroethane	64	66
Chloroform	83	85
Chloromethane	50	52
Chlortrifluoroethene	116	118
cis-1,3-Dichloropropene	75	77

TABLE 7 Characteristic Ions of Volatile Organic Compounds		
Cyclohexane	56	84, 69
Cyclopentene	67	68, 68, 53
Dibromochloromethane	129	208,206
Dibromomethane	93	95, 174
Dichlorodifluoromethane	85	87
Dimethylnaphthalene (total)	141	156, 155
Epichlorohydrin	57	62, 49
Ethanol	46	45
Ethyl Acetate	70	61, 43
Ethyl Acrylate	55	56
Ethyl Ether	59	74, 75
Ethylbenzene	106	91,
Ethyl methacrylate	69	41, 99
Freon TF	101	103, 151, 85
Hexachlorobutadiene	225	223
Hexane	56	57, 86
Indan	117	118, 58
Iodomethane (methyl iodide)	142	127
Isobutyl Alcohol (Isobutanol)	43	41, 42
Isoprene	67	53, 59
Isopropanol	45	59
Isopropyl Acetate	43	61, 87
Isopropyl Ether (DIPE)	45	87
Isopropylbenzene	105	120
Methyl Acetate	43	74
Methyl Acrylate	55	85, 42
Methyl cyclohexane	83	55, 98
Methyl Methacrylate	100	69
Methyl tert-butyl ether (MTBE)	73	57
Methylene chloride	84	49,51,86
Methylnaphthalene (total)	142	141, 115
Naphthalene	128	--
n-Butanol	56	41, 43
n-Butylbenzene	91	92, 134
n-Heptane	57	43, 71
n-Pentane	72	57
N-Propanol	60	59
n-Propylbenzene	91	120
P-Isopropyltoluene`	119	134, 91
Propyl Acetate	43	61, 73
Propionitrile	54	52, 54

TABLE 7 Characteristic Ions of Volatile Organic Compounds		
sec-Butylbenzene	105	134
Styrene	104	78,103
Tert-Amyl Methyl Ether	73	55, 87
Tert-butyl Alcohol	59	--
Tert-Butyl Ethyl Ether	59	87
Tert-Butylbenzene	119	91, 134
Tetrachloroethene	164	129,131,166
Tetrahydrofuran	42	72, 71
Toluene	92	91
Total Xylenes	106	91
trans,-1,3-Dichloropropene	75	77
Trans-1,4-dichloro-2-butene	53	75
Trichloroethene	130	95,97,132
Trichlorofluoromethane	101	103
Vinyl acetate	43	86
Dichlorofluoromethane	67	69
Chlorotrifluoroethene	116	118
1,2-tetrachlorodifluoroethane	101	103,167
1,2-Dichlorotrifluoroethane	67	117
Vinyl chloride	62	64
Isooctane	57	41, 56
1- Chlorohexane	91	93, 55, 56
1,2,4,5-Tetramethylbenzene	119	134, 91
4-EthylToluene	105	120, 77
Chlorotrifluoroethylene	66	116,118,85
Freon 114	85	87,135,137
t-Amyl Alcohol	59	55, 73, 43
1,4-Difluorobenzene	114	63
1,4-Diethylbenzene	119	105,134
Freon 123a	67	69, 117, 119
Butadiene	54	53, 39
4-Bromofluorobenzene (sur)	95	174,176
1,2-Dichloroethane-d4 (sur)	65	102, 104
Toluene-d8 (sur)	98	70,100
Fluorobenzene (istd)	96	77
Chlorobenzene-d5 (istd)	117	82,119
1,4-Dichlorobenzene-d4 (istd)	152	115,150

Title: Trace Metals Analysis by Inductively Coupled Plasma Emission Spectroscopy by SW846 Method 6010B, 6010C & 6010D

Once printed, this is considered an uncontrolled document

Approvals (Signature/Date):

Laura Demone	06/29/18	Donald Evans	06/29/18
Laura Demone	Date	Donald Evans	Date
Department Manager		Operations Manager	
	06/29/18		06/29/18
Carl Armbruster	Date	Mark Acierno	Date
Quality Assurance Manager		Laboratory Director	
	07/02/18		
Dan Helfrich	Date		
Health & Safety Manager Coordinator			

Copyright Information:
This documentation has been prepared by TestAmerica Laboratories, Inc. and its affiliates ("TestAmerica"), solely for their own use and the use of their customers in evaluating their qualifications and capabilities in connection with a particular project. The user of this document agrees by its acceptance to return it to TestAmerica upon request and not to reproduce, copy, lend, or otherwise disclose its contents, directly or indirectly, and not to use it for any other purpose other than that for which it was specifically provided. The user also agrees that where consultants or other outside parties are involved in the evaluation process, access to these documents shall not be given to said parties unless those parties also specifically agree to these conditions.

THIS DOCUMENT CONTAINS VALUABLE CONFIDENTIAL AND PROPRIETARY INFORMATION. DISCLOSURE, USE OR REPRODUCTION OF THESE MATERIALS WITHOUT THE WRITTEN AUTHORIZATION OF TESTAMERICA IS STRICTLY PROHIBITED. THIS UNPUBLISHED WORK BY TESTAMERICA IS PROTECTED BY STATE AND FEDERAL LAW OF THE UNITED STATES. IF PUBLICATION OF THIS WORK SHOULD OCCUR THE FOLLOWING NOTICE SHALL APPLY:

©COPYRIGHT 2018 TESTAMERICA LABORATORIES, INC. ALL RIGHTS RESERVED.

1.0 Scope and Application

1.1. Analytes, Matrix(s), and Reporting Limits

Method 6010B, 6010C and 6010D determines trace elements in solution using inductively coupled plasma-atomic emission spectrometry (ICP-AES). The method is applicable to all of the elements listed in Table 1. All matrices are digested prior to analysis (i.e. 3050B or 3010A).

Detection limits, sensitivity, and the optimum and linear concentration ranges of the elements can vary with the wavelength, spectrometer, matrix and operating conditions. Elements listed in Table 1 may be analyzed by this method if performance at the concentration levels of interest is demonstrated. The laboratory's reporting limits (RL) are listed in Table 1.

Table 1: Reporting Limit for Soil and Water

Element	Aqueous Reporting Limit (Non-TCLP) (ug/L)		Aqueous Reporting Limit (TCLP) *adjusted for typical 5X-dilution* (ug/L)		Soil Reporting Limit, *adjusted for typical 4X-dilution* (mg/Kg)	
	6010B	6010C & 6010D	6010B	6010C & 6010D	6010B	6010C & 6010D
Al	200	200	1000	1000	40	40
Sb	10	20	50	100	2	4
As	5	15	25	75	1	3
Ba	200	200	1000	1000	40	40
Be	2	2	10	10	0.4	0.4
Bo	50	50	250	250	10	10
Cd	5	4	25	20	1	0.8
Ca	5000	5000	25000	25000	1000	1000
Cr	10	10	50	50	2	2
Co	50	50	250	250	10	10
Cu	25	25	125	125	5	5
Fe	150	150	750	750	30	30
Pb	5	10	25	50	1	2
Mn	15	15	75	75	3	3
Mg	5000	5000	25000	25000	1000	1000
Mo	20	20	100	100	4	4
Ni	40	40	200	200	8	8
K	5000	5000	25000	25000	1000	1000
Se	10	20	50	100	2	4
Ag	10	10	50	50	2	2
Na	5000	5000	n/a	n/a	1000	1000
Sr	20	20	100	100	4	4
Tl	10	20	50	100	2	4
Sn	50	50	250	250	10	10

Ti	20	20	100	100	4	4
V	50	50	250	250	10	10
Zn	30	30	150	150	6	6

On occasion clients may request modifications to this SOP. These modifications are handled following the procedures outlined in Sections 7 (Review of Work Request) and 19 (Test Methods and Method Validation) of TestAmerica Edison's Quality Assurance Manual (ED-QA-LQM).

2.0 Summary of Method

Samples are digested with the appropriate digestion technique prior to sample analysis. SW846 Method 3050B (TestAmerica Edison SOP No. ED-MTP-005) is the procedure utilized for digesting metals in soil samples; SW846 Method 3010A (TestAmerica Edison SOP No. ED-MTP-003) is the procedure utilized for digesting metals in water samples. Following the sample preparation/digestion, a multi-elemental analysis of elements in solution is performed via Inductively Coupled Plasma- Atomic Emission Spectrometry (ICP-AES). Elemental constituents are determined simultaneously. Samples are nebulized and the aerosol is transported to the plasma torch where excitation occurs. Element specific spectra are produced by radio-frequency inductively coupled plasma. The spectra are dispersed by a grating and the intensities are measured. Background correction points and/or inter-element correction factors must be determined at instrument and method set-up. This laboratory semi-annually investigates the need for such factors and copies of the data are available. The points and correction factors determined are used throughout the year and verified on a daily basis. If a discrepancy exists, the situation is remedied.

3.0 Definitions

For a complete list of definitions refer to Appendix 2 in the most current revision of TestAmerica Edison's Quality Assurance Manual (ED-QA-LQM).

4.0 Interferences

4.1. Most interference are eliminated or greatly reduced during the acid digestion of the sample matrix. High salt concentrations can cause analyte signal suppressions and confuse interference tests. Dilute the sample if necessary.

4.2. Spectral interferences encountered on the instrument are corrected for by using baseline correction points and/or by applying background correction factors. Background correction points are determined by scanning the area on either side of the wavelength and recording the apparent intensity from all other method analytes. Use single element solutions at or near the upper linear range of each element. Interelement Correction Factors are determined by analyzing single element solutions and recording the apparent analyte concentration. All interfering elements must be analyzed at the same time as the elements of interest.

4.3. Physical interferences are effects associated with sample nebulization and transport. They are reduced or eliminated with use of a peristaltic pump and internal standards. If these methods are insufficient to reduce the interference, they must be reduced by diluting the sample.

4.4. Memory interferences result when analytes in a previous sample contribute to the signals measured in a new sample. The necessary rinse times must be estimated prior to sample analysis. Until required rinse times are determined a suggested rinse time of 60 seconds shall be used. If a memory interference is suspected the sample must be reanalyzed.

4.5. Chemical interferences include molecular compound formation, ionization effects, and solute vaporization effects. Normally, these effects are not significant with the ICP technique. If observed, changing the operation conditions (e.g., forward power) and/or buffering the sample (adding 2% Lithium to the internal standard solution) should reduce or eliminate the interference.

5.0 Safety

Employees must abide by the policies and procedures in the Corporate Environmental Health and Safety Manual (CW-E-M-001) and this document. This procedure may involve hazardous material, operations and equipment. This SOP does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of the method to follow appropriate safety, waste disposal and health practices under the assumption that all samples and reagents are potentially hazardous. Safety glasses, gloves, lab coats and closed-toe, nonabsorbent shoes are a minimum.

5.1. Specific Safety Concerns or Requirements

The ICP plasma emits strong UV light and is harmful to vision. All analysts must avoid looking directly at the plasma.

5.2. Primary Materials Used

The following is a list of the materials used in this method, which have a serious or significant hazard rating. **Note: This list does not include all materials used in the method. The table contains a summary of the primary hazards listed in the MSDS for each of the materials listed in the table.** A complete list of materials used in the method can be found in the reagents and materials section. Employees must review the information in the MSDS for each material before using it for the first time or when there are major changes to the MSDS.

Material (1)	Hazards	Exposure Limit (2)	Signs and symptoms of exposure
Hydrochloric Acid	Corrosive Poison	5 ppm-Ceiling	Inhalation of vapors can cause coughing, choking, inflammation of the nose, throat, and upper respiratory tract, and in severe cases, pulmonary edema, circulatory failure, and death. Can cause redness, pain, and severe skin burns. Vapors are irritating and may cause damage to the eyes. Contact may cause severe burns and permanent eye damage.
Nitric Acid	Corrosive Oxidizer Poison	2 ppm-TWA 4 ppm-STEL	Nitric acid is extremely hazardous; it is corrosive, reactive, an oxidizer, and a poison. Inhalation of vapors can cause breathing difficulties and lead to pneumonia and pulmonary edema, which may be fatal. Other symptoms may include coughing, choking, and irritation of the nose, throat, and respiratory tract. Can cause redness, pain, and severe skin burns. Concentrated solutions cause deep ulcers and stain skin a yellow or yellow-brown color. Vapors are irritating and may cause damage to the eyes. Contact may cause severe burns and permanent eye damage.
1 – Always add acid to water to prevent violent reactions. 2 - Exposure limit refers to the OSHA regulatory exposure limit.			

6.0 Equipment and Supplies

6.1. Instrumentation

- iCAP 6500 Duo View ICP-OES Spectrometer with a solid state 27.12MHz RF generator. The nebulizer, plasma, and auxiliary gas to the sample introduction system are supplied and controlled via 3 independent mass flow controllers. Vacuum purged spectrophotometer with an axial and radial plasma torch. Purchased from ThermoFisher Scientific.
  Note: Operating conditions must be established by the analyst according to the instrument manufacturer's specification and must meet conditions satisfying the analytical and quality assurance requirements.

6.2. Supplies

- Volumetric Flasks (Class A): 50 mLs, 100 mLs, 500mls & 1000mls
- Eppendorf & Fisher Pipettes, varying volumes
- Polypropylene tubes
- Argon supply – 99.5%

7. Reagents and Standards

7.1. Reagents

7.1.1. 18 megohm Reagent grade Type II water

7.1.2. Concentrated distilled nitric acid--Trace Grade or Equivalent; store at room temperature; for stability information, refer to manufacturer's instructions.

7.1.3. Concentrated distilled hydrochloric acid--Trace Grade or Equivalent; store at room temperature; for stability information, refer to manufacturer's instructions.

7.1.4. 5% HNO3 + 5% HCl: Add 1 liter of concentrated HNO3 and 1 liter of concentrated HCl to deionized water and bring to 20 liter volume with deionized water. Note: Always add acid to water. Record preparation in TALS Reagent Module. Prepare every 12 months or refer to manufacturer's expiration date; store at room temperature.

7.1.5. 5% HNO3: Add 1 liter of concentrated HNO3 to deionized water and bring to 20 liter volume with deionized water. Note: Always add acid to water. Record preparation in TALS Reagent Module. Prepare every 12 months or refer to manufacturer's expiration date; store at room temperature.

7.2. Standards

Storage requirements: All standards are stored at room temperature. Standards containing Silver and HCl must be stored in amber containers or covered when not in use to minimize exposure to light.

Shelf-life: Stock standards – refer to manufacturer's instructions
Intermediate standards – 12 months
Working standards – 12 month
(Note: expiration date must not go beyond the expiration date of the source stock).

Concentration: see Attachment 3 for example certificates of analysis (COA) for all of the standards mixes listed below. The COA lists the manufacturer's part number, certified concentration and shelf life.

Standards must be prepared every 12 months or sooner if needed or required. "If needed" means the standard has been exhausted; "if required" means that the standard does not meet the QC criteria. **Exception Note:** *ICV for Method 6010D should be prepared fresh daily.*
All standards must be prepared in (5%HNO3/5%HCl) or when noted, 5%HNO3 (see Sec. 7.1.4 and 7.1.5).

7.2.1. Calibration stock standards - CLPP-CAL-1, CLPP-CAL-3, STLNJ-CAL-1A, STLNJ-CAL-1B, STLNJ-CAL-2, TANJ-STD-3 and TANJ-STD-4; 1000ppm Sb; 10000 ppm each of: Al, As, Pb, Se, Tl, Si; purchased from Inorganic Ventures, Christiansburg, VA. See Attachment 3 for details.

7.2.2. Working Calibration Standards: see Attachment 1 for final elemental concentration.

7.2.2.1. CAL1: add 100 ml of 5%HNO_3/5%HCl to a clean 1000 ml volumetric flask. Add 10 ml of ME_Cal1-int (see 7.2.2.1.1). Bring to volume with 5%HNO_3/5%HCl.

7.2.2.1.1. ME_Cal1-int: add 100 ml of 5%HNO3/5%HCl to a clean 1000 ml volumetric flask. Using 10,000 ppm stock standards, add 0.05 ml each of As, Pb, Se; add and 0.1 ml of Tl; Using 1,000 ppm stock standard, add 1 mL Sb. Bring to volume with 5%HNO3/5%HCl.

7.2.2.2. CAL2/LLICV/LLCCV: add 100 ml of 5%HNO3/5%HCl to a clean 1000 ml volumetric flask. Add 10 ml each of TANJ-STD-3 and TANJ-STD-4. Add 10 ml of ME_Cal2-int (see 7.2.2.2.1). Bring to volume with 5%HNO3/5%HCl.

7.2.2.2.1. ME_Cal2-int: add 100 ml of 5%HNO3/5%HCl to a clean 1000 ml volumetric flask. Using 10,000 ppm stock standards, add 0.1 ml each of As, Tl; add 0.05 mL of Pb; add 0.15 mL of Se. Using 1,000 ppm stock standard, add 1 mL of Sb. Bring to volume with 5%HNO3/5%HCl.

7.2.2.3. CAL3: add 100 ml of 5%HNO3/5%HCl to a clean 1000 ml volumetric flask. Add 1 ml each of CLPP-CAL-1, STLNJ-CAL-1A, and STLNJ-CAL-1B, 2 ml of STLNJ-CAL-2, 2.3 ml of 10000 ppm Aluminum, and 0.5 ml of CLPP-CAL-3. Add 0.2 ml of 10,000ppm Si. Bring to volume with 5%HNO3/5%HCl.

7.2.2.4. CAL4/CCV: add 100 ml of 5%HNO3/5%HCl to a clean 1000 ml volumetric flask. Add 5 ml each of CLPP-CAL-1, STLNJ-CAL-1A, and STLNJ-CAL-1B, 10 ml of STLNJ-CAL-2, 11.5 ml of 10000 ppm Aluminum, and 2.5 ml of CLPP-CAL-3. Add 1 ml of 10,000ppm Si. Bring to volume with 5%HNO3/5%HCl.

7.2.2.5. CAL5: add 100 ml of 5%HNO3/5%HCl to a clean 1000 ml volumetric flask. Add 10 ml each of CLPP-CAL-1, STLNJ-CAL-1A, and STLNJ-CAL-1B, 20 ml of STLNJ-CAL-2, 23 ml of 10000 ppm Aluminum, and 5 ml of CLPP-CAL-3. Add 2 ml of 10,000ppm Si. Bring to volume with 5%HNO3/5%HCl.

7.2.3. Calibration Verification Stock Standards – Part No. SM-606-122 (Duo_CCV-int) Solutions: A, B, C, and D; purchased from High-Purity Standards. See attachment 1 for final elemental concentrations.

7.2.4. Working Calibration Verification Standards**:**

7.2.4.1. ICV: add 50 ml of 5%HNO3/5%HCl to a clean 100 ml volumetric flask. Add 1.0 ml each of Part No. SM-606-122 (Duo_CCV-int) Solutions: A, B, C, and D. Bring to volume with 5%HNO3/5%HCl.

7.2.4.2. CCV**:** see CAL4 (Sec. 7.2.2.4)

7.2.5. Stock Spike Standards:

7.2.5.1. Solutions A, B, C, and D (ICP-Spk), Part No. 4400-100419DD01: commercially purchased from CPI International. Store at room temperature; for stability information, refer to manufacturer's instructions. See Attachment 3 for the standards certified concentrations and catalog numbers.

7.2.5.2. Solutions A, B, C, and D (TCLP-Spk), Part No. SM-606-114: commercially purchased from High-Purity Standards. Store at room temperature; for stability information, refer to manufacturer's instructions. See Attachment 3for the standards certified concentrations and catalog numbers.

7.2.5.3. Working Spike Standards

7.2.5.3.1. ME_LCS-int (for ICP prep) - Add the following to a 1000 ml volumetric flask and bring to volume using 5% HNO_3: 50 ml each of Part No. 4400-100419DD01 Solutions A, B, C, and D. Record standard preparations in TALS Reagent module. **Note**: Standard must not exceed the earliest expiration date of any one of its components.

7.2.5.3.2. ME_TCLPspk (for TCLP prep) – Add the following to a 200 mL volumetric flask and bring to a volume using 5% HNO_3: 40 ml each of TCLP-Spk Solutions: A, B, C, and D- Part No. SM-606-114. Record standard preparations in TALS Reagent module. **Note**: Standard must not exceed the earliest expiration date of any one of its components.

7.2.6. Spectral Interference Check (SIC) Solutions: – CLPP-ICS-A, IV-7, IV-19, 10,000 ppm each of: Na, K, Co, Si, Sn, Sr, V, Cr, Cu, Mn, Mo, Ni, Ti, B, Ba, Ca, Fe, Mg, Se, Zn, Al, As, Pb; purchased from Inorganic Ventures, Christiansburg, VA.

7.2.7. Working SIC Standard A (ICSA): add 500 ml of 5%HNO3/5%HCl to a clean 1000 ml volumetric flask. Add 100 ml of CLPP-ICS-A stock standard solution. Bring to volume with 5%HNO3/5%HCl. See Attachment 1 for final elemental concentration.

7.2.8. Working Interference Check Standard AB (ICSAB) For 6010B and 6010C: add 500 ml of 5%HNO3/5%HCl to a clean 1000 ml volumetric flask. Add

100 ml CLPP-ICS-A, 1 ml each of IV-7, IV-19, and LCSW-III (Sec 7.2.8.1), 0.9 ml each of 10000 ppm Sodium and 10000 ppm Potassium. Bring to volume with 5%HNO3/5%HCl. See Attachment 1 for final elemental concentration.

7.2.8.1. LCSW-III: add 100 ml of 5% HNO_3 to a clean 200 ml volumetric flask. Add 18 ml of 10,000 ppm Sodium; add 2 ml each of 10,000 ppm Tin and 10,000 ppm Strontium. Bring the volume with 5% HNO_3.

7.2.9. Int-A For 6010B and 6010C: add 500 ml of 5%HNO3/5%HCl to a clean 1000 ml volumetric flask. Using 10000 ppm standards, add the following: 1 ml of Si and 0.5 ml of V. Bring to volume with 5%HNO3/5%HCl. Final concentration in solution: 10 ppm for Si and 5ppm for V.

7.2.10. Int-B For 6010B and 6010C: add 500 ml of 5%HNO3/5%HCl to a clean 1000 ml volumetric flask. Using 10000 ppm standards, add the following:1 ml each of Cr, Mn, and Ti. Bring to volume with 5%HNO3/5%HCl. Final concentration in solution: 10 ppm for Cr, Mn, Ti..

8. Sample Collection, Preservation, Shipment and Storage

Sample container, preservation techniques and holding times may vary and are dependent on sample matrix, method of choice, regulatory compliance, and/or specific contract or client requests. Listed below are the holding times and the references that include preservation requirements.

Matrix	Sample Container	Min. Sample Size	Preservation	Holding Time [1]	Reference
Waters	Plastic, glass	100 mLs	HNO_3, pH < 2 prior to shipment; if not, acidify upon receipt in lab [2,3]	180 days	SW846 Method 6010B 40 CFR Part 136.3
Soils	Plastic, Glass	3 grams	Cool 4 ± 2°C	180 days	SW846 Method 6010B

[1] Inclusive of digestion and analysis.
[2] Acid preservation may be omitted for shipping; however, acid must be added upon receipt in the lab. Following acidification, mix the sample and hold for at least 24 hours. Just prior to digestion or direct analysis, verify pH<2. If pH≥ 2, repeat steps (i.e., add acid, hold for 24hrs, verify ph<2).
[3] Aqueous samples may be stored at room temperature.

Note: Samples for dissolved metals should be filtered in the field before acid is added to the sample. If the sample is to be filtered in the lab, no preservative is added to the sample until the sample is filtered.

9. Quality Control

9.1. **Sample QC** - The following quality control samples are prepared with each batch of samples.

Quality Control	Frequency	Control Limit
Method Blank (MB)	1 in 20 or fewer samples	For 6010B and 6010C: <MDL; or <5% of Reg. Limit; or <5% of the measured concentration in the sample. For 6010D: <1/2 the RL; or project specific requirements; or <10% of the measured concentration in the sample
Laboratory Control Sample (LCS) for *aqueous samples*	1 in 20 or fewer samples	80-120%
Laboratory Control Sample (LCS) in soil	1 in 20 or fewer samples	Vendor's certified limits
Matrix Duplicate [1]	1 in 20 or fewer samples	If original sample and dup are both ≥ 5X RL, then 20% RPD. If original sample and duplicate are less than the RL, the RPD is not calculated; otherwise ±RL.
Matrix Spike (MS)[1]	1 in 20 or fewer samples	75-125%
Matrix Spike Duplicate (MSD)[1]	When requested by client	75-125%; If MS and MSD are both ≥ 5X RL, then 20% RPD. If MS and MSD are less than the RL, the RPD is not calculated; otherwise ±RL.
Serial Dilution (1/5 dilution) [1]	1 per batch of 20 or fewer samples	See sec 9.1.6.
Post Digestion Spike[1]	1 per batch of 20 or fewer samples	Method 6010B & 6010C: 80-120% Method 6010D: 75-125%

[1] The sample for DU, MS, MSD, SD, and PDS is randomly selected unless specifically requested by a client; Use the same environmental sample whenever possible. If insufficient sample amount is available, another environmental sample may be used.
[2] Statistical control limits are updated annually and are updated into lab database.

9.1.1 Method Blank (MB): One laboratory method blank will be analyzed with each batch of samples prepared together (not to exceed 20 samples). The method blank is used to identify possible contamination during acid digestion.

- For methods 6010B & 6010C: results must be less than the highest of: (1) MDL, (2) 5% of the regulatory limit, (3) 5% of the measured concentration of the sample.
- For method 6010D: results must be less than the highest of: (1) ½ the RL, (2) project specific requirements, or (3) 10% of the measured concentration of the sample. If any analyte concentration in the blank is above the control limit, the batch must be prepared again for the element in question and the samples reanalyzed.

9.1.2. Laboratory Control Sample (LCS): A laboratory control sample must be analyzed with each group of samples digested. Results of the aqueous LCS must fall within ±20% of the true value. For solid matrices, a vendor supplied solid matrix with certified values is carried through the same preparation procedure as the samples. The results of the solid LCS must fall within the 'QC Performance acceptance limits' of the reference material used for that sample. If not, all samples prepared in association with the LCS must be redigested and reanalyzed. For dissolved aqueous samples not undergoing a preparation procedure, the CCV may serve as the LCS.

9.1.3. Matrix Duplicate (DU): A duplicate is analyzed for each batch of samples. If original sample and duplicate are both ≥ RL (reporting limit), then 20% RPD. If original sample and duplicate are less than the RL, the RPD is not calculated; otherwise, ±RL.

9.1.4. Matrix Spike (MS): A matrix spike is prepared and analyzed for each batch of samples digested. A recovery of 75-125% is required. An exception to this occurs if the sample concentration exceeds the spike concentration by a factor of four or more. If the recovery is not within specified limits a post digestion spike is required to be analyzed (see Sec 9.1.5). See Attachment 2 for list of the elemental concentrations.

9.1.5. Post-Digestion Spike: A post-digestion spike is analyzed for each batch of samples digested. Prepare the sample by mixing 0.20 ml of the working standard ME_LCS-int (Sec. 7.2.5.3.1.) and 9.80 ml of the sample digestate. If sample requires dilution, adjust the sample as appropriate, (e.g. sample at 4X dilution, add 0.20 ml of the standard spike to 2.5 ml of sample digestate, and add 7.3 ml of the 5%HNO3/5%HCl). For Method 6010B and 6010C the acceptable range is 80-120% recovery. For Method 6010D the acceptance range is 75-125% recovery. If both the MS and post digestion spike fail, then matrix effects are confirmed.

9.1.6. Serial Dilution (SD): A 1/5 dilution is prepared and analyzed on one sample per batch to determine if matrix interferences are present.

9.1.6.1. Select a sample and dilute the digestate by a factor of 5 (DF=5).

9.1.6.2. Analyze the dilution using the same procedures used for the un-diluted aliquot.

9.1.6.3. Compare the results of the diluted and un-diluted aliquots of sample digestate.

9.1.6.4. For Method 6010B & 6010C:If the analyte concentration is minimally a factor of 10 above the RL (report limit), then the results of the dilution should be within ±10% of the result of the undiluted sample. If not, then a chemical or physical interference effect should be suspected.

9.1.6.5. For Method 6010D: If the analyte concentration is minimally a factor of 25 above the RL (Reporting Limit), then the results of the dilution should be within ±20% of the result of the undiluted sample (original sample). If not, then a chemical or physical interference effect should be suspected.

9.1.7. Matrix Spike Duplicate (MSD): When requested by the client, a matrix spike duplicate is prepared and analyzed. A recovery of 75-125% is required. An exception to this occurs if the sample concentration exceeds the spike concentration by a factor of four or more. See Attachment 2 for list of the elemental concentrations. If the recovery is not within specified limits a post digestion spike is required (see Sec 9.1.5). If the MS and MSD are both ≥ RL (reporting limit), then 20% RPD. If MS and MSD are less than the RL, the RPD is not calculated; otherwise, ±RL.

9.2. Instrument QC

9.2.1. Initial Calibration Verification (ICV): Instrument calibration is verified using an independent standard at the midpoint of the calibration curve. The ICV source must be different from the calibration standards. Results must be within 10% of the true value and the replicate measurements must be less than 5% RSD. If results are outside of the specified limits, terminate the analysis, correct the problem, prepare new sets of ICAL standards if needed and recalibrate the instrument.

9.2.2. Continuing Calibration Verification (CCV) - The calibration must be verified every 10 samples and at the end of the analysis run by analyzing the CCV standard. The same solution used for the calibration standard 4 is used for the CCV standard. The concentration of the CCV standard must be at or near mid-range levels of the calibration. The final concentrations of the various elements are listed in Attachment 1. The results for the target compounds must be within +/-10% of the true value. If results are outside of the specified limits, terminate the analysis, correct the problem, recalibrate the instrument, and reanalyze the samples following the last good CCV. See CAL4/CCV of Sec 7.2.2.4 for the preparation instructions.

9.2.3. Initial and Continuing Calibration Blank (ICB/CCB):

9.2.3.1. Method 6010B & 6010C: ICB and CCB must be analyzed after the calibration curve, every 10 samples and at the end of the analytical run. The absolute value of the calibration verification blank must not exceed the reporting limit. If it does, terminate the analysis, correct the problem, recalibrate and reanalyze the samples following the last good CCB. The calibration verification blank is the same blank solution as used for the calibration blank.

9.2.3.2. Method 6010D:

9.2.3.2.1. Initial Calibration Blank (ICB) must be analyzed after the calibration curve. The value of the initial calibration verification blank must not exceed ½ reporting limit. If it does, terminate the analysis, correct the problem, and recalibrate. The initial calibration verification blank is the same blank solution as used for the calibration blank.

9.2.3.2.2. Continuing Calibration Blank (CCB) must be analyzed after the calibration curve, every 10 samples and at the end of the analytical run. The absolute value of the calibration verification blank must not exceed the reporting limit. If it does, terminate the analysis, correct the problem, recalibrate and reanalyze the samples following the last good CCB. The calibration verification blank is the same blank solution as used for the calibration blank.

9.2.4. Daily Interference Check Solution: Verify the inter-element and background corrections by analyzing the interference check solutions (ICSA for 6010D and ICSA/ICSAB for 6010B and 6010C) daily at the beginning of the analysis run. The ICS is analyzed in order to demonstrate that proper corrections are being utilized for known interferences. Analyst must evaluate these solutions in order to detect trends that require corrective actions. Pay particular attention to false positives and false negatives for elements not present in the interference check solutions.

9.2.4.1. Method 6010B & 6010C: Results should fall within the control limit of +/-20% of the true value or +/-2 times the RL of the true value, whichever is greater. See Attachment 1 for list of elements and the corresponding concentrations. See Sec 7.2.7 and Sec 7.2.8 for preparation instructions.

9.2.4.2. Methods 6010D: Results should fall within the control limit of +/-20% of the true value or +/-1 times the LLOQ of the true value, whichever is greater. See Attachment 1 for list of

elements and the corresponding concentrations. See Sec 7.2.7 and Sec 7.2.8 for preparation instructions.

9.2.5. Int-A & Int-B for 6010B & 6010C: Int-A and Int-B samples are analyzed for each analytical run. Results should fall within the control limit of +/-20% of the true value or +/-2 times the RL of the true value, whichever is greater; if not, problem must be identified and corrected. Analyst must evaluate this solution in order to detect trends that require corrective actions. Pay particular attention to false positives and false negatives for elements not present in the solution. Refer to Sec 7.2.9 and Sec 7.2.10 for preparation instructions and final elemental concentrations (mg/L).

9.2.6. Low-Level Initial/Continuing Calibration Verification Standard (LLICV/LLCCV; for method 6010C only): At the beginning of the analysis run, after every 10 samples, and at the end of the run, verify the accuracy at the Lower-Limit of Quantitation (LLQ) by analyzing a solution at the LLQ. The LLICV and LLCCV are analyzed to demonstrate that the ICP is capable of detecting the target analyte at the reporting limit (RL). The determined concentration must be within +/-30% of the true concentration. See Attachment 1 for the list of LLICV/LLCCV standard concentrations in ug/L. Refer to Sec 7.2.2.2 for preparation instructions.

9.2.7. Linear Range Check Standard (LRC): the LRC standard is prepared from single element stock standards of each metal obtained from a commercial source. The LRC is prepared by taking the appropriate volume of each stock and adding it to a 1L volumetric flask partially filled with 5%HNO_3/5%HCl and diluted to the mark after all elements have been added. The Table below lists the volume of each stock solution of each metal, volume used and final concentrations:

Elements	Stock Conc (mg/L)	Volume of stock (mL)	Final Conc (mg/L)
B	10000	0.5	5
As, Mo, Ni	10000	1	10
Mn, Zn	10000	1.5	15
Cr	10000	2	20
Si	10000	3	30
Ba, Cu, Pb	10000	5	50
K	10000	30	300
Na, Fe	10000	40	400
Al, Ca, Mg	10000	50	500

The LRC standard is analyed after each calibration. If the results fall within the control limit of +/-10% of the true value, then the linear range at that concentration has been verified; if not, the linear range is the highest point in the calibration.

9.3. Instrument Performance Criteria

Prior to the analysis of any samples the following must be performed.

9.3.1. Instrument Detection Limits (IDLs): The IDL for each analyte must be determined for each wavelength used on each instrument.

9.3.1.1. **Method 6010B and 6010C**: The Instrument Detection Limit (IDL) is defined as 3.14 times the average of the standard deviations of three runs on three non-consecutive days from the analysis of a reagent blank solution with seven consecutive measurements per day. IDLs should be determined at least every three months.

9.3.1.2. **Method 6010D:** The IDLs for each analyte must be determined for each prep method. The IDL is determined by preparing 10 method blanks (see ED-MTP-003 & ED-MTP-005 for MB preparation instructions). Analyze the ten MB's and calculate the standard deviation and the mean (use zero for the mean if the mean is negative). The IDL = mean + (3 x Std Dev). The IDL must be determined when the instrument is initially set up, when the instrument is adjusted in any way that may affect the IDL (e.g., detector replacement), and annually.

9.3.2. Linear Dynamic Range (LDR):

Determine the linear dynamic range of the instrument by analyzing a minimum of three succeeding higher standards at the upper limit of the instrument. The analytically determined concentration of these standards must be within 10% of the true value. The linear ranges should be re-determined semi-annually for Method 6010B and 6010C or if the instrument is significantly changed.

9.3.2.1. Prepare the standard at concentrations that are expected to define the linear range of the instrument. The calibration standards and the linear range standards should be matrix matched; that is, they have the same percentage of hydrochloric and nitric acids.

9.3.2.2. Analyze the standard(s) after the initial calibration is validated.

9.3.2.3. Compare the concentration of the linear range standard with its true concentration using the following equation:

$$\text{Percent Difference} = \left|\frac{Ccal - Ctrue}{Ctrue}\right| \otimes 100$$

Where:

Ccal = concentration determined from analysis
Ctrue = true concentration of the standard

Note: If the percent difference is less than or equal to 10%, the linear range is confirmed at that concentration. If the percent difference is greater than 10%, repeat the analysis with a lower concentration. For elements validated in this manner you may report data up to 90% of that linear range before a dilution is required.

9.3.3. Linear Range Check (LRC) for Method 6010D: see Sec 9.2.7

9.3.4. Single Element Interference Checks:

Sixteen single-element interference check standards should be prepared and analyzed semi-annually (see table below). Each single-element solution is prepared using a clean 100 ml volumetric flask. See Sec 7.2.6 for single element stock standard information. The absolute value of the concentration observed for any unspiked analyte in the single-element interference checks must be less than two times the analytes' LLOQ.

Elements	Stock Conc (mg/L)	Volume of stock (mL)	Final Conc (mg/L)
Al	10000	5	500
B	10000	0.5	50
Ba	10000	0.5	50
Ca	10000	5	500
Cu	10000	0.5	50
Fe	10000	4	400
Mg	10000	5	500
Mn	10000	0.5	50
Mo	10000	0.2	20
Na	10000	10	1000
Ni	10000	0.2	20
Se	10000	0.2	20
Si	10000	1	100
Sn	10000	0.2	20
V	10000	0.2	20
Zn	10000	0.2	20

10. Procedure

10.1. Sample Preparation

Prior to sample analysis, digest samples following sample digestion procedures: aqueous samples are digested using SW846 Method 3010A. Soil samples are digested using Method 3050B. Refer to TestAmerica Edison SOP Nos.ED-MTP-003

and ED-MTP-005 for the digestion procedure (see Section 15 for complete references).

10.2. Set up the instrument according to the instrument's operating parameters.

10.2.1. TJA 6500 Duo ICP

10.2.1.1. Set up the instrument with the operating parameters recommended by the manufacturer as specified in the iCAP 6000 Series ICP-OES Spectrometer Operator Manuals (see Sec 15.8 for reference). Allow the instrument to become thermally stable, minimally 30min. Perform a torch alignment by aspirating a 2 ppm Zn standard. *Note*: The following elements are analyzed using the axial viewing of the plasma: As, Ag, Ba, Ca, Cd, Co, Cr, Cu, Fe, Mg, Mn, Ni, Pb, Sb, Se, Tl, V, Zn, B, Mo, Sn and Ti. The following elements are analyzed using the radial viewing of the plasma: Al, Be, K, Na, Sr and Si.

10.2.1.2. Background correction points must be determined during the initial set-up of the instrument. Refer to the iTEVA Software Manual located in the Operator Manuals for instructions.

10.2.1.3. Interelement Correction factors must be verified daily. Refer to the iTEVA Software Manual located in the Operator Manuals for instructions. Criteria for determining IEC's is an apparent positive or negative concentration for the analyte that falls outside of one reporting limit from zero.

10.3. Calibration

10.3.1. Profile and calibrate the instrument according to the instrument manufacturer's instructions using a blank (5%HNO3/5%HCl) and 5 standards, see Section 7.2.2 for details on calibration standards preparation.

10.3.2. The instrument must be calibrated daily or once every 24 hours and each time the instrument is set up. The correlation coefficient (r) of the calibration curve for Method 6010B & 6010D must be ≥0.995 and for Method 6010C the correlation coefficient (r) must be ≥0.998. If not, the problem must be corrected, and the instrument must be recalibrated.

10.3.3. Calibration Curve Read-Back (Method 6010D):

10.3.3.1. Low-level readback - The low-level calibration standard (Cal 2, at the RL) must recover within 80-120% of the true value; if not, stop the analysis and recalibrate.

10.3.3.2. Mid-level readback - The mid-level calibration standard (Cal 4)

must recover within 90-110% of the true value; if not, stop the analysis and recalibrate.

10.4. Sample Analysis

10.4.1. Following a sample preparation/digestion procedure, the samples are ready for instrumental analysis. It is advisable to investigate each matrix for any complexities, which might adversely affect the acquisition of valid data.

10.4.1.1. Soil samples and relating QC samples (except soil MB) are typically diluted 4X prior to analysis. Soil MB are diluted 2X prior to analysis.

10.4.1.2. TCLP samples and relating QC samples (except TCLP MB and LCS) are diluted 5X prior to analysis. TCLP MB and LCS are not diluted.

10.4.1.3. Aqueous (non-TCLP) samples are typically analyzed without dilution.

10.4.2. Flush the instrument between standards and samples, minimally 60 seconds, using the calibration blank.

10.4.3. The following analytical run sequence is currently used for samples run under the SW846 6010B/6010C protocol:

Typical Analytical Sequence
INSTRUMENT WARM-UP (approx. 30 minutes)
Align torch using 2 ppm Zn std (Duo)
STANDARDIZATION/CALIBRATION (Blank, 5 Cal Stds)
ICV
ICB
LLICV (for 6010C)
ICSA
ICSAB (for 6010B and 6010C)
Int-A
Int-B
LRC
CCV
CCB
LLCCV (for 6010C)
10 Samples
CCV
CCB
LLCCV (for 6010C)
10 Samples
CCV
CCB
LLCCV (for 6010C)

10.4.4. A minimum of two exposures for each standard, sample and blank is required. The average of the exposures is reported.

10.4.5. Dilute and reanalyze all samples for which the required analytes exceed its linear range (see Sec 10.3.2 & 10.3.3) as well as samples which interfering elements exceeds its linear range.

11.0. Calculations / Data Reduction

11.1. Accuracy:

$$\text{ICV / CCV, LCS \% Recovery} = \frac{\text{observed concentration}}{\text{known concentration}} \times 100$$

$$\text{MS \% Recovery} = \frac{\text{(spiked sample) - (unspiked sample)}}{\text{spiked concentration}} \times 100$$

11.2. Precision (RPD):

$$\text{Matrix Duplicate (MD)} = \frac{|\text{orig. sample value - dup. sample value}|}{[(\text{orig. sample value + dup. sample value})/2]} \times 100$$

11.3. Final results calculation in soil samples:

$$\text{Concentration = mg/kg} = \frac{C \times V \times D}{W}$$

Where:
C= Element concentration from Instrument (ppb)
V= Final volume of sample digested (in liters)
D= Dilution performed on sample
W= Initial dry weight of sample digested (in gram)

NOTE: All dry weight corrections are made in LIMS at the time the final report is prepared.

11.4. Final results calculation in aqueous samples :

$$\text{Concentration = mg/ L} = \frac{C \times V1 \times D}{V2}$$

Where:
C= Element concentration from instrument (ppb)
V1= Final volume of sample digested (in liters)
D= Dilution performed on sample)

V2= Initial volume of sample digested
(in liters).

11.5. Data Reduction:

11.5.1. All data is recorded directly in TALS' Analyst Desktop II program.
11.5.2. Record standard preparations in TALS Reagent module.
11.5.3. Sample and standard preparations must be documented in the Analyst Desktop II program located in TestAmerica Laboratory System (TALS). The analyst must enter the following information: Source standard, initial and final sample volume, spike name and amount used, all reagents and their corresponding lot numbers, creation date and expiration date.
11.5.4. Complete the Data Review Checker (DRC) in TALS: Prior to data submission (i.e., prior to 1st leveling data in the Analyst Desktop II module in TALS), the analyst must execute the DRC (Data Review Checker) program in the applicable analytical batch.

11.5.4.1. Open the analytical batch and click on the Edit tab above to enter the Edit Mode.
11.5.4.2. Press F8 command key on the computer keyboard or right click anywhere on the batch worksheet then click on 'Run checklist.'
11.5.4.3. Acknowledge by filling in responses to all unacknowledged findings.
11.5.4.3.1. Highlight the checklist with findings, then highlight the associated list of findings; right click and choose 'Acknowledge Item.'
11.5.4.3.2. Fill in appropriate comments in the response box, then hit 'OK.'
11.5.4.3.3. Acknowledge all Finding items in the 'Manual Batch Checklist' except for the *"2nd Level review complete?"* this is to be completed by the 2nd level reviewer.

.

12.0. Method Performance

12.1. Method Detection Limit Study (MDL)

The method detection limit (MDL) is the lowest concentration that can be detected for a given analytical method and sample matrix with 99% confidence that the analyte is present. The MDL is determined according to the laboratory's MDL procedure in Section 19 (Test Methods and Method Validation) of TestAmerica Edison's Quality Assurance Manual (ED-QA-LQM). MDLs reflect a calculated (statistical) value determined under ideal laboratory conditions in a clean matrix, and may not be achievable in all environmental matrices. The laboratory maintains MDL studies for analyses performed; these are verified at least annually unless method requirements require a greater frequency.

12.2. Demonstration of Capabilities

For DOC procedure refer to Section 19 in the most current revision of TestAmerica Edison's Quality Assurance Manual (ED-QA-LQM).

12.3. Training Requirements

Refer to TestAmerica SOP No. ED-GEN-022, Training, for the laboratory's training program.

12.4. Lower-Limit of Quantitation Check (LLQC) for method 6010C:

This check sample is prepared at the same concentrations as CAL 2 (see Sec 7.2.2.2 for the preparation procedures) except LLQC is carried through the entire preparation and analytical procedure. The LLQC is verified when all analytes are detected within +/- 30% of their true value. This check should be preformed annually or if the instrument is altered in a way that might significantly change the detection limits.

12.5. Lower-Limit of Quantitation (LLOQ) for method 6010D:

12.5.1. LLOQ verification: prior to analyzing samples under method 6010D, after any change that may affect the LLOQ, and quarterly, the LLOQ must be verified. The LLOQ is verified by the analysis of 7 replicate samples spiked at the LLOQ. Use the same concentrations as CAL 2 (see Sec 7.2.2.2 for preparation procedures). The 7 replicates must be digested prior to analysis. See SOPs ED-MTP-003 and ED-MTP-005 for digestion instructions. The LLOQ is verified when the mean recovery of the 7 replicates is +/-35% of the true value and the RSD $\leq$ 20%.

13.0. Pollution Control

It is TestAmerica's policy to evaluate each method and look for opportunities to minimize waste generated (i.e., examine recycling options, ordering chemicals based on quantity needed, preparation of reagents based on anticipated usage and reagent stability). Employees must abide by the policies in Section 13 of the Corporate Environmental Health and Safety Manual (CW-E-M-001) for "Waste Management and Pollution Prevention."

14.0. Waste Management

14.1. Waste management practices are conducted consistent with all applicable rules and regulations. Excess reagents, samples and method process wastes are disposed of in an accepted manner. Waste description rules and land disposal restrictions are followed. Waste disposal procedures are incorporated by reference to TestAmerica SOP No. ED-SPM-008 (Laboratory Waste Disposal Procedures).

14.2. The following waste streams are generated as a result of this analysis:

- Digested Samples: Corrosive Acid- Materials that are not above regulatory limits will be submitted for elementary neutralization with 50% sodium hydroxide solution (Siedler Chemical SC-1824-03). Major concern is heat generated from the neutralization process. Initial volume of acid waste to be

neutralized should be no more than 15 gallons. Finished neutralization with sodium bicarbonate (Siedler Chemical SC-0219-25) to a pH of 6 – 9 in the primary tank. Once pH has been established the primary tank is transferred through filter housing to a secondary tank. The pH is rechecked. If the pH is within specifications, the secondary tank is released to the municipal sewer system.

- Samples above regulatory limits and expired RCRA metals standards (Waste Corrosive Liquid, Acidic, Inorganic, n.o.s.) are collected in satellite accumulation and sent off site through a Waste disposal vendor.
  Onyx Profile WIP Number: 590598
  Teris Profile Number 50016653

- Soil Retain Samples - These samples if not flagged in the system for any hazardous constituents are transferred to poly-lined cubic yard boxes. These boxes when full are sent to stabilization or incineration. These materials are sent out as hazardous for lead and chromium

  Teris Profile Number (incineration): 50016710
  Onyx Profile Number: (stabilization) 402535

15.0. References / Cross-References

15.1. Test Methods for Evaluating Solid Waste Physical/Chemical Methods (SW846), Third Edition, September 1986, Final Update I, July 1992, Final Update IIA, August 1993, Final Update II, September 1994; Final Update IIB, January 1995; Final Update III, December 1996; SW-846, Method 6010B.

15.2. TestAmerica Edison SOP ED-MTP-005, *Hot Block Digestion of Sediments, Sludges and Soils, SW846 Method 3050B,* most current revision.

15.3. TestAmerica Edison SOP ED-MTP-003, *Digestion of Water and Wastewater Samples for analysis by ICP, SW846 Method 3010A,* most current revision.

15.4. TestAmerica Edison Document No. ED-QA-LQM, *Laboratory Quality Manual*, most current revision.

15.5. TestAmerica Edison SOP ED-GEN-022, *Training*, most current revision.

15.6. TestAmerica Corporate SOP No. CA-Q-S-005, *Calibration Curves (General)*, most current revision.

15.7. iCAP 6000 Series ICP-OES Spectrometer Operator Manuals (8499 400 90001), Thermo Electron Corporation, 2005.

15.8. TestAmerica Corporate Environmental Health and Safety Manual, Document No. CW-E-M-001, most current revision.

16.0. Method Modifications:

None

17.0. Attachments

Attachment 1: Working Standard Concentration for ICP Elements in ug/L
Attachment 2: Matrix spike concentration for soil and aqueous samples
Attachment 3: Certified Values of each elements in multi-element stock standard

18.0. Revision History

- Revision 12, dated 29 June 2018

  - Sec 1.1: Added Method 6010D to the list of methods applicable to this SOP
  - Sec 1, Table 1: Updated Table to include RL's for 6010D
  - Throughout the SOP removed all references to Reagent Water and replaced with 5%HNO_3/5%HCl
  - Sec 7.2: Added ICV preparation instructions and frequency for method 6010D.
  - Sec 7.2.2.4 & 7.2.4.2: Added the CCV preparation instructions in both sections; removed the CCV preparation instructions in Section 7.2.4.1.
  - Sec 7.2.6: Added B, Ba, Ca, Fe, Mg, Se, Zn, Al, As, Pb to the list of stock standards.
  - Sec 7.2.6 & 7.2.7: Renamed "Stock Interference Check Standard" and 'Working Interference Check Standards" to Spectral Interference check solution (SIC).
  - Sec 7.2.9 & 7.2.10: revised the preparation instructions for Int-A and Int-B.
  - Sec 9.1 Table: Added MB and PDS control limit criteria for method 6010D
  - Sec 9.1.1: Added MB control limit criteria for method 6010D
  - Sec 9.1.5: Added PDS control limit criteria for method 6010D
  - Sec 9.1.6.5: Added serial dilution control limit criteria for method 6010D
  - Sec 9.2.1: Revised to remove CCV procedure (ICV standard source separate from CCV source).
  - Sec 9.2.2: Added CCV criteria; subsequent sections adjusted accordingly.
  - Sec 9.2.3.2: Added ICB & CCB control limit criteria for method 6010D
  - Sec 9.2.4.2: Added ICSA control limit criteria for method 6010D
  - Sec 9.2.7: Added LRC preparation and control limit criteria for method 6010D
  - Sec 10.2.1.1: Added axial/radial plasma view information in this section; deleted this information in Sec 12.
  - Sec 10.2.1.3: Replaced the semi-annual linear range verification with daily verification.
  - Sec 9.3.1.2: Added the IDL procedure for Method 6010D
  - Sec 9.3.3: Added Linear Range check (LRC) information; Revised the analytical sequence to analyze first CCV prior to regular samples.
  - Sec 9.3.4: Added Single Element Interference Check preparation procedure and criteria information
  - Sec 10.3.2: Added calibration curve correlation coefficient criteria for method 6010D.
  - Sec 10.3.3: Added calibration curve read-back instructions for method 6010D.
  - Sec 10.4.5: Added dilution instructions for analytes and interfering analytes that exceed their linear range.
  - Sec 12.6: Added LLOQ standard preparation and criteria information.
  - Attachment 1: Added LLQC and LLOQ on this Table.

- Revision 11, dated 27 April 2018
  - Attachment 1: Corrected the working standard concentration for CAL2 specifically Sr, Sn, Ti, V, Zn and Si.
  - Sec 9.1.2: Revised to clarify the acceptance criteria used in the COA when evaluating recoveries for LCS.
  - Sec 12.4: Revised to include LDR as a QC method performance. Specified which elements are viewed at axial plasma and radial plasma.
  - Attachment 1: Added the analytical wavelength (nm) for each of the elements.

- Revision 10, dated 06 October 2017
  - Sec 10.1: Deleted reference to SOP# ED-MTP-002, not applicable to this SOP.
  - Sec 11.5.4: Replaced Data Review Checklist (CA-Q-WI-043) with TALS program Data Review Checker (DRC).
  - Sec 15.7: Deleted reference Data Review Checklist (CA-Q-WI-043); subsequent sections adjusted accordingly.

- Revision 9, dated 29 September 2015
  - Combined SOP ED-MT-033 with SOP ED-MT-004; retired SOP ED-MT-033 at the effective date of this SOP.
  - Sec 1.1: Added 6010C to the list of methods covered in this SOP. Remove the exception for filtered groundwaters not needing a digestion step.
  - Sec 1, Table 1: Updated table to include RL's for 6010C.
  - Sec 1, 2.0 & 10.1: Removed reference to method 3005A.
  - Sec 7.1.4 & Sec 7.1.5: Replaced ICP Reagent Dilution logbook with TALS Reagent Module for the documentation of Standard/Reagent preparation.
  - Sec 7.2: Added instructions for the storage of standards which contain both Ag and HCl.
  - Sec 7.2.1: Updated the list of stock standards to reflect actual lab practices.
  - Sec 7.2.2.1- 7.2.2.2: Updated the standard preparation.
  - Sec 7.2.2.1.1 – 7.2.2.2.1: Added preparation instructions for Cal2.
  - Sec 7.2.6: Updated the list of standards.
  - Sec 7.2.9: Replaced Int-10 with Int-A; removed Int-10 Table and added preparation instructions for Int-A.
  - Sec 7.2.10: Added preparation instructions for Int-B.
  - Sec 9.1: Added MSD to the list of Sample QC. Updated the control limits for PDS to reflect method requirement.
  - Sec 9.1.7: Added MSD and quality control information for MSD.
  - Sec 9.2.4: Replaced Int-10 with Int-A & Int-B.
  - Sec 9.2.5: Added Low-Level Initial/Continuing Calibration Verification Standard (LLICV/LLCCV) for method 6010C.
  - Sec 10.3.1: Updated IDL study frequency and definition to reflect method requirements.
  - Sec 10.3.3: Added LLQC instructions for method 6010C.
  - Sec 10.4.2: Added correlation coefficient requirements for Method 6010C.
  - Sec 10.5.1.3: Added dilution instructions for typical aqueous (non-tclp) samples.
  - Sec 10.5.3: Updated to include Method 6010C; updated Table to include LLICV, LLCCV, Int-A & Int-B.
  - Sec 11.5.4: Replaced Metals ICAL Data Review checklist EDS-WI-124 with the standardized ICP data review checklist CA-Q-WI-043.

- Sec 15: Removed reference to SOP ED-MTP-002 (method 3005A) in Sec 15.4; subsequent sections adjusted accordingly. Sec 15.7: Deleted Data review checklist WI# EDS-WI-007, checklist is referenced in the applicable digestion SOP. Added the standardized Data review checklist WI# CA-Q-WI-043. Sec 15.10: Deleted reference WI# EDS-WI-124.
- Attachment 1: Updated elemental concentrations for Cal1 and Cal2, LLICV/LLCCV.
- Attachment 3: Updated COAs for all standards.

- Revision 8, dated 03 June 2013
  - Sec 1 and 12: Updated LQM section references to reflect the most current LQM.
  - Sec 6.1: Deleted TJA 61E ICP, the lab no longer uses this instrument. All references to this instrument have been removed from this SOP.
  - Sec 7.2.1: Added Cu, Ni, Fe, Si and Zn to the calibration stock standards.
  - Sec 7.2.2.1.: Revised the preparation procedure for standard CAL1 to reflect actual lab practices.
  - Sec 7.2.2.1.1: Added the preparation instructions of the intermediate standard ME_Cal1-int.
  - Sec 7.2.2.2-7.2.2.4: Sections revised to include Si standard.
  - Sec 7.2.2.5: Added the preparation instructions for standard CAL5.
  - Sec 7.2.3 & 7.2.4.1: Replaced Part No SM-606-098 with SM-606-122.
  - Sec 7.2.9: Revised to include Si to INT-10 standard.
  - Sec 9.1 & 9.1.1: Revised the control limit of Method blank to reflect SW846 criteria.
  - Sec 11.5.4: Added WI# EDS-WI-124, Initial Calibration Data Review, to metals data review checklist.
  - Sec 15: Deleted SOP reference ED-MT-013, not applicable; added EDS-WI-124 to the list of references for this SOP.
  - Attachment 1: Revised Table to update concentrations for standard CAL1.
  - Attachment 3: Revised COA attachments for SM-606-122.

- Revision 7, dated 25 April 2011
  - Sec 1.1: Revised Table 1 to include the reporting limits for Aqueous TCLP/Non-TCLP and soils; included the information in Table 2 to Table 1; deleted Table 2.
  - Sec 3: Updated the LQM reference for the definitions.
  - Sec 4.0: Expanded the possible interferences for this method.
  - Sec 6.1: Added iCAP 6500 Duo View ICP-OES to the list of instruments.
  - Sec 6.2: Revised to include Argon supply to the list of supplies; deleted argon supply previously listed in Sec 7.1.4. Deleted Nitrogen which is not used by this method. Subsequent sections adjusted accordingly.
  - Sec 7.1.5: Added 5% HNO3 as the reagent for the analysis of undigested samples
  - Sec 7.2.1: Added TANJ-STD-3 and TANJ-STD-4; deleted standards Zn and La
  - Sec 7.2.2: Updated the preparation of CAL1, CAL2, CAL3, and CAL4
  - Sec 7.2.3: Added Solutions: A, B, C, and D, Part No. SM-606-098 (Duo_CCV-int)
  - Sec 7.2.4.1: Updated the preparation of ICV/CCV
  - Sec 7.2.5.1: Added Solutions: A, B, C, and D, Part No. SM-606-098 (Duo_CCV-int).
  - Sec 7.2.5.2: Added Solutions A, B, C, and D, Part No. SM-606-114 (TCLP-Spk).
  - Sec 7.2.5.3.1: Updated the preparation of ME_LCS-int.
  - Sec 7.2.5.3.2: Added the preparation of ME_TCLPspk.
  - Deleted RepLim-INT, previously Sec 7.2.9, subsequent sections adjusted accordingly.
  - Sec 7.2.9: Replaced INT-20 with INT-10.

- Sec 8: Added footnotes 2 & 3.
- Sec 9: Deleted all entries of the Blank Spike (BS) and LCSD for this SOP.
- Sec 9.1.1: Updated pass/fail criteria for the MB.
- Deleted the following QC samples: HSA (previously sec 9.2.3), Replim and Replim2 (previously sec 9.2.6 & 9.2.7), subsequent sections adjusted accordingly.
- Sec 9.2.4: Updated pass/fail criteria for ICSA/ICSAB
- Sec 10.2: Added the TJA 6500 Duo instrument set up procedure.
- Sec 11.5: Updated Data Reduction section in accordance with TALS.
- Sec 15.0: Added applicable references.
- Updated Attachment 1 & 2 and removed Attachment 3 (Reporting Limt Check Standard – RepLim) and Attachment 4 (Interferent-20, Int-20). Attachment 5 (COAs) is now referred to as Attachment 3.

- Revision 1, dated June 26, 2008
  - Revised SOP format in accordance with TestAmerica Corporate Quality SOP No. CW-QS-002 (Rev 0) Writing a Standard Operating Procedure (SOP).
  - Added Table 2, reporting limits for soil and water by method 6010B.
  - Section 2: Included applicable digestion technique for method 6010B.
  - Section 7 Reagents and Standards: Added Reagent water (5% HNO3+5% HCl) in the list of reagents; included stability and storage information of all reagents and standards.
  - Section 7.2 Standards: Added standards (ICP-PSPK, RepLim-INT & INT-20) in the list of instrument QC samples performed on this method.
  - Section 8: Updated in Table format and have included sample container, sample size requirements and method reference.
  - Section 9 Quality Control: Updated to include all Sample and Instrument QC for both matrices (soil and water).
  - Section 9.1.6, Added Post-digestion spike and defined its spiking procedure, recovery limits and frequency.
  - Section 9.2 Instrument QC: Added instrument QCs such as RepLim, RepLim-2, INT-20 and described their frequency, control limits and spiking procedure.
  - Section 10 Procedure: Clarified steps performed during instrument operation including referencing TestAmerica SOP No. ED-MT-013.
  - Section 15 References: Added references applicable to the SOP.
  - Section 17 Attachment: Added attachments 2, 3 & 4 to list the elemental concentrations of BS, MS (for water and soil), RepLim, RepLim-2 and INT-20 in Table format.
  - Attachment 5: included certificate of analysis of stock standards to list concentration of each element in the mix.

Attachment 1

Working Standard Concentration for ICP Elements in ug/L

Element (wavelength, nm)	CAL1	CAL2 (LLICV/ LLCCV LLQC LLOQ)	CAL3	CAL4 ICV CCV	CAL5	ICSA	ICSAB (6010B 6010C)
Al (396.152)	n/a	200	25000	125000	250000	500000	500000
Sb (206.833)	10	20	200	1000	2000	n/a	100
As (189.042)	5	15	500	2500	5000	n/a	100
Ba (233.527)	n/a	200	2000	10000	20000	n/a	100
Be (313.042)	n/a	2	200	1000	2000	n/a	100
B (208.959)	n/a	50	200	1000	2000	n/a	100
Cd (226.502)	n/a	4	250	1250	2500	n/a	100
Ca (318.128)	n/a	5000	25000	125000	250000	500000	500000
Cr (267.716)	n/a	10	1000	5000	10000	n/a	100
Co (228.616)	n/a	50	500	2500	5000	n/a	100
Cu (324.754)	n/a	25	2500	12500	25000	n/a	100
Fe (271.441)	n/a	150	20000	100000	200000	200000	200000
Pb (220.353)	5	10	1500	7500	15000	n/a	100
Mn (257.610)	n/a	15	1000	5000	10000	n/a	100
Mg (279.079)	n/a	5000	25000	125000	250000	500000	500000
Mo (202.030)	n/a	20	500	2500	5000	n/a	100
Ni (231.604)	n/a	40	500	2500	5000	n/a	100
K (766.490)	n/a	5000	10000	50000	100000	n/a	10000
Se (196.090)	5	20	500	2500	5000	n/a	100
Ag (328.068)	n/a	10	250	1250	2500	n/a	100
Na (589.592)	n/a	5000	25000	125000	250000	n/a	10000
Sr (407.771)	n/a	20	1000	5000	10000	n/a	100
Tl (190.856)	10	20	500	2500	5000	n/a	100
Sn (189.989)	n/a	50	200	1000	2000	n/a	100
Ti (334.941)	n/a	20	2000	10000	20000	n/a	100
V (292.402)	n/a	50	500	2500	5000	n/a	100
Zn (206.200)	n/a	30	500	2500	5000	n/a	100
Si (288.158)	n/a	n/a	2000	10000	20000	n/a	100

Attachment 2

Matrix Spike Final Concentration in Solution (ug/L)			
ELEMENT	Aqueous (Non-TCLP)	TCLP	Soil
Aluminum	2000	5000	4000
Antimony	500	1000	1000
Arsenic	2000	5000	4000
Barium	2000	10000	4000
Beryllium	50	1000	100
Cadmium	50	1000	100
Calcium	20000	20000	40000
Chromium	200	5000	400
Cobalt	500	1000	1000
Copper	250	1000	500
Iron	1000	1000	2000
Lead	500	5000	1000
Manganese	500	1000	1000
Magnesium	20000	20000	40000
Nickel	500	1000	1000
Potassium	20000	20000	40000
Selenium	2000	1000	4000
Silver	50	500	100
Sodium	20000	20000	40000
Thallium	2000	1000	4000
Vanadium	500	500	1000
Zinc	500	1000	1000
Boron	500	1000	1000
Molybdenum	500	1000	1000
Tin	500	1000	1000
Titanium	500	1000	1000
Strontium	500	1000	1000

Attachment 3

INORGANIC VENTURES

CERTIFICATE OF ANALYSIS

300 Technology Drive
Christiansburg, VA 24073 • USA
inorganicventures.com

tel: 800.669.6799 • 540.585.3030
fax: 540.585.3012
info@inorganicventures.com

1.0 ACCREDITATION / REGISTRATION

INORGANIC VENTURES is accredited to ISO Guide 34, "General Requirements for the Competence of Reference Material Producers" and ISO/IEC 17025, "General Requirements for the Competence of Testing and Calibration Laboratories". Inorganic Ventures is also an ISO 9001 registered manufacturer (SAI Global File Number 010105).

ACCREDITED ACCREDITED

2.0 PRODUCT DESCRIPTION

Product Code:	Multi Analyte Custom Grade Solution
Catalog Number:	CLPP-CAL-1
Lot Number:	J2-MEB578011
Matrix:	5% (v/v) HNO3
Value / Analyte(s):	5 000 µg/mL ea: Ca, K, Mg, Na,
	2 000 µg/mL ea: Al, Ba,
	1 000 µg/mL ea: Fe,
	500 µg/mL ea: Co, Mn, Ni, V, Zn,
	250 µg/mL ea: Ag, Cu,
	200 µg/mL ea: Cr3,
	50 µg/mL ea: Be

3749900
ID: ME_CAL_1_00023
Exp: 08/15/18 Prpd: CDC
CLPP-CAL-1

rec'd 6/23/15

3.0 CERTIFIED VALUES AND UNCERTAINTIES

ANALYTE	CERTIFIED VALUE	ANALYTE	CERTIFIED VALUE
Aluminum, Al	1 999 ± 10 µg/mL	Barium, Ba	1 999 ± 10 µg/mL
Beryllium, Be	49.98 ± 0.27 µg/mL	Calcium, Ca	4 998 ± 23 µg/mL
Chromium+3, Cr3	199.9 ± 1.0 µg/mL	Cobalt, Co	499.8 ± 2.5 µg/mL
Copper, Cu	249.9 ± 1.2 µg/mL	Iron, Fe	1 000 ± 5 µg/mL
Magnesium, Mg	4 998 ± 23 µg/mL	Manganese, Mn	499.8 ± 2.2 µg/mL
Nickel, Ni	499.6 ± 2.4 µg/mL	Potassium, K	4 998 ± 22 µg/mL

INORGANIC VENTURES™
300 Technology Drive
Christiansburg, VA 24073 • USA
inorganicventures.com

tel: 800.669.6799 • 540.585.3030
fax: 540.585.3012
info@inorganicventures.com

CERTIFICATE OF ANALYSIS

1.0 ACCREDITATION / REGISTRATION

INORGANIC VENTURES is accredited to ISO Guide 34, "General Requirements for the Competence of Reference Material Producers" and ISO/IEC 17025, "General Requirements for the Competence of Testing and Calibration Laboratories". Inorganic Ventures is also an ISO 9001 registered manufacturer (SAI Global File Number (010105).

ACCREDITED Testing Laboratory Certificate 83101 ACCREDITED Reference Material Producer Certificate 83102

2.0 PRODUCT DESCRIPTION

Product Code:	Multi Analyte Custom Grade Solution
Catalog Number:	CLPP-CAL-3
Lot Number:	G2-MEB501091
Matrix:	7% (v/v) HNO3
Value / Analyte(s):	1 000 µg/mL ea: As, Pb, Se, Tl, 500 µg/mL ea: Cd

3749920
ID: ME_CAL-3_00023
Exp: 11/26/16 Prpd: CDC
CLPP-CAL-3, calibration s

rec'd 6/23/15

3.0 CERTIFIED VALUES AND UNCERTAINTIES

ANALYTE	CERTIFIED VALUE	ANALYTE	CERTIFIED VALUE
Arsenic	1 000 ± 7 µg/mL	Cadmium	500.0 ± 3.2 µg/mL
Lead	1 000 ± 7 µg/mL	Selenium	1 000 ± 6 µg/mL
Thallium	1 000 ± 7 µg/mL		

Certified Density: 1.045 g/mL (measured at 20 ± 1 °C)

Assay Information:

ANALYTE	METHOD	NIST SRM#	SRM LOT#
As	Calculated		See Sec. 4.2
As	ICP Assay	3103a	100818
Cd	ICP Assay	3108	060531
Cd	EDTA	928	928
Pb	ICP Assay	3128	101026
Pb	EDTA	928	928
Se	Calculated		See Sec. 4.2
Se	ICP Assay	3149	100901
Tl	Calculated		See Sec. 4.2
Tl	ICP Assay	3158	993012

The following equations are used in the calculation of the certified value and the uncertainty. Reported uncertainties represent expanded uncertainties expressed at approximately the 95% confidence level using a coverage factor of k = 2.

INORGANIC VENTURES™
300 Technology Drive
Christiansburg, VA 24073 · USA
inorganicventures.com

CERTIFICATE OF ANALYSIS

tel: 800.669.6799 · 540.585.3030
fax: 540.585.3012
info@inorganicventures.com

1.0 **INORGANIC VENTURES** is an ISO Guide 34 "General Requirements for the Competence of Reference Material Producers" and ISO 9001 registered manufacturer. Our manufacturing laboratory is accredited to ISO/IEC 17025 "General Requirements for the Competence of Testing and Calibration Laboratories."

ACCREDITED Testing Laboratory Certificate 883.01

ACCREDITED Reference Material Producer Certificate 883.02

rec'd 3/31/14 LD

2.0 **DESCRIPTION OF CRM** **Custom Solution**

Catalog No.: STLNJ-CAL-1A

Lot Number: **H2-MEB516143**

Matrix: 3% HNO3(v/v)

2855554
ID: ME_CAL_1A_00009
Exp: 05/01/15 Prpd: LBO
STLNJ-CAL-1A

2,250 µg/mL ea:
Cu,

1,000 µg/mL ea:
Pb, Sr,

800 µg/mL ea:
Cr3,

500 µg/mL ea:
Mn,

200 µg/mL ea:
B,

150 µg/mL ea:
Be

3.0 CERTIFIED VALUES AND UNCERTAINTIES

ELEMENT	CERTIFIED VALUE	ELEMENT	CERTIFIED VALUE	ELEMENT	CERTIFIED VALUE
Beryllium, Be	150.0 ± 0.8 µg/mL	Boron, B	200.0 ± 1.3 µg/mL	Chromium+3, Cr3	800 ± 5 µg/mL
Copper, Cu	2,250 ± 15 µg/mL	Lead, Pb	1,000 ± 7 µg/mL	Manganese, Mn	500.2 ± 2.2 µg/mL
Strontium, Sr	1,000 ± 6 µg/mL				

Certified Density: 1.032 g/mL (measured at 20 ± 1° C)

INORGANIC VENTURES™
300 Technology Drive
Christiansburg, VA 24073 · USA
inorganicventures.com

CERTIFICATE OF ANALYSIS

tel: 800.669.6799 540.585.3030
fax: 540.585.3012
info@inorganicventures.com

1.0 **INORGANIC VENTURES** is an ISO Guide 34 "General Requirements for the Competence of Reference Material Producers" and ISO 9001 registered manufacturer. Our manufacturing laboratory is accredited to ISO/IEC 17025 "General Requirements for the Competence of Testing and Calibration Laboratories."

ACCREDITED Testing Laboratory Certificate 853.01
ACCREDITED Reference Material Producer Certificate 853.02

rec'd 3/31/14 LD

2.0 **DESCRIPTION OF CRM**

	Custom Solution
Catalog No.:	STLNJ-CAL-1B
Lot Number:	**H2-MEB516144**
Matrix:	3% HNO3(v/v), tr. HF

2855556
ID: ME_CAL_1B_00009
Exp: 05/01/15 Prpd: LBO
STLNJ-CAL-1B

2,000 µg/mL ea:
Ti,

500 µg/mL ea:
Mo,

200 µg/mL ea:
Sb, Sn

3.0 **CERTIFIED VALUES AND UNCERTAINTIES**

ELEMENT	CERTIFIED VALUE	ELEMENT	CERTIFIED VALUE	ELEMENT	CERTIFIED VALUE
Antimony, Sb	200.0 ± 1.6 µg/mL	Molybdenum, Mo	500.0 ± 2.5 µg/mL	Tin, Sn	200.1 ± 1.4 µg/mL
Titanium, Ti	2,000 ± 13 µg/mL				

Certified Density: 1.022 g/mL (measured at 20 ± 1° C)

The following equations are used in the calculation of the certified value and the uncertainty. Reported uncertainties represent expanded uncertainties expressed at approximately the 95% confidence level using a coverage factor of k = 2.

Certified Value $(\bar{x}) = \frac{\Sigma x_i}{n}$

Uncertainty $(\pm) = 2\,[\,\Sigma(s_i)^2\,]^{1/2}$

($\bar{x}$) = mean
x_i = individual results
n = number of measurements
2 = the coverage factor.
$[\,\Sigma(s_i)^2\,]^{1/2}$ = The square root of the sum of the squares of the most common errors (where 's' stands for the standard deviation) from instrumental measurement, density, NIST SRM uncertainty, weighing, dilution to volume, homogeneity, long term stability and short term stability.

INORGANIC VENTURES™
300 Technology Drive
Christiansburg, VA 24073 · USA
inorganicventures.com

CERTIFICATE OF ANALYSIS

tel: 800.669.6799 · 540.585.3030
fax: 540.585.3012
info@inorganicventures.com

1.0 **INORGANIC VENTURES** is an ISO Guide 34 "General Requirements for the Competence of Reference Material Producers" and ISO 9001 registered manufacturer. Our manufacturing laboratory is accredited to ISO/IEC 17025 "General Requirements for the Competence of Testing and Calibration Laboratories."

ACCREDITED Testing Laboratory Certificate 853.01

ACCREDITED Reference Material Producer Certificate 853.02

rec'd 3/31/14 LD

2.0 **DESCRIPTION OF CRM** **Custom Solution**

Catalog No.: STLNJ-CAL-2

Lot Number: **H2-MEB516145**

Matrix: 3% HNO3(v/v)

2855552
ID: ME_CAL_2_00010
Exp: 05/01/15 Prpd: LBD
STLNJ-CAL-2

10,000 µg/mL ea:
Ca, Mg, Na,

9,500 µg/mL ea:
Fe,

2,500 µg/mL ea:
K

3.0 **CERTIFIED VALUES AND UNCERTAINTIES**

ELEMENT	CERTIFIED VALUE	ELEMENT	CERTIFIED VALUE	ELEMENT	CERTIFIED VALUE
Calcium, Ca	10,000.0 ± 70.0 µg/mL	Iron, Fe	9,500.0 ± 40.0 µg/mL	Magnesium, Mg	10,000.0 ± 60.0 µg/mL
Potassium, K	2,500 ± 11 µg/mL	Sodium, Na	10,000.0 ± 40.0 µg/mL		

Certified Density: 1.157 g/mL (measured at 20 ± 1° C)

The following equations are used in the calculation of the certified value and the uncertainty. Reported uncertainties represent expanded uncertainties expressed at approximately the 95% confidence level using a coverage factor of k = 2.

Certified Value $(\bar{x}) = \frac{\Sigma x_i}{n}$

($\bar{x}$) = mean
x_i = individual results
n = number of measurements

Uncertainty $(\pm) = 2\,[\,\Sigma(s_i)^2\,]^{1/2}$

2 = the coverage factor.
$[\,\Sigma(s_i)^2\,]^{1/2}$ = The square root of the sum of the squares of the most common errors (where 's' stands for the standard deviation) from instrumental measurement, density, NIST SRM uncertainty, weighing, dilution to volume, homogeneity, long term stability and short term stability.

INORGANIC VENTURES™
300 Technology Drive
Christiansburg, VA 24073 • USA
inorganicventures.com

Rev'd 4/30/15
a.c.

CERTIFICATE OF ANALYSIS

tel: 800.669.6799 • 540.585.3030
fax: 540.585.3012
info@inorganicventures.com

1.0 ACCREDITATION / REGISTRATION

INORGANIC VENTURES is accredited to ISO Guide 34, "General Requirements for the Competence of Reference Material Producers" and ISO/IEC 17025, "General Requirements for the Competence of Testing and Calibration Laboratories". Inorganic Ventures is also an ISO 9001 registered manufacturer (SAI Global File Number 010105).

ACCREDITED Testing Laboratory Certificate 863.01
ACCREDITED Reference Material Producer Certificate 863.02

2.0 PRODUCT DESCRIPTION

3647218
ID: ME-TANJ-STD-3_00009
Exp: 05/01/16 Prpd: CDC
REPORTING LIMIT INT

Product Code:	Multi Analyte Custom Grade Solution
Catalog Number:	TANJ-STD-3
Lot Number:	H2-MEB546087
Matrix:	5% (v/v) HNO3
Value / Analyte(s):	500 µg/mL ea:

Ca,	K,	Mg,
Na,		

20 µg/mL ea:

Al,	Ba,

15 µg/mL ea:

Fe,

5 µg/mL ea:

Co,	V,

4 µg/mL ea:

Ni,

3 µg/mL ea:

Zn,

2.5 µg/mL ea:

Cu,

2 µg/mL ea:

Sr,

1.5 µg/mL ea:

Mn,

1 µg/mL ea:

Ag,	Cr3,	Tl,

0.5 µg/mL ea:

As,	Pb,	Se,

0.4 µg/mL ea:

Cd,

0.2 µg/mL ea:

Be

3.0 CERTIFIED VALUES AND UNCERTAINTIES

INORGANIC VENTURES™
300 Technology Drive
Christiansburg, VA 24073 • USA
inorganicventures.com

Rev'd 4/30/15
c.c.

CERTIFICATE OF ANALYSIS

tel: 800.669.6799 • 540.585.3030
fax: 540.585.3012
info@inorganicventures.com

1.0 ACCREDITATION / REGISTRATION

INORGANIC VENTURES is accredited to ISO Guide 34, "General Requirements for the Competence of Reference Material Producers" and ISO/IEC 17025, "General Requirements for the Competence of Testing and Calibration Laboratories". Inorganic Ventures is also an ISO 9001 registered manufacturer (SAI Global File Number 010105).

ACCREDITED ACCREDITED

2.0 PRODUCT DESCRIPTION

Product Code:	Multi Analyte Custom Grade Solution
Catalog Number:	TANJ-STD-4
Lot Number:	H2-MEB546088
Matrix:	5% (v/v) HNO3 tr. HF
Value / Analyte(s):	5 µg/mL ea: B, Sn, 2 µg/mL ea: Mo, Ti, 1 µg/mL ea: Sb

3647220
ID: ME-TANJ-STD-4_00009
Exp: 05/01/16 Prpd: CDC
REPORTING LIMIT INT

3.0 CERTIFIED VALUES AND UNCERTAINTIES

ANALYTE	CERTIFIED VALUE	ANALYTE	CERTIFIED VALUE
Antimony, Sb	1.000 ± 0.008 µg/mL	Boron, B	5.001 ± 0.032 µg/mL
Molybdenum, Mo	2.000 ± 0.018 µg/mL	Tin, Sn	5.000 ± 0.032 µg/mL
Titanium, Ti	2.000 ± 0.016 µg/mL		

Certified Density: 1.025 g/mL (measured at 20 ± 1 °C)

Assay Information:

ANALYTE	METHOD	NIST SRM#	SRM LOT#
B	Calculated		See Sec. 4.2
B	ICP Assay	3107	070514
Mo	Calculated		See Sec. 4.2
Mo	ICP Assay	3134	891307
Sb	Calculated		See Sec. 4.2
Sb	ICP Assay	3102A	061229
Sn	Calculated		See Sec. 4.2
Sn	ICP Assay	3161a	070330
Ti	Calculated		See Sec. 4.2
Ti	ICP Assay	3162a	060808

The following equations are used in the calculation of the certified value and the uncertainty. Reported uncertainties represent expanded uncertainties expressed at approximately the 95% confidence level using a coverage factor of k = 2.

HIGH-PURITY STANDARDS
www.highpuritystandards.com

P.O. Box 41727
Charleston, SC 29423
Phone (843) 767-7900
Fax (843) 767-7906

Certificate of Analysis

Product Description:

Part Number:	**SM-606-122 Solution A**
Lot Number:	**1505111**
Matrix:	5% HNO_3
Purity:	99.98% - 99.999%

3548489
ID: ME_Duo_CCV_A_00007
Exp: 03/02/16 Prpd: CDC
Int. CCV Soln A for Duo

Certified Values:

Element	(µg/mL)	SRM ID	SRM Lot#	Element	(µg/mL)	SRM ID	SRM Lot#
Ca	12500.0 ± 62.5	3109a	050825	K	5000 ± 25	*	
Mg	12500.0 ± 62.5	3131a	050302	Na	12500.0 ± 62.5	3152a	120715

The Certified values are based on gravimetric and volumetric preparation, and verified against SRM 3100 series developed by National Institute of Standards and Technology (NIST) via inductively coupled plasma optical emission spectrometry (ICP-OES) using an internal laboratory developed method. The uncertainty in the certified value is calculated for a 95% confidence interval and coverage factor *k* is about 2
* Refer to Traceability Information, Section d

Preparation Information:

The standard is generally prepared from single element standard solutions that are ISO Guide 34 certified reference materials. Highest purity source materials were purchased from qualified vendors per ISO 9001:2008 guidelines and assayed by ICP-OES for conformity prior to use. Sub-boiling distilled high-purity acid has been used to place the materials in solution and to stabilize the standard. The matrix is as noted above in 18 megaohm deionized water.

Traceability Information:

The traceability of this standard is maintained through an unbroken chain of comparisons to appropriate standards with suitable procedure and measurement uncertainties. The maintenance of the base and derived units of International System of Units (SI) with traceability of measurement results (contemporary metrology) to SI ensures their comparability over time as follows.

a. **Standard Weight and Analytical Balance**
The standard weights (NBS weights Inventory No 20231A) are calibrated every two years by South Carolina Metrology Laboratory that is a participant in "NIST Weights and Measures Measurement Assurance Program" with a certificate of measurement traceability to NIST primary standards.
The balances are calibrated yearly by the ISO 17025 accredited metrology service, and are verified weekly by an in-house method using standard weights.

b. **Volumetric Device**
The calibration of volumetric vessels is checked annually using the ASTM method E542

Company Confidential & Proprietary

HIGH-PURITY STANDARDS
www.highpuritystandards.com

P.O. Box 41727
Charleston, SC 29423
Phone (843) 767-7900
Fax (843) 767-7906

Certificate of Analysis

Product Description:

3548491
ID: ME_Duo_CCV_B_00007
Exp: 03/02/16 Prpd: CDC
Int. CCV Soln B for Duo

Part Number:	**SM-606-122 Solution B**
Lot Number:	**1505112**
Matrix:	10% HNO_3
Purity:	99.99% - 99.9995%

Certified Values:

Element	(µg/mL)	SRM ID	SRM Lot#	Element	(µg/mL)	SRM ID	SRM Lot#
Al	12500.0 ± 62.5	3101a	060502	Fe	10000 ± 50	3126a	140812

The Certified values are based on gravimetric and volumetric preparation, and verified against SRM 3100 series developed by National Institute of Standards and Technology (NIST) via inductively coupled plasma optical emission spectrometry (ICP-OES) using an internal laboratory developed method. The uncertainty in the certified value is calculated for a 95% confidence interval and coverage factor *k* is about 2
* Refer to Traceability Information, Section d

Preparation Information:

The standard is generally prepared from single element standard solutions that are ISO Guide 34 certified reference materials. Highest purity source materials were purchased from qualified vendors per ISO 9001:2008 guidelines and assayed by ICP-OES for conformity prior to use. Sub-boiling distilled high-purity acid has been used to place the materials in solution and to stabilize the standard. The matrix is as noted above in 18 megaohm deionized water.

Traceability Information:

The traceability of this standard is maintained through an unbroken chain of comparisons to appropriate standards with suitable procedure and measurement uncertainties. The maintenance of the base and derived units of International System of Units (SI) with traceability of measurement results (contemporary metrology) to SI ensures their comparability over time as follows.

a. **Standard Weight and Analytical Balance**
The standard weights (NBS weights Inventory No 20231A) are calibrated every two years by South Carolina Metrology Laboratory that is a participant in "NIST Weights and Measures Measurement Assurance Program" with a certificate of measurement traceability to NIST primary standards.
The balances are calibrated yearly by the ISO 17025 accredited metrology service, and are verified weekly by an in-house method using standard weights.

b. **Volumetric Device**
The calibration of volumetric vessels is checked annually using the ASTM method E542

Company Confidential & Proprietary

HIGH-PURITY STANDARDS
www.highpuritystandards.com
P.O. Box 41727
Charleston, SC 29423
Phone (843) 767-7900
Fax (843) 767-7906

Certificate of Analysis

Product Description:

Part Number:	**SM-606-122 Solution C**
Lot Number:	**1505113**
Matrix:	5% HNO_3
Purity:	99.98% - 99.9999%

3548499
ID: ME_Duo_CCV_C_00007
Exp: 03/02/16 Prpd: CDC
Int. CCV Soln C for Duo

Certified Values:

Element	(µg/mL)	SRM ID	SRM Lot#	Element	(µg/mL)	SRM ID	SRM Lot#
As	250.0 ± 2.5	3103a	100818	Pb	750.0 ± 4.5	3128	101026
Ba	1000 ± 6	*		Mn	500 ± 5	3132	050429
Be	100 ± 1	3105a	090514	Ni	250.00 ± 1.25	3136	120619
B	100.0 ± 0.5	3107	070514	Se	250.0 ± 2.5	3149	100901
Cd	125.00 ± 0.63	3108	130116	Sr	500.0 ± 2.5	3153a	990906
Cr	500.0 ± 2.5	3112a	030730	Tl	250.00 ± 1.25	3158	993012
Co	250.00 ± 1.25	3113	000630	V	250.0 ± 1.5	3165	992706
Cu	1250.00 ± 6.25	3114	121207	Zn	250.00 ± 1.25	3168a	120629

The Certified values are based on gravimetric and volumetric preparation, and verified against SRM 3100 series developed by National Institute of Standards and Technology (NIST) via inductively coupled plasma optical emission spectrometry (ICP-OES) using an internal laboratory developed method. The uncertainty in the certified value is calculated for a 95% confidence interval and coverage factor *k* is about 2
* Refer to Traceability Information, Section d

Preparation Information:

The standard is generally prepared from single element standard solutions that are ISO Guide 34 certified reference materials. Highest purity source materials were purchased from qualified vendors per ISO 9001:2008 guidelines and assayed by ICP-OES for conformity prior to use. Sub-boiling distilled high-purity acid has been used to place the materials in solution and to stabilize the standard. The matrix is as noted above in 18 megaohm deionized water.

Traceability Information:

The traceability of this standard is maintained through an unbroken chain of comparisons to appropriate standards with suitable procedure and measurement uncertainties. The maintenance of the base and derived units of International System of Units (SI) with traceability of measurement results (contemporary metrology) to SI ensures their comparability over time as follows.

a. **Standard Weight and Analytical Balance**

Company Confidential & Proprietary

HIGH-PURITY STANDARDS
www.highpuritystandards.com
P.O. Box 41727
Charleston, SC 29423
Phone (843) 767-7900
Fax (843) 767-7906

Certificate of Analysis

Product Description:

Part Number:	**SM-606-122 Solution D**
Lot Number:	**1505115**
Matrix:	Tr HF + 2% HNO_3
Purity:	99.95% - 99.999%

3548503
ID: ME_Duo_CCV_D_00007
Exp: 03/02/16 Prpd: CDC
Int. CCV Soln D for Duo

Certified Values:

Element	(µg/mL)	SRM ID	SRM Lot#	Element	(µg/mL)	SRM ID	SRM Lot#
Sb	100.0 ± 0.6	*		Ag	125.00 ± 0.63	3151	992212
Mo	250.0 ± 1.5	3134	891307	Sn	100.0 ± 0.6	3161a	070330
Si	1000 ± 10	*		Ti	1000 ± 6	3162a	130925

The Certified values are based on gravimetric and volumetric preparation, and verified against SRM 3100 series developed by National Institute of Standards and Technology (NIST) via inductively coupled plasma optical emission spectrometry (ICP-OES) using an internal laboratory developed method. The uncertainty in the certified value is calculated for a 95% confidence interval and coverage factor *k* is about 2
* Refer to Traceability Information, Section d

Preparation Information:

The standard is generally prepared from single element standard solutions that are ISO Guide 34 certified reference materials. Highest purity source materials were purchased from qualified vendors per ISO 9001:2008 guidelines and assayed by ICP-OES for conformity prior to use. Sub-boiling distilled high-purity acid has been used to place the materials in solution and to stabilize the standard. The matrix is as noted above in 18 megaohm deionized water.

Traceability Information:

The traceability of this standard is maintained through an unbroken chain of comparisons to appropriate standards with suitable procedure and measurement uncertainties. The maintenance of the base and derived units of International System of Units (SI) with traceability of measurement results (contemporary metrology) to SI ensures their comparability over time as follows.

a. **Standard Weight and Analytical Balance**
The standard weights (NBS weights Inventory No 20231A) are calibrated every two years by South Carolina Metrology Laboratory that is a participant in "NIST Weights and Measures Measurement Assurance Program" with a certificate of measurement traceability to NIST primary standards.
The balances are calibrated yearly by the ISO 17025 accredited metrology service, and are verified weekly by an in-house method using standard weights.

b. **Volumetric Device**
The calibration of volumetric vessels is checked annually using the ASTM method E542

Company Confidential & Proprietary

Certificate of Analysis

Expiry: 14-Jun-16

Rev'd 1/8/15
a.a.

Part Number: 4400-100419DD01
Lot Number: 14L124
Shelf Life: 18 Months

3453180
ID: ME_LCSspkA_00009
Exp: 06/14/16 Prp: CDC
LCS & MS spk Solution A

SOLN A
DE
9-8-15

TestAmerica/Edison
Custom Standard
5% HNO3

Concentrations in ug/mL ± 0.5%

Fe	1000	Zn	500
Se	2000	Mn	500
TL	2000	Ni	500
Be	50		
Cd	50		
Cr	200		
Co	500		
Cu	250		
Pb	500		
Ag	50		
Sr	500		
V	500		

This standard solution was prepared using high-purity starting materials, high-purity acid (if required) and 18-megaohm de-ionized water. The starting materials were weighed to five significant figures and diluted in volumetric glassware calibrated to five significant figures.

Starting materials were analyzed at 1000µg/mL by ICP-MS for trace impurities. The standard solution concentrations were certified instrumentally against the National Institute of Standards and Technology's SRM 3100 series, NIST approved second source and/or gravimetrically.

Accuracy and stability are guaranteed to within plus or minus 0.5% of the certified value for the stated shelf life from the date of shipment. The solution should be kept tightly capped and stored under normal laboratory conditions. See attached MSDS for proper handling information.

For questions or comments please call 1-800-878-7654 in the USA, +31 20 638 05 97 in Europe or visit our web-site at www.cpiinternational.com.

CPI INTERNATIONAL
www.cpiinternational.com

USA
5580 Skylane Boulevard, Santa Rosa, CA 95403
Phone: 1 (800) 878-7654 • Fax: 1 (707) 545-7901
E-mail: sales@cpiinternational.com

Europe
PO Box 2704, 1000 CS Amsterdam
Phone: +31 20 638 05 97 • Fax: +31 20 420 28 36
E-mail: sales@cpiinternational.com

• ISO • 9001:2008 REGISTERED 17025 ACCREDITED

Certificate of Analysis

Rev'd 1/8/15
C.C.

Expiry: 18-Jun-16

Part Number: 4400-100419DD01
Lot Number: 14L126
Shelf Life: 18 Months

Solution B

3453212
ID: ME_LCSspkB_00009
Exp: 06/18/16 Prpd: CDC
LCS & MS spk Solution B

TestAmerica/Edison
Custom Standard
10% HNO3

Concentrations in ug/mL ± 0.5%

Al	2000
Mg	20000
K	20000
Ca	20000
Na	20000

This standard solution was prepared using high-purity starting materials, high-purity acid (if required) and 18-megaohm de-ionized water. The starting materials were weighed to five significant figures and diluted in volumetric glassware calibrated to five significant figures.

Starting materials were analyzed at 1000µg/mL by ICP-MS for trace impurities. The standard solution concentrations were certified instrumentally against the National Institute of Standards and Technology's SRM 3100 series, NIST approved second source and/or gravimetrically.

Accuracy and stability are guaranteed to within plus or minus 0.5% of the certified value for the stated shelf life from the date of shipment. The solution should be kept tightly capped and stored under normal laboratory conditions. See attached MSDS for proper handling information.

For questions or comments please call 1-800-878-7654 in the USA, +31 20 638 05 97 in Europe or visit our web-site at www.cpiinternational.com.

Certifying Officer

CPI INTERNATIONAL
www.cpiinternational.com

USA
5580 Skylane Boulevard, Santa Rosa, CA 95403
Phone: 1 (800) 878-7654 • Fax: 1 (707) 545-7901
E-mail: sales@cpiinternational.com

Europe
PO Box 2704, 1000 CS Amsterdam
Phone: +31 20 638 05 97 • Fax: +31 20 420 28 36
E-mail: sales@cpiinternational.com

• ISO • 9001:2008 REGISTERED 17025 ACCREDITED

Certificate of Analysis

Expiry: 14-Jun-16

Rev'd 1/8/15 C.C.

Part Number: **4400-100419DD01** **Solution C**
Lot Number: **14L127**
Shelf Life: **18 Months**

3453214
ID: ME_LCSspkC_00010
Exp: 06/14/16 Prpd: CDC
LCS & MS spk Solution C

TestAmerica/Edison
Custom Standard
5% HNO3 + 2% HF

Concentrations in ug/mL ± 0.5%

Sb	500
Mo	500
Sn	500
Ti	500

This standard solution was prepared using high-purity starting materials, high-purity acid (if required) and 18-megaohm de-ionized water. The starting materials were weighed to five significant figures and diluted in volumetric glassware calibrated to five significant figures.

Starting materials were analyzed at 1000µg/mL by ICP-MS for trace impurities. The standard solution concentrations were certified instrumentally against the National Institute of Standards and Technology's SRM 3100 series, NIST approved second source and/or gravimetrically.

Accuracy and stability are guaranteed to within plus or minus 0.5% of the certified value for the stated shelf life from the date of shipment. The solution should be kept tightly capped and stored under normal laboratory conditions. See attached MSDS for proper handling information.

For questions or comments please call 1-800-878-7654 in the USA, +31 20 638 05 97 in Europe or visit our web-site at www.cpiinternational.com.

CPI INTERNATIONAL
www.cpiinternational.com

USA
5580 Skylane Boulevard, Santa Rosa, CA 95403
Phone: 1 (800) 878-7654 • Fax: 1 (707) 545-7901
E-mail: sales@cpiinternational.com

Europe
PO Box 2704, 1000 CS Amsterdam
Phone: +31 20 638 05 97 • Fax: +31 20 420 28 36
E-mail: sales@cpiinternational.com

• ISO • 9001:2008 REGISTERED 17025 ACCREDITED

Certificate of Analysis

Expiry: 14-Jun-16

Revd 1/8/15
c.c.

Part Number: **4400-100419DD01** **Solution D**
Lot Number: **14L128**
Shelf Life: **18 Months**

3453216
ID: ME_LCSspkD_00005
Exp: 06/14/16 Prpd: CDC
LCS & MS spk Solution D

TestAmerica/Edison
Custom Standard
2% HNO3

Concentrations in ug/mL ± 0.5%

As	2000
Ba	2000
B	500

This standard solution was prepared using high-purity starting materials, high-purity acid (if required) and 18-megaohm de-ionized water. The starting materials were weighed to five significant figures and diluted in volumetric glassware calibrated to five significant figures.

Starting materials were analyzed at 1000µg/mL by ICP-MS for trace impurities. The standard solution concentrations were certified instrumentally against the National Institute of Standards and Technology's SRM 3100 series, NIST approved second source and/or gravimetrically.

Accuracy and stability are guaranteed to within plus or minus 0.5% of the certified value for the stated shelf life from the date of shipment. The solution should be kept tightly capped and stored under normal laboratory conditions. See attached MSDS for proper handling information.

For questions or comments please call 1-800-878-7654 in the USA, +31 20 638 05 97 in Europe or visit our web-site at www.cpiinternational.com.

CPI INTERNATIONAL
www.cpiinternational.com

USA
5580 Skylane Boulevard, Santa Rosa, CA 95403
Phone: 1 (800) 878-7654 • Fax: 1 (707) 545-7901
E-mail: sales@cpiinternational.com

Europe
PO Box 2704, 1000 CS Amsterdam
Phone: +31 20 638 05 97 • Fax: +31 20 420 28 36
E-mail: sales@cpiinternational.com

• ISO • 9001:2008 REGISTERED 17025 ACCREDITED

HIGH-PURITY STANDARDS
www.highpuritystandards.com

P.O. Box 41727
Charleston, SC 29423
Phone (843) 767-7900
Fax (843) 767-7906

Certificate of Analysis

Product Description:

Name:	TCLP-Spk
Part Number:	**SM-606-114**
	Solution A
Lot Number:	**1505632**
Matrix:	4% HNO_3
Purity:	99.99% - 99.9995%

3548481
ID: ME_TCLP_A_00011
Exp: 03/02/18 Prpd: CDC
TCLP solution A stock

Certified Values:

Element	(µg/mL)	SRM ID	SRM Lot#	Element	(µg/mL)	SRM ID	SRM Lot#
Ba	5000 ± 30	*		Mn	500 ± 5	3132	050429
Be	500 ± 5	3105a	090514	Ni	500.0 ± 2.5	3136	120619
Cd	500.0 ± 2.5	3108	130116	Se	500 ± 5	3149	100901
Co	500.0 ± 2.5	3113	000630	Sr	500.0 ± 2.5	3153a	990906
Cu	500.0 ± 2.5	3114	121207	Tl	500.0 ± 2.5	3158	993012
Fe	500.0 ± 2.5	3126a	140812	Zn	500.0 ± 2.5	3168a	120629

The Certified values are based on gravimetric and volumetric preparation, and verified against SRM 3100 series developed by National Institute of Standards and Technology (NIST) via inductively coupled plasma optical emission spectrometry (ICP-OES) using an internal laboratory developed method. The uncertainty in the certified value is calculated for a 95% confidence interval and coverage factor *k* is about 2

* Refer to Traceability Information, Section d

Preparation Information:

The standard is generally prepared from single element standard solutions that are ISO Guide 34 certified reference materials. Highest purity source materials were purchased from qualified vendors per ISO 9001:2008 guidelines and assayed by ICP-OES for conformity prior to use. Sub-boiling distilled high-purity acid has been used to place the materials in solution and to stabilize the standard. The matrix is as noted above in 18 megaohm deionized water.

Traceability Information:

The traceability of this standard is maintained through an unbroken chain of comparisons to appropriate standards with suitable procedure and measurement uncertainties. The maintenance of the base and derived units of International System of Units (SI) with traceability of measurement results (contemporary metrology) to SI ensures their comparability over time as follows.

Company Confidential & Proprietary

Rec'd 7/7/14 CC.

HIGH-PURITY STANDARDS
www.highpuritystandards.com

P.O. Box 41727
Charleston, SC 29423
Phone (843) 767-7900
Fax (843) 767-7906

Certificate of Analysis

Product Description:

3548483
ID: ME_TCLP_B_00008
Exp: 03/02/16 Prpd: CDC
TCLP solution B stock

Name:	TCLP-Spk
Part Number:	**SM-606-114 Solution B**
Lot Number:	**1505633**
Matrix:	5% HNO_3
Purity:	99.98% - 99.999%

Certified Values:

Element	(µg/mL)	SRM ID	SRM Lot#	Element	(µg/mL)	SRM ID	SRM Lot#
Ca	10000 ± 50	3109a	050825	K	10000 ± 50	*	
Mg	10000 ± 50	*		Na	10000 ± 50	3152a	120715

The Certified values are based on gravimetric and volumetric preparation, and verified against SRM 3100 series developed by National Institute of Standards and Technology (NIST) via inductively coupled plasma optical emission spectrometry (ICP-OES) using an internal laboratory developed method. The uncertainty in the certified value is calculated for a 95% confidence interval and coverage factor *k* is about 2
* Refer to Traceability Information, Section d

Preparation Information:

The standard is generally prepared from single element standard solutions that are ISO Guide 34 certified reference materials. Highest purity source materials were purchased from qualified vendors per ISO 9001:2008 guidelines and assayed by ICP-OES for conformity prior to use. Sub-boiling distilled high-purity acid has been used to place the materials in solution and to stabilize the standard. The matrix is as noted above in 18 megaohm deionized water.

Traceability Information:

The traceability of this standard is maintained through an unbroken chain of comparisons to appropriate standards with suitable procedure and measurement uncertainties. The maintenance of the base and derived units of International System of Units (SI) with traceability of measurement results (contemporary metrology) to SI ensures their comparability over time as follows.

a. **Standard Weight and Analytical Balance**
The standard weights (NBS weights Inventory No 20231A) are calibrated every two years by South Carolina Metrology Laboratory that is a participant in "NIST Weights and Measures Measurement Assurance Program" with a certificate of measurement traceability to NIST primary standards.
The balances are calibrated yearly by the ISO 17025 accredited metrology service, and are verified weekly by an in-house method using standard weights.

Company Confidential & Proprietary

HIGH-PURITY STANDARDS
www.highpuritystandards.com

P.O. Box 41727
Charleston, SC 29423
Phone (843) 767-7900
Fax (843) 767-7906

Certificate of Analysis

Product Description:

Name:	TCLP-Spk
Part Number:	**SM-606-114**
	Solution C
Lot Number:	**1505634**
Matrix:	2% HNO_3 + 0.5% HF
Purity:	99.95% - 99.999%

3548485
ID: ME_TCLP_C_00005
Exp: 03/02/16 Prpd: CDC
TCLP solution C stock

Certified Values:

Element	(µg/mL)	SRM ID	SRM Lot#	Element	(µg/mL)	SRM ID	SRM Lot#
Sb	500 ± 3	*		Sn	500 ± 3	3161a	070330
B	500.0 ± 2.5	3107	070514	Ti	500 ± 3	3162a	130925
Mo	500 ± 3	3134	891307	V	250.0 ± 1.5	3165	992706
Ag	250.0 ± 1.3	3151	992212				

The Certified values are based on gravimetric and volumetric preparation, and verified against SRM 3100 series developed by National Institute of Standards and Technology (NIST) via inductively coupled plasma optical emission spectrometry (ICP-OES) using an internal laboratory developed method. The uncertainty in the certified value is calculated for a 95% confidence interval and coverage factor *k* is about 2
* Refer to Traceability Information, Section d

Preparation Information:

The standard is generally prepared from single element standard solutions that are ISO Guide 34 certified reference materials. Highest purity source materials were purchased from qualified vendors per ISO 9001:2008 guidelines and assayed by ICP-OES for conformity prior to use. Sub-boiling distilled high-purity acid has been used to place the materials in solution and to stabilize the standard. The matrix is as noted above in 18 megaohm deionized water.

Traceability Information:

The traceability of this standard is maintained through an unbroken chain of comparisons to appropriate standards with suitable procedure and measurement uncertainties. The maintenance of the base and derived units of International System of Units (SI) with traceability of measurement results (contemporary metrology) to SI ensures their comparability over time as follows.

a. **Standard Weight and Analytical Balance**
The standard weights (NBS weights Inventory No 20231A) are calibrated every two years by South Carolina Metrology Laboratory that is a participant in "NIST Weights and Measures Measurement Assurance Program" with a certificate of measurement traceability to NIST primary standards.
The balances are calibrated yearly by the ISO 17025 accredited metrology service, and are verified weekly by an in-house method using standard weights.

Company Confidential & Proprietary

HIGH-PURITY STANDARDS
www.highpuritystandards.com

P.O. Box 41727
Charleston, SC 29423
Phone (843) 767-7900
Fax (843) 767-7906

Certificate of Analysis

Product Description:

Name:	TCLP-Spk
Part Number:	**SM-606-114**
	Solution D
Lot Number:	**1505635**
Matrix:	5% HNO_3
Purity:	99.98% - 99.9999%

3548487
ID: ME_TCLP_D_00005
Exp: 03/02/18 Prpd: CDC
TCLP solution D stock

Certified Values:

Element	(µg/mL)	SRM ID	SRM Lot#	Element	(µg/mL)	SRM ID	SRM Lot#
Al	2500.0 ± 12.5	3101a	060502	Cr	2500.0 ± 12.5	3112a	030730
As	2500 ± 25	3103a	100818	Pb	2500 ± 15	3128	101026

The Certified values are based on gravimetric and volumetric preparation, and verified against SRM 3100 series developed by National Institute of Standards and Technology (NIST) via inductively coupled plasma optical emission spectrometry (ICP-OES) using an internal laboratory developed method. The uncertainty in the certified value is calculated for a 95% confidence interval and coverage factor *k* is about 2
* Refer to Traceability Information, Section d

Preparation Information:

The standard is generally prepared from single element standard solutions that are ISO Guide 34 certified reference materials. Highest purity source materials were purchased from qualified vendors per ISO 9001:2008 guidelines and assayed by ICP-OES for conformity prior to use. Sub-boiling distilled high-purity acid has been used to place the materials in solution and to stabilize the standard. The matrix is as noted above in 18 megaohm deionized water.

Traceability Information:

The traceability of this standard is maintained through an unbroken chain of comparisons to appropriate standards with suitable procedure and measurement uncertainties. The maintenance of the base and derived units of International System of Units (SI) with traceability of measurement results (contemporary metrology) to SI ensures their comparability over time as follows.

a. **Standard Weight and Analytical Balance**
The standard weights (NBS weights Inventory No 20231A) are calibrated every two years by South Carolina Metrology Laboratory that is a participant in "NIST Weights and Measures Measurement Assurance Program" with a certificate of measurement traceability to NIST primary standards.
The balances are calibrated yearly by the ISO 17025 accredited metrology service, and are verified weekly by an in-house method using standard weights.

Company Confidential & Proprietary

Rev'd 6/10/14
C.G.

INORGANIC VENTURES™

300 Technology Drive
Christiansburg, VA 24073 - USA
inorganicventures.com

tel 800.669.6799 - 540.585.3030
fax 540.585.3012
info@inorganicventures.com

CERTIFICATE OF ANALYSIS

1.0 ACCREDITATION / REGISTRATION

INORGANIC VENTURES is accredited to ISO Guide 34, "General Requirements for the Competence of Reference Material Producers" and ISO/IEC 17025, "General Requirements for the Competence of Testing and Calibration Laboratories". Inorganic Ventures is also an ISO 9001 registered manufacturer (SAI Global File Number (010105).

ACCREDITED Testing Laboratory Certificate 853.01
ACCREDITED Reference Material Producer Certificate 853.02

2995856
ID: ME_ICSA_00044
Exp: 07/01/15 Prpd: CDC
ICP ICSA standard

2.0 PRODUCT DESCRIPTION

Product Code:	Multi Analyte Custom Grade Solution
Catalog Number:	CLPP-ICS-A
Lot Number:	G2-MEB491156
Matrix:	2% (v/v) HNO3
Value / Analyte(s):	5,000 µg/mL ea: Al, Ca, Mg, 2,000 µg/mL ea: Fe

3.0 CERTIFIED VALUES AND UNCERTAINTIES

ELEMENT	CERTIFIED VALUE	ELEMENT	CERTIFIED VALUE	ELEMENT	CERTIFIED VALUE
Aluminum, Al	4,996 ± 31 µg/mL	Calcium, Ca	4,996 ± 31 µg/mL	Iron, Fe	1,998 ± 10 µg/mL
Magnesium, Mg	4,996 ± 32 µg/mL				

Certified Density: 1.084 g/mL (measured at 20 ± 1 °C)

Assay Information:

ELEMENT	METHOD	NIST SRM#	SRM LOT#
Al	ICP Assay	3101a	060502
Al	EDTA	928	928
Ca	ICP Assay	3109a	050825
Ca	EDTA	928	928
Fe	ICP Assay	3126a	051031
Fe	EDTA	928	928
Mg	ICP Assay	3131a	050302
Mg	EDTA	928	928

The following equations are used in the calculation of the certified value and the uncertainty. Reported uncertainties represent expanded uncertainties expressed at approximately the 95% confidence level using a coverage factor of k = 2.

Certified Value $(\bar{x}) = \frac{\Sigma x_i}{n}$ ($\bar{x}$) = mean
x_i = individual results
n = number of measurements

Uncertainty (±) = $2\,[\,\Sigma(s_i)^2\,]^{1/2}$ 2 = the coverage factor.
$[\,\Sigma(s_i)^2\,]^{1/2}$ = The square root of the sum of the squares of the most common errors (where 's' stands for the standard deviation) from instrumental measurement, density, NIST SRM uncertainty, weighing, dilution to volume, homogeneity, long term stability and short term stability.

4.0 TRACEABILITY TO NIST

- This product is traceable to NIST via an unbroken chain of comparisons. The uncertainties for each certified value are reported, taking into account the SRM/RM uncertainty error and the measurement, weighing and volume dilution errors. In rare cases where no NIST SRM/RM are available, the term 'in-house std.' is specified.

INORGANIC VENTURES™
300 Technology Drive
Christiansburg, VA 24073 · USA
inorganicventures.com

CERTIFICATE OF ANALYSIS

tel: 800.669.6799 · 540.585.3030
fax: 540.585.3012
info@inorganicventures.com

1.0 ACCREDITATION / REGISTRATION

INORGANIC VENTURES is accredited to ISO Guide 34, "General Requirements for the Competence of Reference Material Producers" and ISO/IEC 17025, "General Requirements for the Competence of Testing and Calibration Laboratories". Inorganic Ventures is also an ISO 9001 registered manufacturer (SAI Global File Number 010105).

ACCREDITED Testing Laboratory Certificate 863.01 ACCREDITED Reference Material Producer Certificate 863.02

2.0 PRODUCT DESCRIPTION

Product Code:	Multi Analyte Custom Grade Solution
Catalog Number:	IV-7
Lot Number:	J2-MEB556130
Matrix:	7% (v/v) HNO3 tr. HF
Value / Analyte(s):	1 000 µg/mL ea: K, 100 µg/mL ea: Ag, Al, B, Ba, Na, 50 µg/mL ea: Si

3749922
ID: ME_IV_7_00011
Exp: 01/12/18 Prpd: CDC
IV_7

rec'd 6/23/15

Second Source: Whenever possible, this solution was manufactured from a second set of concentrates in our manufacturing facility.

3.0 CERTIFIED VALUES AND UNCERTAINTIES

ANALYTE	CERTIFIED VALUE	ANALYTE	CERTIFIED VALUE
Aluminum, Al	100.0 ± 0.7 µg/mL	Barium, Ba	100.0 ± 0.7 µg/mL
Boron, B	100.0 ± 0.7 µg/mL	Potassium, K	1 000 ± 4 µg/mL
Silicon, Si	50.01 ± 0.33 µg/mL	Silver, Ag	100.0 ± 0.6 µg/mL
Sodium, Na	100.0 ± 0.4 µg/mL		

Certified Density: 1.038 g/mL (measured at 20 ± 1 °C)

Assay Information:

INORGANIC VENTURES™
300 Technology Drive
Christiansburg, VA 24073 • USA
inorganicventures.com

tel: 800.669.6799 • 540.585.3030
fax: 540.585.3012
info@inorganicventures.com

CERTIFICATE OF ANALYSIS

1.0 ACCREDITATION / REGISTRATION

INORGANIC VENTURES is accredited to ISO Guide 34, "General Requirements for the Competence of Reference Material Producers" and ISO/IEC 17025, "General Requirements for the Competence of Testing and Calibration Laboratories". Inorganic Ventures is also an ISO 9001 registered manufacturer (SAI Global File Number (010105).

ACCREDITED Testing Laboratory Certificate 853.01 ACCREDITED Reference Material Producer Certificate 853.02

2.0 PRODUCT DESCRIPTION

Product Code:	Multi Analyte Custom Grade Solution
Catalog Number:	IV-19
Lot Number:	J2-MEB556129
Matrix:	7.14% (v/v) HNO3 tr. HF
Value / Analyte(s):	100 µg/mL ea:

As,	Be,	Ca,
Cd,	Co,	Cr3,
Cu,	Fe,	Mg,
Mn,	Mo,	Ni,
Pb,	Sb,	Se,
Ti,	Tl,	V,
Zn		

3749896
ID: ME_IV-19_00013
Exp: 01/12/18 Prpd: CDC
IV-19

rec'd 6/23/15

Second Source: Whenever possible, this solution was manufactured from a second set of concentrates in our manufacturing facility.

3.0 CERTIFIED VALUES AND UNCERTAINTIES

ANALYTE	CERTIFIED VALUE	ANALYTE	CERTIFIED VALUE
Antimony	100.0 ± 0.8 µg/mL	Arsenic	100.0 ± 0.8 µg/mL
Beryllium	100.0 ± 0.5 µg/mL	Cadmium	100.0 ± 0.7 µg/mL
Calcium	100.0 ± 0.6 µg/mL	Chromium+3	100.0 ± 0.7 µg/mL
Cobalt	100.0 ± 0.6 µg/mL	Copper	100.0 ± 0.7 µg/mL
Iron	100.0 ± 0.7 µg/mL	Lead	100.0 ± 0.7 µg/mL
Magnesium	100.0 ± 0.6 µg/mL	Manganese	100.0 ± 0.6 µg/mL
Molybdenum	100.0 ± 0.5 µg/mL	Nickel	100.1 ± 0.7 µg/mL
Selenium	100.0 ± 0.6 µg/mL	Thallium	100.0 ± 0.8 µg/mL
Titanium	100.0 ± 0.7 µg/mL	Vanadium	100.0 ± 0.5 µg/mL
Zinc	100.0 ± 0.7 µg/mL		

Certified Density: 1.040 g/mL (measured at 20 ± 1 °C)

Assay Information:

Title: Mercury Analysis for Water and Wastewater using EPA 245.1 and SW846 7470A; Mercury in Drinking Water using EPA 245.1; Leeman Mercury Analyzer (Cold Vapor Technique)

Once printed, this is considered an uncontrolled document

Laura Demone	04/25/18	Dan Helfrich	04/23/18
Laura Demone	Date	Dan Helfrich	Date
Department Manager		Health & Safety Manager Coordinator	
Carl Armbruster	04/23/18	Mark Acierno	04/23/18
Carl Armbruster	Date	Mark Acierno	Date
Quality Assurance Manager		Laboratory Director	

Copyright Information:
This documentation has been prepared by TestAmerica Laboratories, Inc. and its affiliates ("TestAmerica"), solely for their own use and the use of their customers in evaluating their qualifications and capabilities in connection with a particular project. The user of this document agrees by its acceptance to return it to TestAmerica upon request and not to reproduce, copy, lend, or otherwise disclose its contents, directly or indirectly, and not to use it for any other purpose other than that for which it was specifically provided. The user also agrees that where consultants or other outside parties are involved in the evaluation process, access to these documents shall not be given to said parties unless those parties also specifically agree to these conditions.

THIS DOCUMENT CONTAINS VALUABLE CONFIDENTIAL AND PROPRIETARY INFORMATION. DISCLOSURE, USE OR REPRODUCTION OF THESE MATERIALS WITHOUT THE WRITTEN AUTHORIZATION OF TESTAMERICA IS STRICTLY PROHIBITED. THIS UNPUBLISHED WORK BY TESTAMERICA IS PROTECTED BY STATE AND FEDERAL LAW OF THE UNITED STATES. IF PUBLICATION OF THIS WORK SHOULD OCCUR THE FOLLOWING NOTICE SHALL APPLY:

©COPYRIGHT 2018 TESTAMERICA LABORATORIES, INC. ALL RIGHTS RESERVED.

1.0 Scope and Application

1.1. Analytes, Matrix(s), and Reporting Limits

EPA Method 245.1 and SW846 Method 7470A are applicable to the determination of mercury in water matrices. Mercury may be found in water in both inorganic and organic forms. Organomercury compounds must first be broken down to respond to the cold vapor atomic absorption technique.

The typical detection limit using a 30 ml sample size is 0.2ug/L Hg.

On occasion clients may request modifications to this SOP. These modifications are handled following the procedures outlined in Sections 7 (Review of Work Request) and 19 (Test Methods and Method Validation) of TestAmerica Edison's Quality Assurance Manual (ED-QA-LQM).

2.0 Summary of Method

A digested sample is analyzed using cold vapor atomic absorption. The mercury is reduced to the elemental state and aerated from solution in a closed system. The mercury vapor passes through a cell positioned in the light path of an atomic absorption spectrophotometer. Absorbance (peak height) is measured as a function of mercury concentration.

3.0 Definitions

For a complete list of definitions refer to Appendix 2 in the most current revision of TestAmerica Edison's Quality Assurance Manual (ED-QA-LQM).

4.0 Interferences

4.1 The addition of potassium persulfate during the digestion step can eliminate the possible interference from sulfide in the sample without affecting the recovery of inorganic mercury.

4.2 Copper may also be a potential interference although no effect has been observed for samples containing up to 10 mg/l total copper.

4.3 Samples that contain high levels of chloride have a potential to interfere due to a reaction that takes place during the oxidation step. During this step chloride is converted to free chlorine which absorbs light at 253.7 nm. The analyst must not allow the chlorine to be swept into the optical cell. The possibility of the chlorine interfering with the analysis can be minimized by using an excess of up to 7.5 ml hydroxylamine sulfate.

5.0 Safety

Employees must abide by the policies and procedures in the Corporate Environmental Health and Safety Manual (CW-E-M-001) and this document. This procedure may

involve hazardous material, operations and equipment. This SOP does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of the method to follow appropriate safety, waste disposal and health practices under the assumption that all samples and reagents are potentially hazardous. Safety glasses, gloves, lab coats and closed-toe, nonabsorbent shoes are a minimum.

5.1 Specific Safety Concerns or Requirements

Samples that contain high concentrations of carbonates or organic material or samples that are at elevated pH can react violently when acids are added.

5.2 Primary Materials Used

The following is a list of the materials used in this method, which have a serious or significant hazard rating. **Note: This list does not include all materials used in the method. The table contains a summary of the primary hazards listed in the MSDS for each of the materials listed in the table.** A complete list of materials used in the method can be found in the reagents and materials section. Employees must review the information in the MSDS for each material before using it for the first time or when there are major changes to the MSDS.

Material (1)	Hazards	Exposure Limit (2)	Signs and symptoms of exposure
a) Mercury b) (1,000 PPM in Reagent)	Oxidizer Corrosive Poison	0.1 mg/M^3 Ceiling (Mercury Compounds)	Extremely toxic. Causes irritation to the respiratory tract. Causes irritation. Symptoms include redness and pain. May cause burns. May cause sensitization. Can be absorbed through the skin with symptoms to parallel ingestion. May affect the central nervous system. Causes irritation and burns to eyes. Symptoms include redness, pain, and blurred vision; may cause serious and permanent eye damage.
Sulfuric Acid	Corrosive Oxidizer Dehydrator Poison	1 mg/M^3-TWA	Inhalation produces damaging effects on the mucous membranes and upper respiratory tract. Symptoms may include irritation of the nose and throat, and labored breathing. Symptoms of redness, pain, and severe burn can occur. Contact can cause blurred vision, redness, pain and severe tissue burns. Can cause blindness.

Material (1)	Hazards	Exposure Limit (2)	Signs and symptoms of exposure
Nitric Acid	Corrosive Oxidizer Poison	2 ppm-TWA 4 ppm-STEL	Nitric acid is extremely hazardous; it is corrosive, reactive, an oxidizer, and a poison. Inhalation of vapors can cause breathing difficulties and lead to pneumonia and pulmonary edema, which may be fatal. Other symptoms may include coughing, choking, and irritation of the nose, throat, and respiratory tract. Can cause redness, pain, and severe skin burns. Concentrated solutions cause deep ulcers and stain skin a yellow or yellow-brown color. Vapors are irritating and may cause damage to the eyes. Contact may cause severe burns and permanent eye damage.
Hydrochloric Acid	Corrosive Poison	5 PPM-Ceiling	Inhalation of vapors can cause coughing, choking, inflammation of the nose, throat, and upper respiratory tract, and in severe cases, pulmonary edema, circulatory failure, and death. Can cause redness, pain, and severe skin burns. Vapors are irritating and may cause damage to the eyes. Contact may cause severe burns and permanent eye damage.
Potassium Permanganate	Oxidizer	5 Mg/M^3 for Mn Compounds	Causes irritation to the respiratory tract. Symptoms may include coughing, shortness of breath. Dry crystals and concentrated solutions are caustic causing redness, pain, severe burns, brown stains in the contact area and possible hardening of outer skin layer. Diluted solutions are only mildly irritating to the skin. Eye contact with crystals (dusts) and concentrated solutions causes severe irritation, redness, and blurred vision and can cause severe damage, possibly permanent.
1 – Always add acid to water to prevent violent reactions.			
2 – Exposure limit refers to the OSHA regulatory exposure limit.			

6.0 Equipment and Supplies

6.1 Instrumentation

6.1.1. Leeman Laboratories Inc. Hydra AA Automated Hg Analyzer

6.1.2. Computer and Printer with Leeman WinHg software

6.1.3. Block digestor (Environmental Express or SCP Science): Adjustable and capable of maintaining a temperature of 90 -95°C.

6.2 Supplies

6.2.1. 50 ml Hot Block Digestion Cups

6.2.2. 100 ml graduated cylinder

6.2.3. Eppendorf Pipettes and tips in various sizes

6.2.4. 100 ml volumetric flasks

6.2.5. 15 ml sample cups

6.2.6. 8 liter carboy container

6.2.7. Pump tubing:

- Sample, viton, blue tab
- Reductant, red tab
- Drain, blue tab
- Rinse, black tab

6.2.8. Drying Tube – Purchased pre-packed with Magnesium Perchlorate from Leeman Labs. Located prior to the optical cell.

6.2.9. Nitrogen or Argon supply - capable of producing 80 PSI.

7. Reagents and Standards

7.1. Reagents

Storage requirements: store at room temperature

Life of Reagent: - Concentrated acids: refer to manufacturer's instructions
- Laboratory prepared reagents and diluted acids: one year

Document prepared reagents in the Reagent module located in the TestAmerica Laboratory System (TALS).

7.1.1 Sulfuric acid - Concentrated (Trace Grade or Equivalent); store at room temperature; for stability information, refer to manufacturer's instructions.

7.1.2 Nitric acid - Concentrated (Trace Grade or Equivalent)

7.1.3 Hydrochloric acid-Concentrated (Trace Grade or Equivalent)

7.1.4 Potassium Permanganate (ACS Grade); for stability information, refer to manufacturer's instructions.

7.1.5 Sodium Chloride (analytical reagent grade); for stability information, refer to manufacturer's instructions.

7.1.6 Hydroxylamine Hydrochloride (ACS Grade); for stability information, refer to manufacturer's instructions.

7.1.7 Stannous Chloride (ACS Grade); for stability information, refer to manufacturer's instructions.

7.1.8 Potassium Persulfate (ACS Grade); for stability information, refer to manufacturer's instructions.

7.1.9 Deionized water - 18 megohm minimum

7.1.10 10% Hydrochloric Acid- Add approximately 5 liters of deionized water into an 8 liter carboy container. Cautiously add 800 ml of concentrated HCl and bring the final volume up to 8 liters with deionized water.

7.1.11 10% Stannous chloride solution - Add 50 g of $SnCl_2$ to 500 ml 10% HCl solution.

7.1.12 Sodium chloride/Hydroxylamine Hydrochloride solution - Dissolve 120 g of NaCl and 120 g of hydroxylamine hydrochloride in deionized water and dilute to 1 liter using deionized water.

7.1.13 Potassium Permanganate ($KMnO_4$) 5% solution w/v - Dissolve 100 g of $KMnO_4$ in deionized water and dilute to 2 liters using deionized water.

7.1.14 0.15% Nitric Acid- Add approximately 5 liters of deionized water into an 8 liter carboy container. Cautiously add 12mL of concentrated HNO_3 and bring the final volume up to 8 liters with deionized water.

7.1.15 Potassium Persulfate ($K_2O_8S_2$) 5% solution w/v: Dissolve 50 g of potassium persulfate in 1 liter of deionized water.

7.2 Standards

Storage requirements: all standards are stored at room temperature

Shelf-life: Stock standards – refer to manufacturer's instructions
Intermediate standards – made fresh daily
Working standards – made fresh daily
(Note: expiration date must not go beyond the expiration date of the source stock).

Concentration: see Attachment 1 for example certificates of analysis (COA) for all of the standards mixes listed below. The COA lists the manufacturer's part number, certified concentration and shelf life.

Document standard preparation in TALS (see Section 11.4.2).

7.2.1 Stock Mercury Calibration (10 ppm Hg) - Purchase from SCP Science; store at room temperature; for stability information, refer to manufacturer's instructions.

7.2.2. Stock Mercury Quality Control Standard (10 ppm Hg) - Purchase from Inorganic Ventures; store at room temperature; for stability information, refer to manufacturer's instructions. This stock standard must be purchased from a second source vendor.

7.2.3. Intermediate Calibration Standard (DCAL-Int): Dilute 1 ml of Stock Mercury Calibration (Sec 7.2.1) to 100 ml with 0.15% HNO_3. The resulting solution will contain 100ppb Hg.

7.2.4. Intermediate Quality Control Standard (DQCS-Int): Dilute 1 ml of Stock Mercury Calibration Verification Standard (Sec 7.2.2) solution to 100 ml with 0.15% HNO_3. The resulting solution will contain 100ppb Hg.

7.2.5. Calibration Standard Preparation: Use six 50 ml hot block digestion cups to prepare the standards. Add small portion of 0.15% HNO_3 to each cup. Working in increasing order, spike the appropriate cup with 0.0, 0.06, 0.3, 0.6, 1.5, and 3.0 ml of working solution DCAL-Int (Sec 7.2.3). Bring to final volume of 30 ml and mix thoroughly. The corresponding concentrations are 0.0ppb, 0.2ppb, 1.0ppb, 2.0ppb, 5.0ppb, and 10.0ppb mercury respectively. For drinking water analysis, the 2.0ppb standard is also analyzed as the Maximum Contaminant Level (MCL) standard

7.2.6. Quality Control Standard (QCS) Preparation: Add a small portion of 0.15% HNO_3 to a 30 ml hot block digestion cup and spike 1.5 ml of DQCS-Int (Sec 7.2.4). Bring up to final volume and mix thoroughly. The resulting solution will contain 5.0 ppb of Hg.

7.2.7. Initial Calibration Verification (ICV) standard: Add a small portion of 0.15% HNO3 to a 30 ml hot block digestion cup and spike 1.5 ml of DQCS-Int (Sec 7.2.4). Bring up to final volume and mix thoroughly. The resulting solution will contain 5.0 ppb of Hg.

7.2.8. Continuing Calibration Verification (CCV) standard 5.0ppb: The 5.0 ppb prepared from the calibration standards (Sec 7.2.5) is used as the Continuing Calibration Verification standard (CCV).

7.2.9. Maximum Contaminant Level (MCL) standard 2.0 ppb *for drinking water analysis:* The 2.0ppb standard prepared from the calibration standards (Sec 7.2.5) is analyzed as the Maximum Contaminant Level (MCL) standard.

7.2.10. Reporting limit check standard (CRI) 0.2 ppb: Use the 0.2 ppb prepared

from the calibration standards (Sec 7.2.5) as the reporting limit check standard (CRI).

8.0 Sample Collection, Preservation, Shipment and Storage

Sample container, preservation techniques and holding times may vary and are dependent on sample matrix, method of choice, regulatory compliance, and/or specific contract or client requests. Listed below are the holding times and the references that include preservation requirements.

Matrix	Sample Container	Min. Sample Size	Preservation	Holding Time [2]	Reference
Waters	P. FP, G[1]	500 ml	HNO_3, pH < 2 prior to shipment; if not, acidify upon receipt in lab; Cool 4±2°C	28 Days	40 CFR Part 136.3

[1] Polyethylene, fluoropolymer, glass
[2] Inclusive of digestion and analysis
[3] Acid preservation may be omitted for shipping; however, acid must be added upon receipt in the lab. Following acidification, mix the sample and hold for at least 16 hours for method 245.1 drinking water and 24 hours for non-potable water. Just prior to digestion or direct analysis, verify pH<2. If pH≥2, repeat steps (i.e., add acid, hold for 24hrs, verify ph<2).

9.0 Quality Control

9.1. Sample QC - The following quality control samples are prepared with each batch of samples.

Quality Controls	Frequency	Control Limit
Method Blank (MB)	1 in 20 or fewer samples	**245.1**: < 2.2 times MDL or; <10%sample concentration (whichever is greater) **7470A**: < MDL; **or;** < 5% of the reg limit; **or,** < 5% of the measured sample concentration (whichever is greater)
Laboratory Control Sample (LCS)	1 in 20 or fewer samples	**245.1**: 85-115% **7470A**: 80-120%
Matrix Duplicate (DUP)[1]	1 in 20 or fewer samples	20% RPD
Matrix Spike (MS)[1]	**245.1**: 1, if 10 samples or less; 2 if 11-20 samples **7470A**: 1 in 20 or fewer samples	**245.1**: 70-130% **7470A**: 75-125%

Quality Controls	Frequency	Control Limit
Serial Dilution (SD) for 7470A	1 in 20 or fewer samples	±10%

[1] The sample for DUP and MS are randomly selected, unless specifically requested by a client. Use the same environmental sample for the matrix spike and matrix duplicate sample whenever possible. If insufficient sample amount is available, another environmental sample may be used for the duplicate sample.

9.1.1 **Method Blank:** One laboratory method/preparation blank will be analyzed with each batch of samples prepared together (not to exceed 20 samples). Preparation blank is used to identify possible contamination during acid digestion. If the Mercury concentration in the MB is above the specified control limit, the batch must be prepared again and the samples reanalyzed.

- For 7470A, results must be less than: MDL, 5% of the regulatory limit for that analyte, or 5% of the measured concentration in the sample, whichever is greater.
- For 245.1, results must be less than 2.2 times the method detection limit (MDL) or less than 10% of the determined Mercury concentration in the sample, whichever is greater.

9.1.2 **Laboratory Control Sample (LCS):** A laboratory control sample must be analyzed with each batch of samples digested. A blank is spiked with 0.1 ug of mercury (0.30 ml of standard DCAL-Int). This is equivalent to 1.0 ppb Hg if a 30 ml portion of sample is digested. Results of the aqueous LCS must fall within ±15% of the true value for Method 245.1 and ±20% for Method 7470A.

9.1.3 **Matrix Duplicate (DUP):** A duplicate is analyzed for each batch of samples digested. The relative percent difference between the results of the sample and the duplicate must fall within 20 % Relative Percent Difference (RPD) for samples greater than five times the reporting limit.

9.1.4 **Matrix Spike (MS):** A matrix spike is prepared and analyzed for each batch of samples. A portion of sample is spiked with 0.1 ug of mercury (0.30 ml of standard DCAL-Int). This is equivalent to 1.0 ppb Hg if a 30 ml portion of sample is digested. A recovery of 70-130% for Method 245.1 and 75-125% for Method 7470A is required.

9.1.5 **Serial Dilution (SD):** For method 7470A, a five fold serial dilution must be performed on one sample per batch. The sample should contain a sufficiently high concentration; minimally a factor of 25 times above the estimated detection limit. Dilute the sample by a minimum of five fold (1+4) and reanalyze. Results must agree within 10% of the original determination. If not, a chemical or physical effect should be suspected.

9.2 Instrument QC

9.2.1. **Initial Calibration Verification (ICV):** Initial calibration is verified after calibration. The ICV solution should be prepared using a second source

vendor; see section 7.2.7 for preparation instructions. Use a concentration of mercury at the midpoint of the calibration range (5.0ppb). The Cal standard containing 5ppb is analyzed as the ICV; see Sec 7.2.7 for preparation instructions. For 245.1, the results must not differ from the true value by more than 5%. For 7470A, the results must be within 10% of the true value. If it's outside of the acceptable limits, terminate the analysis, correct the problem and recalibrate the instrument.

9.2.2. **Continuing Calibration Verification (CCV):** Calibration verification is performed after the calibration, after every 10 samples, and at the end of the run. The CCV solution should be prepared from the same CAL standard as used to prepare the calibration solutions. Use a concentration of mercury at the midpoint of the calibration range (5.0ppb). The Cal standard containing 5ppb is analyzed as the CCV.

For 245.1, CCV must not differ from the true value by more than 10%. For 7470A, CCV values must be within 20%. If not, stop the analysis and recalibrate. Re-analyze the previous ten samples following the last good calibration verification.

9.2.3. **Initial and Continuing Calibration Blank (ICB/CCB):** ICB and CCB must be analyzed after the calibration curve, every 10 samples and at the end of the analytical run. For methods 245.1 and 7470A, the absolute value of the calibration verification blank must not exceed the reporting limit. If it does, terminate the analysis, correct the problem, recalibrate and reanalyze the samples following the last good CCB. The calibration verification blank is the same blank solution as used for the calibration blank.

9.2.4. **Maximum Contaminant Level (MCL):** For drinking water analysis, one MCL standard shall be analyzed per calibration. The 2.0ppb standard prepared from the calibration standards (Sec 7.2.5) is analyzed as the Maximum Contaminant Level (MCL) standard. The result must be within 50% of the true value. If it's outside of the acceptable limit, terminate the analysis, correct the problem, and recalibrate the instrument.

9.2.5. **Quality Control Standard (QCS):** The calibration is verified after calibration using a second source vendor; see Sec 7.2.6 for preparation instructions. For 245.1 and 7470A, the results must not differ from the true value by more than 10%. If it's outside of the acceptable limits, terminate the analysis, correct the problem and recalibrate the instrument.

9.2.6. **Reporting limit check standard (CRI):** At the beginning of the analysis, verify the accuracy at the reporting limit (RL) by analyzing a solution at the RL level. RL check solution is analyzed to demonstrate that the mercury analyzer is capable of detecting the target analyte at the reporting limit (RL). Laboratory limits are 50-150% of the true value.

10.0 Procedure

10.1 Sample Preparation

10.1.1 Filtration Procedure for Dissolved Mercury not filtered in the Field

10.1.1.1 The unpreserved sample must be filtered through a 0.45um filter unit as soon as practical after collection.

10.1.1.2 Collect the required volume of filtrate by using a 0.45um filter unit and a vacuum pump.

10.1.1.3 Acidify the filtrate with 1:1 HNO_3 to a pH of <2.

10.1.1.4 The method blank (MB) must be filtered and digested under the same conditions as the lab filtered samples.

10.1.2 If sample is preserved in laboratory, hold sample for 16 hours for method 245.1 drinking water and 24 hours for wastewater following acidification. Verify and record if sample pH is < 2.0 after 16 or 24 hours (depending on the analysis- drinking water or wastewater) and prior to digestion/analysis. If pH is > 2.0, add HNO3, hold for 24 hours and verify pH.

10.1.3 Transfer 30 ml sample (DI water for MB and LCS) or standard, or an aliquot diluted to 30 ml, to an appropriately identified 50 ml hot block digestion cup. For QA samples, transfer 3 aliquots of 30 ml sample to three digestion cups labeled as SAMPLE, DUP and MS. Spike LCSW and MS with 0.1 µg of mercury (0.3 ml of DCAL-INT standard).

10.1.3.1. Due to insufficient sample volume or an unusual matrix (e.g., samples with potentially high interferences), it may be necessary to perform a pre-digestion dilution. For example, a 10X pre-digestion dilution can be performed by transferring 3 mL of sample to 50 mL hotblock cup. Add 27 ml of deionized water. Record 3 mL initial volume and 30 mL final volume in TALS (see Sec 11.4.2). Continue to section 10.1.3.

10.1.4 Add 1.5 ml concentrated H_2SO_4 and 0.75 ml concentrated HNO_3 mixing well after each addition.

10.1.5 Add 4.5 ml of potassium permanganate solution to each bottle. Mix well and let stand for 15 minutes (minimum); if the color has disappeared, add additional $KMnO_4$ until the purple color persists for at least 15 minutes (document in the Sample preparation log any additional amount of $KMnO_4$ added). The same amount of $KMnO_4$ must be added to the standards and samples.

10.1.6 Add 2.4 ml potassium persulfate solution to each bottle.

10.1.7 Heat for 2 hours in a 95° C hot block digestor. Remove from block digestor and cool.

10.1.8 Add 1.8 ml Sodium chloride - Hydroxylamine hydrochloride solution to reduce the excess permanganate. Mix well; solution should become

colorless. If necessary additional Sodium chloride - Hydroxylamine HCl solution may be added.

10.1.9 Due to differing rates of evaporation, the final volume of the standards/samples maybe different. The standards and samples, including all QC, must be at the same final volume before analysis; if not, find the sample or standard with the highest final volume (e.g., 41 mL) and add deionized water to any sample or standard to bring them to the same final volume . Mix well. Wait at least 30 seconds after decolorization before analyzing.

10.2 Calibration

10.2.1. The instrument must be calibrated daily or once every 24 hours and each time the instrument is set up. The instrument is calibrated according to the manufacturer's specifications and must contain at least four standards and a blank. The laboratory currently uses five standards and a blank. The correlation coefficient of the calibration curve must be ≥0.995. If it does not, the problem must be corrected, and the instrument must be recalibrated. Standard preparations must be documented in TALS (see Sec 11.4.2).

10.2.2. Prepare five calibration standards, blank and calibration verification standard as detailed in sections 7.2.5 and 7.2.6.

10.3 Sample Analysis

10.3.1 Following a sample digestion procedure, the samples are ready for instrumental analysis. It is advisable to investigate each matrix for any complexities, which might adversely affect the acquisition of valid data.

10.3.2 The following analytical run sequence is currently used:

Instrument Calibration (Blank and five standards)

ICV
ICB
CRI
QCS
MCL (for drinking water analysis)
9 Samples
CCV
CCB
10 Samples
CCV
CCB
Repeat until run is completed
CCV
CCB

10.3.3 Instrument Operation:

10.3.3.1. Turn on Computer, Printer and Monitor.

10.3.3.2. Plumbing the Reagent Lines:

10.3.3.2.1. One at a time, feed each of the pump tubes into a pump cassette, sliding the tube through the plastic clips at the bottom until the plastic tab is secure. Then, holding the tube taut, slide the loaded cassette onto the pump head and click the clamp, lever up. The tab end of the tube should be located at the front of the pump head.

10.3.3.2.2. Reductant (Red): Connect tab end of tube to the reductant tubing that is connected to the reductant bottle and the other end to the mixing tee.

10.3.3.2.3. Sample (Blue): Connect tab end of tube to the autosampler probe and the other end to the mixing tee.

10.3.3.2.4. Drain (Blue): Connect the tab end of tube to the sample discharge tube connected on the Liquid/Gas separator and the other end to the waste line.

10.3.3.2.5. Rinse (Black): Connect tab end of tube to rinse tubing that is connected to the rinse bottle. Connect the other end to the rinse tubing leading to the rinse cup.

10.3.3.3. Preparation of Reagents:

10.3.3.3.1. Pour the 10% $SnCl_2$ solution into the reductant bottle.

10.3.3.3.2. Pour the 10% HCl solution into the rinse reservoir bottle.

10.3.3.4. Start the Program:

10.3.3.4.1. Double click WinHg icon on the desktop. This will open the WinHg Runner 1.5 window.

10.3.3.5. Select the Protocol/Method:

10.3.3.5.1. Go to the Protocol box in the upper left hand corner and click on the "▼" button.

10.3.3.5.2. Scroll down to the desired protocol and click on it, (i.e. 245_7470).

10.3.3.6. Create a New Data Set/File Name:

10.3.3.6.1. From the drop down file menu, click on 'New Data

Set'. Type in name of Data Set (i.e. 15990HG1), and click Ok, this opens new batch name box. Type in Batch name (i.e. 15990HG1), and click OK. The new Data Set and batch name should now appear in the respective locations.

10.3.3.7. Open the Data Base Window:

10.3.3.7.1. From the tool bar click on the "DB↓" button. Check to ensure that the correct protocol is open in the Protocol Box (in upper left corner) of the Data Base window. The Dataset/Protocol in the upper right hand corner, should match that in the Runner Window, (i.e. 5990HG1/DW200). Report specs should also have the same protocol open.

10.3.3.7.2. Left click the Apply button. Left click the Cal Curve tab to view calibration values.

10.3.3.8. Turn on Instrument (Lamp/Gas/Pump):

10.3.3.8.1. Return to the Win Hg Runner window by clicking anywhere inside of runner window.

10.3.3.8.2. In main 'WinHg Runner' window, click on Control tab. Under Lamp click "ON" to turn lamp on. Lamp should warm up for approximately 5 minutes before analyzing any samples or standards.

10.3.3.8.3. Under Pump click "ON" to turn pump on. Click on "Standby" to keep lamp on while instrument is not running, otherwise lamp will automatically shut off after 15-20 minutes. Before analysis, click the "On" button for the pump and gas.

10.3.3.9. Setting up rack:

10.3.3.9.1. In 'WinHg Runner' window, click on icon that looks like rack. This will open up the rack editor.

10.3.3.9.2. Type in the Sample IDs under the "Extended ID" column, starting with sequence 1 and follow down until sequence 44. The dilution factor for the sample is typed in under the "Extended ID" column. "Weight" and "Volume" column should always have a value of 1.00 for all samples; the actual weight and volume will be included later in the calculation and data processing. The last column is the macro column. This is where the analyst tells the instrument to automatically go to the check standard cups (i.e. C3 C1 C2 CP). Click on "File", "Save As;" use the prep

batch number as the save file name.

Cup	SampleID	ExtendedID	Weight	Volume	Macros
1	MBW011505		1.00	1.00	C3 C1 C4 CP
2	LCSW		1.00	1.00	
3	610544		1.00	1.00	
4	610544D		1.00	1.00	
5	610544MS		1.00	1.00	
	……..				
10	610545@2			1.00	1.00 C2 C1

- The Macro command C3 C1 C4 CP tells the instrument to run the check standards before running the sample cup.
- The Macro command C2 C1 tells the instrument to run the sample cup first then the check standards. Make sure the CCV/CCB is run so no more than 10 samples run between check standards.

10.3.3.10. Activating and Running Standards:

10.3.3.10.1. Click on the Standards tab in the Runner Window.

10.3.3.10.2. Click on button to activate standard cup S1(0.0ppb), S2(0.2 ppb), S3(1.0ppb), S4(2.0ppb), S5(5.0ppb), S6(10.0ppb) and Rep1. Fill cups appropriately.

10.3.3.10.3. Click on C1<CCB>, C2<AICV>, C3<ACCV>, C4<ACCV>.

10.3.3.10.4. Click on New Cal Reset.

10.3.3.10.5. Click on Standard Auto to run standards.

10.3.3.10.6. Check the calibration curve in the 'Database' window. If the correlation coefficient (Rho) of the calibration is ≥0.995, it is acceptable; Click Accept. Recalibrate if Rho is <0.995.

10.3.3.10.7. Print the calibration curve by hitting the printer icon.

10.3.3.10.8. In the 'WinHg Runner 1.5' window, open File from the drop down menu, choose Page eject, to print the standards on their own separate page.

10.3.3.11. Analyzing Samples:

10.3.3.11.1. Click on the Sample tab in the 'WinHg Runner 1.5' window.

10.3.3.11.2. Select the rack you want to run by clicking the "▼" button for Rack Name and highlighting the desired

rack, for station 1 and station 2. If you're only running one rack, make sure you highlight the blank space at the top of the rack list for the unused station.

10.3.3.11.3. Enter the start cup and the end cup values. If you're only running one rack, make sure the start and end cup are blank for the empty station.

10.3.3.11.4. Real time print should be checked.

10.3.3.11.5. Pour samples into polypropylene test tubes and place them in the appropriate sequence noted on rack. Rack sequence is from right to left i.e. (…. 3, 2, 1).

10.3.3.11.6. Click Run Auto to begin analyzing samples.

10.3.3.11.7. If the sample concentration is greater than or equal to the linear range (i.e., ≥10 ug/L), the sample digestate must be diluted and reanalyzed. For example, if the analytically determined result is 12 ug/L, a 2X post-digestion dilution should be performed. Using a sample cup, add 5 mL of the digested calibration blank and 5 mL of the sample. Mix well. Sample is ready for re-analysis. In the sequence, enter "@2" to indicate a 2X post-digestion dilution (see Sec 10.3.3.9.2, cup 10).

10.3.3.12. Printing Report and Post-Run Report:

10.3.3.12.1. In the 'WinHg Runner 1.5' window, click File, Page eject to print out last page of data.

10.3.3.12.2. In the 'WinHg Data Base' window, click on the "Report tab."

10.3.3.12.3. Mark the box next to the Batch ID with a check (✓) by clicking on the box. The sample IDs should now show up under the Record ID column.

10.3.3.12.4. Select the IDs you wish to print by checking on the box in the far left column of the desired sample, a check mark (✓) on the box will include data in report, a box without a checkmark will exclude the sample data from the report. You can select all of them by checking the ALL button, or only a few by checking the NONE button and then clicking the box next to the desired sample.

10.3.3.12.5. Under the Report Specs, click the "▼" button to the right of the box and select the report format you wish

to use.

e.g. 245_7470 = results in concentration, and 245_INT = results in intensities

10.3.3.12.6. Click the include button under the Select Records heading.

10.3.3.12.7. Click the Generate Report button at the lower right hand corner. This will activate the 'Generate Report' window.

10.3.3.12.8. To send report to the printer, choose the 'report option', under the format heading along with the printer option under 'destination.' Click Generate Tab.

10.3.3.13. Creating a PRN File:

10.3.3.13.1. Choose the PRN option under format.

10.3.3.13.2. Choose the Disk File option under destination (should default to this option).

10.3.3.13.3. The output file path and name is: F:\INORG\LEEMAN#\DATASET NAME. You must delete C:\ before typing in path. Click Generate Tab.

10.3.3.14. Shutting Down the Instrument:

10.3.3.14.1. Close the 'WinHg Database' window.

10.3.3.14.2. Open the control tab in the 'WinHg Runner 1.5' window.

10.3.3.14.3. Click the "Off" button for the lamp, pump, and gas.

10.3.3.14.4. Close the WinHg Runner 1.5 window.

11.0 Calculations / Data Reduction

11.1 Accuracy:

$$\text{ICV / CCV, LCS \% Recovery} = \frac{\text{observed concentration}}{\text{known concentration}} \times 100$$

$$\text{MS \% Recovery} = \frac{\text{(spiked sample) - (unspiked sample)}}{\text{spiked concentration}} \times 100$$

11.2 Precision (RPD):

$$\text{Matrix Duplicate (MD)} = \frac{|\text{orig. sample value - dup. sample value}|}{[(\text{orig. sample value + dup. sample value})/2]} \times 100$$

11.3. Final results calculation in aqueous samples :

$$\text{Concentration} = \text{mg/ L} = \frac{C \times V1 \times D}{V2}$$

Where:
C= Element concentration from instrument (ppb)
V1= Final volume of sample digested (in liters)
D= Dilution performed on sample

V2= Initial volume of sample digested (in liters).

11.4. Data Processing:

11.4.1. All data is recorded directly in TALS' Analyst Desktop II program.

11.4.2. Record standard/sample preparations in the Analyst Desktop II program located in the TestAmerica Laboratory System (TALS) Reagent module.

11.4.3. Import Data from WinHG Leeman to TALS.

11.4.3.1. Start WinHg and Select 'DataBase'
11.4.3.2. Select Report Tab and Batch ID
11.4.3.3. Select 'Generate Report,' and PRN File format
11.4.3.4. Select Output File Path under c:\Hg\RawData\TodaysDate.PRN
11.4.3.5. Select 'Generate'

11.4.4. Metals Data Review checklist (EDS-WI-007) and the TestAmerica Edison Metals Initial Calibration Data Review Mercury checklist (EDS-WI-125) must be filled out prior to data submission.

12. Method Performance

12.1. Method Detection Limit Study (MDL)

The method detection limit (MDL) is the lowest concentration that can be detected for a given analytical method and sample matrix with 99% confidence that the analyte is present. The MDL is determined according to the laboratory's MDL procedure in Section 19 (Test Methods and Method Validation) of TestAmerica Edison's Quality Assurance Manual (ED-QA-LQM). MDLs reflect a calculated (statistical) value determined under ideal laboratory conditions in a clean matrix, and may not be achievable in all environmental matrices. The laboratory maintains MDL studies for

analyses performed; these are verified at least annually unless method requirements require a greater frequency.

12.2 Instrument Detection Limit:

The IDL for each analyte must be determined for each wavelength used on each instrument. The IDL must be determined annually or if the instrument is adjusted in any way that may affect the IDL. For 245.1, the IDL is determined by multiplying the average of the standard deviations obtained from the analysis of 10 reagent blanks by 3. For 7470A, the IDL is determined by multiplying the average of the standard deviations obtained from the analysis of 7 reagent blanks by 3.14

12.3 Linear Dynamic Range (LDR)

The upper limit of the LDR must be established. It must be determined from a linear calibration prepared from a minimum of three different concentration standards, one of which is close to the upper limit of the linear range. The LDR should be determined by analyzing succeedingly higher standard concentrations of mercury until the observed analyte concentration is no more than 10% below the stated concentration of the standard. The determined LDR must be documented and kept on file. The LDR which may be used for the analysis of samples should be judged by the analyst from the resulting data. Determined sample analyte concentrations that are greater than 90% of the determined upper LDR limit must be diluted and reanalyzed. The LDR should be verified annually or whenever, in the judgement of the analyst, a change in analytical performance caused by either a change in instrument hardware or operating conditions would dictate they be redetermined.

12.4 Demonstration of Capabilities

For DOC procedure refer to Section 19 in the most current revision of TestAmerica Edison's Quality Assurance Manual (ED-QA-LQM).

12.5 Training Requirements

Refer to TestAmerica SOP No. ED-GEN-022, Training, for the laboratory's training program.

13. Pollution Control

It is TestAmerica's policy to evaluate each method and look for opportunities to minimize waste generated (i.e., examine recycling options, ordering chemicals based on quantity needed, preparation of reagents based on anticipated usage and reagent stability). Employees must abide by the policies in Section 13 of the Corporate Environmental Health and Safety Manual (CW-E-M-001) for "Waste Management and Pollution Prevention."

14. Waste Management

14.1. Waste management practices are conducted consistent with all applicable rules and regulations. Excess reagents, samples and method process wastes are disposed of in an accepted manner. Waste description rules and land disposal restrictions are followed. Waste disposal procedures are incorporated by reference to TestAmerica SOP No. ED-SPM-008 (Laboratory Waste Disposal Procedures).

14.2. The following waste streams are produced when this method is carried out.

- Digested Samples: Corrosive Acid- Materials that are not above regulatory limits will be submitted for elementary neutralization with 50% sodium hydroxide solution (Siedler Chemical SC-1824-03). Major concern is heat generated from the neutralization process. Initial volume of acid waste to be neutralized should be no more than 15 gallons. Finished neutralization with sodium bicarbonate (Siedler Chemical SC-0219-25) to a pH of 6 – 9 in the primary tank. Once pH has been established the primary tank is transferred through filter housing to a secondary tank. The pH is rechecked. If the pH is within specifications, the secondary tank is released to the municipal sewer system.

- Samples above regulatory limits and expired RCRA metals standards (Waste Corrosive Liquid, Acidic, Inorganic, n.o.s.) are collected in satellite accumulation and sent off site through a Waste disposal vendor.
  Onyx Profile WIP Number: 590598
  Teris Profile Number 50016653

15. References / Cross-References

15.1. Determination of Mercury in Water by Cold Atomic Absorption Spectrometry, EMSL-Cincinnati, EPA/600/R-94/11, May 1994; Method 245.1 Revision 3.0.

15.2. 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.

15.3. TestAmerica Edison Document No. ED-QA-LQM, *Laboratory Quality Manual*, most current revision.

15.4. TestAmerica Edison SOP ED-GEN-022, *Training*, most current revision.

15.5. TestAmerica Edison SOP No. ED-SPM-008, *Laboratory Waste Disposal Procedures*, current revision.

15.6. Corporate Environmental Health and Safety Manual CW-E-M-001, most current revision.

15.7. Metals Data Review Checklist Work Instruction # EDS-WI-007, most current revision.

15.8. TestAmerica Edison Metals Initial Calibration Data Review Mercury checklist, Work Instruction EDS-WI-125, most current revision.

15.9. Leeman Hydra AA Operating Manual

16. Method Modifications:

Item	Method No.	Modification
Sample Preparation	SW 7470A	Stannous Chloride is automatically added via the instrument versus the manual addition of Stannous Chloride as stated in the method. This is an instrument manufacturer's improvement that will reduce error due to loss of Mercury.
Sample Preparation	SW 7470A EPA 245.1	The hotblock has replaced the hot-water bath for digestion. This modification has been made to reduce cross-contamination (the hotblock tubes are disposable).
Sample Preparation	SW 7470A EPA 245.1	The typical prep sample size is reduced to 30ml (previously 100ml). This modification was made to allow for limited available sample volumes. Reagent volumes were adjusted to maintain sample to reagent volume ratio
Sample Preparation	EPA 245.1	The Initial Calibration Verification (ICV) will be prepared from a secondary source (as required in Method SW7470A), rather than the calibration standard source. Method 7470A and 245.1 will be prepared and analyzed in the same batch which improves efficiency and reduces waste. CCV will be prepared using the same source as the calibration standards.

17. Attachments

Attachment 1: Example Certificate of analysis (10 ppm Hg)

18. Revision History

- Revision 11, dated 25 April 2018
  - Sec 8: Added footnote 3 to clarify instructions for metals sample preservation (verify pH <2.0 prior to digestion).
  - Sec 10.1.2: Added procedure for samples which pH are >2.0 and acid preservation is performed in laboratory.
  - Sec 12.3: Added procedure for the determination of linear dynamic range.

- Revision 10, dated 05 February 2018
  - Sec 7.2.10: Added CRI to the list of standards.
  - Sec 9.2.6: Added CRI to the list of instrument QC.
  - Sec 10.3.2: Revised the instrument sequence list to include CRI.

- Revision 9, dated 09 December 2015
  - Sec 7.2.7: Revised preparation procedure for ICV, standard solution should be obtained from a source different from the calibration standard. Removed reference to CCV.
  - Sec 7.2.8: Added CCV preparation procedure; standard solution same source as the calibration standard. Subsequent sections adjusted accordingly.
  - Sect 9.2.1: Revised - ICV solution should be prepared from a separate source.
  - Section 16: Added Method Modifications for using the secondary source when

preparing the ICV standard for method EPA 245.1.

- Revision 8, dated 13 January 2014
  - Sec 7.1.8: Added the stock standard Potassium Persulfate to the list of reagents; subsequent sections adjusted accordingly.
  - Sect 7.1.15: Added preparation instructions for the 5% Potassium Persulfate solution.
  - Sec 7.1: Revised to include recording of reagent prep information in TALS and discontinue the use of Mercury Prep Logbook.
  - Sec 7.2.5 & 7.2.6: Revised the spiking amount to add when preparing calibration standards in 30 ml final volume; also replaced the 100 ml flasks with 50 ml digestion cups.
  - Sec 9.0: The matrix spike (MS) frequency for Method 245.1 has been revised to 1 per 10 samples to comply with the method.
  - Sec 11.4.4: Added the Mercury calibration checklist (EDS-WI-125).
  - Sec 15.8: Added WI# ED-WI-125 on the list of references; subsequent sections adjusted accordingly.
  - Sec 17: Updated the attached COA for the Hg standards.

- Revision 7, dated 02 December 2011
  - Sec 1.1: Revised detection limit to 0.2 ug/L Hg to reflect actual laboratory limits.
  - Sec 1 & 12: Revised the LQM reference for DOC and Test Methods and Method Validation to Section 19.
  - Sec 3: Revised LQM reference for the definitions.
  - Sec 5: Revised in accordance with TestAmerica Corporate Quality SOP No. CW-QS-002, Writing a Standard Operating Procedure (SOP), most current revision.
  - Sec 7.1: Added requirements that all prepared reagents are recorded in TestAmerica Edison Mercury Std Prep Log.
  - Sec 7.2 & 10.2.1: Revised the documentation of standard preparation from logbook to TALS.
  - Sec 7.2.5: Replaced cal std 0.1 ug/L (Hg) with 0.2 ug/L.
  - Sec 9.1 and 9.1.1: Expanded MB control limit for 245.1 to reflect method's criteria.
  - Replaced PB (preparation blank) with MB (method blank) in various sections where applicable.
  - Sec 10.1.1: Added procedure for filtering dissolved samples in the lab.
  - Sec 10.1.2.1: Added procedure for pre-digestion dilutions.
  - Sec 10.1.8: Added procedure for visually checking and adjusting final volumes for all standards and samples.
  - Sec 10.3.3.11.7: Added procedure for post-digestion dilutions.

- Revision 6, dated 03 September 2009

  - Revised SOP format in accordance with TestAmerica Corporate Quality SOP No. CW-QS-002 (Rev 0) Writing a Standard Operating Procedure (SOP).
  - Combined SOP ED-MT-014 and ED-MT-015 with SOP ED-MT-017; retired SOP ED-MT-014 and ED-MT-015 at the effective date of this SOP.
  - Sec 6.1.3: Replaced water bath with block digestor.
  - Sec 6.2.1: Replaced 300 ml BOD bottles with 50 ml hot block digestion cups
  - Sec 6.2.3. Added 'Rinse Black' and changed Drain 'Black' to Drain 'Blue' in the list of Reagent lines
  - Sec 7.1: Added 0.15% HNO3 to list of reagents. Deleted Potassium Persulfate,

Magnesium Perchlorate, 0.5N H2SO4 & 5% Potassium Persulfate solution to list of reagents; Reagents deleted are not applicable to this method.

- Sec 7.2 Standards: Revised the Hg stock standard concentrations and preparation of standards.
- Sec 7.1.9 & 7.1.13: Revised preparation procedure to reflect actual lab practices.
- Sec 7.2.6: Renamed the second source standard 'Initial Calibration Verification standard' to 'Quality control standard.' QCS is added in the Instrument QC and analytical run sequence.
- Section 8: Updated the section into Table format and have included the sample container, sample size requirements and method reference.
- Changed the verification standard concentration of ICV (3ppb) to 5ppb; CCV concentration remains 5ppb.
- Revised control limits to comply with Method 245.1
- Sec 9.1.1: Clarified QC limits for the Preparation Blank
- Sec 9.1.2: Revised the LCS limits for wastewater samples analyzed via Method 245.1 from ±20% to ±15%.
- Sec 9.1.4: Revised the MS limits for wastewater samples analyzed via Method 245.1 from ±20% to ±30%.
- Sec 9.1.5: Added Serial Dilution (L) in Sample QC
- Sec 9.2.1: Clarified the recovery limits of ICV for method 245.1; % Rec limits were revised from 10% to 5% to reflect actual laboratory practices.
- Sec 10.3.2: Added MCL in the analytical run sequence.
- Sec 10.3.3.2: Added 'Rinse Black' and changed Drain 'Black' to Drain 'Blue' in the list of Reagent lines.
- Sample size reduced from 100 ml to 30ml; preparation of the LCS (Sec 9.1.2) and MS (Sec 9.1.4) were revised to reflect this change in sample volume.
- Sec 10.1: Adjusted reagent volume to maintain sample to reagent ratios.
- Sec 11: Updated data processing in accordance with the new TALS.
- Sec 15: Added applicable references.
- Sec 16: Described the elimination of stannous chloride in the sample preparation and replacement of hot water bath as a method modification.

Attachment 1

SCP SCIENCE
Providing Innovative Solutions to Analytical Chemists

ACCREDITED

Certificate of Analysis

1.0 DESCRIPTION : ***Plasma*** **CAL – Custom Standard**

Catalogue Number : **141-110-11X**
Lot Number : **S130418021**
Matrix: 10.0% HNO3
Expiration Date : **July 2014**

2691783
ID: ME_Hg_1st_00013
Exp: 07/01/14 Prod: DLE
Mercury Calibration Stand

2.0 CERTIFIED VALUES AND ASSOCIATED UNCERTAINTY:

Method of Analysis: Inductively Coupled Plasma Atomic Emission Spectroscopy (ICP-AES)

Traceability: Applicable NIST Standard Reference Materials (see list below):

3101a	Al	3109a	Ca	3117a	Eu	3126a	Fe	3134	Mo	3142a	Pr	3151	Ag	3159	Th	3167a	Y
3102a	Sb	3110	Ce	3118a	Gd	3127a	La	3135a	Nd	3143	Re	3152a	Na	3160a	Tm	3168a	Zn
3103a	As	3111a	Cs	3119a	Ga	3128	Pb	3136	Ni	3144	Rh	3153a	Sr	3161a	Sn	3169	Zr
3104a	Ba	3112a	Cr	3120a	Ge	3129a	Li	3137	Nb	3145a	Rb	3154	S	3162a	Ti		
3105a	Be	3113	Co	3121	Au	3130a	Lu	3138	Pd	3147a	Sm	3155	Ta	3163	W		
3106	Bi	3114	Cu	3122	Hf	3131a	Mg	3139a	P	3148a	Sc	3156	Te	3164	U		
3107	B	3115a	Dy	3123a	Ho	3132	Mn	3140	Pt	3149	Se	3157a	Tb	3165	V		
3108	Cd	3116a	Er	3124a	In	3133	Hg	3141a	K	3150	Si	3158	Tl	3166a	Yb		

Certified Concentrations:

Hg 9.96 ± 0.06 µg/ml

Note: The uncertainty of the certified value has been calculated from applicable uncertainty contributors (u_i) such as the SRM inherited uncertainty, weighing and dilution errors and instrument variability. The combined uncertainty ($u_c = \sqrt{2u_i^2}$) has been multiplied by a coverage factor (k) of 2 to provide a 95% confidence interval.

3.0 REFERENCE VALUES:

Density: **1.051 g/ml** @ **20.9 °C**

4.0 APPROVAL AND DATE OF CERTIFICATION:

Certification Approval: Yaling Sui, Chemist Yaling Sui
Certification Date: **April 19, 2013**

INORGANIC VENTURES
300 Technology Drive
Christiansburg, VA 24073 • USA
inorganicventures.com

c.c.

CERTIFICATE OF ANALYSIS

tel: 800.669.6799 • 540.585.3030
fax: 540.585.3012
info@inorganicventures.com

1.0 **INORGANIC VENTURES** is an ISO Guide 34 "General Requirements for the Competence of Reference Material Producers" and ISO 9001 registered manufacturer. Our manufacturing laboratory is accredited to ISO/IEC 17025 "General Requirements for the Competence of Testing and Calibration Laboratories."

ACCREDITED Testing Laboratory Certificate 863.01

ACCREDITED Reference Material Producer Certificate 863.02

2.0 **DESCRIPTION OF CRM** **10 µg/mL Mercury in 10% (v/v) HCL**

2035815
ID: ME_Hg_00019
Exp: 04/01/14 Prod: CDC
Mercury Calibration Stand

Catalog Number:	MSHG-10PPM
Lot Number:	F2-HG02097
Starting Material:	Hg metal
Starting Material Purity (%):	100.0000
Starting Material Lot No:	R307HGA1
Matrix:	10% (v/v) HCL

3.0 **CERTIFIED VALUES AND UNCERTAINTIES**

Certified Concentration: 9.990 ± 0.074 µg/mL

Certified Density: 1.026 g/mL (measured at 20 ± 1°C)

The following equations are used in the calculation of the certified value and the uncertainty. Reported uncertainties represent expanded uncertainties expressed at approximately the 95% confidence level using a coverage factor of k = 2.

Certified Value $(\bar{x}) = \frac{\Sigma x_i}{n}$ ($\bar{x}$) = mean

x_i = individual results

n = number of measurements

Uncertainty (±) = $2\,[\,\Sigma(s_i)^2\,]^{1/2}$ 2 = the coverage factor.

$[\,\Sigma(s_i)^2\,]^{1/2}$ = The square root of the sum of the squares of the most common errors (where 's' stands for the standard deviation) from instrumental measurement, density, NIST SRM uncertainty, weighing, dilution to volume, homogeneity, long term stability and short term stability.

4.0 **TRACEABILITY TO NIST AND VALUES OBTAINED BY INDEPENDENT METHODS**

- "Property of the result of a measurement or the value of a standard whereby it can be related to stated references, usually national or international standards, through an unbroken chain of comparisons all having stated uncertainties." (ISO VIM, 2nd ed.,

Title: Trace Metals Analysis for Water, Wastewater, Soil, Sediment and Leachate Samples by ICP-MS Method No(s). EPA Method 200.8 and SW-846 Method 6020

Once printed, this is considered an uncontrolled document

Approvals (Signature/Date):

Laura Demone	04/26/2018	Dan Helfrich	04/26/2018
Laura Demone	Date	Dan Helfrich	Date
Department Manager		Health & Safety Manager Coordinator	
Carl Armbruster	04/26/2018	Mark Acierno	04/26/2018
Carl Armbruster	Date	Mark Acierno	Date
Quality Assurance Manager		Laboratory Director	

Copyright Information:
This documentation has been prepared by TestAmerica Laboratories, Inc. and its affiliates ("TestAmerica"), solely for their own use and the use of their customers in evaluating their qualifications and capabilities in connection with a particular project. The user of this document agrees by its acceptance to return it to TestAmerica upon request and not to reproduce, copy, lend, or otherwise disclose its contents, directly or indirectly, and not to use it for any other purpose other than that for which it was specifically provided. The user also agrees that where consultants or other outside parties are involved in the evaluation process, access to these documents shall not be given to said parties unless those parties also specifically agree to these conditions.

THIS DOCUMENT CONTAINS VALUABLE CONFIDENTIAL AND PROPRIETARY INFORMATION. DISCLOSURE, USE OR REPRODUCTION OF THESE MATERIALS WITHOUT THE WRITTEN AUTHORIZATION OF TESTAMERICA IS STRICTLY PROHIBITED. THIS UNPUBLISHED WORK BY TESTAMERICA IS PROTECTED BY STATE AND FEDERAL LAW OF THE UNITED STATES. IF PUBLICATION OF THIS WORK SHOULD OCCUR THE FOLLOWING NOTICE SHALL APPLY:

©COPYRIGHT 2018 TESTAMERICA LABORATORIES, INC. ALL RIGHTS RESERVED.

1.0 Scope and Application

1.1 Analytes, Matrix(s), and Reporting Limits

This SOP describes the procedures used to determine the concentration of various elements in surface, drinking, and ground water, wastewater, soil, sediment, leachate (ASTM, SPLP or TCLP), and waste samples by Inductively Coupled Plasma - Mass Spectrometry (ICP-MS) using EPA Method 200.8 and EPA SW-846 Method 6020.

The routine target analytes and reporting limits are as follows:

Table 1

Analyte List and Method Reporting Limits (RL)

Element	Drinking Water RL	Digested Water RL (adjusted for typical 5X dilution)	Soil RL (adjusted for typical 20X dilution)
	(200.8) (ug/L)	(200.8 NPDES & 6020) (Prep: 200.8/3010) (ug/L)	(6020) (Prep: 3050B) (mg/Kg)
Aluminum (Al)	25	50	10
Antimony (Sb)	2.5	2.5	0.5
Arsenic (As)	1	2.5	0.5
Boron (B)	20	100	20
Barium (Ba)	2.5	5	1
Beryllium (Be)	2.5	1.0	0.2
Cadmium (Cd)	2.5	2.5	0.5
Calcium (Ca)	250	250	50
Cobalt (Co)	2.5	5	1
Chromium (Cr)	2.5	5	1
Copper (Cu)	2.5	5	1
Iron (Fe)	250	150	30
Lead (Pb)	2.0	1.5	0.3
Magnesium (Mg)	250	250	50
Manganese (Mn)	25	10	2
Molybdenum (Mo)	2.5	5	1
Nickel (Ni)	2.5	5	1
Potassium (K)	250	250	50
Selenium (Se)	2.5	2.5	0.5
Silver (Ag)	2.5	5	1
Sodium (Na)	250	250	50
Strontium (Sr)	2.5	5	1
Thallium (Tl)	0.5	1	0.2
Tin (Sn)	4	20	4
Titanium (Ti)	2.5	5	1
Vanadium (V)	2.5	5	1
Zinc (Zn)	4	20	4

On occasion clients may request modifications to this SOP. These modifications are handled following the procedures outlined in Sections 7 (Review of Work Request) and 19 (Test Methods and Method Validation) of TestAmerica Edison's Quality Assurance Manual (ED-QA-LQM).

2.0 Summary of Method

Prior to analysis by ICP-MS, the sample must be solubilized or digested using the sample preparation method appropriate to the matrix. Sample digestates are aspirated and nebulized into a spray chamber. A stream of argon gas carries the sample aerosol through the innermost of three concentric tubes and injects it into the middle of the donut-shaped plasma. The sample elements are dissociated, atomized, and excited to a higher energy level. The ions that are produced are entrained in the plasma and introduced, by means of an interface, into a mass spectrometer. The ions are sorted according to their mass to charge ratios and quantified with a channel mass spectrometer.

Aqueous samples are digested using Method 200.8 and SW846 3010. Soil samples are digested using Method 3050B. Drinking water samples with turbidity $\geq$ 1 NTU must undergo digestion prior to analysis. Refer to TestAmerica Edison SOP Nos. ED-MTP-001, ED-MTP-003 and ED-MTP-005 and for the digestion procedure (see Section 15.0 for complete references).

3.0 Definitions

For a complete list of definitions refer to Appendix 2 in the most current revision of TestAmerica Edison's Quality Assurance Manual (ED-QA-LQM).

4.0 Interferences

4.1. Isobaric elemental interferences are caused by isotopes of different elements which form singly or doubly charged ions of the same nominal mass-to-charge ratio and which cannot be resolved by the mass spectrometer in use. Isobaric polyatomic/molecular ion interferences are caused by ions consisting of more than one atom which have the same nominal mass-to-charge ratio as the isotope of interest and which cannot be resolved by the mass spectrometer in use. Most isobaric interferences that could affect the ICP-MS analysis for elements in this SOP have been identified. These can be managed by the selection of an alternate isotope, the use of the collision/reaction cell (used for non-potable samples only), or by the use of elemental interference equations when the collision/reaction cell is not in use.

4.2. For Drinking Water analysis (standards, samples, and relating QC) the collision/reaction cell can not be used; therefore, the following interference equations should be used to reduce interferences:

Mass	Equation
6:	(6)*1 - (7)*0.082
44:	(44)*1 - (88)*0.015
51:	(51)*1 – (53)*3 + (52)*0.34

66: (66)*1 – (69)*0.00141
75: (75)*1 – (77)*2.9 + (82)*2.23 – (83)*2.23
82: (82)*1 – (83)*1
111: (111)*1 – (108)*1.073 + (106)*0.712
115 (115)*1 – (118)*0.016
208: (208)*1 + (206)*1 + (207)*1 (used for both drinking and non-drinking water analysis)

The basic elemental interference equations are based on natural isotopic abundances. The most precise coefficients for an instrument must be determined from the ratio of the net isotope signals that are observed for a known standard solution at a concentration sufficient to produce suitable counting statistics.

4.3. Krypton affects the determination of both arsenic and selenium but can be greatly reduced with the use of high purity Krypton free argon. Krypton must be analyzed for every sample and standard.

4.4. Physical interferences are effects associated with the sample nebulization and transport processes as well as ion-transmission efficiencies. Changes in viscosity can cause significant inaccuracies, especially in samples containing high concentrations of dissolved solids or high acid concentrations. These changes in matrix can cause significant signal suppression or enhancement. Dissolved solids can deposit on nebulizer tips and interface cones (reducing the orifice size and the instrument's performance). Internal standards can be used to correct for physical interferences if they are carefully matched to the analyte so that both elements react similarly to the matrix changes.

4.5. Memory interferences can occur when analytes from a previous sample contribute to signals measured from subsequent samples. The memory effects can result from analyte deposition of sample on the sample tubing, joints, nebulizer, spray chamber, torch, and/or interface cones. Routine maintenance on the sample introduction system is necessary in order to minimize the memory interferences. The memory effects must be taken into account when setting up a suitable rinse times. The evaluation of a minimum of three replicate integrations will help to determine memory problems.

4.6. Contamination of the sample can occur when the preparation glassware and/or reagents contain the target elements. Reagent blanks (method blanks) must be analyzed as a check on contamination due to the sample digestion.

5.0 Safety

Employees must abide by the policies and procedures in the Corporate Environmental Health and Safety Manual (CW-E-M-001) and this document. This procedure may involve hazardous material, operations and equipment. This SOP does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of the method to follow appropriate safety, waste disposal and health practices under the assumption that all samples and reagents are potentially hazardous. Safety glasses, gloves, lab coats and closed-toe, nonabsorbent shoes are a minimum.

5.1. Specific Safety Concerns or Requirements

The ICP plasma emits strong UV light and is harmful to vision. All analysts must avoid looking directly at the plasma.

5.2. Primary Materials Used

The following is a list of the materials used in this method, which have a serious or significant hazard rating. **Note: This list does not include all materials used in the method. The table contains a summary of the primary hazards listed in the MSDS for each of the materials listed in the table.** A complete list of materials used in the method can be found in the reagents and materials section. Employees must review the information in the MSDS for each material before using it for the first time or when there are major changes to the MSDS.

Material	Hazards	Exposure Limit (1)	Signs and symptoms of exposure
Hydrochloric Acid	Corrosive Poison	5 ppm-Ceiling	Inhalation of vapors can cause coughing, choking, inflammation of the nose, throat, and upper respiratory tract, and in severe cases, pulmonary edema, circulatory failure, and death. Can cause redness, pain, and severe skin burns. Vapors are irritating and may cause damage to the eyes. Contact may cause severe burns and permanent eye damage.
Nitric Acid	Corrosive Oxidizer Poison	2 ppm-TWA 4 ppm-STEL	Nitric acid is extremely hazardous; it is corrosive, reactive, an oxidizer, and a poison. Inhalation of vapors can cause breathing difficulties and lead to pneumonia and pulmonary edema, which may be fatal. Other symptoms may include coughing, choking, and irritation of the nose, throat, and respiratory tract. Can cause redness, pain, and severe skin burns. Concentrated solutions cause deep ulcers and stain skin a yellow or yellow-brown color. Vapors are irritating and may cause damage to the eyes. Contact may cause severe burns and permanent eye damage.
1 – Exposure limit refers to the OSHA regulatory exposure limit.			

6.0 Equipment and Supplies

6.1. Instrumentation

- Agilent 7500CE, inductively coupled plasma mass spectrometer with data system (microprocessor, monitor, printer) and autosampler. Resolution 0.011 nm on a holographically grooved grating (2400 grooves/mm). Vacuum purged spectrophotometer with an axial plasma torch. Collision/Reaction Cell.

- Heat exchanger – Polyscience Model 3370 air cooled recirculator

- Autosampler – CETAC ASX 520

6.2. Supplies

- Reagent Water -18 megohm Reagent grade Type II water
- Volumetric Flasks (Class A): 50 mLs, 100 mLs, 500mls & 1000mls
- Eppendorf & Fisher Pipettes, varying volumes
- Polypropylene tubes

7.0. Reagents and Standards

7.1. Reagents

7.1.1. Concentrated Nitric Acid (HNO3) - Trace Grade or Equivalent; store at room temperature; for stability information, refer to manufacturer's instructions. The assay sheet of each lot of acid received into the lab must be reviewed to ensure the quality of the acid is sufficient for trace analysis of metals.

7.1.2. Concentrated Hydrochloric Acid (HCL) - Trace Grade or Equivalent; store at room temperature; for stability information, refer to manufacturer's instructions. The assay sheet of each lot of acid received into the lab must be reviewed to ensure the quality of the acid is sufficient for trace analysis of metals.

7.1.3. Argon supply - 99.9% (Liquid)

7.1.4. Helium and Hydrogen supply – 99.999% (Gas)

7.1.5. 18 megohm Reagent grade Type II water.

7.1.6. Reagent water, 2% HNO3 + 0.5% HCl by volume: Add 400ml of concentrated HNO3 and 100ml of concentrated HCl to deionized water and bring to 20 liter volume with deionized water. For potable samples not requiring digestion, the reagent water is prepared without HCL. Note: Always add acid to water. Record preparation in the TALS Reagent Module Prepare every 12 months or refer to manufacturer's expiration date; store at room temperature.

7.2. Standards

Calibration and spike solutions are prepared from either certified stock solutions or from stock solutions purchased from vendors. Certificates of analysis or purity must be received with all neat compounds or stock solutions. Certificate of analysis are filed in Metals managers office.

Storage requirements: all standards are stored at room temperature

Shelf-life: Stock standards – refer to manufacturer's instructions
Intermediate standards – 6 months
Working cal standards – two weeks
Working Initial calibration verification standard – quarterly
Interference Check Standards (A&B) - weekly
(Note: expiration date must not go beyond the expiration date of the source stock).

Concentration: Final concentrations for the calibration standards are given in Attachment 1. All standards must be prepared in reagent water (Sec. 7.1.6).

Standards must be prepared in accordance to the shelf life (Sec 7.2) "or sooner if needed or required." "If needed" means the standard has been exhausted; "if required" means that the standard does not meet the QC criteria.

All standards must be prepared in reagent water (Sec. 7.1.6). Final concentrations for the calibration standards are given in Attachment 1.

7.2.1. Calibration Standards: ICPMS-Cal (Custom Calibration Standard Stock Solution) purchased from Inorganic Ventures. Refer to manufacturer's instructions for stability and storage information.

TANJ-STD-1 contains: 20ug/mL B
10 ug/mL Mo, Sb, Sn and Ti

TANJ-STD-2 contains: 1000 ug/mL Ca, Fe, K, Mg, & Na
100 ug/mL Al & Mn
10 ug/mL Ag, As, Ba, Be, Cd, Co, Cr3, Cu, Ni, Pb, Se, Sr, V & Zn
2.0 ug/mL Tl

TA-88 contains: 10,000 ug/L Ca, K, Mg, Na
6,000 ug/L Fe
4,000 ug/L B
2,000 ug/L Al
800 ug/L Sn, Zn
500 ug/L Se
400ug/L Mn
200 ug/L Ba, Co, Cr, Cu, Mo, Ni, Sr, Ti, V
100 ug/L Ag, As, Cd, Sb
60 ug/L Pb
40 ug/L Be, Tl

STLNJ-STD-1 contains: 5000 ug/L Ca, K, Mg, Na
3,000 ug/L Fe
2,000 ug/L B
1,000 ug/L Al
400 ug/L Zn
200ug/L Mn
100 ug/L Ag, Ba, Co, Cr, Cu, Ni, Sr, V
50 ug/L As, Cd, Se

30 ug/L Pb
20 ug/L Be, Tl

STLNJ-STD-2 contains: 400 ug/L Sn
100 ug/L Mo, Ti
50 ug/L Sb

TA-72REV2 contains: 5000 ug/L Ca, Fe, K, Mg, Na
2500 ug/L Al, Mn,
2000 ug/L B
400 ug/L Sn, Zn,
250 ug/L Ag, Ba, Be, Cd, Co, Cr, Cu, Mo, Ni, Sb, Se, Sr, Ti, V
Pb 200 ug/L
As 100 ug/L
Tl 50 ug/L

7.2.2. Working Calibration Standards:

For Non-Drinking Waters:

CAL5: Add 100ml of reagent water (Sec 7.1.6) to a clean 200ml flask. Add 2ml each of TANJ-STD-1 and TANJ-STD-2. Bring to volume with reagent water (Sec 7.1.6).

CAL4/CCV: Add 200ml of reagent water to a clean 500ml flask. Add 2.5ml each of TANJ-STD-1 and TANJ-STD-2. Bring to volume with reagent water (Sec 7.1.6).

CAL3: Add 100ml of reagent water to a clean 200mL flask. Add 0.2ml each of TANJ-STD-1 and TANJ-STD-2. Bring to volume with reagent water (Sec 7.1.6).

CAL2: Add 100ml of reagent water to a clean 200ml flask. Add 2.0ml of TA-88. Bring to volume with reagent water (Sec 7.1.6).

CAL1: Add 100ml of reagent water to a clean 200ml flask. Add 2.0ml each of STLNJ-STD-1 (TALS reagent name - ME_ICMSRL1 and STLNJ-STD-2 (TALS reagent name - ME_ICMSRL2). Bring to volume with reagent water (Sec 7.1.6). This standard is used as the Reporting Limit standard.

For Drinking Waters:

CAL4: Add 100ml of reagent water (Sec 7.1.6) to a clean 200ml flask. Add 2ml each of TANJ-STD-1 and TANJ-STD-2. Bring to volume with reagent water (Sec 7.1.6).

CAL3/CCV: Add 100ml of reagent water to a clean 200ml flask.

Add 1 mL each of TANJ-STD-1 and TANJ-STD-2. Bring to volume with reagent water (Sec 7.1.6).

CAL2: Add 100ml of reagent water to a clean 200mL flask. Add 0.2ml each of TANJ-STD-1 and TANJ-STD-2. Bring to volume with reagent water (Sec 7.1.6).

CAL1: Add 100ml of reagent water to a clean 200mL flask. Add 2 mL of TA-72REV2. Bring to volume with reagent water (Sec 7.1.6). This standard is used as the Reporting Limit standard.

Note: see Attachment 1 for final elemental concentrations.

7.2.3. Initial Calibration Verification standard: purchased from CPI International. Refer to manufacturer's instructions for stability and storage information. Standards must be from a different source than those used for the calibration standards.

Standard	Element	Conc. µg/mL
ICV1ICPMS	Be	5
	Na	500
	Mg	500
	Al	50
	K	500
	Ca	500
	V	5
	Cr	5
	Mn	50
	Fe	500
	Co	5
	Ni	5
	Cu	5
	Zn	5
	As	5
	Se	5
	Sr	5
	Ag	5
	Cd	5
	Ba	5
	TL	1
	Pb	5
ICV2ICPMS	B	10
	Ti	5
	Mo	5
	Sn	5
	Sb	5

7.2.4. ICV Working Standard: The ICV Working Standard must be at a concentration other than the concentrations used for the CCV working standard. Prepare by adding 300ml of reagent water (Sec 7.1.6) to a clean 500ml flask. Add 4ml each: ICV1ICPMS and ICV2ICPMS. Bring to volume with reagent water (Sec 7.1.6). The final concentrations of the various elements are listed in Attachment 1. This standard is prepared fresh quarterly or as needed and store at room temperature.

7.2.5. Continuing Calibration Verification standard, Non-potable: see CAL4/CCV (Sec. 7.2.2)see CAL4 (Sec. 7.2.2).

7.2.6. Continuing Calibration Verification standard, Potable: see CAL3/CCV(Sec. 7.2.2).

7.2.7. Internal Standards and EPA Tune Check:

The following elements are used as the internal standards: Bi, In, Li6, Sc, Tb, and Ge.

7.2.7.1. Internal Standards Stock Solutions**:**

7.2.7.1.1. Testamerica-1 (Custom Calibration Standard Stock Solution) purchased from Inorganic Ventures. Refer to manufacturer's instructions for stability and storage information. Contains: 250 mg/L each: Li6, Sc; 50 mg/L each: Bi, In, Tb.

7.2.7.1.2. MSGE-100PPM (Custom Calibration Standard Stock Solution) purchased from Inorganic Ventures. Refer to manufacturer's instructions for stability and storage information. Ge 100 mg/L.

7.2.7.1.3. Triton X-100 (Standard Stock Solution: Non-ionic Surfactant) purchased from Fisher Scientific, p/n BP151-100. Refer to manufacturer's instructions for stability and storage information. According to the ICPMS manufacturer (Agilent Technologies), Triton-X 100 will reduce the 3 replicate %RSD during analysis.

7.2.7.2. Working Internal Standard Solution: add 100ml of reagent water to a clean 1000ml flask. Add 20 mL of Testamerica-1 and 10ml of MSGE-100PPM, and 1ml of Triton X-100. Bring to volume with reagent water. This solution contains 1 mg/L of Bi, Ge, In, Tb and 5 mg/L of Li6 and Sc.

The working internal standard is added in-stream by the use of a T-fitting. The resulting concentration (in-stream) of the standards are ~50 ug/L of Bi, Ge, In, and In and ~250 ug/L of Li6 and Sc.

The following are the recommended analysis masses, tune steps, and internal standards:

Element	IS
Be9	Li6
B11	Li6
Na23	Sc45
Mg24	Sc45
Al27	Sc45
K39	Sc45
Ca44	Sc45
Ti47	Sc45
V51	Sc45
Cr52	Sc45
Mn55	Sc45
Fe56	Sc45
Co59	Sc45
Ni60	Sc45
Cu63	Sc45
Zn 66	Sc45
As75	Ge74
Se78	Ge74
Sr88	In115
Kr83**	(none)
Mo95	In115
Ag107	In115
Cd111	In115
Sn118	In115
Sb121	In115
Ba137	Tb159
Tl205	Bi209
Pb 208*	Bi209

*Pb 208 = Pb 208 + Pb 207 + Pb 206
**Kr83 is used for monitoring Krpyton levels only.

Different masses and internal standards may be utilized, as matrix issues deem necessary. Non-drinking waters are analyzed using collision/reaction cell technology (helium/hydrogen gas mode). Typical non-drinking water Collision/Reaction Cell usage: Selenium is analyzed in the Hydrogen reaction mode; all other elements are analyzed in the helium collision mode. For drinking water analyses, collision cell technology is not allowed; therefore, the no-gas mode is utilized. Use masses Fe57, Cu65, and Se82 instead of their respective masses listed above.

Tune check criteria:

Instrument tunes must be performed daily, before calibration. A solution at ~100ppb of Be, Mg, Co, In, and Pb is analyzed, and the precision, mass calibration, and resolution are checked. Resolution is checked by analyzing Magnesium isotopes 24, 25, 26 and Lead isotopes 206, 207, 208.

The following limits are used to evaluate mass calibration, resolution, and instrument stability:

The following limits are used to evaluate the tune:

Mass calibration: +/-0.1 amu
Resolution check: ≤0.9 amu at 10% peak height. Note: This also satisfies Method 200.8, 1.0 amu at 5% peak height)
Stability (5reps): <5%

7.2.7.3. EPA Tune Check Stock Standard: 2008TS purchased from Inorganic Ventures. Refer to manufacturer's instructions for stability and storage information. Contains: 10 ug/mL each: Be, Co, In, Mg, Pb.

7.2.7.4. Working EPA Tune Check Standard: add 100ml of reagent water to a clean 1000ml flask. Add 10 mL of 2008TS. Bring to volume with reagent water. This solution contains 100 ug/L of Be, Co, In, Mg, and Pb

7.2.8. Reporting Limit (CRI) Check standard:

For Non-Drinking Water: See Section 7.2.2, CAL1
For Drinking Water: See Section 7.2.2, CAL1.

7.2.9. ICP Interference Check Solutions: Stock solutions for ICSA and ICSAB, purchased from Inorganic Ventures, contains the following elemental concentrations:

6020ICS-8A contains: 18,000ppm Chloride, 3000ppm Ca, 2500ppm ea: Fe, Na, 2000ppm Carbon, 1000ppm ea: Al, K, Mg, P, S, 20 ppm ea: Mo, Ti

6020ICS-8B contains: 20ppm ea: Ag, Co, Cr3, Cu, Mn, Ni, V
10ppm ea: As, Cd, Se, Zn

7.2.10. Working Interference Standards (ICSA/ICSAB):

For Non-Drinking Waters:

ICSA: Add 50ml of reagent water (Sec 7.1.6) to a clean 100ml flask. Add 5ml of 6020ICS-8A stock solution. Bring to volume with reagent water (Sec 7.1.6). This standard is made fresh weekly; store at room temperature. Refer to Attachment 2 for final elemental concentrations.

ICSAB: Add 50ml of reagent water (Sec 7.1.6) to a clean 100ml flask. Add 5ml of 6020ICS-8A and 1ml of 6020ICS-8B stock solutions. Bring to volume with reagent water (Sec 7.1.6). This standard is made fresh weekly; store at room temperature. Refer to Attachment 2 for final elemental concentrations.

For Drinking Waters:

ICSA: Add 50ml of reagent water (Sec 7.1.6) to a clean 100ml flask. Add 2.5ml of 6020ICS-8A stock solution. Bring to volume with reagent water (Sec 7.1.6). This standard is made fresh weekly; store at room temperature. Refer to Attachment 2 for final elemental concentrations.

ICSAB: Add 50ml of reagent water (Sec 7.1.6) to a clean 100ml flask. Add 2.5ml of 6020ICS-8A and 0.5ml of 6020ICS-8B stock solutions. Bring to volume with reagent water (Sec 7.1.6). This standard is made fresh weekly; store at room temperature. Refer to Attachment 2 for final elemental concentrations.

7.2.11. ICP-MS Matrix Spiking Solution, ICPMS LCS/SPK: purchased from High-Purity Standards. Refer to manufacturer's instructions for stability and storage information. Refer to attachment 2 for final elemental concentrations. Stock solution contains the following elemental concentrations:

Element	Conc. Of Stock (mg/L)
Aluminum (Al)	500
Antimony (Sb)	5
Arsenic (As)	10
Barium (Ba)	10
Beryllium (Be)	5
Boron (B)	100
Cadmium (Cd)	5
Calcium (Ca)	500
Chromium (Cr)	10
Cobalt (Co)	5
Copper (Cu)	10
Iron (Fe)	500
Lead (Pb)	5
Magnesium(Mg)	500
Manganese (Mn)	50
Molybdenum (Mo)	10
Nickel (Ni)	10
Potassium (K)	500
Selenium (Se)	10
Silver (Ag)	5
Sodium (Na)	500
Strontium (Sr)	10
Thallium (Tl)	4
Tin (Sn)	10
Titanium (Ti)	10
Vanadium (V)	10
Zinc (Zn)	50

8.0. Sample Collection, Preservation, Shipment and Storage

Sample container, preservation techniques and holding times may vary and are dependent on sample matrix, method of choice, regulatory compliance, and/or specific contract or client requests. Listed below are the holding times and the references that include preservation requirements.

Matrix	Sample Container	Min. Sample Size[2]	Preservation	Holding Time[1]	Reference
Metals Waters except Hg	Polyethylene or Glass	500 mLs	HNO_3 to pH < 2 prior to shipment; if not, acidify upon receipt in lab [3,4]	6 months	40 CFR Part 136.3
Soils except Hg	8 oz or 16 oz.Polyethylene or Glass	10 grams	Cool 4 ± 2°C	6 months	SW846 Method 6020

[1] Inclusive of digestion and analysis.
[2] Drinking water samples may require 1000ml sample volume.
[3] Acid preservation may be omitted for shipping; however, acid must be added upon receipt in the lab. Following acidification, mix the sample and hold for at least 24 hours. Just prior to digestion or direct analysis, verify pH<2. If pH≥2, repeat steps (i.e., add acid, hold for 24 hrs, verify ph<2).
[4] Aqueous samples may be stored at room temperature.

Note: If drinking water samples are properly preserved with nitric acid to a pH<2 and the turbidity is <1 NTU, then acid digestion is not required. The laboratory must maintain documentation of the turbidity reading.

Samples for dissolved metals should be filtered in the field before acid is added to the sample. If the sample is to be filtered in the lab, no preservative is added to the sample until the sample is filtered.

Leachates are transferred to a plastic container after the applicable leachate extraction procedure. The sample is preserved with HNO3 to a pH<2. Leachate samples must be analyzed within 6 months of the extraction.

9.0. Quality Control

Note: If a batch of samples requires digestion, then the relating QC samples must be carried through the entire digestion process.

9.1. **Sample QC** - The following quality control samples are prepared with each batch of samples.

Quality Control	Frequency	Control Limit

Quality Control	Frequency	Control Limit
Preparation blank	One per batch of twenty samples or less	**200.8**: < 2.2 times MDL or <10% sample concentration. **6020**: either <MDL, or <5% of the reg limit, or <5% of the measured concentration in the sample
Lab Control Sample Water (LCSW)	One per batch of twenty samples or less	**200.8**: +/- 15% **6020**: +/- 20%
Matrix Duplicate	One in 20 or fewer samples	20% RPD
Matrix Spike (200.8)[1]	One, if 10 or less samples in batch; two, if 11 or more samples	70-130%
Matrix Spike (SW846)[1]	One per batch of twenty samples or less	75-125%
Matrix Spike Duplicate	When requested by client	**200.8**: 70-130%; **6020**: 75-125% For both **200.8** & **6020**: If MS and MSD are both ≥ 5X CRDL, then 20% RPD. If MS and MSD are less than the CRDL, the RPD is not calculated; otherwise ±CRDL.
Post Digestion Spike	One per batch of twenty samples or less	See Section 9.1.6.4
Laboratory Control Sample Soil (LCSS)	1 in 20 or fewer samples	Vendor's certified limits

[1] The sample for MS is randomly selected, unless specifically requested by a client.

9.1.1. Preparation Blank/Method Blank: One laboratory method/preparation blank will be analyzed with each batch of samples prepared together (not to exceed 20 samples). Preparation blank is used to identify possible contamination during acid digestion. For 200.8: results must be less than 2.2 times the MDL or less than 10% of the determined analyte concentration for a sample. For 6020: results must be less than the highest of: (1) MDL, (2) 5% of the regulatory limit, (3) 5% of the measured concentration of the sample. If any analyte concentration in the blank is above the control limit, the batch must be prepared again for the element in question and the samples reanalyzed.

9.1.2. Laboratory Control Sample Water (LCS): A laboratory control sample must be analyzed with each group of samples digested. Refer to Water Matrix Spike concentration in Attachment 1 for final elemental concentrations. Results must be within the acceptable control limits, if not, all samples prepared in association with the LCS must be redigested and reanalyzed.

- Method 200.8: wastewater samples - prepare the LCS by adding 0.50 ml of ICP-MS LCS/SPK (Sec. 7.2.11) in 100 ml deionized water. Results must be within ± 15% of the true value.
- Method 200.8: drinking water samples - prepare the LCS by adding 0.10 ml of ICP-MS LCS/SPK (Sec. 7.2.11) in 100 ml deionized water. Results must be within ± 15% of the true value.
- Method 6020: wastewater samples - prepare the LCS by adding 0.50 ml of ICP-MS LCS/SPK (Sec. 7.2.11) in 100 ml deionized water. Results must be within ± 20% of the true value.
- LCS for solid matrices (LCSS) is obtained from a vendor supplied solid matrix and is carried through the same preparation procedure as the samples. The results of the solid LCS must fall within the QC Performance acceptance limits' of the reference material used for that sample. If not, all samples prepared in association with the LCS must be redigested and reanalyzed.
- For dissolved aqueous samples not undergoing a preparation procedure, the CCV may serve as the LCS.

9.1.3. Matrix Duplicate: A duplicate is analyzed for each batch of samples digested. The relative percent difference between the results of the sample and the duplicate must fall within 20% RPD for samples greater than five times the reporting limit.

9.1.5. Matrix Spike (MS): A matrix spike is prepared and analyzed for each batch of samples digested.

- For aqueous MS sample, spike 0.50 ml of ICP-MS LCS/SPK (Sec 7.2.11) into 100 ml sample.
- For drinking water MS sample spike 0.10 ml of ICP-MS LCS/SPK into 100 ml sample.
- For soil MS sample spike 1.0 ml of ICP-MS LCS/SPK into 1.0 gram of sample.
- Method 200.8: a recovery of 70-130% is required.
- Method 6020: a recovery of 75-125% is required.
  An exception to this occurs if the sample concentration exceeds the spike concentration by a factor of four or more. If the recovery is not within specified limits a post digestion spike is required to be analyzed at a concentration between 10 to 100 times the instrument detection limit. If the Post digestion spike recovery is not recovered within 75-125% for 6020 and 85-115% for 200.8, a matrix effect should be suspected. See Attachment 1 for final Matrix spike concentration for water and soil.

9.1.6. Post-Digestion Spike: To check for possible matrix interference, analyze a post digestion spike on a representative sample (minimum of 1 per batch). Post-Digestion Spike sample should be evaluated if serial dilution fails.

9.1.6.1. Transfer 10mL of a digestate to a suitable vial.

9.1.6.2. Spike the sample with 0.01ml of ICP-MS LCS/SPK. Analyze the

spiked aliquot and an un-spiked aliquot (the un-spiked may have been analyzed previously and does not need to be reanalyzed). The final concentration of the post digestion spike is as follows:

Element	Post spike concentration (ug/L)
Aluminum (Al)	500
Antimony (Sb)	5
Arsenic (As)	10
Barium (Ba)	10
Beryllium (Be)	5
Boron (B)	20
Cadmium (Cd)	5
Calcium (Ca)	500
Chromium (Cr)	10
Cobalt (Co)	5
Copper (Cu)	10
Iron (Fe)	500
Lead (Pb)	5
Magnesium(Mg)	500
Manganese (Mn)	50
Mercury (Hg)	0.5
Molybdenum (Mo)	10
Nickel (Ni)	10
Potassium (K)	500
Selenium (Se)	10
Silver (Ag)	5
Sodium (Na)	500
Strontium (Sr)	10
Thallium (Tl)	4
Tin (Sn)	10
Titanium (Ti)	10
Vanadium (V)	10
Zinc (Zn)	10

9.1.6.3. Calculate the percent recovery of the post digestion spike as follows:

$$\%REC = \frac{Cps - Cs}{C2} \otimes 100$$

Where: Cps = concentration of post digestion spike (ug/L)
Cs = concentration of un-spiked sample (ug/L)
C2 = concentration of spike (ug/L

9.1.6.4. Limits for post digestion spike recoveries: 85-115% for method 200.8 and 75-125% for method 6020. Results must be within the acceptable limits, if not, repeat analysis and/or re-prepare the sample and reanalyze. If necessary dilute the QC sample

(DU, MS, and PDS) to compensate for the matrix effect and reanalyze.

The post digestion spike must not be applied to samples analyzed at a dilution that produces a significant negative response. The analyst must use good judgment when evaluating data where the sample response is negative. Where a significant negative response is present, the digestate should be diluted and reanalyze to determine the extent of the matrix interferences. If necessary, adjust the interference corrections and reanalyze the samples.

9.1.7. Matrix Spike Duplicate (MSD): A matrix spike duplicate is prepared and analyzed when requested by the client. See Sec 9.1.5 for spiking procedures and acceptance limits.

- If matrix spike sample and matrix spike duplicate are both ≥ CRDL, then 20% RPD. If matrix spike sample and matrix spike duplicate are less than the CRDL, the RPD is not calculated; otherwise, ±CRDL.

9.2. Instrument QC

Table 2 below describes the frequency, criteria, and corrective actions for the calibration and Quality control samples.

Table 2: Calibration, Quality control and Corrective Action Summary

QC Item	Frequency	Criteria	Corrective Action
Tune validation-Mass Calibration	SW6020/200.8-Daily	Within 0.1amu from mass unit	Terminate analysis, fix problem and repeat
Tune validation-Resolution Check	SW6020/200.8-Daily	≤0.9amu full width at 10% peak height	Terminate analysis, fix problem and repeat
Instrument Stability Check	200.8-Daily	<5% RSD for five replicates	Terminate analysis, fix problem and repeat
Initial Calibration: Multi-point-minimum 3 stds and 1 blank	Daily	Correlation ≥0.995	Recalibrate
Initial Calibration Verification Standard (ICV)	At the beginning of the analysis	+/- 10%	Recalibrate
Continuing Calibration Verification Standard (CCV)	At the beginning and end of the analysis, and every 10 samples	Within +/- 10% of the true value	Fix the problem and reanalyze the previous 10 samples.
Calibration Blank (ICB/CCB)	After ICV and every CCV	Absolute value of the calibration blank must be less than the RL.	Terminate the analysis, correct the problem and reanalyze the previous 10 samples
Interference check standards (ICSA/ICSAB)	At the beginning of an analysis and after every 12 hours of analysis	Pay attention to false positives and false negatives for elements not present	If an element is consistently out of the +/- 20% range in the ICSAB solution, the problem

QC Item	Frequency	Criteria	Corrective Action
		in the solutions. Results of the spiked elements should fall within +/- 20%.	should be investigated.
Serial Dilution (1/5 dilution)	One per batch of twenty samples or less	See Section 9.2.6	
Reporting Limit Check Standard (CRI)	At the beginning of analysis run	For *non-drinking water* analysis: Recovery +/- 50% of the true concentration. For *drinking water* analysis: Recovery +/- 15% of the true concentration	Stop the analysis, fix the problem and reanalyze the affected samples
Internal Standards	Analyzed with every standard and sample	6020- 30-120% 200.8- 60-125%	Standard-terminate the analysis and recalibrate Sample- dilute (The analyst should check the IS response for the bracketing CCV/CCBs to see if there is a similar response to the sample. Recalibration may be required.)

9.2.1. Initial Calibration Verification (ICV): Analyze an initial calibration verification solution at the beginning of the run. ICV solution must have the same acid matrix as the calibration standard and it must come from a source other than the calibration standard source. The final concentrations of the various elements are listed in Attachment 1. The results for the target elements in the initial calibration verification (ICV) must be within +/-10% of the true value. If results are outside of the specified limits, terminate the analysis, correct the problem and recalibrate the instrument. See Sec 7.2.4 for the ICV standard preparation instructions.

9.2.2. Continuing Calibration Verification (CCV): The calibration of the ICP-MS must be verified every 10 samples and at the end of the analysis run by analyzing the QC Check Solutions (CCV). The same solution used for the calibration standard 4 (CAL4- drinking water; CAL3 for non-drinking water) is used for the CCV standard. The concentration of the CCV standard must be at or near mid-range levels of the calibration. The final concentrations of the various elements are listed in Attachment 1. The results for the target compounds must be within +/-10% of the true value. If results are outside of the specified limits, terminate the analysis, correct the problem, recalibrate the instrument, and reanalyze the samples following the last good CCV.

9.2.3. Initial and Continuing Calibration Blank (ICB/CCB): ICB and CCB must be analyzed after the calibration curve, every 10 samples and at the end of

the analytical run. The absolute value of the calibration verification blank must not exceed the reporting limit. If it does, terminate the analysis, correct the problem, recalibrate and reanalyze the samples following the last good CCB. Use reagent water for the blank solution.

9.2.4. Reporting Limit Check Standard (CRI): At the beginning of the analysis run verify the accuracy at the RL by analyzing a solution at the RL. RL/PQL Check Solution is analyzed to demonstrate that the ICPMS is capable of detecting the target compounds at the reporting limit (RL). For non-drinking water analysis, the determined concentration must be within +/-50% of the true concentration. For drinking water analysis, the determined concentration must be within +/-15% of the true concentration. See Attachment 1 for list of Rep Lim standard concentration in ug/L. Refer to Sec 7.2.8 for the RL/PQL standard preparation instructions.

9.2.5. Interference Check Sample (ICSA & ICSAB): Verify the inter-element and background corrections by analyzing the interference check solutions (ICSA & ICSAB) at the beginning of the analysis run and after every 12 hours of analysis. The ICS is analyzed in order to demonstrate that proper corrections are being utilized for known interferences. Analyst must evaluate these solutions in order to detect trends that require corrective actions. Pay particular attention to false positives and false negatives for elements not present in the interference check solutions. Results should be within 20% of the true value for each element. See Attachment 2 for list of elements and the corresponding concentrations in ppb.

9.2.6. Serial Dilution QC Check: A 1/5 dilution is prepared and analyzed on one sample per batch (one for every ten samples for Method 200.8, or whichever is greater) to determine if matrix interferences are present.

9.2.6.1. Select a sample digestate that contains one or more target analytes at concentrations greater than 10X the reporting limit.

9.2.6.2. Dilute the digestate by a factor of 5 (DF=5) and analyze the dilution using the same procedures used for the un-diluted aliquot.

9.2.6.3. Compare the results of the diluted and un-diluted aliquots of sample digestate.

9.2.6.4. If the results of the dilution are within ±10% of the results of the undiluted sample, no matrix interference is present. If not, matrix interference should be suspected and the sample digestate should be subjected to a post-digestion spike.

9.2.7. Determination of Linear Range of the ICP-MS

Determine the linear dynamic range of the instrument by analyzing a minimum of three high standards at the upper limit of the instrument. The analytically determined concentration of these standards must be within 10% of the true value. The linear ranges should be redetermined annually or if the instrument is significantly changed.

9.2.7.1. Prepare the standard at concentrations that are expected to define the linear range of the instrument. The calibration standards and the linear range standards should be matrix matched; that is, they have the same percentage of hydrochloric and nitric acids. See Attachment 1 for Linear Range concentrations in ug/L.

9.2.7.2. Analyze the standard(s) after the initial calibration is validated.

9.2.7.3. Compare the concentration of the linear range standard with its true concentration using the following equation:

$$\text{Percent Difference} = \left|\frac{Ccal - Ctrue}{Ctrue}\right| \otimes 100$$

Where:
Ccal = concentration determined from analysis
Ctrue = true concentration of the standard

Note: If the percent difference is less than or equal to 10%, the linear range is confirmed at that concentration. If the percent difference is greater than 10%, repeat the analysis with a lower concentration. For elements validated in this manner you may report data up to 90% of that linear range before a dilution is required.

10.0. Procedure

10.1. Sample Preparation

10.1.1. For non-potable samples to be analyzed by Method 200.8, follow TestAmerica SOP No. ED-MTP-001.

10.1.2. For Drinking water samples, if turbidity is ≥1, follow the digestion procedure for Total recoverable in TestAmerica SOP No. ED-MTP-001.

10.1.3. For samples to be analyzed by Method 6020, follow digestion procedure in TestAmerica SOP No. ED-MTP-005 (soil) or ED-MTP-003 (water).

10.2. Calibration

10.2.1. Turn the ICP-MS on and initiate the tune screen. Start the tune screen to allow the instrument to become thermally stable before beginning to analyze the calibration standards. While the instrument is warming up, aspirate the interference check solution (or similar solution) to precondition the cones.

10.2.2. Check the tune parameters for the proper sensitivities, precision, oxides, and double charged values following the instrument manufacturer's recommendations. For ICPMS1, refer to section 4 (Tuning) of the

Operator's manual (*Agilent 7500 ICP-MS ChemStation (G1834B) Operator's Manual).* For ICPMS2, refer to section 1 (Tuning) of the Tuning & Application Handbook (*Agilent 7500 Series ICP-MS, p/n G3270-90132).*

10.2.3. Prior to calibration, run an EPA Tune check solution. Refer to Sec. 7.2.7 for the Tune check criteria.

10.2.4. The instrument is required to be calibrated daily or once every 24 hours. Using method 2008.M or 2008_DW.M (programs in the ChemStation and Mass Hunter Workstation software containing calibration standards, check tables, etc.), calibrate the instrument using a blank and at least four standards. Method 2008_DW.M is used for drinking waters only. For ICPMS1, refer to section 5 (Creating a Method) of the instrument manufacturer's instructions (*Agilent 7500 ICP-MS ChemStation (G1834B) Operator's Manual*) for calibration instructions. For ICPMS2, refer to section 4 (Creating a Method) of the instrument manufacturer's instructions (*Agilent 7500 series ICP-MS, MassHunter Workstation (G7200A) Operator's Manual*).

10.2.5. Internal standards are added to the calibration standards, blank and samples in-stream by the use of T-fitting.

10.2.6. Analyze the calibration standards and calibrate the ICP-MS in accordance with the manufacturer's recommendations and the TestAmerica Corporate SOP No. CA-Q-S-005 Calibration Curves (General).

10.3. Sample Analysis

10.3.1. All sample measurements must be made within within 90% of the linear range of the instrument. Preparation of all reference materials used for calibration must be documented.

10.3.2. Dilute samples prior to analysis. Dilutions must be prepared in reagent water (Sec 7.1.6).

10.3.2.1. Non-potable samples and relating QC samples are diluted 5x prior to analysis.

10.3.2.2. Soil samples and relating QC samples are diluted 20x prior to analysis.

10.3.3. The samples are analyzed only after the ICB/CCB and ICV/CCV criteria are met.

10.3.4. The samples are analyzed in a sequence as follows:

Typical Analytical Sequence
INSTRUMENT WARM-UP (approx. 30 minutes)
TUNE CHECK
INITIAL CALIBRATION (STANDARDIZATION/CALIBRATION OF THE ICPMS)

Typical Analytical Sequence
INITIAL CALIBRATION VERIFICATION (ICV)
INITIAL CALIBRATION BLANK (ICB)
REPORTING LIMIT STANDARD (CRI)
ICPMS INTERFERENCE CHECK SOLUTION A (ICSA)
ICPMS INTERFERENCE CHECK SOLUTION AB (ICSAB)
CONTINUING CALIBRATION VERIFICATION (CCV)
CONTINUING CALIBRATION BLANK (CCB)
10 SAMPLES
CONTINUING CALIBRATION VERIFICATION (CCV)
CONTINUING CALIBRATION BLANK (CCB)
10 SAMPLES
CCV
CCB
10 SAMPLES
CCV
CCB
10 SAMPLES
CCV
CCB
~after 12 hrs of analysis~
ICPMS INTERFERENCE CHECK SOLUTION A (ICSA)
ICPMS INTERFERENCE CHECK SOLUTION AB (ICSAB)
CONTINUING CALIBRATION VERIFICATION (CCV)
CONTINUING CALIBRATION BLANK (CCB)

Note: The analytical sequence must end with the analysis of the CCV and CCB. The 10 samples include all QC samples/standards with the exception of CCVs and CCBs.

10.3.5. Determine the concentration of the samples and QC items using the procedures of Section 11.

10.3.5.1. If the sample concentration is greater than 90% of the linear range, the sample digestate must be diluted and reanalyzed.

10.3.5.2. The amount of sample digestate needed to prepare the desired dilution is determined from the following equation:

$$\text{V digest} = \frac{Vf\ vol}{DF}$$

Where:
Vf vol = final volume of diluted sample (mL)
V digest = volume of sample digestate used to make the dilution (mL)
DF = Dilution Factor

10.3.5.3. The dilution factor is calculated as follows:

$$DF = \frac{Vf\ vol}{V\ digest}$$

Where: Vf vol = final volume of diluted sample extract (mL)
V digest = volume of sample extract used to make the dilution (mL)

Note: The following examples are based on a final volume of 100mL. It may be more convenient to prepare dilutions at smaller final volumes.

EXAMPLE

A sample digestate is analyzed and one of the target analytes exceeds the linear range of the ICP-MS. 1.0mL of the digestate is added to a 100mL volumetric flask and the extract brought up to volume with reagent water. What is the dilution factor?

$$DF = \frac{100ml}{1.0ml} = 100$$

Some samples may require multiple dilutions; that is, a dilution of a dilution will have to be made. In this case, the final dilution factor is the product of the individual dilutions.

11.0. Calculations / Data Reduction

11.1. Accuracy:

$$\text{ICV / CCV, LCS \% Recovery} = \frac{\text{observed concentration}}{\text{known concentration}} \times 100$$

$$\text{MS \% Recovery} = \frac{\text{(spiked sample) - (unspiked sample)}}{\text{spiked concentration}} \times 100$$

11.2. Precision (RPD):

$$\text{Matrix Duplicate (MD)} = \frac{|\text{orig. sample value - dup. sample value}|}{[(\text{orig. sample value + dup. sample value})/2]} \times 100$$

11.3. Aqueous and Leachate Samples:

Leachate samples are routinely reported in mg/L while the ICP-MS is routinely calibrated in ug/L. If the results are reported in ug/L, the conversion factor is omitted from the calculation.

11.3.1. The concentration of the target analyte in liquid samples is calculated as follows:

Concentration (mg/L) = $ug/L(\,from\ pr\,\mathrm{int}\,out)\otimes\frac{F}{V}\otimes DF\otimes\frac{1mg}{1000ug}$

Where: F = final volume of the sample digestate (L) -usually 50mL
V= volume of sample digested (L)
DF = dilution factor

11.3.2. The Reporting Limit (RL) of the target analyte in liquid samples is calculated as follows:

Concentration(mg/L) = $RL_{table1}\otimes\frac{F}{V}\otimes DF\otimes\frac{1mg}{1000ug}$

Where: $RL_{table\ 1}$ = reporting limit from this SOP (ug/L)
F = final volume of the sample digestate (L)
V= volume of sample digested (L)
DF = dilution factor

The Table 1 Reporting Limits assumes:
F = 50ml
V = 50ml
DF = 1

11.4. Soil/Solid Samples

Soils and solids are routinely reported in mg/kg while the ICP-MS is routinely calibrated in ug/L. If the results are reported in ug/kg, the conversion factor is omitted from the calculation.

11.4.1. The concentration of the target analyte in soil and solid samples is calculated as follows:

Concentration (mg/kg,dw) = $ug/L(\,from\ pr\,\mathrm{int}\,out)\otimes\frac{F}{W\otimes solids}\otimes DF\otimes\frac{1mg}{1000ug}$

Where: F = final volume of the sample digestate (L)
W = volume of sample digested (kg)
DF = dilution factor
solids = decimal equivalent of the percent solids (percent solids/100)

For example, if the percent solid is 85%, the decimal equivalent is 0.85; if the %solid is 100%, the decimal equivalent is 1.0.

11.4.2. The Reporting Limit (RL) of the target analyte in soil/solid samples is calculated as follows:

Concentration(mg/kg,dw) = $RL_{table1}\otimes\frac{0.0010kg}{W\otimes solids}\otimes\frac{F}{0.100L}\otimes DF$

Where:
$RL_{table\ 1}$= reporting limit from Table 1 of this SOP
W = weight of sample digested (kg)
F = final volume of the sample digestate (L)
V = volume of sample digested (L)
DF = dilution factor
solids = decimal equivalent of the percent solids (percent solids/100)
dw = dry weight

The Table 1 Reporting Limit assumes:
F = 0.100L
DF = 1
W = 0.0010kg (1.0g)
solids = 1.0

NOTE: All dry weight corrections are made in LIMS at the time the final report is prepared.

11.5. Data Reduction

11.5.1. All data is recorded directly into the TALS Analyst Desktop II program.

11.5.2. Standard and standard preparations must be documented in the Analyst Desktop II program located in TestAmerica Laboratory System (TALS). The analyst must enter the following information: Source standard, Initial and final sample volume, spike name and amount used, all reagents and their corresponding lot numbers.

11.5.3. Generate Raw Data PDF file for TALS

11.5.3.1. Using a scanner, scan all raw data, i.e., instrument printouts. This includes, the calibration, calibration curve info, and all samples analyzed (includes instrument and batch QC). Attach the pdf to the Docs section of the analytical batch in TALS (ADII).

11.5.4. Metals Data Review checklist (EDS-WI-146) must be filled out prior to data submission.

12.0. Method Performance

12.1. Method Detection Limit Study (MDL)

The method detection limit (MDL) is the lowest concentration that can be detected for a given analytical method and sample matrix with 99% confidence that the analyte is present. The MDL is determined according to the laboratory's MDL procedure in Section 19 (Test Methods and Method Validation) of TestAmerica Edison's Quality Assurance Manual (ED-QA-LQM). MDLs reflect a calculated (statistical) value

determined under ideal laboratory conditions in a clean matrix, and may not be achievable in all environmental matrices. The laboratory maintains MDL studies for analyses performed; these are verified at least annually unless method requirements require a greater frequency.

The IDL for EPA Method 6020 is defined as 3.14 times the standard deviation of seven replicate analyses of a reagent blank solution and should be determined every three months. The IDL for EPA Method 200.8 is defined as the concentration equivalent to the analyte signal which is equal to 3 times the standard deviation of a series of ten replicate measurements of the calibration blank signal at the selected mass(es) and should be determined annually.

12.2. Demonstration of Capabilities

For DOC procedure refer to Section 19 in the most current revision of TestAmerica Edison's Quality Assurance Manual (ED-QA-LQM).

12.3. Training Requirements

Refer to TestAmerica SOP No. ED-GEN-022, Training, for the laboratory's training program.

13.0. Pollution Control

It is TestAmerica's policy to evaluate each method and look for opportunities to minimize waste generated (i.e., examine recycling options, ordering chemicals based on quantity needed, preparation of reagents based on anticipated usage and reagent stability). Employees must abide by the policies in Section 13 of the Corporate Environmental Health and Safety Manual (CW-E-M-001) for "Waste Management and Pollution Prevention."

14.0. Waste Management

14.1. Waste management practices are conducted consistent with all applicable rules and regulations. Excess reagents, samples and method process wastes are disposed of in an accepted manner. Waste description rules and land disposal restrictions are followed. Waste disposal procedures are incorporated by reference to TestAmerica SOP No. ED-SPM-008 (Laboratory Waste Disposal Procedures).

14.2. The following waste streams are produced when this method is carried out.

- Digested Samples: Corrosive Acid- Materials that are not above regulatory limits will be submitted for elementary neutralization with 50% sodium hydroxide solution (Siedler Chemical SC-1824-03). Major concern is heat generated from the neutralization process. Initial volume of acid waste to be neutralized should be no more than 15 gallons. Finished neutralization with sodium bicarbonate (Siedler Chemical SC-0219-25) to a pH of 6 – 9 in the primary tank. Once pH has been established the primary tank is transferred through filter housing to a secondary tank. The pH is rechecked. If the pH is within specifications, the secondary tank is released to the municipal sewer system.

- Samples above regulatory limits and expired RCRA metals standards (Waste Corrosive Liquid, Acidic, Inorganic, n.o.s.) are collected in satellite accumulation and sent off site through a Waste disposal vendor.
  Onyx Profile WIP Number: 590598
  Teris Profile Number 50016653

- Expired Standards – The vials are collected in a 1 gallon polyethylene bucket. These vials are then transferred to an open top 55 gallon steel or polyethylene waste drum. These drums are transported to a waste facility for proper disposal.

- Soil Retain Samples - These samples if not flagged in the system for any hazardous constituents are transferred to poly-lined cubic yard boxes. These boxes when full are sent to stabilization or incineration. These materials are sent out as hazardous for lead and chromium

  Teris Profile Number (incineration): 50016710
  Onyx Profile Number: (stabilization) 402535

15.0. References / Cross-References

15.1. Methods for the Determination of Metals in Environmental Samples; US EPA Office of Research and Development. Washington, DC, May 1994. Method 200.8, Revision 5.4.

15.2. Test Methods for Evaluating Solid Waste, Third Edition; U.S. EPA Office of Solid Waste and Emergency Response: Washington, D.C., November 1986. Method 6020, Revision 0, September 1994.

15.3. Agilent 7500 ICP-MS Chemstation (G1834B) Operator's Manual, Agilent Technologies, Inc. 2005.

15.4. TestAmerica Edison SOP ED-MTP-005, *Hot Block Digestion of Sediments and Sludges and Soils Using SW846 Method 3050B,* most current revision.

15.5. TestAmerica Edison SOP No. ED-MTP-003, *Digestion of Water and Wastewater Samples for Analysis by ICP and ICP-MS, SW846 Method No. 3010A,* most current revision.

15.6. TestAmerica Corporate SOP No. CA-Q-S-005, *Calibration Curves (General)*, most current revision.

15.7. TestAmerica Edison Document No. ED-QA-LQM, *Laboratory Quality Manual*, most current revision.

15.8. TestAmerica Edison SOP ED-GEN-022, *Training*, most current revision.

15.9. TestAmerica Corporate Environmental Health and Safety Manual, Document No. CW-E-M-001, most current revision.

15.10. TestAmerica Edison SOP ED-MTP-001, *Digestion of Aqueous Samples for Analysis by ICP and ICPMS USEPA Method No(s). 200.2, 200.7 (Rev 4.4), 200.8 & CLP SOW ISM01.2*, most current revision.

15.11. Agilent 7500 Series ICP-MS, Tuning & Application Handbook, Rev. A, May 2008, p/n G3270-90132.

15.12. Agilent 7500 Series ICP-MS, MassHunter Workstation (G7200A) Operator's Manual, Rev. A, May 2008.

15.13. ICPMS Data Review Checklist, CA-Q-WI-044, most current revision.

16.0. Method Modifications:

None

17.0. Attachments

Attachment 1: Standards and Matrix Spiking concentrations – Drinking water and non Drinking water.
Attachment 2: Interference Check standard elemental concentrations - Drinking water and non Drinking water.

18.0. Revision History

- **Revision 11, dated 26 April 2018**
  - Sec 7.2.7.4: Added Magnesium as one of the working tune check solution's constituents.
  - Sec 9.1.2: Revised to clarify the acceptance criteria used in the COA when evaluating recoveries for LCS in soil matrix.

- **Revision 10, dated 15 November 2016**

  - Sec 1.1, Table 1: Changed the Drinking water RL from 2.5 ug/L to 2 ug/L. Removed reference to Method 3005.
  - Sec 2.0: Removed reference to method 3005 and replaced SOP ED-MTP-007 with SOP ED-MTP-001.
  - Sec 7.1.6: Reagent recording in ICPMS Reagent Dilution logbook is replaced with TALS Reagent Module.
  - Sec 7.2.1: Replaced stock standard TA-72 with standard TA-72REV2.
  - Sec 7.2.2: Replaced standard TA-72 with TA-72REV2 for CAL1 standard-Drinking Water.
  - Sec 7.2.7.2: Tune check criteria: updated the resolution check criteria to reflect laboratory practices.
  - Sec 7.2.10: Added the preparation instructions for ICSA & ICSAB standards used when analyzing drinking water samples.
  - Sec 9.2, Table 2: Updated Tune validation-Resolution Check criteria to reflect laboraotry practices

- Sec 10.1: Replaced SOP ED-MTP-007 reference with SOP ED-MTP-001.
- Sec 11.5.4: Replaced checklist CA-Q-WI-004 with Work Instruction EDS-WI-146.
- Attachment 1, Drinking water: Replaced the Calibration concentration and RL Std.Conc. of Pb from 2.5 to 2 ug/L.
- Attachment 2: Added ICSA & ICSAB elemental concentrations for Drinking water analysis.

- **Revision 9, dated 02 April 2015**

  - Throughout the SOP: Removed all reference to SOP# ED-MTP-002.
  - Sec 7.2.1: Added calibration standards TA-88 and TA-72.
  - Sec 7.2.2: Added the new low-level cal std (CAL2) for non-potable water and updated the preparation instructions for the potable cal standards.
  - Sec 7.2.7.1.3: Added Triton X-100 to the list of internal standard stock solutions.
  - Sec 7.2.7.2: Updated the preparation instruction of the working internal standard solution to include Triton X-100; Revised note to state that Selenium is now analyzed under the Hydrogen reaction mode.
  - Sec 7.2.8: CAL1 is now used as the RL check standard for drinking water analysis.
  - Sec 9.1.6.4: Added PDS recovery limits (85-115%) for method 200.8. Clarified the procedure when PDS is outside the acceptable limits.
  - Sec 9.1 and 9.1.7: Added Matrix Spike Duplicate (MSD) to the list of Sample QC.
  - Sec 11.5.4: Updated the metals data review checklist form number.
  - Sec 15.13: Added TestAmerica Corp form No. CA-Q-WI-044 to the list of references.
  - Attachment 1 (non-Drinking Waters): Updated the calibration concentrations for all metals; updated the matrix spike concentration for Zinc (water & soil).
  - Attachment 1 (Drinking Waters): Updated the calibration concentrations for As, B,Sn and Zn.

- **Revision 8, dated 05 December 2012**

  - Sec 1 and 12: Updated LQM section references to reflect the most current LQM revision.
  - Sec 2.0: Deleted the exception that silver analysis does not undergo digestion. Silver may be directly analyzed for drinking waters provided the turbidity <1.
  - Sec 7.1.6: Revised to include the reagent water prep for potable samples not requiring digestion. Revised preparation frequency to 12 months to reflect actual laboratory practices.
  - Sec 7.2.2 & 7.2.3: Sections deleted to remove Hg stock standard info and the preparation instructions of Hg intermediate standard; subsequent sections adjusted accordingly.
  - Sec 7.2.2 (formerly Sec 7.2.4): Removed all references to Hg. Clarified M1CAL3 as the reporting limit for Drinking Water standard. Clarified M1CAL2 is not the reporting limit for Boron.
  - Sec 7.2.3 and 7.2.4 (formerly Sec 7.2.5 & 7.2.6): Hg references have been removed.
  - Sec 7.2.6: Section added; subsequent sections adjusted accordingly.
  - Sec 7.2.7 (formerly Sec 7.2.8): Added ‘EPA Tune Check’ to the section header. Removed the use of gold standard from this SOP. Hg is not analyzed by this method.
  - Sec 7.2.7.1. Added internal standard stock solutions.
  - Sec 7.2.7.2: Added internal standard preparation instructions. Also clarified that drinking water samples are analyzed without the use of collision cell technology.
  - Sec 7.2.7.3: Added Tune Check stock standard

- Sec 7.2.7.4: Added preparation instructions of the EPA Tune Check standard.
- Sec 7.2.11 (formerly Sec 7.2.12): Changed standard vendor for the Matrix spike stock solution to High-Purity Standards.
- Sec 8: Revised the wait time for sample preparation after in-lab preservation to 24 hours to comply with the updated 40CFR Part 136.
- Sec 9.1: Preparation Blank's control limits have been updated to reflect method requirements.
- Sec 9 and 10: Revised section references of standard solutions where applicable.
- Sec 10.1.2: Revised SOP number reference to reflect actual laboratory practices.
- Sec 10.2.2: Included Tuning Handbook reference for ICPMS2.
- Sec 10.2.4: Included Operation Manual for Masshunter software to ICPMS2.
- Sec 11.5: Added instructions for data recording, raw data PDF creations and Metals Data Review Checklist.
- Sec 15.4: Deleted SOP No. ED-MTP-007 reference, not applicable; subsequent sections adjusted accordingly.
- Sec 15.11 through 15.13: Added applicable references.
- Sec 17.0: Attachments 3 and 4 have been removed.
- Attachment 1: Deleted 'Linear range conc.' column; new linear range studies are updated on instruments. Updated MS concentration for Boron.

- **Revision 7, dated 16 July 2010**

  - Sec 1.1 Table 1: Revised Reporting Limits for drinking water and Soil.
  - Sec 3: Revised definitions reference to Appendix 2 of ED-QA-LQM.
  - Sec 7.2.1: Added STLNJ-STD-1 and STLNJ-STD-2 to the list of Calibration Stds
  - Sec 7.2.4: Revised working calibration standard including preparation procedure to reflect actual lab practices.
  - Sec 7.2.9: Revised prep procedure for RL check standard. Also deleted RL/PQL working standard, previously section 7.2.10. Subsequent sections adjusted accordingly.
  - Sec 18.0 Attachment 1: Table updated to reflect correct calibration standard concentrations and drinking water reporting limits.

- **Revision 6, dated 11 September 2009**

  - Sec 9.2 Table 2 & Sec. 9.2.4: Revised Reporting limit criteria for drinking water analysis to ± 15%.

- **Revision 5, dated 08 July 2009**

  - Sec 4.1: Expanded section to include the isobaric polyatomic/molecular ion interferences; clarified using collision/reaction cell for non-potable samples only.
  - Sec 4.2: Added equations used for drinking water analysis when collision/reaction cell can not be used.
  - Sec 4.3: Added Krypton interference which affects arsenic and selenium, and the use of high purity Krypton free argon.
  - Sec 6.1 Instrumentation: Add collision/reaction cell.
  - Sec 7.2.4: Added a low calibration standard (M1CAL1) increasing the number of calibration standards to five; five cal standards will only be analyzed for elements Be, Fe, Pb and Mn.
  - Sec 7.2.8: Added Kr83 in the list of elements in order to monitor Kr levels.

- Added text in Tune check criteria, Sec 7.2.8: *Resolution is checked by analyzing Magnesium isotopes 24, 25, 26 and Lead isotopes 206, 207, 208.*
- Sec 8: Clarified holding time and procedure for acidifying unpreserved samples.
- Sec 9: Added text: *If a batch of samples requires digestion, then the relating QC samples must be carried through the entire digestion process.*
- Sec 9.1 & 9.1.2: Changed LCS control limit for Method 6020 from ± 25% to ± 20%.
- Sec 9.2.2: CCV standard- changed solution to M1CAL4.
- Sec 10.2.4: Calibration- changed the number of standards from three to four standards.
- Sec 10.3.2: Added in the procedure the requirement for diluting samples 5x (water) and 20x (soil) prior to analysis; subsequent section numbers were adjusted accordingly.
- Sec 10.3.5.1: revised to include that samples will be diluted if the concentration is >90% of the linear range.
- Attachment 1: Revised calibration standard concentration specifically Cal std 2 to reflect the correct concentration. Additionally, a fifth cal standard was added to the four elements (Be, Fe, Pb, and Mn) so the RL will be at or above the low level standard; Reporting limits were also revised for these elements.
- Attachment 3: Revised dilution requirement for soil analysis from 10x to 20x.

- **Revision 4, dated 24 March 2009**
  - Revised SOP format in accordance with TestAmerica Corporate Quality SOP No. CW-QS-002 (Rev 0) Writing a Standard Operating Procedure (SOP).
  - Section 1.1: Relocated Table 1 to this section. Updated 6020 Soil RLs. Revised several RLs for Drinking Water and Digested Water
  - Sec. 2: Added the method and SOP references for digestion of samples to be analyzed for metals via ICPMS.
  - Sec 6.1: Added Heat exchanger and autosampler to the list of instruments.
  - Sec 7.1.6: Added reagent water and its prep instructions to the list of reagents.
  - Sec 7.2 Standards: Included vendor and storage information of standards.
  - Sec 7.2.2: Revised the vendor source and stock concentration of the Hg standard from 1000 ppm to 10 ppm; revised preparation instructions of the Intermediate standard (Hg-Cal-Int).
  - Sec 7.2.4: Revised prep instructions for the working calibration standards (i.e. amount of spiking solution and final volume).
  - Sec 7.2.5: Deleted ICV stock standards STLNJ-Cal 6 & 7 and added ICV stock standards ICVICPMS and ICV2ICPMS.
  - Hg Secondary stock solution in Sec 7.2.5: Revised the vendor source and stock concentration of the Hg secondary standard from 1000 ppm to 10 ppm; revised preparation instructions of the Intermediate standard (Hg-ICV-Int).
  - Sec. 7.2.8: added statement: “Drinking Water Analysis: All tune steps = 3 (thereby eliminating the use of the Collision/Reaction Cell.) Use masses Fe57, Cu65, and Se82 instead of their respective masses listed above.”
  - Sec 7.2.9: Deleted ICPMS RL stock standard and added custom standards (STLNJ-STD-1 & 2) for Reporting Limit Check standard.
  - Sec 7.2.10: Revised prep instructions for the RL/PQL Working standard.
  - Sec 7.2.11: Revised prep instructions for the Working Interference check standards (final volume reduced from 100ml to 50ml; spiking amount adjusted).
  - Sec 8: Sample collection, preservation and storage: Reformat section into Table format and included minimum sample size and reference information.
  - Sec. 9.1: Added prep instruction to the QC samples (MB, LCS, BS and MS).

- Deleted Single point method of standard addition; previously identified Sec.9.2.8.
- Sec 10.1: Clarified the digestion procedure utilized for digesting different matrices (wastewater, drinking water and soil).
- Sec 15: Added applicable references.
- Sec 17: Added Attachment 2 (Interference Check Standard elemental concentrations).

- **Revision 3, dated March 2007**
  - Section 16. Attachment 1. Modified the 'Linear Range Concentration' of 11 elements (Sb, Be, B, Cd, Co, Pb, Se, Ag, Sr, Tl, V) to reflect the most recent LDR study.

- **Revision 2**, **dated February 2007**
  - Revised various sections of the SOP to include the specific sections in the Operator's manual where applicable information can be found. These sections are Sec. 5.3, 10.1.2., 10.1.3, Appendix A – Analytical Sequence Table.
  - Section 6.3 Calibration Standards. Modified the frequency for the preparation of calibration standards from six months to two weeks as per Method 200.8.
  - Section 6.4. Linearity Check Standards. Revised the HNO3 acid concentration used in the preparation of linearity check solutions to '2% by volume' to reflect actual laboratory practices.
  - Section 6.5 (Internal Standards). Revised the concentration of the element Au, both spiking and resulting concentrations.

    '*A solution of 1 mg/L (internal standard elements) and 2 mg/L of Au is prepared and added in-stream by the use of a T-fitting. The resulting concentration (in-stream) of the standard is ~100 ug/L (Au) and ~50 ug/L (internal standard elements).'*

  - Section 7.4. Section deleted; contents not applicable to this SOP.
  - Section 8.1.1 (MDL and IDL determination). Modified the solution used in the analysis of IDL to 'reagent blank solution' to reflect actual laboratory practices.
  - Section 8.1.2 (MDL and IDL determination). Added an SOP reference to further describe MDL determination.
  - Section 10.3.2. The name of the Reporting limit solution in the analysis sequence was changed from 'Detection Limit Check Solution' to 'Reporting Limit Standard (RL).'
  - Section 15.1, deleted; reference not applicable. Subsequent section numbers adjusted accordingly.
  - Section 15.1 (previously identified as section 15.2), was revised to include the specific method in the reference information, in this case method 6020.
  - Section 15.2 (previously identified as section 15.3), was revised to include the specific method in the reference information, in this case method 200.8.
  - Section 15.6. Added Operator's instructions manual in the list of references. The section previously identified as 15.6 is now 15.5.
  - Section 16. Attachment 1. Modified the 'Linear Range Conc. (ug/L)' to reflect the most recent LDR study. Updated the 'RL Std. conc.' for Boron, Iron, Tin and Zinc to reflect actual laboratory's reporting limits for these elements.
  - Appendix A (Sample Preparation). Specified the name of the SOP used in the digestion of samples.
  - Appendix A (Calibration, Quality Control and Corrective action summary). Revised the criteria for the ICSA/ICSAB to clarify that ICSA when used as linear range

sample the determined analyte concentrations >90% must be diluted and reanalyzed.
- Appendix B (ICV Solution). Added the frequency for the preparation of the ICV solution.

- **Revision 1, dated January 2007**

  - Specified Operator's manual '*Agilent 7500 ICP-MS Chemstation (G1834B)*' in various sections: Sections 5.3, 10.1.2, 10.1.3, Appendix A – Analytical Sequence section.
  - All references to '6020A' are updated to '6020' to reflect actual method reference (i.e. sections 1.1, 2.2, 8.1.2 etc.).
  - Section 10.1.3. Filename '2008.M' is specified as the program/filename used in the ChemStation software.
  - Section 16 Table 1. Updated Reporting limits of 8 elements (As, Cd, Co, Cu, Ni, Ag, Sr, Sn) to reflect current standard concentrations and laboratory's reporting limits.
  - Section 16 Attachment 1. Revised Matrix spike concentrations for water and soil to include the actual final expected concentrations of the matrix spike.
  - Appendix B – Calibration, Quality Control and Corrective action summary. Criteria for internal standards was changed from >30% to 30-120% to reflect actual method criteria limits.
  - Appendix B – ICP Interference Check Solutions for SW846 6020. Added the frequency of preparation for the Working Interference Standards ICSA and ICSAB, 'these standards are made fresh weekly.'
  - Appendix B. Added data qualifiers for ICP-MS
  - Section 15.4. Added USEPA CLP ILM05.3 to the list of references.

Attachment 1

STANDARDS AND MATRIX SPIKING CONCENTRATIONS
For Non-Drinking Waters

Element	Mass	Calibration Conc. (ug/L) (stds: 5,4,3,2,1)	ICV/CCV Conc. (ug/L)	RL Std. Conc. (ug/L)	Matrix Spike Conc. *see note	
					Water (ug/L)	Soil (mg/kg)
Aluminum (Al)	27	1000,500,100,20,10	400/500	10	2500	500
Antimony (Sb)	121	100,50,10,1, 0.5	40/50	0.5	25	5
Arsenic (As)	75	100,50,10,1, 0.5	40/50	0.5	50	10
Barium (Ba)	137	100,50,10,2,1	40/50	1	50	10
Beryllium (Be)	9	100,50,10, 0.4, 0.2	40/50	0.2	25	5
Boron (B)	11	200,100, 20, 40, 20	80/100	20	500	100
Cadmium (Cd)	111	100,50,10,1, 0.5	40/50	0.5	25	5
Calcium (Ca)	44	10000,5000,1000, 100, 50	4000/5000	50	2500	500
Chromium (Cr)	52	100,50,10,2,1	40/50	1	50	10
Cobalt (Co)	59	100,50,10,2,1	40/50	1	25	5
Copper (Cu)	63	100,50,10,2,1	40/50	1	50	10
Iron (Fe)	56	10000,5000,1000, 60, 30	4000/5000	30	2500	500
Lead (Pb)	208	100,50,10, 0.6, 0.3	40/50	0.3	25	5
Magnesium(Mg)	24	10000,5000,1000, 100, 50	4000/5000	50	2500	500
Manganese (Mn)	55	1000,500,100, 4, 2	400/500	2	250	50
Molybdenum (Mo)	95	100,50,10,2,1	40/50	1	50	10
Nickel (Ni)	60	100,50,10,2,1	40/50	1	50	10
Potassium (K)	39	10000,5000,1000, 100, 50	4000/5000	50	2500	500
Selenium (Se)	78	100,50,10, 5, 0.5	40/50	0.5	50	10
Silver (Ag)	107	100,50,10,1,1	40/50	1	25	5
Sodium (Na)	23	10000,5000,1000, 100, 50	4000/5000	50	2500	500
Strontium (Sr)	88	100,50,10,2,1	40/50	1	50	10
Thallium (Tl)	205	20,10,2, 0.4, 0.2	8/10	0.2	20	4
Tin (Sn)	118	100,50,10,8,4	40/50	4	50	10

Titanium (Ti)	47	100,50,10,2,1	40/50	1	50	10
Vanadium (V)	51	100,50,10,2,1	40/50	1	50	10
Zinc (Zn)	66	100,50,10,8,4	40/50	4	250	50

**Note: All non-potable samples and relating QC samples (i.e., MB, LCS, Dup, MS,MSD, Serial Diln, Post Spk.) are diluted (5X) prior to analysis. All soil samples and relating QC samples (i.e., MB, LCS, Dup, MS, MSD, Serial Diln, Post Spk.) are diluted (20X) prior to analysis.*

STANDARDS AND MATRIX SPIKING CONCENTRATIONS
For Drinking Waters

Element	Mass	Calibration Conc. (ug/L) (stds: 4,3,2,1)	ICV/CCV Conc. (ug/L)	RL Std. Conc. (ug/L)	LCS/MS Conc. (ug/L)
Aluminum (Al)	27	1000,500,100,25	400/500	25	500
Antimony (Sb)	121	100,50,10, 2.5	40/50	2.5	5
Arsenic (As)	75	100,50,10, 1	40/50	1	10
Barium (Ba)	137	100,50,10, 2.5	40/50	2.5	10
Beryllium (Be)	9	100,50,10, 2.5	40/50	2.5	5
Boron (B)	11	200,100,20,20	80/100	20	100
Cadmium (Cd)	111	100,50,10, 2.5	40/50	2.5	5
Calcium (Ca)	44	10000,5000,1000, 250	4000/5000	250	500
Chromium (Cr)	52	100,50,10, 2.5	40/50	2.5	10
Cobalt (Co)	59	100,50,10, 2.5	40/50	2.5	5
Copper (Cu)	65	100,50,10, 2.5	40/50	2.5	10
Iron (Fe)	57	10000,5000,1000, 250	4000/5000	250	500
Lead (Pb)	208	100,50,10, 2	40/50	2	5
Magnesium(Mg)	24	10000,5000,1000, 250	4000/5000	250	500
Manganese (Mn)	55	1000,500,100, 25	400/500	25	50
Molybdenum (Mo)	95	100,50,10, 2.5	40/50	2.5	10
Nickel (Ni)	60	100,50,10, 2.5	40/50	2.5	10
Potassium (K)	39	10000,5000,1000, 250	4000/5000	250	500
Selenium (Se)	82	100,50,10, 2.5	40/50	2.5	10
Silver (Ag)	107	100,50,10, 2.5	40/50	2.5	5
Sodium (Na)	23	10000,5000,1000, 250	4000/5000	250	500
Strontium (Sr)	88	100,50,10, 2.5	40/50	2.5	10
Thallium (Tl)	205	20,10,2, 0.5	8/10	0.5	4
Tin (Sn)	118	100,50,10, 4	40/50	4	10
Titanium (Ti)	47	100,50,10, 2.5	40/50	2.5	10

Vanadium (V)	51	100,50,10, 2.5	40/50	2.5	10
Zinc (Zn)	66	100,50,10, 4	40/50	4	50

Attachment 2

Interference Check Standard (ICSA & ICSAB) Elemental Concentrations				
ELEMENT	**Non-Potable**		**Potable**	
	ICSA (ug/L)	**ICSAB (ug/L)**	**ICSA (ug/L)**	**ICSAB (ug/L)**
Sodium	125000	125000	62500	62500
Magnesium	50000	50000	25000	25000
Aluminum	50000	50000	25000	25000
Potassium	50000	50000	25000	25000
Calcium	150000	150000	75000	75000
Titanium	1000	1000	500	500
Iron	125000	125000	62500	62500
Molybdenum	1000	1000	500	500
Vanadium		200		100
Chromium		200		100
Manganese		200		100
Cobalt		200		100
Nickel		200		100
Copper		200		100
Zinc		100		50
Arsenic		100		50
Selenium		100		50
Molybdenum		1000		500
Silver		200		100
Cadmium		100		50

Title: Digestion of Water and Wastewater Samples for Analysis by ICP and ICP-MS, SW846 Method 3010A

Once printed, this is considered an uncontrolled document

Approvals (Signature/Date):

Laura Demone — 11/04/15
Laura Demone — Date
Department Manager

David W. Helfrich — 08/03/2016
Dan Helfrich — Date
Health & Safety Manager/Coordinator

Carl Armbruster — 11/04/15
Carl Armbruster — Date
Quality Assurance Manager

11/04/15
Mark Acierno — Date
Laboratory Director

Donald Evans — 11/04/15
Donald Evans — Date
Operations Manager

Copyright Information:
This documentation has been prepared by TestAmerica Laboratories, Inc. and its affiliates ("TestAmerica"), solely for their own use and the use of their customers in evaluating their qualifications and capabilities in connection with a particular project. The user of this document agrees by its acceptance to return it to TestAmerica upon request and not to reproduce, copy, lend, or otherwise disclose its contents, directly or indirectly, and not to use it for any other purpose other than that for which it was specifically provided. The user also agrees that where consultants or other outside parties are involved in the evaluation process, access to these documents shall not be given to said parties unless those parties also specifically agree to these conditions.

THIS DOCUMENT CONTAINS VALUABLE CONFIDENTIAL AND PROPRIETARY INFORMATION. DISCLOSURE, USE OR REPRODUCTION OF THESE MATERIALS WITHOUT THE WRITTEN AUTHORIZATION OF TESTAMERICA IS STRICTLY PROHIBITED. THIS UNPUBLISHED WORK BY TESTAMERICA IS PROTECTED BY STATE AND FEDERAL LAW OF THE UNITED STATES. IF PUBLICATION OF THIS WORK SHOULD OCCUR THE FOLLOWING NOTICE SHALL APPLY:

©COPYRIGHT 2016 TESTAMERICA LABORATORIES, INC. ALL RIGHTS RESERVED.

Facility Distribution No. Electronic **Distributed To:** Facility Intranet

1.0 Scope and Application

1.1. Analytes, Matrix(s), and Reporting Limits

Method SW846 3010A covers the preparation procedures for the determination of elemental constituents in water samples, wastewater samples and leachates by inductively coupled plasma (SW846 Method 6010B/6010C) and inductively coupled plasma – mass spectrometry (SW846 Method 6020/6020A).

The elements analyzed for by this method and the laboratory's reporting limits (RLs) are summarized below:

Analytical Method Reporting Limits (ug/L)				
Element	**Analytical Method: 6010B**	**Analytical Method: 6010C**	**Analytical Method: 6020**	**Analytical Method: 6020A**
Al	200	200	50	20
Sb	10	20	2.5	1
As	5	15	2.5	1
Ba	200	200	5	2
Be	2	2	1	0.4
B	50	50	100	40
Cd	5	4	2.5	1
Ca	5000	5000	250	100
Cr	10	10	5	2
Co	50	50	5	2
Cu	25	25	5	2
Fe	150	150	150	60
Pb	5	10	1.5	0.6
Mn	15	15	10	4
Mg	5000	5000	250	100
Mo	20	20	5	2
Ni	40	40	5	2
K	5000	5000	250	100
Se	10	20	2.5	5
Ag	10	10	5	2
Na	5000	5000	250	100
Sr	20	20	5	2
Tl	10	20	1	0.4
Sn	50	50	20	8
Ti	20	20	5	2
V	50	50	5	2
Zn	30	30	20	8

On occasion clients may request modifications to this SOP. These modifications are handled following the procedures outlined in Sections 7 (Review of Work Request) and 19 (Test Methods and Method Validation) of TestAmerica Edison's Quality Assurance Manual (ED-QA-LQM).

2.0 Summary of Method

A volume of sample is refluxed with nitric acid until the digestate is light in color in a sample cup using a hot-block. After the digestate is reduced to a low volume, it is refluxed with hydrochloric acid and brought to a final volume for inductively coupled plasma (ICP) by SW846 Method 6010B/6010C or inductively coupled plasma-mass spectrometry (ICP-MS) analysis by SW846 Method 6020/6020A.

3.0 Definitions

For a complete list of definitions refer to Appendix 2 in the most current revision of TestAmerica Edison's Quality Assurance Manual (ED-QA-LQM).

4.0 Interferences

4.1 Whenever reflux is required or sample is to be reduced, NEVER BOIL THE SAMPLE. Boiling could result in a significant loss of sample constituents.

4.2 See the current analytical SOPs for further information related to interferences: TestAmerica SOP Nos. ED-MT-004 *Trace Metals Analysis by Inductively Coupled Plasma Emission Spectroscopy by using SW846 Method 6010B, 6010C;* SOP No. ED-MT-029, *Trace Metals Analysis for Water, Wastewater, Soil, Sediment and Leachate Samples by ICP-MS Using EPA Methods 200.8 and SW-846 Method 6020*; and ED-MT-034, *SW-846 Method 6020A, Trace Metals Analysis of Water, Wastewater, Soil, Sediment and Leachate Samples by ICP-MS*.

5.0 Safety

Employees must abide by the policies and procedures in the Corporate Environmental Health and Safety Manual (CW-E-M-001) and this document. This procedure may involve hazardous material, operations and equipment. This SOP does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of the method to follow appropriate safety, waste disposal and health practices under the assumption that all samples and reagents are potentially hazardous. Safety glasses, gloves, lab coats and closed-toe, nonabsorbent shoes are a minimum.

5.1. Specific Safety Concerns or Requirements

Samples that contain high concentrations of carbonates or organic material or samples that are at elevated pH can react violently when acids are added.

5.2. Primary Materials Used

The following is a list of the materials used in this method, which have a serious or significant hazard rating. **Note: This list does not include all materials used in the method. The table contains a summary of the primary hazards listed in the MSDS for each of the materials listed in the table.** A complete list of materials used in the method can be found in the reagents and materials section. Employees must review the information in the MSDS for each material before using it for the first time or when there are major changes to the MSDS.

Material [1]	Hazards	Exposure Limit [2]	Signs and symptoms of exposure
Hydrochloric Acid	Corrosive Poison	5 ppm-Ceiling	Inhalation of vapors can cause coughing, choking, inflammation of the nose, throat, and upper respiratory tract, and in severe cases, pulmonary edema, circulatory failure, and death. Can cause redness, pain, and severe skin burns. Vapors are irritating and may cause damage to the eyes. Contact may cause severe burns and permanent eye damage.
Nitric Acid	Corrosive Oxidizer Poison	2 ppm-TWA 4 ppm-STEL	Nitric acid is extremely hazardous; it is corrosive, reactive, an oxidizer, and a poison. Inhalation of vapors can cause breathing difficulties and lead to pneumonia and pulmonary edema, which may be fatal. Other symptoms may include coughing, choking, and irritation of the nose, throat, and respiratory tract. Can cause redness, pain, and severe skin burns. Concentrated solutions cause deep ulcers and stain skin a yellow or yellow-brown color. Vapors are irritating and may cause damage to the eyes. Contact may cause severe burns and permanent eye damage.
1 – Always add acid to water to prevent violent reactions.			
2 – Exposure limit refers to the OSHA regulatory exposure limit.			

6.0 Equipment and Supplies

6.1. Instrumentation

- Block Digestor: Adjustable and capable of maintaining a temperature of 90°C-95°C (Environmental Express or equivalent).

6.2. Supplies

- watch glasses
- 75 mm funnels
- 50 ml and 100 ml Hot Block Digestion Cups
- Whatman # 41 filter paper or equivalent
- 0.45 micron filter units and vacuum pump
- Pipettes varying volumes: Eppendorfs & Fisher

7.0 Reagents and Standards

7.1. Reagents

7.1.1. Nitric Acid, Concentrated - Trace Grade or equivalent; store at room

temperature; for stability information, refer to manufacturer's instructions.

7.1.2. Hydrochloric Acid, Concentrated - Trace Grade or equivalent; store at room temperature; for stability information, refer to manufacturer's instructions.

7.1.3. Reagent Grade Water 18 Megohm Minimum

7.2. Standards

7.2.1 Stock ICP Spike Standards:

7.2.1.1 Solutions A, B, C, and D (ICP-Spk), Part No. 4400-100419DD01: commercially purchased from CPI International. Store at room temperature; for stability information, refer to manufacturer's instructions. See Attachment 1 for the standards certified concentrations and catalog numbers.

7.2.1.2 Solutions A, B, C, and D (TCLP-Spk), Part No. SM-606-114: commercially purchased from High-Purity Standards. Store at room temperature; for stability information, refer to manufacturer's instructions. See Attachment 1 for the standards certified concentrations and catalog numbers.

7.2.2 Stock ICP-MS Spike Standards:

7.2.2.1 ICPMS LCS/SPK, Part No. SM-606-111: commercially purchased from High-Purity Standards. Store at room temperature; for stability information, refer to manufacturer's instructions. This stock standard doesn't require any pre-dilutions and is ready to be spiked directly into the QC samples. See Attachment 1 for the standards certified concentrations and catalog numbers.

7.3 Working Spike Standards:

7.3.1. ME_LCS-int (for ICP prep) - Add the following to a 1000 ml volumetric flask and bring to volume using 5% HNO_3: 50 ml each of Part No. 4400-100419DD01 Solutions A, B, C, and D (Sec 7.2.1.1). Record standard preparations in TALS Reagent module. **Note**: Standard must not exceed the earliest expiration date of any one of its components.

7.3.2. ME_TCLPspk (for TCLP prep) – Add the following to a 200 mL volumetric flask and bring to a volume using 5% HNO_3: 40 ml each of Part No. SM-606-114 (TCLP-Spk) Solutions: A, B, C, and D (Sec 7.2.1.2). Record standard preparations in TALS Reagent module. **Note**: Standard must not exceed the earliest expiration date of any one of its components.

8.0 Sample Collection, Preservation, Shipment and Storage

Sample container, preservation techniques and holding times may vary and are dependent on sample matrix, method of choice, regulatory compliance, and/or specific contract or client requests. Listed below are the holding times and the references that include preservation requirements.

Matrix	Sample[4] Container	Min. Sample Size	Preservation	Holding Time[1]	Reference
Water	Polyethylene, glass	50 ml	HNO_3 to pH < 2 prior to shipment; if not, acidify upon receipt in lab [2,3]	180 Days	40 CFR Part 136.3

[1] Inclusive of digestion and analysis.
[2] Acid preservation may be omitted for shipping; however, acid must be added upon receipt in the lab. Following acidification, mix the sample and hold for at least 24 hours. Just prior to digestion or direct analysis, verify pH<2. If pH≥ 2, repeat steps (i.e., add acid, hold for 24hrs, verify ph<2).
[3] Aqueous samples may be stored at room temperature.
[4] All containers must be pre-washed with detergents, acids and water.

9.0 Quality Control

9.1. Sample QC - The following quality control samples are prepared with every 20 samples or every batch of samples digested whichever comes first.

Quality Controls	Frequency	Control Limit
Method Blank (MB)	1 in 20 or fewer samples	See the Quality Control Section of the referring analytical SOP (i.e., SOP No. ED-MT-004, ED-MT-029 and ED-MT-034)
Laboratory Control Sample (LCS)	1 in 20 or fewer samples	
Matrix Spike (MS)[1]	1 in 20 or fewer samples	
Sample Duplicate (DUP)[1]	1 in 20 or fewer samples	
Matrix Spike Duplicate (MSD)	When requested by the client	

[1] The sample selection for MS and Sample Duplicate is random unless specifically requested by a client. Quality control samples prepared with each set of samples digested include Matrix Spike, Sample Duplicate, Method Blank, Laboratory Control Sample and Matrix Spike Duplicate (MSD: when requested by the client). Use the same environmental sample for the matrix spike and duplicate sample whenever possible. If insufficient sample volume is available, another environmental sample may be used for the duplicate sample.

9.2 Instrument QC

None

10.0 Procedure

10.1. Sample Preparation

10.1.1. Filtration Procedure for Dissolved Metals not filtered in the Field

10.1.1.1. The unpreserved sample must be filtered through a 0.45um filter unit as soon as practical after collection.

10.1.1.2. Collect the required volume of filtrate by using a 0.45um filter unit and a vacuum pump.

10.1.1.3. Acidify the filtrate with 1:1 HNO_3 to a pH of <2.

10.1.1.4. The method blank (MB) must be filtered and digested under the same conditions as the lab filtered samples.

10.1.2. During digestion, at least 2-3 ml of sample solution must be maintained in the digestion cup. Sample must never be allowed to go to dryness. If sample should go to dryness, discard sample and redigest.

10.1.3. Pour out 50 ml of well-mixed sample into a 50 ml digestion cups *[note:100 mL hot block digestion cups can be used, except use the same sample volume (50 mL) for the entire preparation batch].* Label the cup with the sample number using a permanent marker. Prepare the QC samples as follows.

10.1.3.1. Method Blank (MB): Pour out 50 ml of deionized reagent water into the 50 ml digestion cup. Label the cup MB mm/dd/yy (this being the date the sample was prepared) and batch number. The Method Blank is carried through the complete digestion process.

10.1.3.2. Matrix Duplicate (DU): Label the digestion cup assigned for the duplicate sample with sample number and suffix 'DU'. Pour 50 ml of the well-mixed sample.

10.1.3.3. Matrix Spike Sample (MS): Measure 50 ml of well-mixed sample into the appropriately labeled 50 ml digestion cups (cup is labeled with the sample number and suffix 'MS'). The resulting elemental concentrations are listed in Attachments 2 & 3.

- For ICP preps (non-TCLP): spike the ICP Matrix Spike sample with 1 ml of ME_LCS-int (Sec 7.3.1).
- For ICP and ICPMS preps (TCLP): spike the Matrix sample with 0.5 ml ME_TCLPspk (Sec 7.3.2).
- For ICP-MS preps (non-TCLP): spike the sample with 0.25 ml ICPMS LCS/SPK (Sec 7.2.2.1).

10.1.3.4. Laboratory Control Sample (LCS): Pour out 50 ml of deionized

reagent water sample into the appropriately labeled 50 ml digestion cups. Label the cup 'LCS' and the batch number. Spike the ICP-LCS and ICPMS- LCS (as appropriate) in the same way as the Matrix Spike Sample, see Sec. 10.1.3.3. Refer to Attachments 2 & 3 for final elemental concentrations.

10.1.4. Add 1.5 ml concentrated HNO_3, cover with ribbed watch glass, place digestion cup on hot block and reduce volume to approximately 5 ml. Do not allow sample to boil or dry.

10.1.5. Cool digestion cup and add another 1.5 ml concentrated HNO_3, cover cup with a watch glass, and return to hot block allowing a gentle reflux action.

10.1.6. Continue heating, adding additional HNO_3 until the digestate is light in color or does not change in appearance. Additional acid must not exceed to more than 1.5 ml. Allow the sample to cool down

10.1.7. Add 5 ml of 1:1 HCl and warm cup gently for 15 minutes to dissolve any precipitate or residue. Allow sample to cool down.

10.1.8. Wash down watchglass and digestion cup walls with deionized water. If necessary, accurately label a new 50 ml sample cup and filter samples through a #41 Whatman filter paper to remove any silicates and other insoluble material that could clog the nebulizer on the instrument.

10.1.9. Bring the sample volume up to 50 ml.

10.1.10. Enter all batch/sample information into TALS (see Sec 11.4. for instructions). Print the sample labels from the batch and attach to the corresponding sample cups. These labels contain information such as: Job & sample number, Batch number and container number.

10.2. Calibration

10.2.1. The volume of the 50ml and 100 ml hotblock digestion cups are verified for each lot received. The 50 ml and 100 ml volume verification with the appropriate Lot number is documented in the 'Metals Labware Volume Verification Logbook.'

10.3. Sample Analysis

10.3.1. Refer to the Analytical SOP, TestAmerica Edison SOP No. ED-MT-004, SOP No. ED-MT-029 and SOP No. ED-MT-034.

11.0. Calculations / Data Reduction

11.1. Accuracy:

$$\text{ICV / CCV, LCS \% Recovery} = \frac{\text{observed concentration}}{} \times 100$$

known concentration

$$\text{MS \% Recovery} = \frac{\text{(spiked sample) - (unspiked sample)}}{\text{spiked concentration}} \times 100$$

11.2. Precision (RPD):

$$\text{Matrix Duplicate (MD)} = \frac{|\text{orig. sample value - dup. sample value}|}{[(\text{orig. sample value + dup. sample value})/2]} \times 100$$

11.3. Concentration:

Refer to TestAmerica SOP No. ED-MT-004, ED-MT-029, ED-MT-033, and ED-MT-034.

11.4. Documenting and Reporting

11.4.1. Sample preparations must be documented in the Analyst Desktop II program located in TestAmerica Laboratory System (TALS).

11.4.1.1. Double click the TALS icon on the computer desktop. Enter your username and password and select Login.

11.4.1.2. Under TALS Menu, click the 'plus' sign next to Analyst. Double click Analyst Desktop II. Click the 'plus' sign next to Methods.

11.4.1.3. Right click the prep method that you are using to prep the samples. Select Create New Batch-From Scratch.

11.4.1.4. The analyst must enter the following information: Sample names (use scanners), initial and final sample volume, spike name and amount used, and all reagents and their corresponding lot numbers.

11.4.1.5. After saving the batch information in TALS, select Print-Batch Sheets (which must be submitted to metals analytical dept. along with the samples) and select Print-Batch Labels (which must be attached to the corresponding final digestate sample cups in the batch).

11.4.1.6. The Metals Data Review checklist (EDS-WI-007) must be completed prior to data submission.

12.0. Method Performance

12.1. Method Detection Limit Study (MDL)

The method detection limit (MDL) is the lowest concentration that can be detected for a given analytical method and sample matrix with 99% confidence that the analyte is present. The MDL is determined according to the laboratory's MDL procedure in Section 19 (Test Methods and Method Validation) of TestAmerica Edison's Quality

Assurance Manual (ED-QA-LQM). MDLs reflect a calculated (statistical) value determined under ideal laboratory conditions in a clean matrix, and may not be achievable in all environmental matrices. The laboratory maintains MDL studies for analyses performed; these are verified at least annually unless method requirements require a greater frequency.

12.2. Demonstration of Capabilities

For DOC procedure refer to Section 19 in the most current revision of TestAmerica Edison's Quality Assurance Manual (ED-QA-LQM).

12.3. Training Requirements

Refer to TestAmerica SOP No. ED-GEN-022, Training, for the laboratory's training program.

13.0. Pollution Control

It is TestAmerica's policy to evaluate each method and look for opportunities to minimize waste generated (i.e., examine recycling options, ordering chemicals based on quantity needed, preparation of reagents based on anticipated usage and reagent stability). Employees must abide by the policies in Section 13 of the Corporate Environmental Health and Safety Manual (CW-E-M-001) for "Waste Management and Pollution Prevention."

14.0. Waste Management

14.1. Waste management practices are conducted consistent with all applicable rules and regulations. Excess reagents, samples and method process wastes are disposed of in an accepted manner. Waste description rules and land disposal restrictions are followed. Waste disposal procedures are incorporated by reference to TestAmerica SOP No. ED-SPM-008 (Laboratory Waste Disposal Procedures).

14.2. The following waste streams are produced when this method is carried out:

- Digested Samples: Corrosive Acid- Materials that are not above regulatory limits will be submitted for elementary neutralization with 50% sodium hydroxide solution (Siedler Chemical SC-1824-03). Major concern is heat generated from the neutralization process. Initial volume of acid waste to be neutralized should be no more than 15 gallons. Finished neutralization with sodium bicarbonate (Siedler Chemical SC-0219-25) to a pH of 6 – 9 in the primary tank. Once pH has been established the primary tank is transferred through filter housing to a secondary tank. The pH is rechecked. If the pH is within specifications, the secondary tank is released to the municipal sewer system.

- Samples above regulatory limits and expired RCRA metals standards (Waste Corrosive Liquid, Acidic, Inorganic, n.o.s.) are collected in satellite accumulation and sent off site through a Waste disposal vendor.
  Onyx Profile WIP Number: 590598
  Teris Profile Number 50016653

- Soil Retain Samples - These samples if not flagged in the system for any hazardous constituents are transferred to poly-lined cubic yard boxes. These boxes when full are sent to stabilization or incineration. These materials are sent out as hazardous for lead and chromium

  Teris Profile Number (incineration): 50016710
  Onyx Profile Number: (stabilization) 402535

15.0. References / Cross-References

15.1. Test Methods for Evaluating Solid Waste Physical/Chemical Methods (SW846), Third Edition, September 1986, Final Update I, July 1992, Final Update IIA, August 1993, Final Update II, September 1994; Final Update IIB, January 1995; Final Update III, December 1996; SW-846, Method 3010A.

15.2. TestAmerica Edison Document No. ED-QA-LQM, *Laboratory Quality Manual*, most current revision.

15.3. TestAmerica Edison SOP ED-GEN-022, *Training*, most current revision

15.4. TestAmerica Edison SOP No. ED-MT-004, *Trace Metals Analysis by Inductively Coupled Plasma Emission Spectroscopy by using SW846 Method 6010B*, 6010C; most current revision.

15.5. TestAmerica Edison SOP No. ED-MT-029, *Trace Metals Analysis for Water, Wastewater, Soil, Sediment and Leachate Samples by ICP-MS Using EPA Methods 200.8 and SW-846 Method 6020*, most current revision.

15.6. TestAmerica Edison SOP No. ED-MT-034, *SW-846 Method 6020A, Trace Metals Analysis of Water, Wastewater, Soil, Sediment and Leachate Samples by ICP-MS,* most current revision.

15.7. TestAmerica Edison SOP No. ED-SPM-008, *Laboratory Waste Disposal Procedures*, current revision.

15.8. TestAmerica Corporate Environmental Health and Safety Manual, Document No. CW-E-M-001, most current revision.

15.9. Metals Preparation Data Review Checklist Work Instruction # EDS-WI-007, most current revision.

16.0. Method Modifications:

None

17.0. Attachments

Attachment 1: Certificate of Analysis of ICP and ICPMS stock standard.
Attachment 2: ICP: Laboratory Control Sample and Matrix Spike Final Concentration in Soln (ug/L), for Non-TCLP and for TCLP
Attachment 3: ICP-MS Matrix Spiking Solution, ICPMS LCS/SPK

18.0. Revision History

- Revision 10, dated 03 August 2016
  - Updated this SOP to reflect the new initial and final sample volumes (50ml) keeping the sample/reagent volume ratio the same; 100 ml initial and final sample volumes have been removed from this SOP.
  - Removed all reference to SOP ED-MT-033 from this SOP.
  - Sec 6.2: Added 50ml hotblock digestion cups to the list of supplies.
  - Sec 7.2.2.1: Added the part number for the ICPMS stock spike standard.
  - Sec 8.0: Revised the minimum sample size to 50ml.
  - Sec 9.1: Added matrix spike duplicate to the list of QC sample.
  - Sec 10.1.3.3: Revised the spiking amount (ml) added to the Matrix spike sample to reflect the 50 ml final volume.
  - Sec 10.14, Sec 10.1.5, Sec 10.1.6, and Sec 10.1.7 Updated the amount (ml) of reagents added to reflect the 50 ml final volume.
  - Sec 10.2.1: Added 50ml hotblock digestion cups.

- Revision 9, dated 19 August 2013
  - Sec 1 & 12: Updated LQM section references to reflect the most current LQM revision.
  - Sec 11.4.1.6. Added requirements to complete the Batch preparation section of the Metals Data Review Checklist (EDS-WI-007) prior to data submission.

- Revision 8, dated 24 August 2011
  - Sec 10.1.1.4: added new section to include procedure for filtering method blanks.

- Revision 7, dated 13 April 2011
  - Sec 1.1: Revised to reference the analytical methods 6010C and 6020A and added their corresponding reporting limits to the Analytical Method Reporting Limits Table.
  - Sec 2.0, 10.3 & 11.3: Revised to reference the analytical methods 6010C and 6020A.
  - Sec 3: Revised LQM reference to the list of definitions.
  - Sec 4.2: Added SOP No. ED-MT-033 and ED-MT-034 for reference.
  - Sec 7.2.1 & 7.3: Added new spike standards for ICP (p/n 4400-100419DD01) and TCLP prep (TCLP-Spk).
  - Sec 8: Added Footnotes.
  - Sec 10.1.3.3: Added TCLP spiking instructions.
  - Sec 11.4: updated documenting and reporting instructions to comply with TALS procedure.
  - Sec 15: added applicable references.
  - Sec 17: Attachment 2: Updated Table to include TCLP spike concentration; deleted internal COC (previously attached as Attachment 4).
  - Updated COAs in attachment 1.

- Revision 6, dated 24 November 2008
  - Revised SOP format in accordance with TestAmerica Corporate Quality SOP No. CW-QS-002 (Rev 0) Writing a Standard Operating Procedure (SOP).
  - Sec 1.1: Deleted '*flame atomic absorption spectroscopy*.'
  - Added information related to ICP-MS (method 6020) where applicable (i.e. Sec

1.1, Sec 7 etc.).

- Added Laboratory's reporting limits in Table format.
- Sec 6: Updated list of equipment and supplies to include current materials used (e.g. added block digestor, deleted hot plate & centrifuge tubes).
- Section 7: Included storage and stability information of reagents and standards. Updated the source of the stock spike standards and the preparation instructions for the ICP working spike standards.
- Section 8: Updated in Table format and have included sample container, sample size requirements and method reference.
- Sec 10: Deleted procedure for acid rinsing glassware; replaced use of beakers with disposable digestion cups
- Deleted the use of 100 ml digestion cup and replaced with 125ml specimen cup.
- Deleted procedure for transferring digestates from digestion cup to specimen cup. Also added information and procedure on internal chain of custody to identify sample digestates.
- Section 15: Added applicable references
- Section 17: Added attachment 1 to include certificate of analysis of stock standards, attachment 3 to include on instrument elemental concentrations of the ICPMS QC samples and attachment 4 to describe the internal chain of custody used for the digested samples.

Attachment 1

USA
5580 Skylane Boulevard P: 707.525.5788
Santa Rosa, CA 95403 P: 800.878.7654
F: 707.545.7901

CPI INTERNATIONAL
Advanced Analytical, Semiconductor and Life Science Solutions

Europe
P.O. Box 2704 P: +31 20 638 05 97
1000 CS Amsterdam F: +31 20 420 28 36
The Netherlands

www.cpiinternational.com
www.calibag.com

SOLN. A
DE
4-4-11

Expiry: 21-Sep-12

Certificate of Analysis

Part Number: **4400-100419DD01**
Lot Number: **11C157**
Shelf Life: **18 Months**

Date Rec'd
03/22/11 MP

TestAmerica/Edison
Custom Standard
5% HNO3

Concentrations in ug/mL ± 0.5%

Fe	1000	Zn	500
Se	2000	Mn	500
TL	2000	Ni	500
Be	50		
Cd	50		
Cr	200		
Co	500		
Cu	250		
Pb	500		
Ag	50		
Sr	500		
V	500		

This standard solution was prepared using high-purity starting materials, high-purity acid (if required) and 18-megaohm de-ionized water. The starting materials were weighed to five significant figures and diluted in volumetric glassware calibrated to five significant figures.

Starting materials were analyzed at 1000µg/mL by ICP-MS for trace impurities. The standard solution concentrations were certified instrumentally against the National Institute of Standards and Technology's SRM 3100 series, NIST approved second source and/or gravimetrically.

Accuracy and stability are guaranteed to within plus or minus 0.5% of the certified value for the stated shelf life from the date of shipment. The solution should be kept tightly capped and stored under normal laboratory conditions. See attached MSDS for proper handling information.

For questions or comments please call 1-800-878-7654 in the USA, +31 20 638 05 97 in Europe or visit our web-site at www.cpiinternational.com.

USA
5580 Skylane Boulevard P: 707.525.5788
Santa Rosa, CA 95403 P: 800.878.7654
P: 707.545.7901

CPI INTERNATIONAL
Advanced Analytical, Semiconductor and Life Science Solutions

Europe
P.O. Box 2704 P: +31 20 638 05 97
1000 CS Amsterdam P: +31 20 420 28 36
The Netherlands

www.cpiinternational.com
www.coilog.com

Expiry: 21-Sep-12

Certificate of Analysis

Part Number: **4400-100419DD01** **Solution B**
Lot Number: **11C157**
Shelf Life: **18 Months**

TestAmerica/Edison
Custom Standard
10% HNO3

Date Rec'd
03/22/11 MP

Concentrations in ug/mL ± 0.5%

Al	2000
Mg	20000
K	20000
Ca	20000
Na	20000

This standard solution was prepared using high-purity starting materials, high-purity acid (if required) and 18-megaohm de-ionized water. The starting materials were weighed to five significant figures and diluted in volumetric glassware calibrated to five significant figures.

Starting materials were analyzed at 1000µg/mL by ICP-MS for trace impurities. The standard solution concentrations were certified instrumentally against the National Institute of Standards and Technology's SRM 3100 series, NIST approved second source and/or gravimetrically.

Accuracy and stability are guaranteed to within plus or minus 0.5% of the certified value for the stated shelf life from the date of shipment. The solution should be kept tightly capped and stored under normal laboratory conditions. See attached MSDS for proper handling information.

For questions or comments please call 1-800-878-7654 in the USA, +31 20 638 05 97 in Europe or visit our web-site at www.cpiinternational.com

USA
5580 Skylane Boulevard P: 707.525.5788
Santa Rosa, CA 95403 P: 800.878.7654
F: 707.545.7901

CPI INTERNATIONAL
Advanced Analytical, Semiconductor and Life Science Solutions

Europe
P.O. Box 2704 P: +31 20 638 05 97
1000 CS Amsterdam F: +31 20 420 28 36
The Netherlands

www.cpiinternational.com
www.calibag.com

Expiry: 21-Sep-12

Certificate of Analysis

Part Number: **4400-100419DD01** **Solution D**
Lot Number: **11C157**
Shelf Life: **18 Months**

Date Rec'd
05/22/11 MP

TestAmerica/Edison
Custom Standard
2% HNO3

Concentrations in ug/mL ± 0.5%

As	2000
Ba	2000
B	500

This standard solution was prepared using high-purity starting materials, high-purity acid (if required) and 18-megaohm de-ionized water. The starting materials were weighed to five significant figures and diluted in volumetric glassware calibrated to five significant figures.

Starting materials were analyzed at 1000µg/mL by ICP-MS for trace impurities. The standard solution concentrations were certified instrumentally against the National Institute of Standards and Technology's SRM 3100 series, NIST approved second source and/or gravimetrically.

Accuracy and stability are guaranteed to within plus or minus 0.5% of the certified value for the stated shelf life from the date of shipment. The solution should be kept tightly capped and stored under normal laboratory conditions. See attached MSDS for proper handling information.

For questions or comments please call 1-800-878-7654 in the USA, +31 20 638 05 97 in Europe or visit our web-site at www.cpiinternational.com

USA
5580 Skylane Boulevard P: 707.525.5788
Santa Rosa, CA 95403 P: 800.878.7654
F: 707.545.7901

CPI INTERNATIONAL
Advanced Analytical, Semiconductor and Life Science Solutions
www.cpiinternational.com
www.calitag.com

Europe
P.O. Box 2704 P: +31 20 638 05 97
1000 CS Amsterdam F: +31 20 420 28 36
The Netherlands

Expiry: 21-Sep-12

Certificate of Analysis

Part Number: **4400-100419DD01** **Solution C**
Lot Number: **11C157**
Shelf Life: **18 Months**

Date Rec'd
03/22/11 MP

TestAmerica/Edison
Custom Standard
5% HNO3 + 2% HF

Concentrations in ug/mL ± 0.5%

Sb	500
Mo	500
Sn	500
Ti	500

This standard solution was prepared using high-purity starting materials, high-purity acid (if required) and 18-megaohm de-ionized water. The starting materials were weighed to five significant figures and diluted in volumetric glassware calibrated to five significant figures.

Starting materials were analyzed at 1000µg/mL by ICP-MS for trace impurities. The standard solution concentrations were certified instrumentally against the National Institute of Standards and Technology's SRM 3100 series, NIST approved second source and/or gravimetrically.

Accuracy and stability are guaranteed to within plus or minus 0.5% of the certified value for the stated shelf life from the date of shipment. The solution should be kept tightly capped and stored under normal laboratory conditions. See attached MSDS for proper handling information.

For questions or comments please call 1-800-878-7654 in the USA, +31 20 638 05 97 in Europe or visit our web-site at www.cpiinternational.com.

HIGH-PURITY STANDARDS
www.highpuritystandards.com

P.O. Box 41727
Charleston, SC 29423
Phone: (843) 767-7900
Fax: (843) 767-7906

Certificate of Analysis

SM-606-111
(ICPMS LCS/SPK)

Lot # 1031503

Source	Source Purity	Matrix	Standard Concentration
High Purity Metals, Salts and Oxides	99.98+%	HNO_3, 4% + Tr HF	µg/mL ± 0.5% See element list on reverse

This spectrometric standard solution has been prepared from high-purity reference materials. Sub-boiling distilled high-purity acid has been used to place the materials in solution and to stabilize the standard. The matrix is as noted above in 18 megaohm deionized water. The reference materials have been assayed by inductively coupled plasma optical emission spectrometry (ICP-OES).

The standard has been prepared gravimetrically by weighing the reference material to 5 significant figures. Volumetric glassware has been calibrated gravimetrically to 5 significant figures. The standard concentration has been verified by ICP-OES against an independent source which is directly traceable to National Institute of Standards and Technology, Standard Reference Material No. 3100 series.

This standard is valid for one year from the shipping date provided the solution is kept tightly capped and stored under normal laboratory conditions.

Theodore C. Rains

Theodore C. Rains, Ph.D.
President

Exp Date: NOV 15 2011
MSDS ATTACHED

SM-606-111
(ICPMS LCS/SPK)
Element List
(µg/mL)

Aluminum	500
Antimony	5
Arsenic	10
Barium	10
Beryllium	5
Boron	100
Cadmium	5
Calcium	500
Chromium	10
Cobalt	5
Copper	10
Iron	500
Lead	5
Magnesium	500
Manganese	50
Molybdenum	10
Nickel	10
Potassium	500
Selenium	10
Silver	5
Sodium	500
Strontium	10
Thallium	4
Tin	10
Titanium	10
Vanadium	10
Zinc	50

HIGH-PURITY STANDARDS
www.highpuritystandards.com

P.O. Box 41727
Charleston, SC 29423
Phone (843) 767-7900
Fax (843) 767-7906

Certificate of Analysis

Rec'd 4-11-11 DE

SM-606-114 (TCLP-Spk) Solution A

Lot # 1109507

Source	Source Purity	Matrix	Standard Concentration
High Purity Metals, Salts and Oxides	99.995+%	HNO_3, 4%	µg/mL ± 0.5% See element list on reverse

This spectrometric standard solution has been prepared from high-purity reference materials. Sub-boiling distilled high-purity acid has been used to place the materials in solution and to stabilize the standard. The matrix is as noted above in 18 megaohm deionized water. The reference materials have been assayed by inductively coupled plasma optical emission spectrometry (ICP-OES).

The standard has been prepared gravimetrically by weighing the reference material to 5 significant figures. Volumetric glassware has been calibrated gravimetrically to 5 significant figures. The standard concentration has been verified by ICP-OES against an independent source which is directly traceable to National Institute of Standards and Technology, Standard Reference Material No. 3100 series.

This standard is valid for one year from the shipping date provided the solution is kept tightly capped and stored under normal laboratory conditions.

Theodore C. Rains

Theodore C. Rains, Ph.D.

APR 0 7 2012

SM-606-114 (TCLP-Spk)

Solution A

Element List

(µg/mL)

Barium	5000
Beryllium	500
Cadmium	500
Cobalt	500
Copper	500
Iron	500
Manganese	500
Nickel	500
Selenium	500
Strontium	500
Thallium	500
Zinc	500

HIGH-PURITY STANDARDS
www.highpuritystandards.com

P.O. Box 41727
Charleston, SC 29423
Phone (843) 767-7900
Fax (843) 767-7905

Certificate of Analysis

Rec'd 4-11-11 DE

SM-606-114 (TCLP-Spk) Solution B

Lot # 1109508

Source	Source Purity	Matrix	Standard Concentration
High Purity Metals, Salts or Oxides	99.98+%	HNO_3, 5%	10,000 µg/mL ± 0.5% Calcium Magnesium Potassium Sodium

This spectrometric standard solution has been prepared from high-purity reference materials. Sub-boiling distilled high-purity acid has been used to place the materials in solution and to stabilize the standard. The matrix is as noted above in 18 megaohm deionized water. The reference materials have been assayed by inductively coupled plasma optical emission spectrometry (ICP-OES).

The standard has been prepared gravimetrically by weighing the reference material to 5 significant figures. Volumetric glassware has been calibrated gravimetrically to 5 significant figures. The standard concentration has been verified by ICP-OES against an independent source which is directly traceable to National Institute of Standards and Technology, Standard Reference Material No. 3100 series.

This standard is valid for one year from the shipping date provided the solution is kept tightly capped and stored under normal laboratory conditions.

Theodore C. Rains

Theodore C. Rains, Ph.D.
President

APR 0 7 2012
Exp Date:
MSDS ATTACHED

HIGH-PURITY STANDARDS
www.highpuritystandards.com

P.O. Box 41727
Charleston, SC 29423
Phone (843) 767-7900
Fax (843) 767-7906

Certificate of Analysis

Rec'd 4-11-11 DE

SM-606-114 (TCLP-Spk) Solution C

Lot # 1109509

Source	Source Purity	Matrix	Standard Concentration
High Purity Metals, Salts and Oxides	99.96+%	HNO_3, 2% HF, 0.5%	µg/mL ± 0.5% See element list on reverse

This spectrometric standard solution has been prepared from high-purity reference materials. Sub-boiling distilled high-purity acid has been used to place the materials in solution and to stabilize the standard. The matrix is as noted above in 18 megaohm deionized water. The reference materials have been assayed by inductively coupled plasma optical emission spectrometry (ICP-OES).

The standard has been prepared gravimetrically by weighing the reference material to 5 significant figures. Volumetric glassware has been calibrated gravimetrically to 5 significant figures. The standard concentration has been verified by ICP-OES against an independent source which is directly traceable to National Institute of Standards and Technology, Standard Reference Material No. 3100 series.

This standard is valid for one year from the shipping date provided the solution is kept tightly capped and stored under normal laboratory conditions.

Theodore C. Rains

Theodore C. Rains, Ph.D.
President

Exp Date: APR 07 2012

SM-606-114 (TCLP-Spk)

Solution C

Element List

(µg/mL)

Anitmony	500
Boron	500
Molybdenum	500
Silver	250
Tin	500
Titanium	500
Vanadium	250

HIGH-PURITY STANDARDS
www.highpuritystandards.com
Charleston, SC 29423
Phone (843) 767-7900
Fax (843) 767-7906

Certificate of Analysis

rec'd 4-11-11 DE

SM-606-114 (TCLP-Spk) Solution D

Lot # 1109510

Source	Source Purity	Matrix	Standard Concentration
High Purity Metals, Salts or Oxides	99.998+%	HNO_3, 5%	2500 µg/mL ± 0.5% Aluminum Arsenic Chromium Lead

This spectrometric standard solution has been prepared from high-purity reference materials. Sub-boiling distilled high-purity acid has been used to place the materials in solution and to stabilize the standard. The matrix is as noted above in 18 megaohm deionized water. The reference materials have been assayed by inductively coupled plasma optical emission spectrometry (ICP-OES).

The standard has been prepared gravimetrically by weighing the reference material to 5 significant figures. Volumetric glassware has been calibrated gravimetrically to 5 significant figures. The standard concentration has been verified by ICP-OES against an independent source which is directly traceable to National Institute of Standards and Technology, Standard Reference Material No. 3100 series.

This standard is valid for one year from the shipping date provided the solution is kept tightly capped and stored under normal laboratory conditions.

Theodore C. Rains

Theodore C. Rains, Ph.D.
President

Exp Date: APR 07 2012
MSDS ATTACHED

Attachment 2

ICP: LCS & Matrix Spike Final concentration in solution (ug/L)		
ELEMENT	**For Non-TCLP**	**For TCLP**
Aluminum	2000	5000
Antimony	500	1000
Arsenic	2000	5000
Barium	2000	10000
Beryllium	50	1000
Cadmium	50	1000
Calcium	20000	20000
Chromium	200	5000
Cobalt	500	1000
Copper	250	1000
Iron	1000	1000
Lead	500	5000
Manganese	500	1000
Magnesium	20000	20000
Nickel	500	1000
Potassium	20000	20000
Selenium	2000	1000
Silver	50	500
Sodium	20000	20000
Thallium	2000	1000
Vanadium	500	500
Zinc	500	1000
Boron	500	1000
Molybdenum	500	1000
Tin	500	1000
Titanium	500	1000
Strontium	500	1000

Attachment 3

ICPMS Matrix Spiking Solution, ICPMS LCS/SPK	
Element	**Matrix Spike Conc. (ug/L)**
Aluminum (Al)	2500
Antimony (Sb)	25
Arsenic (As)	50
Barium (Ba)	50
Beryllium (Be)	25
Boron (B)	500
Cadmium (Cd)	25
Calcium (Ca)	2500
Chromium (Cr)	50
Cobalt (Co)	25
Copper (Cu)	50
Iron (Fe)	2500
Lead (Pb)	25
Magnesium(Mg)	2500
Manganese (Mn)	250
Molybdenum (Mo)	50
Nickel (Ni)	50
Potassium (K)	2500
Selenium (Se)	50
Silver (Ag)	25
Sodium (Na)	2500
Strontium (Sr)	50
Thallium (Tl)	20
Tin (Sn)	50
Titanium (Ti)	50
Vanadium (V)	50
Zinc (Zn)	250

Title: Extraction of Semi-Volatile Organic Compounds in Aqueous Samples and Leachates - Separatory Funnel, SW846 Method 3510C

Once printed, this is considered an uncontrolled document

Approvals (Signature/Date):

Sylvanus Klusey	03/26/2018 Date	Dan Helfrich	03/26/2018 Date
Organics Operations Manager		Health & Safety Manager / Coordinator	
Carl Armbruster	03/26/2018 Date	Mark Acierno	03/26/2018 Date
Quality Assurance Manager		Laboratory Director	

Copyright Information:
This documentation has been prepared by TestAmerica Laboratories, Inc. and its affiliates ("TestAmerica"), solely for their own use and the use of their customers in evaluating their qualifications and capabilities in connection with a particular project. The user of this document agrees by its acceptance to return it to TestAmerica upon request and not to reproduce, copy, lend, or otherwise disclose its contents, directly or indirectly, and not to use it for any other purpose other than that for which it was specifically provided. The user also agrees that where consultants or other outside parties are involved in the evaluation process, access to these documents shall not be given to said parties unless those parties also specifically agree to these conditions.

THIS DOCUMENT CONTAINS VALUABLE CONFIDENTIAL AND PROPRIETARY INFORMATION. DISCLOSURE, USE OR REPRODUCTION OF THESE MATERIALS WITHOUT THE WRITTEN AUTHORIZATION OF TESTAMERICA IS STRICTLY PROHIBITED. THIS UNPUBLISHED WORK BY TESTAMERICA IS PROTECTED BY STATE AND FEDERAL LAW OF THE UNITED STATES. IF PUBLICATION OF THIS WORK SHOULD OCCUR THE FOLLOWING NOTICE SHALL APPLY:

©COPYRIGHT 2018 TESTAMERICA LABORATORIES, INC. ALL RIGHTS RESERVED.

1.0 Scope and Application

1.1. **Analytes, Matrix(s), and Reporting Limits** SW846 Method 3510C describes a procedure for isolating semivolatile organic compounds from aqueous samples and leachates, including concentration techniques suitable for preparing the extract for GC/MS analysis. This SOP is applicable to the isolation and concentration of water-soluble and slightly water-soluble semivolatile organics in preparation for analysis by SW846 Methods 8270C or 8270D.

1.2. For a complete discussion of analytes and reporting limits (RLs) please refer to TestAmerica Edison SOP Nos. ED-MSS-002, *Semivolatile Organic Compounds by Gas Chromatography/Mass Spectrometry (GC/MS) SW846 Method 8270C*, current revision and ED-MSS-009, *Semivolatile Organic Compounds by Gas Chromatography/Mass Spectrometry (GC/MS) SW846 Method 8270D*, current revision

1.3. On occasion clients may request modifications to this SOP. These modifications are handled following the procedures outlined in Section 7 (*Review of Work Request*) and 19 (*Test Methods and Method Validation*) of TestAmerica Edison's Quality Assurance Manual (ED-QA-LQM).

2.0 Summary of Method

2.1. A measured volume of sample (~250 mL) is serially extracted with methylene chloride at a pH less than 2 and again at a pH greater than 11 using separatory funnel extraction. The methylene chloride extract is dried and concentrated to a volume of approximately 2 mL. Nitrogen blowdown is employed as the final concentration step. The extract is subsequently analyzed by SW846 Method 8270C or 8270D (GC/MS) by a large volume injection (LVI) technique. This procedure is referred to throughout as Reduced Volume Extraction (RVE) and Large Volume Injection (LVI).

2.2. An option for preparing aqueous samples using a larger initial volume (~1000 ml) is also described. This procedure is referred to as 'large volume' throughout the document.

3.0 Definitions

For a complete list of definitions refer to Appendix 2 in the most current revision of TestAmerica Edison's Quality Assurance Manual (ED-QA-LQM).

4.0 Interferences

4.1. Solvents, reagents, glassware, and other sample hardware may yield artifacts and/or interferences to sample analysis. All these materials must be demonstrated to be free from interferences under the conditions of the analysis by analyzing method blanks.

4.2. Phthalate esters contaminate many products commonly found in the laboratory. Plastics, in particular, must be avoided, because phthalates are often used as plasticizers and are easily extracted from plastic material.

Phthalate contamination may result at any time if consistent quality control is not practiced.

4.3. The decomposition of some analytes has been demonstrated under basic extraction conditions. Phthalate esters may exchange and phenols may react to form tannates. These reactions increase with increasing pH, and are decreased by shorter reaction times. Performing the initial extraction at an acid pH will optimize the recovery of phenols

5.0 Safety

Employees must abide by the policies and procedures in the Corporate Environmental Health and Safety Manual (CW-E-M-001) and this document. This procedure may involve hazardous material, operations and equipment. This SOP does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of the method to follow appropriate safety, waste disposal and health practices under the assumption that all samples and reagents are potentially hazardous. Safety glasses, gloves, lab coats and closed-toe, nonabsorbent shoes are a minimum.

5.1. Specific Safety Concerns or Requirements

During Kuderna-Danish (KD) concentration, do not allow the extract to boil to dryness. The solvent vapors remaining in the KD apparatus may superheat and create an explosion or fire hazard.

The following analytes have been tentatively classified as known or suspected, human or mammalian carcinogens: benzo(a)anthracene, benzidine, 3,3'dichlorobenzindine, benzo(a)pyrene, alpha-BHC, beta-BHC, gamma-BHC, delta-BHC, dibenz(a,h)anthracene, N-nitrosodimethylamine, 4,4'-DDT, and polychlorinated biphenyl compounds. Primary standards of these toxic compounds should be prepared in hood.

The use of Kevlar gloves is required for the assembly/disassembly of ground glass joints in addition to those tasks that present the potential risk for injury.

The use of separatory funnels to extract aqueous samples with Methylene Chloride creates excessive pressure very rapidly. Initial venting should be done immediately after the sample container has been sealed and inverted, periodic venting may be necessary during the extraction. Vent the funnel into the hood away from people and other samples. This is considered a high-risk activity, the use of a face shield over safety glasses or goggles is recommended. Keep the sash on the fume hood as low as reasonably possible.

5.2. Primary Materials Used

The following is a list of the materials used in this method, which have a serious or significant hazard rating. **Note: This list does not include all materials used in the method. The table contains a summary of the primary hazards listed in the MSDS for each of the materials listed in the table.** A complete list of materials used in the method can be found in

the reagents and materials section. Employees must review the information in the MSDS for each material before using it for the first time or when there are major changes to the MSDS.

Material (1)	Hazards	Exposure Limit (2)	Signs and symptoms of exposure
Methylene Chloride	Carcinogen Irritant	25 ppm-TWA 125 ppm-STEL	Causes irritation to respiratory tract. Has a strong narcotic effect with symptoms of mental confusion, light-headedness, fatigue, nausea, vomiting and headache. Causes irritation, redness and pain to the skin and eyes. Prolonged contact can cause burns. Liquid degreases the skin. May be absorbed through skin.
Sulfuric Acid	Corrosive Oxidizer Dehydra-dator	1 mg/m^3	This material will cause burns if comes into contact with the skin or eyes. Inhalation of vapors will cause irritation of the nasal and respiratory system.
Sodium Hydroxide	Corrosive Poison	2 ppm, 5 mg/m^3	This material will cause burns if comes into contact with the skin or eyes. Inhalation of Sodium Hydroxide dust will cause irritation of the nasal and respiratory system.
Methanol (MeOH)	Flammable Poison Irritant	200 ppm-TWA	A slight irritant to the mucous membranes. Toxic effects exerted upon nervous system, particularly the optic nerve. Symptoms of overexposure may include headache, drowsiness and dizziness. Methyl alcohol is a defatting agent and may cause skin to become dry and cracked. Skin absorption can occur; symptoms may parallel inhalation exposure. Irritant to the eyes.
1 – Always add acid to water to prevent violent reactions.			
2 – Exposure limit refers to the OSHA regulatory exposure limit.			

6.0 Equipment and Supplies

6.1. Instrumentation

- Separatory funnel rotator, APR Machine or equivalent
- Analytical Evaporator (N-Evap) Organomation
- Centrifuge, Varifuge F; Hereaus Sepatech
- Six Position Steam Bath, Fisher 15-496 or equivalent

6.2. Supplies

- 250 ml Erlenmeyer Flask, AMK Glass ERL-0252 or equivalent
- 2000 ml or 500 ml Separatory Funnel, AMK Glass SFC or equivalent
- 100 mm o. d. glass funnels, Fisher or equivalent

- 10 ml jacketed, graduated Concentrator Tubes, AMK Glass KD-0018 or equivalent
- 19/22 Ground Glass Stoppers
- 3 Ball Snyder Columns, TEC Glass TG6-03 or equivalent
- 1 ml Gastight Syringe, Hamilton 81317 or equivalent
- 150 ml Centrifuge Tube
- 100 ml Graduated Cylinder
- Pasteur 5 ¾" Disposable Pipets, Fisher 13-678-20B or equivalent
- Kuderna Danish Flask (500 ml), TEC Glass TG7-01 or equivalent
- Vials, 2ml amber screw cap with Teflon liner
- Glass Wool
- Dessicator
- Standard Taper Clamps (Size 19, blue)
- Boiling Stones, Troemner P/N 133-B or equivalent, rinsed with Methylene Chloride
- pH Paper
- Watch Glass
- Wax Pencil
- 1 Liter Graduated Cylinder
- Marking Tags

7.0 Reagents and Standards

7.1 Reagents

Note: Each lot of Methylene Chloride, Acetone, Methanol and Sulfuric Acid is screened for contaminants before being used for analysis as detailed in TestAmerica Corporate Quality SOP No. CA-Q-S-001 (*Solvent & Acid Lot Testing & Approval*) and TestAmerica Edison SOP No. ED-GEN-023 (*Bulk Solvent Testing and Approval*).

7.1.1 Methylene Chloride – JT Baker Ultra-Resi 9254-03 or equivalent

7.1.2 Acetone, J.T. Baker Ultra-Resi 9264-03 or equivalent

7.1.3 Methanol, J.T. Baker, Pesticide Grade, 9077-02 or equivalent

7.1.4 Concentrated Sulfuric Acid - Baxter 2876-9 or equivalent

7.1.5 Sodium Hydroxide Pellets – Baxter 7708-500NY or equivalent

7.1.6 Sodium Sulfate Crystals – Mallinckrodt MA8024-06 or equivalent (Must be baked in the muffle furnace for four hours at 400°C and serially rinsed with Methylene Chloride prior to use.)

7.1.7 Sodium Hydroxide (10 N) - Fill a precleaned 1000 ml volumetric flask with 500 mls deionized water. Weigh out 400 g NaOH pellets and dispense slowly into the flask. Stir slowly until the pellets dissolve, then add more deionized water until the 1000 ml level is

reached. Be careful as this procedure generates heat. Never add water to the reagent that is to be dissolved.

7.2 Standards

7.2.1 Most stock target analyte standard solutions are purchased as prepared solutions; other standards are prepared in the laboratory using neat compounds (see table below). Most stock solutions are diluted (in volumetric glassware) to working concentration using methylene chloride as the diluent as described below. Stock standards of similar quality from other suppliers may be substituted as required.

NOTE: The standards listed here are used as calibration standards and spiking standards. Separate source calibration verification standards are addressed in the analytical SOPs.

Standard Name	Concentration	Vendor	Catalog #
8270 List 1/ Std#1 MegaMix	500/1000/2000pmm	RESTEK	570666
8270 List 1/ Std#10Benzoic Acid	2000ppm	RESTEK	569731
8270 List 1/ Std#9	2000ppm	RESTEK	569730
Custom SVO Mix	2000ppm	SPEX	SVO-TANJ-16-5
Bisphenol A	1000ppm	SPEX	S-509-MC
8270 List 1/ Std#11	2000ppm	RESTEK	569732
8270 Surrogate Standard	5000 ppm	RESTEK	567685
Aromatic Amines Custom Mix	2000 ppm	Supelco	21467482

7.2.1.1. **Spiking Standard:** For use in spiking aqueous samples including TCLP/SPLP leach being prepared for BNA analysis by SW846 Method 8270. Prepare the second source spiking solution for MS/MSD/Blank Spike (LCS) as detailed in the following table using the applicable standards listed in 7.2.1. Use volumetric glassware. Dilute to the volume marker with methanol.

Standard Name	Concentration	Volume of Standard added to final volume of 200ml (solvent)	Final Concentration
8270 List 1/ Std#1 MegaMix	500/1000/2000pmm	20 ml (methanol)	50/100/200 ppm
8270 List 1/ Std#10	2000ppm	10 ml (methanol)	100 ppm
8270 List 1/	2000ppm	10 ml (methanol)	100 ppm

Standard Name	Concentration	Volume of Standard added to final volume of 200ml (solvent)	Final Concentration
Std#9			
Custom SVO Mix-SPEX	2000ppm	10 ml (methanol)	100 ppm
Bisphenol A-SPEX	1000ppm	10ml (methanol)	50ppm
8270 List 1/ Std#11	2000ppm	20ul (methanol) 100ul(methanol)	Used neat for LVI Used neat for non-LVI
Aromatic Amines Custom Mix**	2000ppm	10 ml (methylene chloride)	100 ppm

*** As needed based on clients requirement.*

Note: Neat spiking/surrogate standards are stored per vendor requirements (either room temp or 4 deg C, as appropriate). All prepared standard solutions are refrigerated at 4 deg C.

7.2.2 **8270 Surrogate Standard Spiking Solution**: For use in spiking all blanks, samples and associated QC prior to extraction. Prepare as detailed in the following table using the applicable standards listed in 7.2.1. Use volumetric glassware. Dilute to the volume marker with methanol.

Standard Name	Concentration	Volume of Standard added to final volume of 1000 ml methanol	Final Concentration
8270 Surrogate Standard	5000 ppm	20 ml	100 ppm

7.2.3 Internal standard is prepared added by the analytical department. For details see analytical SOPs No. ED-MSS-002 (SW846 8270C) or ED-MSS-009 (SW846 8270D).

7.2.4 The preparation of all standards must be documented in TestAmerica LIMS (TALS) or a standard preparation logbook. Information such as standard supplier, lot number, original concentration, and a description of how standard was prepared are required along with a laboratory lot number, analyst's initials, date prepared and verification signature. Standards must be made every 6 months or sooner if signs of degradation appear.

8.0 Sample Collection, Preservation, Shipment and Storage

Sample container, preservation techniques and holding times may vary and are dependent on sample matrix, method of choice, regulatory compliance, and/or specific contract or client requests. Listed below are the holding times and the references that include preservation requirements.

8.1 All samples must be stored at 4ºC (± 2ºC) upon receipt.

8.2 Sample Extract Storage. Samples extracts must be protected from light and refrigerated at 4ºC (± 2ºC) from time of extraction until analysis.

8.3 Sample Extract Holding Time. All sample extracts must be analyzed within 40 days of extraction.

Matrix	Sample Container	Min. Sample Size	Preservation	Holding Time	Reference
Water or Leachate	250ml Amber (RVE)/ Amber glass, 1L	250ml-RVE/ 1000 ml	Cool 4 ± 2°C	7 days to extraction; Analyze within 40 days of extraction	EPA Method SW846 8270C

9.0 Quality Control

9.1. **Sample QC** - The following quality control samples are prepared with each batch of samples. Refer to TestAmerica Edison analytical SOPs No. ED-MSS-002 (SW846 8270C) or ED-MSS-009 (SW846 8270D) for details on analysis and evaluation of these QC elements:

Quality Controls	Frequency	Control Limit
Method Blank (MB)	1 in 20 or fewer samples	< Rpt. Limit
Laboratory Control Sample (LCS)[1]	1 in 20 or fewer samples	Statistical Limits [4]
Matrix Spike (MS)[2]	1 in 20 or fewer samples	Statistical Limits [4]
MS Duplicate (MSD)[2]	1 in 20 or fewer samples	Statistical Limits [4]
Surrogates	every sample[3]	Statistical Limits [4]

[1] LCS Duplicate (LCD) is performed only when insufficient sample is available for the MS/MSD or when requested by the client/project/contract.
[2] The sample selection for MS/MSD are randomly selected, unless specifically requested by a client….predetermined by the extraction lab.
[3] Analytical and QC samples (MB, LCS, MS/MSD, Method Blank)
[4] Statistical control limits are updated annually and are updated into lab reporting software.

9.1.1. **Method blanks** are extracted with every sample batch on each day that samples are extracted.

9.1.2. **Matrix Spike (MS)/Matrix Spike Duplicate (MSD):** A Matrix Spike/Matrix Spike Duplicate (MS/MSD) pair is extracted and analyzed with every batch of 20 environmental samples. (Note: an

LCS/LCSD may be substituted for the MS/MSD if insufficient client environmental sample volume is available).

9.1.3. Laboratory Control Sample (LCS)/Laboratory Control Sample Duplicate (LCSD): A Laboratory Control Sample (LCS) (aka blank spike) must be extracted and analyzed with each batch of 20 environmental samples.

9.1.3.1 A Laboratory Control Sample Duplicate (LCSD) is extracted and analyzed only when insufficient client sample is available for preparation of an MS/MSD pair. The LCS/LSCD is evaluated in the same manner as the MS/MSD (see Section 9.1.2)

9.1.4. Surrogate Standards: All samples, blanks and QC samples are spiked with a six (6) component surrogate standard mix (see Section 7.2.2).

10.0 Procedure

NOTE: The sample preparation procedure for aqueous samples (Section 10.1) contains two options: reduced volume extraction (RVE) (250ml) for large volume injection (LVI) analysis and large volume extraction (1000ml),

10.1. Sample Preparation for Aqueous/ Leachates Samples

10.1.1. Rinse the required number of 500-ml separatory funnels (when RVE is required) or 2000-ml separatory funnels (when large volume extraction is required) and 250-ml Erlenmeyer flasks twice with a 1:1 mixture of Methylene Chloride:Acetone and once with Methylene Chloride.

10.1.2. Place a small amount of glass wool into a 100-mm funnel and fill with pre-baked sodium sulfate crystals. Rinse three (3) times with Methylene Chloride. Also rinse the outside of the funnel stem three (3) times with Methylene Chloride (since the stem is likely to come into contact with the extract). Allow time for all of the rinsate to drain out of the funnel into a waste container.

10.1.3. Record the lab sample numbers on the separatory funnels with red wax pencil.

10.1.4. Make up tags with the following information and place on a 250ml Erlenmeyer flask:

BNAs		**BNs**	**AEs**
Acid Fraction	BN Fraction		
Sample Number	Sample Number	Sample Number	Sample Number
Fraction-Matrix	Fraction-Matrix	Fraction-Matrix	Fraction-Matrix
Date of Extraction	Date of Extraction	Date of Extraction	Date of Extraction

10.1.5. Place the 100ml funnel containing rinsed sodium sulfate crystals onto the flask.

10.1.6. Mark the fluid level on the sample bottles with a black Sharpie. Pour each sample into its corresponding separatory funnel. Fill each sample bottle to the black line with tap water. Pour this into the graduated cylinder used for measuring sample volumes. Note the volume for each sample on the Organic Extraction Data Sheet.

10.1.7. Rinse out a graduated cylinder with lab reagent water two to three times. Using the graduated cylinder obtain 250 ml when RVE is used (or 1000-ml when large volume is required) of lab reagent water from the Millipore filtering apparatus located in the Wet Chemistry laboratory for each of the method blank and the laboratory control sample (LCS) (aka blank spike).

10.1.8. Pour each the reagent water for the method blank and LCS into the corresponding separatory funnels.

10.1.9. Rinse syringes eight (8) to ten (10) times with Methylene Chloride.

10.1.10. If you are performing BNAs add 0.2 ml when RVE is used (or 1.0 ml when large volume is required) of Surrogate Standard Spiking Solution (see Section 7.2.2) to each sample/QC sample and proceed to Section 10.1.11. If you are performing *BN only* extraction, add 0.2 ml when RVE is used (or 1.0 ml when large volume is required) of Surrogate Standard Spiking Solution (see Section 7.2.2) to each sample/QC sample and proceed to Section 10.1.20. If you are performing *AE only* extraction add 0.2 ml when RVE is used (or 1.0 ml when large volume is required) of Surrogate Standard Spiking Solution (see Section 7.2.2) to each sample/QC sample and proceed to Section 10.1.11.

10.1.10.1. If extracting QC samples (MS, MSD, LCS or LCSD), add 0.2 ml when RVE is used (or 1.0 ml when large volume is required of the Spiking (see Section 7.2.1.1) to the appropriate separatory funnel. Note: When spiking the samples, make sure to get all bubbles out of the syringe. In addition, hold the syringe just above the level of the liquid when adding the spike. Do not touch the tip of the syringe to the liquid or the side of the separatory funnel.

10.1.11. Add concentrated sulfuric acid to each sample to adjust the pH to <2. (Usually you only need to add 1ml using small disposable Pasteur pipette). Note: pH adjustments must be documented in the extraction log.

10.1.12. Shake each sample for a short time and check pH using pH paper. The pH must be 2 or less. If the pH has not been lowered sufficiently, add more acid.

10.1.13. Add 15 ml of Methylene Chloride when RVE is used (or 60 ml when large volume is required) to each sample bottle.

10.1.14. Swirl the bottle and add 15 ml of Methylene Chloride when RVE is used (or 60 ml when large volume is required) to its corresponding separatory funnel.

10.1.15. After making sure the funnels are properly secured, start the rotators. Stop the rotator and vent the funnels after about 10 seconds. Resume rotating for 2 minutes.

10.1.16. Stop the rotation and allow the sample to settle.

10.1.17. Drain the bottom (organic) layer from the separatory funnel into the funnel/Erlenmeyer apparatus.

10.1.18. Repeat steps 10.1.13 through 10.1.17 twice, adding the Methylene Chloride directly to separatory, rather than rinsing the sample container as in 10.1.15.

10.1.18.1. If an emulsion forms during extraction, rinse a centrifuge tube well with methylene chloride and then drain the lower layer from the separatory funnel into the tube. Centrifuge for 3 to 5 minutes at 2000 rpm. Make sure the levels (and weights) of the samples directly opposite one another in the centrifuge are approximately equal. After the centrifuging process is finished, there will be two layers: water on top and the methylene chloride on the bottom. A 1 ml disposable pipette should be used to transfer the methylene chloride (bottom) layer from the centrifuge tube to the appropriate Erlenmeyer flask. With this method, care must be taken not to transfer the water (top) layer. The top layer that remains is poured back into the separatory funnel with the rest of the original sample.

10.1.19. If you are preparing the sample for acid extractables analysis only, you are finished and you can now discard the remaining liquid in each separatory funnel and proceed to Section 10.1.27. If you are required to extract base/neutrals, proceed with Section 10.1.20.

10.1.20. Adjust the pH of the sample to >11 by adding 10N sodium hydroxide (NaOH) to the sample in each separatory funnel. NOTE: pH adjustment must be documented in the extraction logbook.

10.1.21. Shake each separatory funnel for a short time and check pH. It should be basic, >pH 11. If the pH is not as high as it should be, add more 10N sodium hydroxide (NaOH).

10.1.22. Add 15 ml of Methylene Chloride when RVE is used (or 60 ml when large volume is required)

10.1.23. After making sure the funnels are properly secured, start the rotators. Stop the rotator and vent the funnels after about 10 seconds. Resume rotating for 2 minutes.

10.1.24. Stop the rotation and allow the sample to settle.

10.1.25. Drain the bottom (organic) layer from the separatory funnel into the funnel/Erlenmeyer apparatus.

10.1.26. Repeat steps 10.1.22 through 10.1.25 twice.

10.1.26.1. If an emulsion forms during extraction, rinse a centrifuge tube well with methylene chloride and then drain the lower layer from the separatory funnel into the tube. Centrifuge for 3 to 5 minutes at 2000 rpm. Make sure the levels (and weights) of the samples directly opposite one another in the centrifuge are approximately equal. After the centrifuging process is finished, there will be two layers: water on top and the methylene chloride on the bottom. A 1 ml disposable pipette should be used to transfer the methylene chloride (bottom) layer from the centrifuge tube to the appropriate Erlenmeyer flask. With this method, care must be taken not to transfer the water (top) layer. The top layer that remains is poured back into the separatory funnel with the rest of the original sample.

10.1.27. Pour the entire methylene chloride extract from the Erlenmeyer flask into a KD concentration tube apparatus (pre-rinsed three times with acetone). Rinse the Erlenmeyer flask from which the sample came twice with methylene chloride and pour both rinsates into the KD apparatus.

10.1.28. Attach Snyder column (pre-rinsed three times with acetone) to the top of the KD apparatus.

10.1.29. For RVE extractions blow the extract directly down to a final volume of 2-ml on the N-Evap. Remove the Snyder column from the top of the KD flask. Remove the blue taper clamp from the ground glass joint and dry the exterior with a Kimwipe. Transfer the concentrator tube with the 5ml extract to the N-Evap and “blow down” the extract until the volume is 2.0 ml.

10.1.30. For large volume (1000 ml) extractions concentrate the extract to approximately 5-ml in Steam Bath Remove the Snyder column from the top of the KD flask. Remove the blue taper clamp from the ground glass joint and dry the exterior with a Kimwipe. Transfer the concentrator tube with the 5ml extract to the N-Evap and “blow down” the extract until the volume is 1.0 ml. Bring the volume of each extract up to 2ml with methylene chloride

10.1.31. Split each extract into two (2) -1ml aliquots and transfer each aliquot to a separate 2ml amber screw cap vial with Teflon liner. Label one vial as 'SIM" and the second vial as 'Total'. Transfer custody of the vials to the Semivolatile GC/MS laboratory for analysis (see TestAmerica Edison analytical SOPs No. ED-MSS-002 (SW846 8270C*)* or ED-MSS-009 (SW846 8270D).

10.2. Required Documentation:

The organic prep technician is responsible for completing the following items.

10.2.1. The Standards Prep Logbook or TALS Reagent Database must be completed in full with the required information whenever standards are logged and/or prepared.

10.2.2. Each time an extraction is performed, the applicable TALS data record must be completed and reviewed the Organic Prep Supervisor or designee. Lot numbers of all reagents and solvents used or added to samples during preparation must be documented in the database.

10.2.3. Each sample extracted must be included in a batch and be recorded in the TALS database.

10.2.4. Following the extraction procedure, the technician must complete all TALS data fields pertaining to the samples extracted.

11.0. Calculations/Data Reduction

n/a

12.0. Method Performance

12.1. Method Detection Limit Study (MDL)

The method detection limit (MDL) is the lowest concentration that can be detected for a given analytical method and sample matrix with 99% confidence that the analyte is present. The MDL is determined according to the laboratory's MDL procedure in Section 19 (Test Methods and Method Validation) of TestAmerica Edison's Quality Assurance Manual (ED-QA-LQM). MDLs reflect a calculated (statistical) value determined under ideal laboratory conditions in a clean matrix, and may not be achievable in all environmental matrices. The laboratory maintains MDL studies for analyses performed; these are verified at least annually unless method requirements require a greater frequency.

12.2. Demonstration of Capabilities

For demonstration of capability procedure refer to Section 19 in the most current revision of TestAmerica Edison's Quality Assurance Manual (ED-QA-LQM).

12.3. Training Requirements

Refer to TestAmerica SOP No. ED-GEN-022, *Training*, for the laboratory's training program.

13.0. Pollution Control

13.1 It is TestAmerica's policy to evaluate each method and look for opportunities to minimize waste generated (i.e., examine recycling options, ordering chemicals based on quantity needed, preparation of reagents based on anticipated usage and reagent stability). Employees must abide by the policies in Section 13 of the Corporate Environmental Health and Safety Manual (CW-E-M-001) for "Waste Management and Pollution Prevention."

14.0. Waste Management

14.1 Waste management practices are conducted consistent with all applicable rules and regulations. Excess reagents, samples and method process wastes are disposed of in an accepted manner. Waste description rules and land disposal restrictions are followed. Waste disposal procedures are incorporated by reference to TestAmerica Edison SOPs Nos. ED-SPM-007 (*Disposal of Samples and Associated Laboratory Waste, current revision)* and ED-SPM-008 (*Laboratory Waste Disposal Procedures, current revision).* The following waste streams are produced when this method is carried out:

- Extractions Waste water. This material is created when 50% Acetone and 50% Methylene Chloride are added to 1 liter of sample water. The water is shaken with the solvent. The solvent is collected with the compounds of interest and the water is discarded into the Extractions Waste Water drum. This drum is removed to the walk-in hood in the waste room. A ½ inch PVC pipe is inserted into the bung hole of the drum and air is passed through the solution over night. The solution is then transferred into the first drum of the neutralization system and neutralized to a pH of 6 – 9. This solution is discharged into the municipal sewer system.

- Mixed Solvent Waste. This material is collected from rinsing and other processes into 5 gallon solvent cans as satellite accumulation. These cans are emptied every 24 hours into a steel drum in the waste room. This drum is kept in the walk in hood until it is full. The full drum is then removed from the hood and placed on secondary containment in the waste room.
  Teris Profile Number: 50016624
  Onyx Profile WIP Number: 545240

- Waste sodium sulfate. This material is collected from various methods which require the removal of water from solvent which carries the analyte (s) of interest. The solvent is passed through the sodium sulfate and the sodium sulfate plus the water is disposed of. The sodium sulfate is collected in buckets inside the hoods. The material is air dried and disposed of in the municipal waste dumpster.

- Waste sulfuric acid. This material is generated from clean up of PCB extracts for sulfur compounds. The acid is collected in satellite accumulation in the hood. The container is removed to the waste room for neutralization with 50 % sodium hydroxide (Siedler Chemical SC-1824-03), water and sodium bicarbonate (Siedler Chemical SC-0219-25). Ice is used to control temperature in the plastic drums of the neutralization system. When neutralization is complete (pH 6 -9) the material is transferred to the municipal sewer system.

15.0. References / Cross-References

15.1 United States Environmental Protection Agency, “Method SW3510C, Separatory Funnel Extraction”, Test Methods for Evaluating Solid Wastes, SW846 Third Edition, Volume 1B: Laboratory Manual, Physical/Chemical Methods, Revision 3, December 1996.

15.2 TestAmerica Edison SOP No. ED-MSS-002, *Semivolatile Organic Compounds by Gas Chromatography/Mass Spectrometry (GC/MS, SW846 Method 8270C*, current revision.

15.3 TestAmerica Edison SOP No. ED-MSS-009, *Semivolatile Organic Compounds by Gas Chromatography/Mass Spectrometry (GC/MS) SW846 Method 8270D*, current revision

15.4 TestAmerica Edison Document No. ED-QA-LQM, *Laboratory Quality Manual*, current revision.

15.5 TestAmerica Environmental Health and Safety Manual, CW-E-M-001.

15.6 TestAmerica Corporate Quality SOP No. CA-Q-S-001 (*Solvent & Acid Lot Testing & Approval*), current revision.

15.7 TestAmerica Edison SOP No. ED-GEN-023 (*Bulk Solvent Testing and Approval*), current revision.

15.8 TestAmerica Edison SOP No. ED-GEN-022, *Training,* current revision

15.9 TestAmerica Edison SOPs Nos. ED-SPM-007 (*Disposal of Samples and Associated Laboratory Waste, current revision)*

15.10 TestAmerica Edison SOP No. ED-SPM-008 (*Laboratory Waste Disposal Procedures, current revision).*

16.0. Method Modifications:

N/A

17.0. Attachments

N/A

18.0. Revision History

Revision 12, effective 03/26/2018

- Updated throughout to clarify that the lab's standard procdure is to use Reduced Volume Extraction (~250 ml initial volume) with an option to use large volume extraction (~1000 ml initial volume).

Revision 10, effective 11/29/2016:

- Sections 7.2.1 and 7.2.1.1 : updated current sources of standards.
- Section 7.2.1.1: added standards storage information as note at end of section.
- Section 8.0: added option for 250 ml amber sample containers (LVI option)

Revision 9, effective 11/21/2014:

- Section 7.2: updated current sources of all standards.
- Throughout document as required: added option for preparation of leachates by LVI.
- Section 10.1.29 through 10.1.31: clarified the concentration techniques for both full volume and LVI extracts.

Revision 8, effective 11/28/2012

- Throughout document: updated references to Lab Quality Manual section numbers.
- Added references as necessary throughout to TestAmerica Edison SOP No. ED-MSS-009 (*Semivolatile Organic Compounds by GC/MS, SW846 Method 8270C*D, current revision.
- Section 2.2 added describing option for analysis of lower initial volume for subsequent analysis using large volume injection (LVI) technique.
- Section 6.2: added 500 ml separatory funnel.
- Sections 7.2.1 and 7.2.1.1: added '5 Compound BNA Custom Mix' and 'Aromatic Amine Custom Mix' to list of standards and prep instructions table.
- Section 10.1 (Sample Prep for Aqueous Samples): revised throughout to include option for extraction of reduced aqueous volume (250ml) for subsequent analysis by LVI technique. Added note as preface to Section 10.1 alerting analyst to the two available extraction volume options (1000ml and 250ml).

Revision 7, effective 12/6/10

- Section 3: revised to reference new location for definitions

- Sections 7.2.1 and 7.2.1.1: Added 4 compounds and additional details to the description of standard preparation of the Spiking Standard.
- Section 7.2.4: added option to document standards preparation within TALS rather than a laboratory notebook.
- Section 10.3: revised to include TALS as the main repository for raw data associated with sample prep.

Revision 6, effective November, 2008

- Revised SOP format in accordance with TestAmerica Corporate Quality SOP No. CW-QS-002 (Rev 0) Writing a Standard Operating Procedure (SOP).
- Revised title to include 'Leachates'.
- Section 1.3: Added reference to Quality Assurance Manual for method modifications.
- Section 3: revised to reference new location for definitions.
- Section 5: Revised to include most up to date corporate health and safety references and information.
- Section 7: added details of the solvent testing and approval program.
- Section 7.2.1: Added additional details to the description of standards and the preparation of the Spiking Standards and Surrogate Standards. Removed references to the Internal Standard which is now added by the analytical group and is discussed in TestAmerica Edison SOP No. ED-MSS-002, *Semivolatile Organic Compounds by Gas Chromatography/Mass Spectrometry (GC/MS), SW846 Method 8270C*, current revision.
- Section 9: Quality Control: added additional details to the discussion of the various QC sample types
- Section 10: Revised and clarified to reflect current procedures. Removed reference to internal standard addition (now completed by analytical group).
- Section 11: Removed reference to Organic Calculation SOP.
- Section 12: updated and revised the MDL requirements to reflect text in the current revision of the TestAmerica Edison Laboratory Quality Manual (LQM).
- Section 15: References: Expanded to include more specific SOP references
- Section 16: Added Section 16 (Method Modifications).
- Section 18: Added this Revision History section

Title: Analysis of Ammonia in Water, Wastewater and Soil- Automated by Methods EPA 350.1, SM 4500-NH3 B plus G-11, SM 4500-NH3 B plus H-11, and QuikChem Method 10-107-06-1-C rev. 05-07-87

Once printed, this is considered an uncontrolled document

Approvals (Signature/Date):

Jasmine Parillo — 04/17/2018	Dan Helfrich — 04/17/2018
Jasmine Parillo — Date	Dan Helfrich — Date
Department Manager	Health & Safety Manager /Coordinator
Carl Armbruster — 04/17/2018	Mark Acierno — 04/17/2018
Carl Armbruster — Date	Mark Acierno — Date
Quality Assurance Manager	Laboratory Director

Copyright Information:
This documentation has been prepared by TestAmerica Laboratories, Inc. and its affiliates ("TestAmerica"), solely for their own use and the use of their customers in evaluating their qualifications and capabilities in connection with a particular project. The user of this document agrees by its acceptance to return it to TestAmerica upon request and not to reproduce, copy, lend, or otherwise disclose its contents, directly or indirectly, and not to use it for any other purpose other than that for which it was specifically provided. The user also agrees that where consultants or other outside parties are involved in the evaluation process, access to these documents shall not be given to said parties unless those parties also specifically agree to these conditions.

THIS DOCUMENT CONTAINS VALUABLE CONFIDENTIAL AND PROPRIETARY INFORMATION. DISCLOSURE, USE OR REPRODUCTION OF THESE MATERIALS WITHOUT THE WRITTEN AUTHORIZATION OF TESTAMERICA IS STRICTLY PROHIBITED. THIS UNPUBLISHED WORK BY TESTAMERICA IS PROTECTED BY STATE AND FEDERAL LAW OF THE UNITED STATES. IF PUBLICATION OF THIS WORK SHOULD OCCUR THE FOLLOWING NOTICE SHALL APPLY:

©COPYRIGHT 2018 TESTAMERICA LABORATORIES, INC. ALL RIGHTS RESERVED.

1.0 Scope and Application

1.1. Methods EPA 350.1, SM 4500 NH3-B +H-11, SM 4500 NH3-B+G-11 and QuikChem Method 10-107-06-1-C rev. 05-07-87 are described in this SOP. The SOP is applicable to the analysis of ammonia in waters, wastewaters, and soils.

1.2. The automated method has a detector that is sensitive to approximately 0.02 mg N/L as NH_3. The laboratory's reporting limit for Ammonia is 0.10 mg N/L for aqueous samples and 8.0 mg N/kg for soil samples.

1.3. On occasion clients may request modifications to this SOP. These modifications are handled following the procedures outlined in Sections 7 (Review of Work Request) and 19 (Test Methods and Method Validation) of TestAmerica Edison's Quality Assurance Manual (ED-QA-LQM).

2.0 Summary of Method

2.1. Ammonia reacts with alkaline phenol and with sodium hypochlorite to form indophenol blue, which is proportional to the amount of ammonia. Samples are distilled at reduced volume via the EASY-dist distillation apparatus and the sample absorbances are measured at 630 nm via the Lachat instrument.

3.0 Definitions

For a complete list of definitions refer to Appendix 2 in the most current revision of TestAmerica Edison's Quality Assurance Manual (ED-QA-LQM).

4.0 Interferences

4.1. All samples must be distilled prior to analysis. Interferences are reduced or eliminated by using the distillation procedure.

4.2. Alkalinity over 500mg/L, acidity over 100mg/L interfere. These are removed by the distillation step.

4.3. Calcium and Magnesium ions in sufficient quantities can cause precipitation during analysis; this can be reduced by adding EDTA and sodium potassium tartrate.

4.4. Color, turbidity, and certain organic species may cause interference. Turbidity may be removed with manual filtration.

4.5. Residual chlorine must be removed by pretreatment of sample with sodium thiosulfate before distillation.

5.0 Safety

Employees must abide by the policies and procedures in the Corporate Environmental Health and Safety Manual (CW-E-M-001) and this document. This procedure may involve hazardous material, operations and equipment. This SOP does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of the method to follow appropriate safety, waste disposal and health practices under the

assumption that all samples and reagents are potentially hazardous. Safety glasses, gloves, lab coats and closed-toe, nonabsorbent shoes are a minimum.

5.1. Specific Safety Concerns or Requirements

Sodium Nitroferricyanide will generate Hydrogen Cyanide (HCN) gas if combined with strong acids. Inhalation of CN gas can cause irritation, dizziness, nausea, unconsciousness and potentially death.

5.2. Primary Materials Used

The following is a list of the materials used in this method, which have a serious or significant hazard rating. **Note: This list does not include all materials used in the method. The table contains a summary of the primary hazards listed in the MSDS for each of the materials listed in the table.** A complete list of materials used in the method can be found in the reagents and materials section. Employees must review the information in the MSDS for each material before using it for the first time or when there are major changes to the MSDS.

Material (1)	Hazards	Exposure Limit (2)	Signs and symptoms of exposure
Phenol	Corrosive	5 ppm-TWA	Breathing vapor, dust or mist results in digestive disturbances. Will irritate, possibly burn respiratory tract. Rapidly absorbed through the skin with systemic poisoning effects to follow. Discoloration and severe burns may occur, but may be disguised by a loss in pain sensation. Eye burns with redness, pain, blurred vision may occur. May cause severe damage and blindness.
Sodium Hydroxide	Corrosive	2 Mg/M3-Ceiling	Severe irritant. Effects from inhalation of dust or mist vary from mild irritation to serious damage of the upper respiratory tract, depending on severity of exposure. Symptoms may include sneezing, sore throat or runny nose. Contact with skin can cause irritation or severe burns and scarring with greater exposures. Causes irritation of eyes, and with greater exposures it can cause burns that may result in permanent impairment of vision, even blindness.
Sodium Nitroferri-cyanide	Poison	5 mg/m^3 as HCN gas	This material may cause irritation if it comes into the contact with the skin. The materials will give off HCN gas if combined with strong acids. Inhalation of HCN gas can cause irritation, dizziness, nausea, unconsciousness and potentially death.

Material (1)	Hazards	Exposure Limit (2)	Signs and symptoms of exposure
Sulfuric Acid	Corrosive Oxidizer Dehydrator Poison Carcinogen	1 Mg/M3-TWA	Inhalation produces damaging effects on the mucous membranes and upper respiratory tract. Symptoms may include irritation of the nose and throat, and labored breathing. Symptoms of redness, pain, and severe burn can occur. Contact can cause blurred vision, redness, pain and severe tissue burns. Can cause blindness.
1 – Always add acid to water to prevent violent reactions.			
2 – Exposure limit refers to the OSHA regulatory exposure limit.			

6.0 Equipment and Supplies

6.1. Instrumentation

- Autoanalyzer: Lachat QuikChem 8000 equipped with:
  - Autosampler
  - Multichannel proportioning pump
  - Reaction Manifold
  - Colorimetric detector
  - Data System
  - Heating Unit
- EASY-Dist distillation apparatus with all associated glassware (Westco Scientific Instruments)
- pH meter- Orion A211
- Analytical balance

6.2. Supplies

- Boiling tube/stopper
- Mini-Allihn condenser/condenser stem
- Sloped T-joint
- Graduated cylinder, 50 ml
- Magnetic stirrer
- Automatic pipettors or volumetric pipets
- Class A volumetric flasks
- Micro-porous boiling stones
- Size 19/22 clips
- 120 ml snap seal sample cups
- Specimen cups
- Transfer pipets
- Potassium Iodide paper

7.0. Reagents and Standards

7.1. Reagents

7.1.1. Ammonia free water.

7.1.2. Sodium Hydroxide, 15N: Dissolve 300g of NaOH in 500ml of deionized water. Prepare fresh every six months and store at room temperature.

7.1.3. Sodium Hydroxide, 11N: Dissolve 220g of NaOH in 500ml of deionized water. Prepare fresh every six months and store at room temperature.

7.1.4. Sodium Hydroxide, 1N: Dissolve 40 g of NaOH in one liter of deionized water. Prepare fresh every six months and store at room temperature.

7.1.5. Sodium Hydroxide, 0.25N: Dissolve 10 g of NaOH in one liter of deionized water. Prepare fresh every six months and store at room temperature.

7.1.6. Sodium Hydroxide, 0.1N: Take 100ml of 1N NaOH (Sec. 7.1.4) and dilute up to 1L with deionized water. Prepare fresh every six months and store at room temperature.

7.1.7. Sodium Hydroxide, 0.02N: Take 20ml of 0.1N NaOH (Sec. 7.1.6) and dilute up to 100ml with deionized water. Prepare fresh every six months and store at room temperature.

7.1.8. Borate Buffer: Dissolve 9.5g of sodium borate decahydrate ($Na_2B_4O_7*10H_2O$) in a 1L volumetric flask containing 800mls of deionized water. Add 88mls of 0.1 N NaOH and dilute to mark with deionized water. Prepare fresh every six months and store at room temperature.

7.1.9. 0.04N H_2SO_4: Dilute 1.12 ml of conc. H_2SO_4 to one liter of deionized water. Prepare fresh every 6 months and store at room temperature.

7.1.10. Dechlorinating agents to remove residual chlorine prior to distillation. Prepare fresh every six months; store at room temperature.

- Sodium Thiosulfate: Dissolve 3.5g $Na_2S_2O_3*5H_2O$ in reagent water and dilute to 1L. Prepare fresh every 6 months and store at room temperature. One mL of this solution will remove 1 mg/L of residual chlorine in 500 mL of sample.
- Sodium sulfite: Dissolve 0.9 g Na2SO3 in reagent water and dilute to 1 L. One mL removes 1 mg/L Cl per 500 mL of sample.

7.1.11. Sodium Phenolate: In a 1L volumetric flask, dissolve 83 g of crystalline phenol in approximately 600ml deionized water. While stirring, slowly add 32 g sodium hydroxide. Cool, dilute to mark, and invert three times. Prepare fresh every month and store at room temperature. Note: Use proper ventilation when preparing this solution.

7.1.12. Sodium Hypochlorite: In a 500ml volumetric flask, dilute 250 ml of 5.65% sodium hypochlorite to the mark with deionized water. Prepare fresh every month and store at room temperature.

7.1.13. Buffer: In 1 L volumetric flask, dissolve 50.0 g disodium ethylenediamine tetraacetate (Na2EDTA) and 5.5 g of Sodium hydroxide in about 900 ml of deionized water. Dilute to mark and invert three times. Prepare fresh every month and store at room temperature.

7.1.14. Sodium Nitroprusside (Nitroferricyanide): In a 1L volumetric flask, dissolve 3.50 g sodium nitroferricyanide and dilute to mark with deionized water. Prepare fresh every month and store at room temperature.

7.2. Standards

7.2.1. Ammonia Primary Stock Standard (1000mg N/L as NH3): pre-made purchased commercially. See manufacturer's instructions for stability and storage information.

7.2.2. Ammonia Secondary Stock Standard (1000mg N/L as NH3) - pre-made purchased commercially See manufacturer's instructions for stability and storage information. The secondary stock must be from a different source (i.e. different manufacturer or different lot #) than the primary source.

To prepare in-house: In a 1L volumetric flask, dissolve 3.819g of ammonium chloride (NH4Cl) that has been dried for two hours at 110 deg C in about 800ml of deionized water. Dilute to mark and invert three times. The secondary stock must be from a different source than the primary source. Prepare fresh every six months and store at 4 deg C.

7.2.3. Ammonia Primary Intermediate Working Standard (100 mg/L): Add 10 ml of the Ammonia Primary stock standard (Sec. 7.2.1) into 100 ml volumetric flask and bring the volume up to the mark with deionized water. Prepare fresh every six months or when QC checks are outside of acceptable limits, whichever comes first, store at 4 deg C.

7.2.4. Ammonia Primary Intermediate Working Standard (10 mg/L): Add 1.0 ml of the Ammonia Primary Stock Standard (Sec. 7.2.1) and 12.5 ml of 0.04N H_2SO_4 (Sec. 7.1.9) into 100 ml volumetric flask and bring up to volume with deionized water. Prepare fresh each time the calibration standards are prepared.

8.0. Sample Collection, Preservation, Shipment and Storage

Sample container, preservation techniques and holding times may vary and are dependent on sample matrix, method of choice, regulatory compliance, and/or specific contract or client requests. Listed below are the holding times and the references that include preservation requirements.

Matrix	Sample Container	Min. Sample Size	Preservation	Holding Time [1]	Reference

Waters	Plastic, glass	50 mLs	H_2SO_4 pH < 2; Cool 4 ±2°C	28 Days	40 CFR Part 136.3
Soils	Glass	5 grams	Cool 4 ±2°C	28 Days	N/A

[1] Inclusive of digestion and analysis.

9.0 Quality Control

9.1 Sample QC: The following quality control samples are prepared daily or with each batch of 20 samples or fewer, whichever is more frequent. Water and soil samples are separated into QC batches.

9.1.1. Method Blank: A blank is set-up each time samples are distilled. The blank consists of ammonia free water and pH adjusted to 9.5. The results must be below the reporting limit or the batch must be redistilled and reanalyzed.

9.1.2. Laboratory Control Sample (LCS), 1.0 mg/L: An LCS is set up daily or with each batch of 20 samples or fewer, whichever is more frequent. Add 0.50 ml of the 100 mg/L Ammonia Primary Intermediate Working Standard (Sec. 7.2.3) into 50 ml deionized water that has been pH adjusted to 9.5. The results must be within laboratory generated control limits, if not, all samples must be re-distilled and re-analyzed.

9.1.3. Matrix Spike/Matrix Spike Duplicate (MS/MSD), 1.0 mg/L: MS/MSD is set up daily or with each batch of 20 samples or fewer, whichever is more frequent. Two portions of the same sample (matrix spike and matrix spike duplicate) are spiked with 50 ug of NH3. Spike 0.50 ml of the 100 mg/L working standard (Sec. 7.2.3) into the 50 ml sample that has been pH adjusted to 9.5. The recovery and relative percent difference must be within laboratory generated control limits.

9.2 Instrument QC

9.2.1 Initial Calibration Verification (ICV), 2.0 mg/L: The curve must be verified by analyzing an Initial Calibration Verification (ICV) solution at the midpoint of the calibration range. Prepare the solution daily by adding 0.20 ml of the 1000 mg/L secondary stock (Sec. 7.2.2) into 100 ml volumetric flask containing 12.5 ml of 0.04N H2SO4 and brought up to volume with deionized water. The value obtained must not differ from the true value by more than 10%. If it does the problem must be corrected, the instrument recalibrated, and the ICV reanalyzed.

9.2.2 Continuing Calibration Verification (CCV), 2.0 mg/L: The validity of the calibration curve must be verified periodically during an analysis. Prepare the solution daily and the same way as the ICV solution. A Continuing Calibration Verification (CCV) must be analyzed following every ten sample analyses. “Samples” include matrix spike, matrix spike duplicate, method blank, LCS, and environmental samples. The value obtained for the CCV must not differ from the true value by more than 10%. If it does, the

problem must be corrected and the previous ten samples reanalyzed following the last good calibration verification.

9.2.3 Initial and Continuing Calibration Blank (ICB/CCB): Following each calibration verification, a calibration blank must be analyzed. Add 12.5 ml of 0.04N H2SO4 to a 100 ml volumetric flask and dilute to volume with deionized water. Use this solution for the blank. The results of this analysis must fall below the reporting limit if not, all samples following the last acceptable calibration blank must be re-analyzed.

10.0. Procedure

10.1. Sample Preparation

10.1.1. Boiling tubes must be treated once a month to verify the absence of contamination. Add 75 ml of deionized water to the boiling tubes along with boiling chips that have been treated with dilute NaOH. Distill and then analyze on Lachat. Results should be less than the reporting limit.

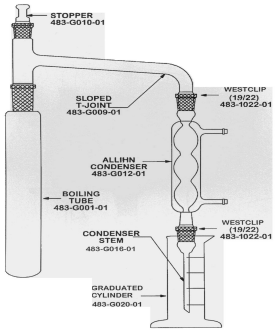


Figure 1: EASY-Dist Ammonia Distillation Apparatus

10.1.2. Distillation Procedure: WATER SAMPLES

10.1.2.1. Sample pretreatment:

10.1.2.1.1. Measure the presence of residual chlorine in each sample by placing a drop or two of sample on a strip of Potassium Iodide paper. The presence of residual chlorine will turn the KI paper blue. Record 'Y' or 'N' under column labeled 'Res Chlor Pres?' in TALS prep batch.

10.1.2.1.2. If residual chlorine is present in the sample, add either sodium thiosulfate solution or Sodium Sulfite (Sec. 7.1.10). One ml of this solution will remove 1mg/L of residual chlorine in 500ml of sample.

10.1.2.1.3. Record under sample 'Notes' that samples were treated prior to distillation. Document Reagent # of the dechlorinating agent in batch information.

10.1.2.2. Pour out 50ml of sample into a specimen cup and add 2.5 ml of the borate buffer. Adjust the pH to 9.5 using the appropriate NaOH concentrations: 15 N NaOH, 11N NaOH, 1N NaOH, 0.25N NaOH, or 0.02N NaOH.

10.1.2.3. Record final pH in TALS worksheet (TALS Method SM4500NH3_B).

10.1.2.4. Add 2-3 boiling stones and assemble distillation apparatus as shown in figure 1.

10.1.2.5. Place 5.0 ml of 0.04N H_2SO_4solution and 2.0ml of deionized water in 50 ml cylinder.

10.1.2.6. Place condenser stems in the graduated cylinder, slide the cylinder and stem under condenser and connect stem to condenser with clip. NOTE: Make sure the condenser tip is submerged in the Sulfuric acid solution. Turn on condenser water.

10.1.2.7. Heat the distillation apparatus to the following settings: Rate1: 15^0C/min; Temp1: 210^0C; Time1: 1.5Hour; Rate2: 0; Temp2: 210^0C; Time2: 0. Note: Insulate glassware if needed, a single long sheet of aluminum foil around the exposed tube and a T-joint glassware surfaces on each side is generally sufficient.

10.1.2.8. Collect 50 ml of distillate.

10.1.2.9. Discontinue heating. REMOVE THE STOPPER FROM THE TOP OF THE T- JOINT WHILE COOLING to prevent the collected distillate from being sucked back into the boiling tube.

10.1.2.10. Disconnect the stem from the condenser and remove the graduated cylinder from the apparatus.

10.1.2.11. If less than 50ml of distillate is collected, dilute the collected distillate to a final volume of 50 ml with deionized water.

10.1.3. Distillation Procedure: <u>SEDIMENT/SOLID SAMPLES</u>

10.1.3.1. Weigh 1.0g or less of wet solid sample into a 100 ml beaker. Add 50 ml DI water. Add 2.5 ml of borate buffer. Adjust pH to 9.5 by adding 1N NaOH dropwise.

10.1.3.2. Record final pH in TALS worksheet (TALS Method SM4500NH3_B).

10.1.3.3. Proceed to section 10.1.2.3 and collect 50 ml of distillate. If less than 50ml of distillate is collected, dilute the collected distillate to a final volume of 50 ml with deionized water.

10.2. Calibration and Sample Analysis:

10.2.1. Prepare calibration standards weekly as shown in the chart below and document preparation in the Standards preparation logbook. Add the respective amount of Ammonia stock standard to a 100ml volumetric flask containing 12.5ml of 0.04N H_2SO_4 and dilute to a final volume of 100ml with deionized water.

Volume (ml) of Stock standard	Conc. of NH3 (mg/L)
0.4 ml of Ammonia 1° Stock Std	4.0
0.2 ml of Ammonia 1° Stock Std	2.0
10 ml of 10 mg/L 1° Working Std	1.0
5 ml of 10 mg/L 1° Working Std	0.5
1 ml of 10 mg/L 1° Working Std	0.1
0 ml	0.0

10.2.2. A correlation coefficient of 0.995 or greater must be obtained.

10.2.3. Use TestAmerica Calibration Spreadsheet CA-Q-WI-13 to back-calculate the concentration of each calibration point. The back-calculated result and true concentrations should agree within 30% for the first calibration standard and within 10% for the rest of the standards. Submit calibration spreadsheet along with the ICAL checklist. Both documents will be scanned and filed after second level review.

10.2.4. System Start-up.

10.2.4.1. Follow manifold scheme for this method; see Attachment 1.

10.2.4.2. Check instrument settings:

- Pump speed: 35
- Cycle period: 40 s
- Load period: 15 s
- Inject period: 25 s
- Inject to start of peak period: 22 s
- Inject to end of peak period: 58 s

10.2.4.3. Turn on heating unit at 60℃ for 15 minutes before analysis.

10.2.4.4. Put all reagent lines into deionized water and turn the pump on. Check for leaks and slow flow rate.

10.2.4.5. Switch over reagent lines to proper reagent. Let reagent run through manifold for 15 minutes before analysis to allow baseline to stabilize.

10.2.5. Setup tray table with all appropriate QC checks; the instrument is programmed to analyze CCV/CCB every 10 samples and at the end of the run.

10.2.6. Place standards and samples into autosampler cups.

10.2.7. The following analytical run sequence must be used when analyzing ammonia using this SOP :

Calibration
AICV
ICB
10 Samples
ACCV1
CCB1
Repeat until complete
ACCV2
CCB2

10.2.8. Run tray.

10.2.9. Make appropriate dilutions, if necessary.

11.0. Calculations / Data Reduction

11.1. Accuracy:

$$\text{ICV / CCV, LCS \% Recovery} = \frac{\text{observed concentration}}{\text{known concentration}} \times 100$$

$$\text{MS \% Recovery} = \frac{\text{(spiked sample) - (unspiked sample)}}{\text{spiked concentration}} \times 100$$

11.2. Precision (RPD):

$$\text{Matrix Duplicate (MD)} = \frac{|\text{orig. sample value - dup. sample value}|}{[(\text{orig. sample value + dup. sample value})/2]} \times 100$$

11.3. Final results calculation in aqueous samples:

Concentration = mg/L

Analyte concentration (mg/L) on instrument; imported directly to TALS

Samples with dilution factors are calculated in TALS for final results

11.4. Concentration : mg N/ kg $= \frac{C \times V \times D \times 100}{W}$ / % solids

Where:
C = sample extract concentration on instrument (mg/L)
V = Volume of extract (50 mL)
D = Dilution factor
W = weight of sample in grams

Note: All dry weight corrections are made in TALS.

11.5. Data Reduction

11.5.1. Instrument data is imported to TALS by following these steps:

11.5.1.1. After the run has finished, click the data icon at the top of the screen. Choose the correct file. Go to 'File" and choose "Export Data' from the drop down menu. A screen will pop up, click 'ok' and then another screen will pop up, click 'ok.'

11.5.2. From the desktop, open the LIMSdata icon. Select the correct file, and then right click. Go to 'Send to-> LimsImport ->' Choose the correct method. The data has now been imported to TALS.

11.5.3. Record special notes and observations on the "worksheet" tab (i.e. sample appearance and notes on why samples were rejected).

11.5.4. Record reagent information in the prep batch information (this can be viewed on the "batch information" page).

11.5.5. All raw data is attached as a pdf file. The raw data includes the instrument report and calibration curve.

11.5.6. Analyst must fill out the Wet Chem Data Review Checklist (WI # EDS-WI-008) during the first level review. After the batch is second level reviewed, the checklist is filed in the Wet Chemistry department.

12.0. Method Performance

12.1. Method Detection Limit Study (MDL)

The method detection limit (MDL) is the lowest concentration that can be detected for a given analytical method and sample matrix with 99% confidence that the analyte is present. The MDL is determined according to the laboratory's MDL procedure in Section 19 (Test Methods and Method Validation) of TestAmerica Edison's Quality Assurance Manual (ED-QA-LQM). MDLs reflect a calculated (statistical) value determined under ideal laboratory conditions in a clean matrix, and may not be achievable in all environmental matrices. The laboratory maintains MDL studies for analyses performed; these are verified at least annually unless method requirements

require a greater frequency. MDLs are typically generated via the TestAmerica LIMS (TALS) control chart module.

12.2. Demonstration of Capabilities

For Demonstration of Capabilities (DOC) procedure refer to Section 19 in the most current revision of TestAmerica Edison's Quality Assurance Manual (ED-QA-LQM). DOCs are typically generated via the TestAmerica LIMS (TALS) control chart module.

12.3. Training Requirements

Refer to TestAmerica SOP No. ED-GEN-022, Training, for the laboratory's training program.

13.0. Pollution Control

It is TestAmerica's policy to evaluate each method and look for opportunities to minimize waste generated (i.e., examine recycling options, ordering chemicals based on quantity needed, preparation of reagents based on anticipated usage and reagent stability). Employees must abide by the policies in Section 13 of the Corporate Environmental Health and Safety Manual (CW-E-M-001) for "Waste Management and Pollution Prevention."

14.0. Waste Management

14.1. Waste management practices are conducted consistent with all applicable rules and regulations. Excess reagents, samples and method process wastes are disposed of in an accepted manner. Waste description rules and land disposal restrictions are followed. Waste disposal procedures are incorporated by reference to TestAmerica SOP No. ED-SPM-008 (Laboratory Waste Disposal Procedures).

14.2. There are no special waste streams produced for this method.

15.0. References / Cross-References

15.1. Methods for Chemical Analysis of Water and Wastes, EMSL-Cincinnati, EPA/600/4-79-020, March 1983 and 1979; Test Methods – 350.1.

15.2. Standard Methods for the Examination of Water and Wastewater, 22th Edition, American Public Health Association, American Washington, DC, 2012, SM 4500 B+H (Editorial Revisions, 2011).

15.3. Standard Methods for the Examination of Water and Wastewater, 22th Edition, American Public Health Association, American Washington, DC, 2012, SM 4500 B+G (Editorial Revisions, 2011).

15.4. QuikChem Method 10-107-06-1-C rev. 05-07-87, Determination of Ammonia (Phenolate) in Potable and Surface Waters.

15.5. TestAmerica Edison Document No. ED-QA-LQM, *Laboratory Quality Manual*, most current revision.

15.6. TestAmerica Edison SOP ED-GEN-022, *Training*, most current revision.

15.7. TestAmerica Edison SOP No. ED-SPM-008, *Laboratory Waste Disposal Procedures*, current revision.

15.8. TestAmerica Corporate Environmental Health and Safety Manual, Document No. CW-E-M-001, most current revision.

15.9. TestAmerica Edison Work Instruction # EDS-WI-008, Wet Chemistry Data Review Checklist, most current revision.

15.10. TestAmerica Calibration Spreadsheet, Work Instruction No. CA-Q-WI-013, (8/10/2011).

16.0. Method Modifications:

Item	Method No.	Modification
Sec. 10.1.2.1; Sec. 10.1.3.1	EPA 350.1; QuikChem Method 10-107-06-1-C	2.5 ml of Borate buffer is added to the sample prior to pH adjustment; this allows for a stable sample pH reading during pH adjustment.
Sec. 7.1.9	QuikChem Method 10-107-06-1-C	Preparation of 0.04N H_2SO_4 follows QuikChem Method 10-107-06-1-C; therefore, 1.12ml of conc. acid is used.
Sec 7.1.13	QuikChem Method 10-107-06-1-C	Preparation of Sodium Nitroprusside follows QuikChem Method 10-107-06-1-C; therefore, 3.5g of sodium Nitroferricyanide is used.

17.0. Attachments

Attachment 1: Manifold Diagram- Ammonia (phenolate)

18.0. Revision History

- Revision 11 dated 16 April 2018
  - Sec. 6.1: Updated pH meter model to reflect current lab equipment.
- Revision 10 dated 04 March 2016
  - Sec. 4.5: Revised to include residual chlorine as method interference.
  - Sec. 6.2: Added potassium iodide paper to the list of supplies

- Sec. 7.1.10: Added dechlorinating agents; subsequent sections adjusted accordingly.
- Sec. 10.1.2.1: Added procedure for residual chlorine check and pretreatment.

- Revision 9 dated January 2015
  - Sec. 9.1 & 9.1.3: Clarified the QC frequency for MS/MSD to daily or with each batch of 20 or fewer samples, whichever is more frequent.
  - Sec 9.1.2: Revised acceptance limits for LCS from 90-110% to laboratory generated control limits; clarified frequency to daily or with each batch of 20 or fewer samples.
  - Sec. 10.2.3: Added criteria to read back and evaluate each calibration standard using the corporate calibration spreadsheet CA-Q-WI-013 to comply with Standard Methods 4020.
  - Sec 15.10: Added applicable reference WI# CA-Q-WI-013.

- Revision 8 dated 28 June 2013

  - Throughout document: Updated Standard Methods reference to currently implemented and accredited methods, SM 4500 NH3-B +H-11 and SM 4500 NH3-B+G-11.
  - Throughout document: updated references to Lab Quality Manual section numbers as necessary.
  - Minor formatting edits throughout document
  - Sec. 9.1.2: Added procedure if LCS is outside of acceptance limits.
  - Sec. 9.2.3: Added procedure is CCB is greater than RL.
  - Sections 12.1 and 12.2: included statements referencing use of TALS control chart in the generation of MDLs and DOCs.

- Revision 7, dated 15 June 2011

  - Sec. 1.1: Added revision 05-07-87 to Lachat method number 10-107-06-1-C
  - Sec. 7.1.3: Revised reagent to sodium nitroferricyanide.
  - Sec 7.1.11: Revised the amount of NaOCl to 250 ml to comply with the method.
  - Sec 9.2.1 & 9.2.2: Added frequency for preparing the ICV and CCV.
  - Sec. 10.2.3.2: Added injection timing settings; subsequent sections adjusted accordingly.
  - Sec. 17: Added Attachment 1.

- Revision 6, dated 12/16/2010

  - SOP title: Revised to reflect added method reference for SM 4500 NH3-B+G (19th/20th Edition)
  - Section 1.1 and throughout document: Revised to reflect added method reference for SM 4500 NH3-B+G (19th/20th Edition)
  - Section 15.0: Revised to reflect added method reference for SM 4500 NH3-B+G (19th/20th Edition)

- Revision 5, dated 09/17/2010

  - Sec. 3: Updated reference for the list of definitions.
  - Sec. 6.0: Updated the list of equipment and supplies:

- Sec. 7.1.4 - 7.1.6: Added several NaOH concentrations to the list of reagents including preparation procedures, storage, and stability information.
- Sec. 7.1.9: Changed amount of H_2SO_4 added from 1.0ml to 1.12ml to reflect lab practice and comply with QuikChem Method 10-107-06-1-C.
- Sec. 7.2.3 & 7.2.4: Added stability and storage information.
- Sec 9.1.3: Revised to include the spiking concentration of the Matrix Spike QC sample.
- Sec. 9.2.3: Added preparation procedure.
- Sec 10.1.1: Clarified the frequency and procedure for treating the boiling tubes.
- Sec 10.1.2 & 10.1.3: Revised to include the addition of buffer prior to pH adjustment and recording the final pH in TALS.
- Sec. 10.1.3.3: Revised distillate amount collected from 30 ml to 50 ml to reflect actual laboratory practices.
- Sec 10.2.1: Clarified the frequency of calibration standard preparation and the spiking volume and source of the stock standard.
- Sec. 10.2.2: Revised correlation coefficient requirement from ≥0.997 to ≥0.995.
- Sec. 11.5: Updated data reduction in accordance with TALS.
- Sec 13 & 14: Revised to comply with the current Method SOP format (CW-QS-002, Writing a Standard Operating Procedure (SOP)).
- Sec 15: Added applicable references.
- Sec 16: Added method modification.

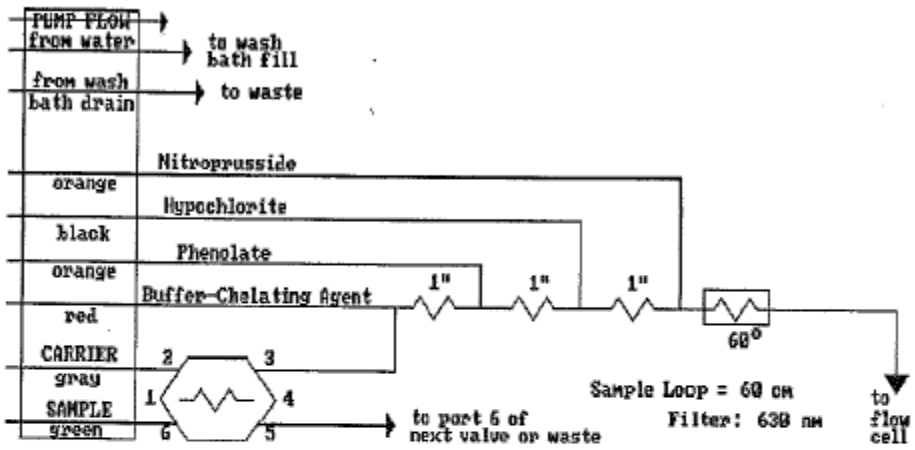
- Revision 4, dated May 30, 2008

  - Revised SOP format in accordance with TestAmerica Corporate Quality SOP No. CW-QS-002 (Rev 0) Writing a Standard Operating Procedure (SOP).
  - Section 1.1. Deleted method 350.2 reference.
  - Section 7. Added the stability and storage information of reagents and standards.
  - Section 7.2.1 Deleted the preparation instructions for the ammonia stock standard and included the purchased of stock solution from vendor.
  - Section 9. Added preparation instructions for the Sample and Instrument QCs.
  - Section 9.1.2 Revised the acceptance limits for Blank spike from a laboratory generated limits to a set limit of 90-110% to reflect actual laboratory practices.
  - Section 10.1.1 Removed the use of Nessler reagent when cleaning the glasswares.
  - Section 15. Deleted references which are not applicable (Methods 350.2 & NH3 D) and added applicable references (ED-QA-LQM, method NH3 B+H).

Attachment 1

QuikChem Method No. 10-107-06-1-C Ammonia (Phenolate)

Manifold Diagram:



CARRIER is helium degassed water.

1"	is	**70.0**	cm of tubing on a 1 in coil support
2"	is	**135**	cm of tubing on a 2 in coil support
2.5"	is	**168**	cm of tubing on a 2.5 in coil support
3"	is	**202**	cm of tubing on a 3 in coil support
4"	is	**255**	cm of tubing on a 4 in coil support
8"	is	**550**	cm of tubing on a 8 in coil support

Heated tubing is shown inside a box with the temperature next to the box. Heated tubing is **650** cm unless otherwise specified.

All manifold tubing is **0.8** mm (0.032 in) i.d. This is 5.2 uL/cm.

Notes:

MANIFOLD DIAGRAM REVISION DATE: 29 July 1986

Title: Analysis of Alkalinity in Water, Wastewater and Soil by Manual Titration or Auto-Titrator, Standard Method 2320 B-11

Once printed, this is considered an uncontrolled document

Approvals (Signature/Date):

Jasmine Parillo — 01/24/2017	Dan Helfrich — 01/24/2017
Jasmine Parillo — Date	Dan Helfrich — Date
Department Manager	Health & Safety Manager /Coordinator
Carl Armbruster — 01/24/2017	Mark Acierno — 01/24/2017
Carl Armbruster — Date	Mark Acierno — Date
Quality Assurance Manager	Laboratory Director

Copyright Information:
This documentation has been prepared by TestAmerica Laboratories, Inc. and its affiliates ("TestAmerica"), solely for their own use and the use of their customers in evaluating their qualifications and capabilities in connection with a particular project. The user of this document agrees by its acceptance to return it to TestAmerica upon request and not to reproduce, copy, lend, or otherwise disclose its contents, directly or indirectly, and not to use it for any other purpose other than that for which it was specifically provided. The user also agrees that where consultants or other outside parties are involved in the evaluation process, access to these documents shall not be given to said parties unless those parties also specifically agree to these conditions.

THIS DOCUMENT CONTAINS VALUABLE CONFIDENTIAL AND PROPRIETARY INFORMATION. DISCLOSURE, USE OR REPRODUCTION OF THESE MATERIALS WITHOUT THE WRITTEN AUTHORIZATION OF TESTAMERICA IS STRICTLY PROHIBITED. THIS UNPUBLISHED WORK BY TESTAMERICA IS PROTECTED BY STATE AND FEDERAL LAW OF THE UNITED STATES. IF PUBLICATION OF THIS WORK SHOULD OCCUR THE FOLLOWING NOTICE SHALL APPLY:

©COPYRIGHT 2017 TESTAMERICA LABORATORIES, INC. ALL RIGHTS RESERVED.

Facility Distribution No. ELECTRONIC **Distributed To:** FACILITY INTRANET

1.0 Scope and Application

1.1 Analytes, Matrix(s), and Reporting Limits

Standard Method 2320 B-11 is applicable to the determination of alkalinity in drinking, surface waters, saline waters, domestic and industrial wastes. The laboratory's reporting limit for aqueous samples is 5.0 mg/L total alkalinity as CaCO3.

Method 2320 B-11 modified is applicable to the determination of alkalinity in soil. The laboratory's reporting limit is 20 mg/kg total alkalinity as CaCO3.

The following forms of alkalinity may be determined using this method: total alkalinity, hydroxide alkalinity, carbonate alkalinity and bicarbonate alkalinity.

On occasion clients may request modifications to this SOP. These modifications are handled following the procedures outlined in Sections 7 (Review of Work Request) and 19 (Test Methods and Method Validation) of TestAmerica Edison's Quality Assurance Manual (ED-QA-LQM).

2.0 Summary of Method

The level of alkalinity contained in a sample is determined by titrating with acid to a pH of 8.3 (if applicable) and then to 4.5. The sample aliquot should be selected such that the titration volume used does not exceed 50ml.

3.0 Definitions

For a complete list of definitions refer to Appendix 2 in the most current revision of TestAmerica Edison's Quality Assurance Manual (ED-QA-LQM).

4.0 Interferences

Dissolved gas can affect alkalinity; prevent undo agitation or exposure to atmosphere as much as possible.

Soaps, oily matter, suspended solids or precipitates may coat the electrode. Allow electrode to come to equilibrium between additions of titrant and clean electrode occasionally. Do not filter, dilute or alter sample.

5.0 Safety

Employees must abide by the policies and procedures in the Corporate Environmental Health and Safety Manual (CW-E-M-001) and this document. This procedure may involve hazardous material, operations and equipment. This SOP does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of the method to follow appropriate safety, waste disposal and health practices under the assumption that all samples and reagents are potentially hazardous. Safety glasses, gloves, lab coats and closed-toe, nonabsorbent shoes are a minimum.

5.1. Specific Safety Concerns or Requirements

There are no specialized safety concerns associated with this method.

5.2. Primary Materials Used

The following is a list of the materials used in this method, which have a serious or significant hazard rating. **Note: This list does not include all materials used in the method. The table contains a summary of the primary hazards listed in the MSDS for each of the materials listed in the table.** A complete list of materials used in the method can be found in the reagents and materials section. Employees must review the information in the MSDS for each material before using it for the first time or when there are major changes to the MSDS.

Material (2)	Hazards	Exposure Limit (1)	Signs and symptoms of exposure
Sulfuric Acid	Corrosive Oxidizer Dehydra-dator	1 mg/m^3	This material will cause burns if comes into contact with the skin or eyes. Inhalation of vapors will cause irritation of the nasal and respiratory system.
1 – Exposure limit refers to the OSHA regulatory exposure limit.			
2- Always add acid to water to prevent violent reactions.			

6.0 Equipment and Supplies

6.1. Instrumentation

- MAN-TECH PC-Titration Plus
- pH meter Thermo Orion, Model 320
- combination electrode: PerpHecT Ross Electrode

6.2. Supplies

- Burette, 10 ml and 25 ml. (Class A)
- Beakers, 150 ml
- Magnetic stirrer and Teflon-coated magnets
- Eppendorf Pipettes, varying volumes
- Volumetric flasks, assorted (Class A)
- Graduated cylinder (Class A)

7. Reagents and Standards

7.1. Reagents

7.1.1. 0.05 N Sodium carbonate solution: Place 0.625 grams Na_2CO_3 (dried 4 hours at 250°C) in a 250 ml volumetric flask and dilute to mark with deionized water. Solution is good for one week; refrigerate until ready to use.

7.1.2. Ross pH electrode filling solution, 3M KCL: Cat. No. 810007; for storage and stability information, see manufacturer's instructions. Note: Do not use any filling solution that contains silver, as silver will damage the electrode.

7.1.3. pH buffer solution of 4, 7, and 10: Purchased commercially; store at room temperature, for stability information refer to manufacturer's instructions.

7.2. Standards

Standardize titrants once a month by the following method and document standardization in the standardization logbook. Use the measured Normality value in sample calculation or prepare and standardize fresh titration reagent as needed.

7.2.1. **0.10 N H_2SO_4**: Dilute 3.0ml conc. H_2SO_4 with de-ionized water to 1000ml. This reagent is stable for 6 months and should be stored at room temperature. Standardize against 0.05 N Na_2CO_3 solution (Sec. 7.1.1).

7.2.1.1. Place 40.0 ml freshly prepared 0.05 N Na_2CO_3 solution into a 500 ml erlenmeyer flask. Add about 60 ml of deionized water.

7.2.1.2. Titrate with 0.1 N H_2SO_4 until pH = 5.0.

7.2.1.3. Cover flask with a watchglass and boil for 3 minutes.

7.2.1.4. Cool and continue titrating to pH 4.5.

7.2.2. **0.02 N H_2SO_4:** Purchased commercially. For stability and storage information, refer to manufacturer's instructions. Standardize against 0.05N Na_2CO_3 solution (Sec. 7.1.1):

7.2.2.1. Same as above except use only 15.0 ml Na_2CO_3 solution and bring up to 100 ml with deionized water.

7.2.2.2. Titrant Concentration (N)= $\frac{2.50 \times A}{53.00 \times B}$

Where: A= vol. Na_2CO_3 solution (ml)
B= vol. titrant (ml)

8. Sample Collection, Preservation, Shipment and Storage

Sample container, preservation techniques and holding times may vary and are dependent on sample matrix, method of choice, regulatory compliance, and/or specific contract or client requests. Listed below are the holding times and the references that include preservation requirements.

Matrix	Sample Container	Min. Sample Size	Preservation	Holding Time[2]	Reference
Waters	P,FP,G[1]	50 mLs	Cool, ≤ 6°C	14 Days	40 CFR Part 136.3
Soils	P,FP,G[1]	50 grams	Cool, ≤ 6°C	14 Days	Not applicable

[1] P' is polyethylene, 'FP' is fluoropolymer, 'G' is glass. Fill container completely, cap tightly and limit headspace. Do not filter, concentrate or alter samples in any way. Avoid sample agitation and prolonged exposure to the air.
[2] Holding time is 14 days but it is strongly urged to analyze as soon as possible.

9. Quality Control

9.1. Sample QC

The following quality control samples are prepared daily or with each batch of 20 samples or less, whichever is more frequent.

Quality Controls	Frequency	Control Limit
Method Blank (MB)	Daily, 1 in 20 or fewer samples	< Reporting Limit
Laboratory Control Sample (LCS)[1] (LCSSRM)	Daily, 1 in 20 or fewer samples	Vendor specified QC limits
Matrix Duplicate (MD)[1]	Daily, 1 in 20 or fewer samples	Statistical Limits [2]

[1] The sample selection for MD is randomly selected, unless specifically requested by a client.
[2] Statistical control limits are updated annually and are updated into LIMS.

9.1.1. Method Blank: A blank must be analyzed each time samples are analyzed. Use deionized water for the blank. If results are at or above the method reporting limit, take immediate corrective action, this may include re-analyzing the sample batch.

9.1.2. Laboratory Control Sample (LCS): The LCS for alkalinity is a whole volume quality control sample purchased from Phenova and is carried through the entire process. It is used to measure method performance on the matrix being analyzed. LCS is analyzed daily; results must be within vendor specified QC limits. If LCS fails, all samples must be re-analyzed following an acceptable LCS.

9.1.3. Matrix Duplicate: A duplicate is analyzed daily or with each batch of 20 samples or fewer, whichever is more frequent, by using a second aliquot of

sample. The relative percent difference (RPD) must be within laboratory generated control limits.

9.2. Instrument QC

None

10. Procedure

10.1. Calibration

10.1.1. Check the flow rate ml/min of the sampler daily or prior to sample analysis as follows:

- Place sample needle (input) in a vial filled with water.
- Set sample pump to 'Forward.'
- Place the sample needle (output) in a graduated cylinder and measure the flow rate in ml/min.
- Repeat the above procedure three times and take the average measurements.
- Set sample back to 'Auto.'
- In 'PC titrate' window, click on 'Interface,' then 'Hardware set-up.'
- Click on 'Digital/Amplifier' tab.
- Click on 'Extended Digital I/O' tab.
- Highlight the row for 'Sample pump'
- Enter the measured average flow rate in 'Flow rate ml/min' box and click 'ok.

10.1.2. Enter the final concentration (N) of the titrant H_2SO_4 in the PC-titrator.

10.1.2.1. From the Main menu, highlight 'Set up.'

10.1.2.2. Click on "Titration Method,' and 'Load.' Select 'Alkalinity,' then click OK.

10.1.2.3. This will bring up H_2SO_4 as the titrant. Type in the obtained concentration (N) value (Sec 7.2.2.2).

10.1.2.4. Click 'SAVE.' A blank box will appear on the screen, type in 'H_2SO_4 standardized on mm/dd/yy,' then click 'Done.'

10.1.3. Calibrate the instrument (PC titrator) daily.

10.1.3.1. Fill vials 1, 2, and 3 with 25 ml of buffer 4, 7, and 10. **Note:** After the last buffer, place a vial filled with diluted buffer 4, this does not need to be typed into the sequence. The diluted buffer 4 will fill the electrode cell after the analysis has been completed and protect the electrodes from drying out.

10.1.3.2. Click twice on the PC-Titrate V3 icon to open the program.

10.1.3.3. Click on the Calibration pH 4-7-10 icon from the list of icons on the bottom on the screen.'

10.1.3.4. Click 'Start' (DO NOT click 'ok'). Instrument will start calibration by measuring the pH of each buffer (4, 7 and 10).

10.1.3.5. After the last buffer (pH 10) is measured, click on 'Calibration Result' tab. If calibration validity is 'True' click 'Ok.' If the calibration validity is 'False', check for crystals around the electrode, check to make sure that the probe has enough filling solution, and recalibrate the instrument.

10.1.3.6. Print Calibration Report:

10.1.3.6.1. Go to 'Titrator.' Select 'Examine Calibrations.'

10.1.3.6.2. From the 'Port' drop down menu, click on '1.'

10.1.3.6.3. From the 'Calibration ID' drop down menu, click 'pH-cal- 4-7-10.'

10.1.3.6.4. Click 'Print this Calibration,' and choose the printer destination. Click 'Done' and 'Ok.'

10.1.4. Calibration for manual titration: see TestAmerica SOP No. ED-WET-060 (Analysis of pH for Waters and Drinking water Measured Electrochemically) for information on calibrating the pH meter.

10.2. Sample Analysis

10.2.1. Manual titration (Water samples):

10.2.1.1. Rinse all glassware well with deionized water.

10.2.1.2. Transfer 50 ml or aliquot of a well-mixed sample to specimen cup with as little agitation as possible.

10.2.1.3. Record initial pH of sample in TALS.

10.2.1.4. Titrate to a pH of 8.3 if carbonate and bicarbonate alkalinity are required, and proceed to next step.

10.2.1.5. Titrate with standardized acid to pH of 4.5 and record volume of acid. Sample may be swirled to mix in acid, but avoid excessive agitation. If alkalinity is <20ppm use 0.02N H_2SO_4.

10.2.1.6. If alkalinity is less than 20 ppm, continue to titrate to 4.20 and note added titrant.

10.2.2. Manual titration (Soil samples):

10.2.2.1. Homogenize sample as per procedure in TestAmerica Edison SOP No. ED-GEN-007 (Subsampling).

10.2.2.2. Rinse all glassware well with deionized water.

10.2.2.3. Weigh 50.0g of sample into a 140ml specimen cup and add 50 ml of deionized water and mix. Avoid excessive agitation.

10.2.2.4. Stir the sample for 5 minutes and let it stand for approximately 30 minutes.

10.2.2.5. Decant as much liquid for titration.

10.2.2.6. Record the initial pH. If total alkalinity is required, titrate to pH =8.3 and proceed to next step. If all other forms of alkalinity is required, see Sec. 10.2.4.

10.2.2.7. Titrate with standardized acid to pH of 4.5 and record volume of acid. Sample may be swirled to mix in acid, but avoid excessive agitation. If alkalinity is <20ppm use 0.02N H_2SO_4.

10.2.2.8. If alkalinity is less than 20 ppm, continue to titrate to 4.20 and note added titrant.

10.2.3. Instrument: MANTECH PC titrator:

10.2.3.1. From the main menu, highlight 'Titrator,' then click 'Run Titration.' This will bring the timetable screen.

10.2.3.2. Determine the number of samples, including MB and LCS, that will be run and add the required number of spaces/rows needed on the timetable by clicking on 'ADD X ROWS'. This will expand the rows in the timetable.

Note: If more rows were added than desired, click on the sample row (this will highlight the sample), then click on 'DELETE THE HIGHLIGHTED SAMPLE.'

10.2.3.3. Double click the first row under the heading SCHEDULE, A pop-up menu will appear, select 'Alkalinity 25 ml.' Do the same for each sample starting on the second row or use the Copy command.

10.2.3.4. Click on the row below the heading ORDER NUMBER then click on AUTO-GENERATE ORDER NUMBER. The order number (i.e. 20060331-1) will appear under the 'Order Number' column.

Do the same for each sample starting on second row or use the Copy command.

10.2.3.5. Under the heading SAMPLE NAME and starting at row 1 (numbers can be seen on the left hand side of the timetable), type in RINSE in row 1, MB in row 2, and type in LCS in row 3. **Note:** Use deionized water for the RINSE sample to flush the system.

10.2.3.6. Continue on to row 3 and enter the Job number and Sample ID in the format 'Job#-XXXXXX.'

10.2.3.7. For QC samples, use the suffix 'DU' on the sample number (i.e. 460-19450-B-2 DU).

10.2.3.8. A new MB and LCS should be analyzed after 20 samples.

10.2.3.9. Enter the VIAL information: Under the heading VIAL, type in the vial number in the sample tray that corresponds to the sample identified in the timetable. Note: Always use vial #1 for sample number 1, this will ensure that vial numbers will match the row numbers.

10.2.3.10. Once the timetable information is complete and accurate, save this information by clicking on SAVE AS. A pop up menu will appear, type in the file ID in the format 'ALKmmddyy' (next to 'ENTER NEW TEXT').

10.2.3.11. Click on 'Create Using Current Sample ID's' then click OK. Pop up menu will close.

10.2.3.12. Fill the vials with sample aliquot using the timetable as a reference. **Note**: After the last sample aliquot, place a vial filled with the diluted buffer 4. This vial does not need to be typed into the sequence but, it will fill the electrode cell with the diluted buffer 4 to protect the electrodes until the instrument is used again.

10.2.3.13. Click 'Start' to begin the run.

10.2.3.14. Once the run is finished, the report will automatically print.

10.2.4. <u>Procedure for All forms of Alkalinity:</u>

10.2.4.1. Follow procedures for Total Alkalinity. If pH of initial sample is greater than 8.3, titrate initially to a pH of 8.3 and record volume of titrant in logbook. Use this volume of titrant to calculate P (or the phenolphthalein alkalinity) according to the calculation in

Sec 11.3. Continue titrating to a pH of 4.5 and record in logbook.

11.0. Calculations / Data Reduction

11.1. Accuracy:

$$\text{LCS \% Recovery} = \frac{\text{observed concentration}}{\text{known concentration}} \times 100$$

11.2. Precision (RPD):

$$\text{Matrix Duplicate (MD)} = \frac{|\text{orig. sample value - dup. sample value}|}{[(\text{orig. sample value + dup. sample value})/2]} \times 100$$

11.3. Total Alkalinity:

$$\text{Alkalinity (mg/l } CaCO_3) = \frac{A \times N \times 50000}{B}$$

A = volume of acid, ml.
N = concentration of acid, N.
B = volume of sample, ml

11.4. $$\text{Alkalinity (mg/Kg } CaCO_3) = \frac{A \times N \times 50000}{B}$$

A = volume of acid, ml.
N = concentration of acid, N.
B = weight of sample in grams

11.5. Total Alkalinity low level, (< 20 mg/l)

$$\text{Alkalinity} = \frac{(2A - B) \times N \times 50000}{C}$$

A = vol. acid to pH = 4.5, ml.
B = vol. acid from pH = 4.5 to 4.2, ml.
C = vol. of sample, ml

11.6. Calculations for All Forms of Alkalinity:

11.6.1. Carbonate alkalinity is present when phenolphthalein alkalinity is not zero but less than total. Hydroxide alkalinity is present if phenolphthalein alkalinity is more than half the total. Bicarbonate alkalinity is present if phenolphthalein alkalinity is zero or less than half the total.

11.6.2. Calculate different forms of alkalinity using relationships in chart below:

Result of	Hydroxide	Carbonate	Bicarbonate

Titration	as CaCO3	as CaCO3	as CaCO3
P = 0	0	0	T
P < 0.5T	0	2P	T - 2P
P = 0.5T	0	2P	0
P > 0.5T	2P - T	2(T - P)	0
P = T	T	0	0

Where P = phenolphthalein alk. and T = total alk

11.7. PC-Titrator Calculation:

Total Alkalinity calculation at pH 4.5 and p-alk at pH 8.3 is automatically performed by the instrument. If alkalinity is < 20 ppm, it will apply the volume used at pH 4.2 in the calculation. Final results are reported in mg/L.

11.8. Exporting Data from PC titrate to TALS:

11.8.1. From the main menu, click "reporting" then click "prepare and/or print shazam reports."

11.8.2. Go to "File," then "open report." In "reports" folder, select "water analysis historical data report.srw." Click "open."

11.8.3. Select "filter on the left side of the grid and the "rundate" column and/or time "runtime" column. Click on each box to open filter screens and select appropriate date/time.

11.8.4. Click "preview report" tab at the top to display the chosen data.

11.8.5. Click "file" then "export." Click on "…" box next to the file name box. Go to desktop icon on left hand side and click on it. Then select 'alk folder results.' Type in the file name in the box at the bottom, then click "open."

11.8.6. Use the drop down menu under "file type" to select "FixedFieldASCII File (*.txt)." Click "ok."

11.8.7. Go to "shortcut to mantechFT.exe" icon on the desktop. Click "…" button next to the raw data box. Choose the appropriate file and click "open." On the left side, choose the appropriate analyte from "available analytes" box (i.e. pH, temperature, FCO2 for carbon dioxide and for Alkalinity select all of the following: pH, temp, palk, talk, bcarb, carb, hydrx).

11.8.8. Click "transfer file." The data will now be in TALS.

11.9. Data Reduction

11.9.1. All reagent information is recorded on the "batch information" page. Use the "worksheet" tab if additional pages are necessary.

11.9.2. Record special notes and observations on the "worksheet" tab (i.e. sample appearance and notes on why samples were rejected or diluted).

11.9.3. All raw data is attached as a pdf file. The raw data includes the instrument report and calibration curve.

11.9.4. The analyst must fill out the Titration Data Review checklist (CA-Q-WI-064) during the first level review. The batch is second level reviewed and the checklist is filed in the Wet Chemistry department.

12.0. Method Performance

12.1. Method Detection Limit Study (MDL)

The method detection limit (MDL) is the lowest concentration that can be detected for a given analytical method and sample matrix with 99% confidence that the analyte is present. The MDL is determined according to the laboratory's MDL procedure in Section 19 (Test Methods and Method Validation) of TestAmerica Edison's Quality Assurance Manual (ED-QA-LQM). MDLs reflect a calculated (statistical) value determined under ideal laboratory conditions in a clean matrix, and may not be achievable in all environmental matrices. The laboratory maintains MDL studies for analyses performed; these are verified at least annually unless method requirements require a greater frequency. MDLs are typically generated via the TestAmerica LIMS (TALS) control chart module. ***MDL study is not required for this method.***

12.2. Demonstration of Capabilities

For Demonstration of Capabilities (DOC) procedure refer to Section 19 in the most current revision of TestAmerica Edison's Quality Assurance Manual (ED-QA-LQM). DOCs are typically generated via the TestAmerica LIMS (TALS) control chart module.

12.3. Training Requirements

Refer to TestAmerica Edison SOP No. ED-GEN-022, Training, for the laboratory's training program.

13.0. Pollution Control

It is TestAmerica's policy to evaluate each method and look for opportunities to minimize waste generated (i.e., examine recycling options, ordering chemicals based on quantity needed, preparation of reagents based on anticipated usage and reagent stability). Employees must abide by the policies in Section 13 of the Corporate Environmental Health and Safety Manual (CW-E-M-001) for "Waste Management and Pollution Prevention."

14.0. Waste Management

14.1. Waste management practices are conducted consistent with all applicable rules and regulations. Excess reagents, samples and method process wastes are disposed of in an accepted manner. Waste description rules and land disposal

restrictions are followed. Waste disposal procedures are incorporated by reference to TestAmerica SOP No. ED-SPM-008 (Laboratory Waste Disposal Procedures).

14.2. The following waste streams are produced when this method is carried out:

- Acidic waste generated by the analysis is collected in a waste container which is periodically dumped down the sink with water.

15.0. References / Cross-References

15.1. Standard Methods for the Examination of Water and Wastewater, 22th Edition, American Public Health Association, American Washington, DC, 2012, SM 2320 B (Editorial Revisions, 2011).

15.2. TestAmerica Edison Document No. ED-QA-LQM, *Laboratory Quality Manual*, most current revision.

15.3. TestAmerica Edison SOP ED-GEN-022, *Training*, most current revision.

15.4. TestAmerica Edison SOP No. ED-SPM-008, *Laboratory Waste Disposal Procedures*, current revision.

15.5. TestAmerica Edison SOP No. ED-GEN-007, *Subsampling*, current revision.

15.6. TestAmerica Corporate Environmental Health and Safety Manual, Document No. CW-E-M-001, most current revision.

15.7. TestAmerica Corp Form No. CA-Q-WI-064, Titration Data Review Checklist, most current revision.

15.8. TestAmerica Edison SOP ED-WET-060, Analysis of pH for Waters and Drinking water Measured Electrochemically, most current revision.

16.0. Method Modifications:

Item	Method No.	Modification
Sec. 10.2.2.3 – 10.2.2.5	SM 2320B	Added procedure for the analysis of alkalinity in soil, following the procedure in SW846 Method 9040C (Analysis of soil and waste pH) where a 1:1 weight to volume (g/ml) is prepared, mixed, decanted and analyzed.

17.0. Attachments

None

18.0. Revision History

- Revision 12, dated 24 January 2017
  - Sec 9.1.2: Updated LCS vendor source from ERA to Phenova.

- Sec.10.2.4.1: Clarified procedure for calculating Phenolpthalein alkalinity
- Sec .11.8: Modified instrument export procedure to reflect actual lab procedure.
- Sec 11.9.4 & 15.7: Updated Data review checklist document number to CA-Q-WI-064 (Titration Data review checklist).

- Revision 11, dated 27 January 2015
  - Sec. 9.1 & 9.1.3: Clarified the QC frequency of Matrix Duplicate to daily or with each batch of 20 or fewer samples.
  - Sec 9.1.1: Revised the method blank criteria to <RL.
  - Sec 9.1.2: Revised to include ERA as the source for the LCS.
  - Sec 7.2: Revised the standardization frequency of titration reagents from quarterly to monthly as per SM 2020B, Rev 2011.
  - Sec 12.1: Added note that this method does not require a MDL study.

- Revision 10, dated 18 November 2013
  - Sec. 9.1.1: Revised the Method Blank criteria from < RL to ≤ ½ RL to comply with MUR.
- Revision 9, dated 28 June 2013
  - Throughout document: Updated Standard Methods reference to currently implemented and accredited methods, SM 2320 B -11.
  - Throughout document: updated references to Lab Quality Manual section numbers as necessary.
  - Minor formatting edits throughout document
  - Sec. 9.1: Changed acceptance limits for LCS from 85-115% to vendor specified QC limits.
  - Sections 12.1 and 12.2: included statements referencing use of TALS control chart in the generation of MDLs and DOCs.

- Revision 8, dated 14 June 2011
  - Sec. 7.1.2: Added Ross pH electrode filling solution to list of reagents.
  - Sec. 7.1.3: Added pH buffers 4, 7 and 10 to list of reagents.
  - Sec. 9.1.1: Revised to include the corrective action taken if MB is greater than RL.
  - Sec. 9.1.2: Revised the corrective action taken if LCS is outside the acceptable range.
  - Sec. 10.1.1: Revised to include the checking of the sampler's flow rate as part of the daily instrument calibration.
  - Sec 10.1.4: Section added.
  - Sec 15: Added SOP No. ED-WET-060 in the list of references.

- Revision 7, dated 17 November 2010
  - Sec 3: Updated the LQM reference for the list of definitions.
  - Sec. 9.1 Table & 9.1.2: Revised LCS control limits to 85-115%.
  - Sec. 11.8: Added information on exporting data to TALS.
  - Sec. 11.9: Added data reduction section in accordance with TALS
  - Sec 15: Added applicable references.

- Revision 6, dated 10 November 2008

- Revised SOP format in accordance with TestAmerica Corporate Quality SOP No. CW-QS-002 (Rev 0) Writing a Standard Operating Procedure (SOP).
- Combined SOP ED-WET-040 (Analysis of Alkalinity in Soil) with this SOP (ED-WET-039). Retired SOP ED-WET-040 at the effective date of this SOP.
- Sec. 1.1: Deleted method EPA 310.1 to comply with the Method Update Rule.
- Added information for soil matrix where applicable (i.e. Sec. 1.1, Sec. 8, etc.).
- Sec. 6: Deleted Erlenmeyer flasks & hotplate; added beakers & graduated cylinder
- Section 7.2: Added shelf life and storage requirements.
- Section 8 & 9: Reformat both sections into Table format.
- Sec 9.1.2: Added the procedure for adjusting the flow rate of the sampler when LCS fails.
- Sec. 10.2.2: Added the soil procedure as written in SOP No. ED-WET-040.
- Sec 10.2.3: Changed the order of instrument operation to reflect actual laboratory procedure
- Sec 10.2.3.5: Revised to include RINSE sample in the analysis log.
- Sec 10.2.3.12: Expanded to include the use of Buffer 4 to fill the electrode cell.
- Section 15: Deleted EPA reference 310.1; added applicable references
- Sec. 16: Added method modification to include reason and reference for the modification.
- Revised SOP Title to include Soil matrix

- Revision 5, dated 02 November 2007
  - Section 6 Standards. Revised the frequency for the standardization of titrants (0.10 N H2SO4 and 0.02N H2SO4 from two weeks to three months.

Title: Analysis of Ferrous Iron in Aqueous Samples by Standard Methods 3500- Fe D - Discreet Analyzer

Once printed, this is considered an uncontrolled document

Approvals (Signature/Date):

Approvals (Signature/Date):

Jasmine Parillo 08/15/17	Dan Helfrich 08/16/2017
Jasmine Parillo Date	Dan Helfrich Date
Department Manager	Health & Safety Manager /Coordinator
Carl Armbruster 08/15/2017	08/15/2017
Carl Armbruster Date	Mark Acierno Date
Quality Assurance Manager	Laboratory Director

Copyright Information:
This documentation has been prepared by TestAmerica Laboratories, Inc. and its affiliates ("TestAmerica"), solely for their own use and the use of their customers in evaluating their qualifications and capabilities in connection with a particular project. The user of this document agrees by its acceptance to return it to TestAmerica upon request and not to reproduce, copy, lend, or otherwise disclose its contents, directly or indirectly, and not to use it for any other purpose other than that for which it was specifically provided. The user also agrees that where consultants or other outside parties are involved in the evaluation process, access to these documents shall not be given to said parties unless those parties also specifically agree to these conditions.

THIS DOCUMENT CONTAINS VALUABLE CONFIDENTIAL AND PROPRIETARY INFORMATION. DISCLOSURE, USE OR REPRODUCTION OF THESE MATERIALS WITHOUT THE WRITTEN AUTHORIZATION OF TESTAMERICA IS STRICTLY PROHIBITED. THIS UNPUBLISHED WORK BY TESTAMERICA IS PROTECTED BY STATE AND FEDERAL LAW OF THE UNITED STATES. IF PUBLICATION OF THIS WORK SHOULD OCCUR THE FOLLOWING NOTICE SHALL APPLY:

©COPYRIGHT 2017 TESTAMERICA LABORATORIES, INC. ALL RIGHTS RESERVED.

1.0 Scope and Application

1.1 Analytes, Matrix(s), and Reporting Limits

Standard Method 3500 Fe D is applicable to the determination of Ferrous iron in drinking, surface, domestic, and industrial waters. The laboratory's reporting limit is 0.10 mg/l for waters.

On occasion clients may request modifications to this SOP. These modifications are handled following the procedures outlined in Sections 7 (Review of Work Request) and 19 (Test Methods and Method Validation) of TestAmerica Edison's Quality Assurance Manual (ED-QA-LQM).

2.0 Summary of Method

Ferrous Iron is determined by a reaction with 1,10 phenanthroline to produce an orange-red complex with a maximum absorbance at 510 nm. The colored solution obeys Beer's Law and its color intensity is independent of pH from 3 to 9.

3.0 Definitions

For a complete list of definitions refer to Appendix 2 in the most current revision of TestAmerica Edison's Quality Assurance Manual (ED-QA-LQM).

4.0 Interferences

4.1 Strong oxidizing agents, cyanides, nitrites and phosphates interfere with this test.

4.2 Cr and Zn in concentrations more than 10x that of Fe can cause interferences. Co and Cu in excess of 5 mg/l and Ni in excess of 2 mg/l can also interfere.

4.3 Bi, Cd, Hg, Molybdate, and Ag precipitate with phenanthroline.

5.0 Safety

Employees must abide by the policies and procedures in the Corporate Environmental Health and Safety Manual (CW-E-M-001) and this document. This procedure may involve hazardous material, operations and equipment. This SOP does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of the method to follow appropriate safety, waste disposal and health practices under the assumption that all samples and reagents are potentially hazardous. Safety glasses, gloves, lab coats and closed-toe, nonabsorbent shoes are a minimum.

5.1. Specific Safety Concerns or Requirements

Samples that contain high concentrations of carbonates or organic material or samples that are at elevated pH can react violently when acids are added.

5.2. Primary Materials Used

The following is a list of the materials used in this method, which have a serious or significant hazard rating. **Note: This list does not include all materials used in the method. The table contains a summary of the primary hazards listed in the MSDS for each of the materials listed in the table.** A complete list of materials used in the method can be found in the reagents and materials section. Employees must review the information in the MSDS for each material before using it for the first time or when there are major changes to the MSDS.

Material (1)	Hazards	Exposure Limit (2)	Signs and symptoms of exposure
Hydrochloric Acid	Corrosive Poison	5 ppm-Ceiling	Inhalation of vapors can cause coughing, choking, inflammation of the nose, throat, and upper respiratory tract, and in severe cases, pulmonary edema, circulatory failure, and death. Can cause redness, pain, and severe skin burns. Vapors are irritating and may cause damage to the eyes. Contact may cause severe burns and permanent eye damage.
1 – Always add acid to water to prevent violent reactions.			
2 – Exposure limit refers to the OSHA regulatory exposure limit.			

6.0 Equipment and Supplies

6.1. Instrumentation

- Discreet Analyzer, Konelab Aqua 20 equipped with Reagent vessels, 20 ml and 60 ml capacities and Sample Segments

6.2. Supplies

- Sample cups, 2.0 ml
- Acrylic multi-cell cuvettes
- Volumetric flasks, varying volumes
- Eppendorf Pipettes, varying volumes
- Transfer pipettes
- Class A graduated cylinder

7.0 Reagents and Standards

7.1. Reagents

7.1.1. Hydrochloric Acid, conc.: ACS grade, store at room temperature; for stability information refer to manufacturer's instructions.

7.1.2. Ammonium Acetate Buffer Solution: Dissolve 125g of ammonium acetate in 75 ml deionized water. Add 350 ml concentrated glacial acetic acid and mix thoroughly. Stable for 6 months. Store at room temperature. Prepare new reference standard with each buffer preparation.

7.1.3. Phenanthroline Solution: Dissolve 100mg 1,10 phenanthroline monohydrate in 100ml DI water and add 2 drops concentrated HCl. Stable for 6 months. Store at room temperature.

7.2. Standards

7.2.1. Primary Ferrous Ammonium Sulfate (FAS) Stock Solution, 200 mg/L: Slowly add 10 ml concentrated sulfuric acid to 25 ml deionized water and dissolve 0.702 g FAS. Dilute to 500 ml with deionized water and mix. Stable for six months. Store at 4ºC.

7.2.2. Secondary Ferrous Ammonium Sulfate (FAS) Stock Solution, 200 mg/L: FAS used to prepare the secondary stock must come from a source different from the FAS used to prepare the primary stock. Slowly add 10 ml concentrated sulfuric acid to 25 ml deionized water and dissolve 0.702 g FAS. Add 0.10N potassium permanganate (KMnO4) dropwise until a faint pink color persists. Dilute to 500 ml with deionized water and mix. Stable for six months. Store at 4ºC.

7.2.3. Primary Ferrous Ammonium Sulfate Working Solution, 10 mg/L: Take 5.0 ml of the primary FAS stock solution (Sec. 7.2.1) and bring it up to a final volume of 100 ml with deionized water. Prepare daily or each time the calibration standards are prepared.

8.0 Sample Collection, Preservation, Shipment and Storage

Sample container, preservation techniques and holding times may vary and are dependent on sample matrix, method of choice, regulatory compliance, and/or specific contract or client requests. Listed below are the holding times and the references that include preservation requirements.

Matrix	Sample Container	Min. Sample Size	Preservation	Holding Time	Reference
Waters	HDPE	50 mLs	Cool 4 ± 2°C	Analyze immediately	Standard Method 3500-Fe D

9.0 Quality Control

9.1. **Sample QC** - The following quality control samples are prepared daily or with each batch of 20 samples or less.

9.1.1. Method Blank: A method blank must be analyzed each time samples are analyzed. The result must be less than the reporting limit. Use 49 ml deionized water plus 1.0 ml of HCl for the blank. The result must be less than the reporting limit. If not, all samples associated with the Method Blank must be re-analyzed.

9.1.2. Laboratory Control Sample, 0.5 mg/l: The LCS is prepared by taking 0.25 ml of the primary FAS solution (Sec. 7.2.1) + 1 ml of HCl and brought up to a final volume of 100 ml with deionized water. The recovery must be within lab generated control limits, otherwise, the analysis must be stopped and the source of the problem corrected, and all samples re-analyzed.

9.1.3. Matrix Duplicate: A duplicate is analyzed by using a second aliquot of sample. The relative percent difference (RPD) must be within laboratory generated control limits.

9.1.4. Matrix Spike, 2.0 mg/l: Spike the sample by taking 0.5 ml of the primary FAS solution (Sec. 7.2.1) + 1.0 ml HCl and brought up to a final volume of 50 ml with sample. The recovery must be between 85-115%.

9.2. Instrument QC

9.2.1. Initial Calibration Verification, 2.0 mg/l: The calibration curve must be verified by analyzing an ICV solution. The ICV is prepared by taking 0.5 ml of the secondary FAS solution (Sec. 7.2.2) and brought up to a final volume of 50 ml with deionized water. The value obtained must be within ±5% of the true value. If the measured value is outside of the acceptance limits, the problem must be corrected, the instrument recalibrated and the ICV reanalyzed.

Note: The ICV and CCV solutions are prepared with the calibration curve and have a one month expiration date. If the ICV/CCV solution is depleted and fresh solution is prepared, the new solution will have an expiration date the same as the existing calibration standards.

9.2.2. Continuing Calibration Verification, 2.0 mg/l: The validity of the curve must be verified periodically during the analysis by analyzing a CCV. The CCV solution is the same solution as the ICV solution. The CCV must be analyzed following every ten samples. “Samples” include method blanks, laboratory control samples, matrix spikes/matrix spike duplicates, and environmental samples. The value obtained for the CCV must be within ±5% of the true value, otherwise the problem must be corrected and the previous ten samples following the last good calibration verification must be reanalyzed.

Note: Use the decision tree below from the corporate guidance document (Doc. No. CA-Q-W-008) to evaluate dual CCVs

Dual CCV Evaluation Decision Tree

Injection	QC Acceptance	Action
CCV 1 CCV 2	Pass Pass	Continue analytical sequence Data acceptable based on calibration
CCV1 CCV2	Pass Fail	Analyses before CCV1 may be accepted Re-analyze all samples that were analyzed after failed CCV2
CCV 1 CCV 2	Fail Pass	Re-analyze all samples that were analyzed before CCV1 and after the previous compliant CCV Analyses after CCV2 may continue
CCV 1 CCV 2	Fail Fail	Re-analyze all samples since the last acceptable CCV. Perform maintenance and/or recalibration prior to re-analysis.

9.2.3. Initial Calibration Blank/Continuing Calibration Blank: Following each calibration verification, a calibration blank (ICB/CCB) must be analyzed. The results must be less than the reporting limit or the previous ten samples must be reanalyzed. Use 49 ml deionized water plus 1.0 ml of HCl for the blank.

10.0 Procedure

10.1. Sample Preparation

10.1.1. Refer to the Discreet Analyzer Instrument Manual to perform all daily maintenance checks to ensure the instrument is functioning properly. Perform a manual QC run to check the validity of the calibration. In the reagent menu, click on the reagents used in the parameter and check the available levels and expiration dates. Change or add if necessary.

10.1.2. Once a day prior to analysis, perform the “Start up” function and “Water blank check.”

10.1.3. Start up function.

10.1.3.1. From the main menu, click on “Start up needed” (located at the uppermost left corner of the screen). Click “ok” to the pop up question. In this procedure, the injecting needle and all tubes

will be rinsed and washed. ANALYSIS WILL NOT START IF "START UP" HAS NOT BEEN PERFORMED.

10.1.4. Run Water blank check after "Start up" is performed.

10.1.4.1. From the main menu, click on the following buttons in this order: F8 (more), F2 (Instr. action), F1 (Perform Water Blk). The instrument will now measure the absorbance of each cuvette for every wavelength and calculates the standard deviation (SD) for each of those wavelengths in mA. This will take approximately 2-3 minutes.

10.1.4.2. Check that the resulting SD (mA) of each wavelength is below 2 mA. If SD is greater than 2 mA, repeat Water blank check (10.1.4.1.) or refer to the instrument manual for proper cleaning maintenance (i.e. clean/wash needle, change DI water supply). If SD (mA) is below 2 mA, then run the calibration standards or the initial calibration checks (ICV/ICB), whichever applies.

10.2. Calibration

10.2.1. Prepare the calibration standards by adding the amount specified below to 50 ml volumetric flask plus 1.0 ml concentrated HCl and then bring to volume with deionized water.

Note: The Calibration standards must be prepared fresh daily or each time the instrument is calibrated. The calibration is good for one month.

Discreet Analyzer Name	10ppm Standard (ml) – Sec 7.2.3	Concentration Fe^{+2} (mg/l)
Fe 0	0.0	0.0
Fe 2	0.50	0.10
Fe 4	2.5	0.50
Fe 5	5.0	1.0
Fe 6	15.0	3.0
Fe 7	25.0	5.0

10.2.2. Load standards, ICV-Fe+2, and ICB-Fe+2 into a segment. In the Sample Entry screen click F8, then the "Sample Segment" button. Begin entering the standards into the correlating positions (use the pull down menu to enter the standard and control names). Once everything has been entered, insert the segment and return to the main menu. Click the

"Calibration/QC Selection" button and highlight "Fe+2." Check the status of all the necessary controls; this is designated by "OK" or "Missing." If the status is "OK" click on the "Calibration" button. The status of your calibration will now change to "pending." Return to the Main screen and push the start button on the keyboard. Note: you can only start an analysis when you are in the Main Screen.

10.2.3. After the calibration has completed, you can either accept or reject/rerun the calibration. A correlation coefficient of 0.995 or greater must be obtained.

10.2.4. If accepted, proceed to sample analysis.

10.3. Sample Analysis

10.3.1. Enter all samples, including any QC samples, and the CCV/CCB to be run in the Sample Entry screen. Print out a sample segment preparation sheet by first clicking F8, then F1 "Print Screen." Repeat this process for each segment.

10.3.2. Shake samples, using transfer pipettes transfer the sample to the appropriate position on the sample segment. Add 1 drop of concentrated HCl to each sample to preserve. If samples are turbid, filter using 0.45 um filter.

10.3.3. Load the segments into the Discreet Analyzer and assign the test parameter (Fe+2). Return to the main page and press start. Continue loading the segments and inserting them into the machine. Once the machine has been started, it is not necessary to hit the start button if more segments are loaded while the analyzer is running.

10.3.4. A CCV and CCB are run at the beginning, end, and every 10 samples. The analyzer is programmed to automatically do this.

NOTE: Prior to sample analysis, at the end of sample sequence and each time samples are selected for re-analysis, the instrument will automatically analyze a CCV/CCB pair. Evaluate the dual CCVs using the decision tree (Sec 9.2.2) from the corporate guidance document.

10.3.5. Dilutions performed by the instrument will be calculated in the software and the final result will be imported to TALS. Note: If a manual dilution is made, the dilution factor must be entered on the same tab as you enter the test parameter in order for it to be applied to the sample result and imported to TALS correctly. Once the data has been imported to TALS, you cannot add dilution factors.

11.0. Calculations / Data Reduction

11.1. Accuracy:

$$\text{ICV / CCV, LCS \% Recovery} = \frac{\text{observed concentration}}{\text{known concentration}} \times 100$$

$$\text{MS \% Recovery} = \frac{\text{(spiked sample) - (unspiked sample)}}{\text{spiked concentration}} \times 100$$

11.2. Precision (RPD):

$$\text{Matrix Duplicate (MD)} = \frac{|\text{orig. sample value - dup. sample value}|}{[(\text{orig. sample value + dup. sample value})/2]} \times 100$$

11.3. Concentration = mg/L = A x B

Where:
A =sample concentration in discreet analyzer (mg/L)
B = dilution factor (if present)

11.4. Ferric Iron Calculation

Ferric Iron (Fe+3) = Total Iron – Ferrous Iron (Fe+2)

Once Total Iron and Ferrous Iron results are entered in TALS and at 2nd level review, create a new batch in TALS under method 3500_Fe+3_D_Cal. Type in the job numbers and TALS will automatically pull in the results for Total Iron and Ferrous Iron and calculate Ferric Iron. Click on the worksheet tab and verify all samples; calc message column will show 'OK.' If any samples have Fe+2 > Fe, then the calc message column will show 'Fe+2>Fe.' In this case, the Ferrous Iron and/or Total Iron should be re-analyzed to confirm results. If Fe+2 < Fe, the Fe+3 calc can be performed. When all samples in the batch are marked 'OK' under calc message, the batch can be put to 2nd level review.

11.5. Data Reduction

11.5.1. Instrument data is imported to TALS by following these steps:

- Select <F4> Reports then <F8> More
- Select <F4> Results to File and Select 1 row per result
- Enter the File Name= KLTodaysDate (KL082806)
- Go to C:\Konelab\results or "shortcut to results" folder on the desktop.
- Highlight the correct file; right click and select "send to" -> "Lims import." The data has now been imported to TALS.

11.5.2. Record special notes and observations in the "worksheet" tab (i.e. sample appearance and notes on why samples were rejected).

11.5.3. Record reagent information in the prep batch information (this can be viewed in the "batch information" page).

11.5.4. The raw data is attached to the batch as a pdf file. This includes the calibration curve, "all results" document which includes rejected and accepted results, and the "accepted results" document.

Print 'All Results' as follows:

- On 'Main Screen', Select <F4> Reports
- From the first drop down menu, choose "Tests"
- From the second drop down menu, choose "All Results"
- From the third drop down menu, choose "Include Rejected"
- Select "Sulfate" from the box on the left hand side of the screen
- Select <F2> Report Selected
- Pdf creator pop box will appear, Click Save.

Print 'Accepted Results' as follows:

- On 'Main Screen', Select <F4> Reports
- From the first drop down menu, choose "Tests"
- From the second drop down menu, choose "All Results"
- From the third drop down menu, choose "Exclude Rejected"
- Select "Sulfate" from the box on the left hand side of the screen
- Select <F2> Report Selected
- Pdf creator pop box will appear, Click Save.

11.5.5. Attach a NCM statement to each sample which is analyzed outside of the 15 minute holding time. Samples are qualified with an 'HF' flag.

11.5.6. Analyst must fill out the Wet Chem Data Review checklist (WI# EDS-WI-008) during the first level review. After the batch is second level reviewed, the checklist is filed in wetchem department.

12.0. Method Performance

12.1. Method Detection Limit Study (MDL)

The method detection limit (MDL) is the lowest concentration that can be detected for a given analytical method and sample matrix with 99% confidence that the analyte is present. The MDL is determined according to the laboratory's MDL procedure in Section 19 (Test Methods and Method Validation) of TestAmerica Edison's Quality Assurance Manual (ED-QA-LQM). MDLs reflect a calculated (statistical) value determined under ideal laboratory conditions in a clean matrix, and may not be achievable in all environmental matrices. The laboratory maintains MDL studies for analyses performed; these are verified at least annually unless method requirements require a greater frequency.

12.2. Demonstration of Capabilities

For DOC procedure refer to Section 19 in the most current revision of TestAmerica Edison's Quality Assurance Manual (ED-QA-LQM).

12.3. Training Requirements

Refer to TestAmerica SOP No. ED-GEN-022, Training, for the laboratory's training program.

13.0. Pollution Control

It is TestAmerica's policy to evaluate each method and look for opportunities to minimize waste generated (i.e., examine recycling options, ordering chemicals based on quantity needed, preparation of reagents based on anticipated usage and reagent stability). Employees must abide by the policies in Section 13 of the Corporate Environmental Health and Safety Manual (CW-E-M-001) for "Waste Management and Pollution Prevention."

14.0. Waste Management

14.1. Waste management practices are conducted consistent with all applicable rules and regulations. Excess reagents, samples and method process wastes are disposed of in an accepted manner. Waste description rules and land disposal restrictions are followed. Waste disposal procedures are incorporated by reference to TestAmerica SOP No. ED-SPM-008 (Laboratory Waste Disposal Procedures).

14.2. There are no specific waste streams generated by this method.

15.0. References / Cross-References

15.1. Standard Methods for the Examination of Water and Wastewater, 18th Edition, American Public Health Association, Baltimore Maryland, 1992, SM 3500-Fe D.

15.2. TestAmerica Edison Document No. ED-QA-LQM, *Laboratory Quality Manual*, most current revision.

15.3. TestAmerica Edison SOP ED-GEN-022, *Training*, most current revision.

15.4. TestAmerica Edison SOP No. ED-SPM-008, *Laboratory Waste Disposal Procedures*, current revision.

15.5. Corporate Environmental Health and Safety Manual CW-E-M-001, most current revision.

15.6. TestAmerica Edison Work Instruction # EDS-WI-008, Wetchem Data Review Checklist, most current revision.

15.7. Corporate Technical Guidance on Use and Evaluation of Replicate Continuing Calibration Verification (CCV); Document No. CA-Q-W-008.

16.0. Method Modifications:

Item	Method	Modification
Sec. 8	SM 3500-Fe D	Holding time: Ferrous iron is determined at the sampling site. This SOP describes procedures for analyzing samples in the laboratory outside of the holding time. The data analyzed outside of the holding time is flagged/qualified and an NCM is reported with the sample.

17.0. Attachments

Attachment 1: Instrument and method condition for ferrous iron

18.0. Revision History

- Revision 9, dated 16 August 2017
  - Section 10.2.1: Removed 0.30 mg/L ICAL standard to reflect actual laboratory practices.

- Revision 8 , dated 19 December 2016
  - Section 11.4: Added procedure for processing Ferric iron calculation in TALS.

- Revision 7 , dated 01 December 2016
  - Sec. 7.2.1: Removed following text from SOP to reflect actual laboratory practices: "Add 0.10N potassium permanganate (KMnO4) dropwise until a faint pink color persists"
  - Section 9.1: added text to indicate that QC is prepared daily during analysis (at minimum).
  - Sec. 10.3.2: Added filtration to the procedure for turbid samples.

- Revision 6, dated 08 August 2014
  - Sec. 9.1.2: Revised LCS control limits from 85-115% to lab generated control limits.
  - Sec 9.1.3: Added QC sample Matrix Duplicate to comply with method requirements.
  - Sec 9.1.4 (previously Sec 9.1.3): Deleted QC sample Matrix Spike Duplicate and revised the acceptance limits of Matrix Spike to 85-115% as defined in the method.

- Revision 5, dated 24 May 2012
  - Sec 1.1 and 12: Revised LQM references to reflect the most current LQM revision.
  - Sec. 9.1.1: Added corrective action procedure when MB result is greater than the RL.
  - Sec 9.2.1: Added stability information for ICV and CCV standards.
  - Sec 9.2.2: Added Decision Tree Table as a guidance for evaluating dual CCVs.
  - Sec 10.3.4: Added notes on dual CCVs.
  - Sec. 11.4.4: Added procedures for creating pdf files for both instrument results - "accepted" and "rejected."
  - Sec 15: Added applicable reference.

- Revision 4, 29 June 2010

  - Revised SOP format in accordance with TestAmerica Corporate Quality SOP No.CW-QS-002 (Rev 0) Writing a Standard Operating Procedure (SOP).

- Section 1: Added reporting limit.
- Sec 7: Added storage information
- Sec 7.1.2: Added stability information: *Prepare new reference standard with each buffer preparation.*
- Sec 7.2.1 & 7.2.2: Revised to include the addition of 0.10N KMNO4 in the preparation of the primary stock, FAS.
- Section 7.2.3: Added 10 ppm primary working standard as the intermediate standard used for preparing the calibration standards.
- Section 9.1.2 & 9.1.3: Included preparation procedure for LCS and MS/MSD; revised LCS acceptance limits to ±15%.
- Section 9.2.1 & 9.2.2: Included preparation procedure for ICV and CCV and revised the ICV/CCV criteria limit from ±10% to ± 5%.
- Sec 10.2.1: Deleted 0.05 mg/l in the calibration standards.
- Section 10.2.3: Revised the correlation coefficient criteria from 0.997 to 0.995.
- Section 10.3.5: Added section regarding dilution factors and Konelab/TALS.
- Section 11.4: Added data reduction section in accordance to TALS.
- Sec 15: Added applicable references.
- Sec 16: Added method modification to include flagging the data analyzed through this SOP.
- Sec 17: Attachment added to specify instrument condition including sample-reagent ratio.
- Added Revision history.

Attachment 1

```
Test definition              Konelab 6.0.2                          Page:    1

FERROUS                      Laboratory
                             Konelab User
Date :   2010-06-22
Time :   10.43
```

Full name						
Online Name			Test In Use	YES		
Test type	Photometric			LOW	HIGH	
			Test limit	0.000	*	mg/l
Result unit	mg/l		Initial absorbance	0.000	*	A
Number of Decim.	3		Dilution limit	*	5.0000	mg/l
			Secondary dil 1+	0.0	4.0	
			Critical limit	*	*	mg/l
			Reflex test limit	*	*	mg/l
			Reflex test			
Acceptance	Manual		Reference class	LOW	HIGH	In Use
Dilution 1+	0.0					
Sample type			Correction factor	1.00		
			Correction bias	0.00	mg/l	
Calibration type	Linear					
Repeat time (d)	0		Abs error (mA)		*	
Points/cal.	Single		Rel error (%)		*	
Acceptance	Manual					
Response limit (mA)	MIN		MAX			
	*		*			
Bias correction in use	NO					
Type of Calibrators	Separate					
Calibrator	Conc.		Dil. ratio			
FE-0	0.000		1+0.0			
FE-0.1	0.100		1+0.0			
FE-0.3	0.300		1+0.0			
FE-0.5	0.500		1+0.0			
FE-1.0	1.000		1+0.0			
FE-3.0	3.000		1+0.0			
FE-5.0	5.000		1+0.0			
Manual QC in Use	YES		Routine QC in Use		YES	
Acceptance	Manual		Interval	Requests	10	
			Additional condition		NO	

Control	Mean	SD	Control	Mean	SD
ICB-FE	0.00	0.05	CCB-FE	0.00	0.05
ICV-FE	2.00	0.20	CCV-FE	2.00	0.20

Rules in Use	1:1.0*SD	Rules in Use	1:1.0*SD
Blank	YES		
		Normal cuvette	
Sample		Volume (ul)	120
Disp. with	Extra	Add. Volume (ul)	40
Dilution with	Water	Wash reagent	None
Reagent	Fe+2 BUFFE	Volume (ul)	20
Disp. with	Water	Add. Volume (ul)	20
Wash reagent	None		
Reagent	WATER	Volume (ul)	20
Disp. with	Extra	Add. Volume (ul)	20
Wash reagent	None		

Test definition Konelab 6.0.2 Page: 2

FERROUS
Laboratory
Konelab User

Date : 2010-06-22
Time : 10.43

Measurement	End point	Blank	
Resp. Min(A)	0.000	Resp. Max(A)	*
Reagent	Fe+2 COLOR	Volume (ul)	50
Disp. with	Extra	Add. Volume (ul)	30
Wash reagent	None		
Incubation		Time (sec)	300
Measurement	End point		
Wavelength (nm)	510 nm	Side wavel. (nm)	None
Meas. type	Normal		

Title: Analysis of Sulfide in Water, Titrimetric by Standard Methods 4500-S^2 F and SW846 9034

Once printed, this is considered an uncontrolled document

Approvals (Signature/Date):

Jasmine Parillo 04/30/18	Dan Helfrich 04/30/18
Jasmine Parillo Date	Dan Helfrich Date
Department Manager	Health & Safety Manager /Coordinator
Carl Armbruster 04/30/18	04/30/18
Carl Armbruster Date	Mark Acierno Date
Quality Assurance Manager	Laboratory Director

Copyright Information:
This documentation has been prepared by TestAmerica Laboratories, Inc. and its affiliates ("TestAmerica"), solely for their own use and the use of their customers in evaluating their qualifications and capabilities in connection with a particular project. The user of this document agrees by its acceptance to return it to TestAmerica upon request and not to reproduce, copy, lend, or otherwise disclose its contents, directly or indirectly, and not to use it for any other purpose other than that for which it was specifically provided. The user also agrees that where consultants or other outside parties are involved in the evaluation process, access to these documents shall not be given to said parties unless those parties also specifically agree to these conditions.

THIS DOCUMENT CONTAINS VALUABLE CONFIDENTIAL AND PROPRIETARY INFORMATION. DISCLOSURE, USE OR REPRODUCTION OF THESE MATERIALS WITHOUT THE WRITTEN AUTHORIZATION OF TESTAMERICA IS STRICTLY PROHIBITED. THIS UNPUBLISHED WORK BY TESTAMERICA IS PROTECTED BY STATE AND FEDERAL LAW OF THE UNITED STATES. IF PUBLICATION OF THIS WORK SHOULD OCCUR THE FOLLOWING NOTICE SHALL APPLY:

©COPYRIGHT 2018 TESTAMERICA LABORATORIES, INC. ALL RIGHTS RESERVED.

Facility Distribution No. Electronic **Distributed To:** Facility Intranet

1.0 Scope and Application

1.1 Analytes, Matrix(s), and Reporting Limits

This SOP is applicable to the measurement of total and dissolved sulfide in non-potable, drinking, surface and saline waters, domestic and industrial waste using Standard Methods, 4500-S^2 F and SW846 9034. The laboratory's reporting limit is 1.0 mg/l for aqueous samples.

On occasion clients may request modifications to this SOP. These modifications are handled following the procedures outlined in Sections 7 (Review of Work Request) and 19 (Test Methods and Method Validation) of TestAmerica Edison's Quality Assurance Manual (ED-QA-LQM).

2.0 Summary of Method

Excess iodine is added to a sample that has been treated with zinc acetate. Under acidic conditions, iodine oxidizes sulfide to sulfur. The excess iodine is then back titrated with sodium thiosulfate.

3.0 Definitions

For a complete list of definitions refer to Appendix 2 in the most current revision of TestAmerica Edison's Quality Assurance Manual (ED-QA-LQM).

4.0 Interferences

4.1 Samples must be taken with minimum aeration to prevent volatilization of sulfide. Dissolved oxygen should not be present in any water used to dilute standards.

4.2 Sulfite, thiosulfate, and hydrosulfite may yield erratic results and volatile iodine consuming substances will give high results.

4.3 If sample is not preserved with zinc acetate and NaOH, the analysis must be started immediately.

5.0 Safety

Employees must abide by the policies and procedures in the Corporate Environmental Health and Safety Manual (CW-E-M-001) and this document. This procedure may involve hazardous material, operations and equipment. This SOP does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of the method to follow appropriate safety, waste disposal and health practices under the assumption that all samples and reagents are potentially hazardous. Safety glasses, gloves, lab coats and closed-toe, nonabsorbent shoes are a minimum.

5.1. Specific Safety Concerns or Requirements

Sodium Sulfide will form Hydrogen Sulfide (HS) gas if combined with water moisture or strong acids. Inhalation of HS gas may be fatal.

5.2. Primary Materials Used

The following is a list of the materials used in this method, which have a serious or significant hazard rating. **Note: This list does not include all materials used in the method. The table contains a summary of the primary hazards listed in the MSDS for each of the materials listed in the table.** A complete list of materials used in the method can be found in the reagents and materials section. Employees must review the information in the MSDS for each material before using it for the first time or when there are major changes to the MSDS.

Material (1)	Hazards	Exposure Limit (2)	Signs and symptoms of exposure
Hydrochloric Acid	Corrosive Poison	5 ppm-Ceiling	Inhalation of vapors can cause coughing, choking, inflammation of the nose, throat, and upper respiratory tract, and in severe cases, pulmonary edema, circulatory failure, and death. Can cause redness, pain, and severe skin burns. Vapors are irritating and may cause damage to the eyes. Contact may cause severe burns and permanent eye damage.
Sodium Hydroxide	Corrosive	2 Mg/M3-Ceiling	Severe irritant. Effects from inhalation of dust or mist vary from mild irritation to serious damage of the upper respiratory tract, depending on severity of exposure. Symptoms may include sneezing, sore throat or runny nose. Contact with skin can cause irritation or severe burns and scarring with greater exposures. Causes irritation of eyes, and with greater exposures it can cause burns that may result in permanent impairment of vision, even blindness.
Sodium Sulfide	Corrosive	10 ppm-TWA 15 ppm-STEL	Will form Hydrogen Sulfide (HS) gas if combined with strong acids. Inhalation of HS gas may be fatal. Symptoms include painful conjunctivitis, headache, nausea, dizziness, coughing and, in extreme cases, pulmonary edema and possible death. Irritant. Contact with skin can produce serious caustic burns with painful inflammation and possible destruction of tissue. Inflammation, tearing and pain may be expected. Severe contact can cause destruction of tissue.
1 – Always add acid to water to prevent violent reactions.			
2 – Exposure limit refers to the OSHA regulatory exposure limit.			

6.0 Equipment and Supplies

6.1. Supplies

- Burette, 25 ml

- Erlenmeyer flasks
- Transfer pipettes
- Specimen cups
- 100 ml class A graduated cylinder
- Eppendorf Pipettes, varying volumes
- pH paper, 0-12 s.u.

7. Reagents and Standards

7.1. Reagents

7.1.1. Zinc Acetate solution, 2N: Dissolve 220g of zinc acetate dihydrate in 1000 ml deionized water. This reagent is used for sample preservation. Store at room temperature. Stable for 6 months.

7.1.2. Hydrochloric Acid, 6N: In a 500 ml flask, add approximately 200 ml deionized water, then slowly add 250 ml of concentrated HCL. Dilute to the mark with deionized water. Store at room temperature. Stable for 6 months.

7.1.3. 0.025N. Iodine solution: purchased commercially. Store at room temperature. Discard after one year of receipt or use manufacturer's expiration, whichever comes first. Standardize weekly against 0.025 N Sodium Thiosulfate.

7.1.3.1. Standardization of 0.025N Iodine solution.

7.1.3.1.1. In a 500 ml Erlenmeyer flask, dissolve 2 g KI in 150 ml of deionized water. Add 20.0 ml of Iodine solution and dilute to 300 ml.

7.1.3.1.2. Titrate with 0.025 N Sodium Thiosulfate until the amber color fades to yellow. Add starch indicator and continue titrating until the disappearance of blue color

7.1.4. 0.025N. Sodium Thiosulfate solution: purchased commercially. Store at room temperature. Discard after one year of receipt or use manufacturer's expiration, whichever comes first. Standardize quarterly.

7.1.4.1. Standardization of 0.025 N Sodium Thiosulfate Solution

7.1.4.1.1. In an Erlenmeyer flask, pipet 5.0 ml of 0.025 N K_2CRO_7 in 85 ml DI water. Add 3 g KI crystals. Add 1.0 ml concentrated H_2SO_4 and let stand in the dark for 10 minutes.

7.1.4.1.2. Titrate with 0.025 N Sodium Thiosulfate until the amber color fades to yellow. Add starch indicator and continue titrating until the disappearance of blue color.

7.1.5. Starch solution: purchased commercially. Store at room temperature. Follow manufacturer's guidelines for stability information.

If prepared in laboratory, dissolve 20g soluble starch powder and 2g salicylic acid in a 100 ml freshly boiled and cooled deionized water. Pour into 900 ml freshly boiled deionized water and stir until homogenous.

7.1.6. 0.025 N Potassium Dichromate: Add 0.123 grams of Potassium Dichromate, dried for an hour at 103-105℃, into a volumetric flask and dilute to 100ml with deionized water. Store at room temperature. Stable for six months. This solution is used for the standardization of the Sodium Thiosulfate solution.

7.2. Standards

7.2.1. Stock sulfide spiking solution: Dissolve 1.875g of sodium sulfide•$9H_2O$ (ACS grade) in 250ml of deionized water. Prepare fresh daily. Determine concentration by titrating the solution with sodium thiosulfate.

Note: The neat standard should not be kept longer than one year.

8. Sample Collection, Preservation, Shipment and Storage

Sample container, preservation techniques and holding times may vary and are dependent on sample matrix, method of choice, regulatory compliance, and/or specific contract or client requests. Listed below are the holding times and the references that include preservation requirements.

Matrix	Sample Container	Min. Sample Size	Preservation	Holding Time	Reference
Waters[1]	P, FP, G[2]	50 mLs	2 ml zinc acetate + NaOH to pH > 9 Cool 4 ± 2°C	7 Days	40 CFR Part 136.3

[1] Avoid agitation when samples are taken.
[2] P- polyethylene, FP – Fluoropolymer, G- glass

9. Quality Control

9.1. **Sample QC** - The following quality control samples are prepared daily or with each batch of 20 samples or less, whichever is more frequent.

9.1.1. Method Blank: A method blank must be analyzed each time samples are analyzed and at a frequency of one per 20 samples. Use deionized water for the blank. If results are at or above the method reporting limit, take immediate corrective action, this may include re-analyzing the sample batch.

9.1.2. Laboratory Control Sample: The LCS is purchased from Environmental Resources Associates (ERA) and is used to measure method performance on the matrix being analyzed. The LCS is run daily or every 20 samples whichever is more frequent. The LCS is prepared by taking 1 ml of the purchased concentrate and diluted to 100 ml with deionized water. The whole volume is then titrated according to the sample procedure. The LCS recovery must be within vendor specified QC limits. If the result is not within the acceptance limits, the problem must be corrected, the LCS reanalyzed, and all samples associated with the LCS are reanalyzed.

9.1.3. Matrix Spike/Matrix Spike Duplicate (MS/MSD): MS/SMD is analyzed daily or with each batch of 20 samples or fewer, whichever is more frequent. Two portions of the same sample are each spike with 1.0 ml of the stock sulfide spiking solution (Sec. 7.2.1) and brought up to 100ml final volume with sample. The stock sulfide spiking solution is titrated daily and the value obtained is used for the MS/MSD true value. The results for the MS/MSD must be within laboratory generated control limits.

9.2. Instrument QC

None

10. Procedure

10.1. Sample Preparation

10.1.1. Use pH paper to verify that the pH of the sample is greater than 9 s.u. Record pH in the appropriate logbook.

10.2. Calibration

Add 1.0 ml of the sulfide spiking solution (Sec 7.2.1) into 100 ml volumetric flask and bring up to volume. Titrate this solution following procedures in Sec 10.3.1 to determine the concentration (mg/l) of the spiking solution. Record results as BS (unextracted) in the appropriate logbook.

10.3. Sample Analysis

10.3.1. For Unprecipitated Samples:

10.3.1.1. Pipet 5.0 ml iodine solution into flask.

10.3.1.2. Add 2 ml 6N HCl and gently swirl.

10.3.1.3. Add approximately 10 ml of de-ionized water.

10.3.1.4. Add 100 ml of sample into flask, discharging sample below surface of the solution.

10.3.1.5. Add more iodine, if necessary to give the characteristic brown iodine color. Record total amount of iodine added.

10.3.1.6. Titrate with 0.025 N sodium thiosulfate solution to a light brown color.

10.3.1.7. Add 1 ml of starch indicator.

10.3.1.8. Continue titrating until the disappearance of the blue color.

10.3.1.9. Record titrant volume used and the current Normality for iodine and sodium thiosulfate.

10.3.2. For Dewatered Samples:(Filtered)

10.3.2.1. Filter 100 ml of sample through glass fiber filter.

10.3.2.2. Return filter with precipitate to original bottle and add 100 ml of de-ionized water.

10.3.2.3. Pipet 5.0 ml iodine solution into flask.

10.3.2.4. Add 2 ml 6N HCl and gently swirl.

10.3.2.5. Add the 100 ml and glass fiber filter into the flask, discharging sample below surface of solution.

10.3.2.6. Add more iodine, if necessary, to give the characteristic brown iodine color. Record total amount of iodine added.

10.3.2.7. Titrate with 0.025 N sodium thiosulfate solution to a light brown color.

10.3.2.8. Add 1ml starch indicator.

10.3.2.9. Continue titrating until the disappearance of the blue color.

10.3.2.10. Note titrant volume used and most recent Normality for iodine and sodium thiosulfate.

11.0. Calculations / Data Reduction

11.1. Accuracy:

$$\text{LCS \% Recovery} = \frac{\text{observed concentration}}{\text{known concentration}} \times 100$$

$$\text{MS \% Recovery} = \frac{\text{(spiked sample) - (unspiked sample)}}{\text{spiked concentration}} \times 100$$

11.2. Precision (RPD):

$$\text{Matrix Duplicate (MD)} = \frac{|\text{orig. sample value - dup. sample value}|}{} \times 100$$

[(orig. sample value + dup. sample value)/2]

11.3. Concentration = mg/L Sulfide = $\frac{(AC-BD) \times 16000}{\text{ml of sample}}$

Where:
A=ml of standard iodine
B=ml of standard sodium thiosulfate
C=normality of standard iodine
D=normality of standard sodium thiosulfate

11.4. Data Reduction

11.4.1. All data is recorded in the appropriate logbook and attached to the batch as a pdf file.

11.4.2. Record special notes and observations in the "worksheet" tab (i.e. sample appearance).

11.5.3. Record reagent information in the "batch information" page. If there is not enough space to record all of the reagent info, utilize the "worksheet" tab under "notes" to record pertinent information.

11.5.4. Analyst must fill out the Wet Chem Data Review checklist (WI# EDS-WI-008) during the first level review. After the batch is second level reviewed, the checklist is filed in wetchem department.

12.0. Method Performance

12.1. Method Detection Limit Study (MDL)

The method detection limit (MDL) is the lowest concentration that can be detected for a given analytical method and sample matrix with 99% confidence that the analyte is present. The MDL is determined according to the laboratory's MDL procedure in Section 19 (Test Methods and Method Validation) of TestAmerica Edison's Quality Assurance Manual (ED-QA-LQM). MDLs reflect a calculated (statistical) value determined under ideal laboratory conditions in a clean matrix, and may not be achievable in all environmental matrices. The laboratory maintains MDL studies for analyses performed; these are verified at least annually unless method requirements require a greater frequency.

12.2. Demonstration of Capabilities

For DOC procedure refer to Section 19 in the most current revision of TestAmerica Edison's Quality Assurance Manual (ED-QA-LQM).

12.3. Training Requirements

Refer to TestAmerica SOP No. ED-GEN-022, Training, for the laboratory's training program.

13.0. Pollution Control

It is TestAmerica's policy to evaluate each method and look for opportunities to minimize waste generated (i.e., examine recycling options, ordering chemicals based on quantity needed, preparation of reagents based on anticipated usage and reagent stability). Employees must abide by the policies in Section 13 of the Corporate Environmental Health and Safety Manual (CW-E-M-001) for "Waste Management and Pollution Prevention."

14.0. Waste Management

14.1. Waste management practices are conducted consistent with all applicable rules and regulations. Excess reagents, samples and method process wastes are disposed of in an accepted manner. Waste description rules and land disposal restrictions are followed. Waste disposal procedures are incorporated by reference to TestAmerica SOP No. ED-SPM-008 (Laboratory Waste Disposal Procedures).

14.2. There are no specific waste streams generated by this method.

15.0. References / Cross-References

15.1. Standard Methods for the Examination of Water and Wastewater , 22nd Edition, American Public Health Association, Washington, DC 2011, Editorial Revision 2011, SM 4500 S^2 F-11.

15.2. United States Environmental Protection Agency, Method SW9034, Test Methods for Evaluating Solid Wastes, Physical/Chemical Methods, Office of Solid Waste and Emergency Response. U.S. Government Printing Office: Washington, DC, SW846 Revision 0, December 1996.

15.3. TestAmerica Edison Document No. ED-QA-LQM, *Laboratory Quality Manual*, most current revision.

15.4. TestAmerica Edison SOP ED-GEN-022, *Training*, most current revision.

15.5. TestAmerica Edison SOP No. ED-SPM-008, *Laboratory Waste Disposal Procedures*, current revision.

15.6. TestAmerica Corporate Environmental Health and Safety Manual, Document No. CW-E-M-001, most current revision.

15.7. TestAmerica Edison Work Instruction # EDS-WI-008, Wetchem Data Review Checklist, most current revision.

16.0. Method Modifications:

N/A

17.0. Attachments

None

18.0. Revision History

- Revision 8, dated 30 April 2018
  - Sec 1.1: Revised to reference method SW846 9034; added non-potable water to list of matrices.
  - Sec 15.2: Added method SW-846 9034 to list of references; subsequent sections adjusted accordingly.
- Revision 7, dated 09 February 2015
  - Sec. 9.1 & 9.1.3: Clarified the QC frequency of MB and MS/MSD to daily or with each batch of 20 or fewer samples, whichever is more frequent.
  - Sec 9.1.1: Revised the method blank criteria to <RL.
  - Sec 9.1.2: Revised to include ERA as the source for the LCS.
- Revision 6, dated 21 January 2014
  - SOP title and throughout document: Revised to reference SM 4500 S2 F (Rev 2011); removed reference to 4500 S2 E (18th ed).
  - Sec. 7.1.4.1.1: Corrected the 0.02N concentration of K2CrO7 to 0.025N to reflect actual laboratory practices.
  - Sec. 7.1.6: Added preparation instruction for 0.025N K2CrO7.
  - Sec 7.2.1: Added stability information on the neat standard (Na_2S x $9H_2O$).
  - Sec. 9.1.1: Revised the acceptance criteria for Method Blank from < RL to ≤ ½ RL to comply with Method 4500 S2 F-11.
  - Sec. 9.1.2: Revised the acceptance limits of LCS from 70-130% to vendor specified QC limits.
- Revision 5, dated 02 May 2012
  - Sec 1.1 and 12: Revised LQM references to reflect the most current LQM revision.
  - Sec. 9.1.1: Added corrective action procedure when MB is greater than the RL.
  - Sec 10.2: Revised to include spiking procedure for the BS (unextracted).
- Revision 4, 28 April 2010
  - Revised SOP format in accordance with TestAmerica Corporate Quality SOP No. CW-QS-002 (Rev 0) Writing a Standard Operating Procedure (SOP).
  - Sec.1.1: Deleted reference to Methods EPA 376.1 to comply with the Method Update Rule; Added laboratory's reporting limit.
  - Sec. 7: Added storage and stability information where applicable.
  - Section 7.1.1: Changed the final volume for the prep of the zinc acetate from 500ml to 1000ml as per the method.
  - Sec 7.2.1: Added preparation frequency for the spike standard.
  - Sec. 9: Expanded Sample and Instrument QC to include QC concentration (mg/L) and spiking procedure.
  - Sec 10.1.1: Added sample pH verification to reflect actual lab practices.
  - Sec. 10.2: Added procedure for determining the concentration of the spike standard.
  - Sec 11.4: Revised data reduction procedures in accordance with new TALS.
  - Sec. 15: Added applicable references.
  - Added this revision history.

ATTACHMENT F

Eurofins

F-1

Laboratory Quality Manual

eurofins	Always check on-line for validity. **Environmental Quality Policy Manual**	Level: **Quality Manual**
Document number: **QA-QM11872**		
Old Reference: **1-P-QM-GDL-9015377; DOD - EQPM**		
Version: **14**		Organisation level: **5-Sub-BU**
Approved by: **UDM6** Effective Date **31-DEC-2015**	Document users: **4_EUUSLA_ELLE_All**	Responsible: **5_EUUSLA_Env Quality Assurance_All**

This documentation has been prepared by Eurofins Lancaster Laboratories Environmental LLC and its affiliates ("Eurofins"), solely for their own use. The user of this document agrees by its acceptance to return it to Eurofins upon request and not to reproduce, copy, lend, or otherwise disclose its contents, directly or indirectly, and not to use if for any other purpose other than that for which it was specifically provided. The user also agrees that where consultants or other outside parties are involved in the evaluation process, access to these documents shall not be given to said parties unless those parties also specifically agree to these conditions.

THIS DOCUMENT CONTAINS VALUABLE CONFIDENTIAL AND PROPRIETARY INFORMATION. DISCLOSURE, USE OR REPRODUCTION OF THESE MATERIALS WITHOUT THE WRITTEN AUTHORIZATION OF EUROFINS IS STRICTLY PROHIBITED. THIS UNPUBLISHED WORK BY EUROFINS IS PROTECTED BY STATE AND FEDERAL LAW OF THE UNITED STATES.

Cover Page
Title Page
Table of Contents
Revision Log
1. INTRODUCTION
1.1 Mission Statement
1.2 Quality Policy
1.3 Statement of Values
1.4 Sample Flow-Through Diagram
Certifications, Accreditations, and Registrations
2.0 ORGANIZATION AND PERSONNEL
2.1 Company Overview and History
2.1.1 Business Continuity and Contigency Plans
2.2 Organizational Structure
2.2.1 Technical Director
2.2.2 Quality Assurance Director
2.3 Management Responsibilities
2.4 Overview of the Quality Assurance Program
2.5 Quality Assurance Responsibilities
2.6 Communication of Quality Issues to Management
2.7 Personnel Qualifications and Responsibilities
2.8 Relationship of Functional Groups and Quality Assurance Program
2.9 Balancing Laboratory Capacity and Workload
2.10 Identification of Approved Signatories
2.11 Personnel Training
2.11.1 New Hire Training
2.11.2 Ongoing Training
2.12 Regulatory Training
2.13 Employee Safety
2.134 Client Services/Project Management Responsibilities
2.15 Confidentiality
2.16 Business Conduct
2.17 Operational Integrity
3 BUILDING AND FACILITIES
3.1 Facility
3.2 Security

3.3 Disaster Recovery
3.4 Environmental Monitoring
3.5 Water Systems
3.6 Housekeeping/Cleaning
3.7 Insect & Rodent Control
3.8 Emergency Power Supply
3.9 Facility Changes
4.0 DOCUMENT CONTROL
4.1 Hierarchy of Internal Operating Procedures
4.1.1 Level 1 - Quality Policy Manual and Company Policies
4.1.2 Level 2 - Standard Operating Procedures
4.1.3 Level 3 - Work Instructions (at a department level)
4.1.4 Level 4 - Quality Records
4.2 Document Approval, Issue, Control, and Maintenance
4.3 Client-Supplied Methods and Documentation
4.4 Laboratory Notebooks, Logbooks, and Forms
4.5 Control of External Documents
5.0 SAMPLE HANDLING
5.1 Sample Collection
5.2 Sample Receipt and Entry
5.2.1 Sample Entry
5.2.2 Sample Entry
5.2.3 Sample Preservation Check
5.2.4 Sample Rejection Policy
5.3 Sample Identification and Tracking
5.4 Sample Storage
5.5 Sample Return/Disposal
5.6 Legal Chain of Custody
5.7 Representativeness of Samples
6 TECHNICAL REQUIREMENTS - TRACEABILITY OF MEASUREMENTS
6.1 Reagents and Solvents
6.2 Media
6.3 Calibration Standards
6.4 Equipment and Instrumentation
6.4.1 General Requirements
6.4.2 Standard Operating Procedures
6.4.3 Maintenance
6.4.4 Calibration
6.5 Computerized Systems and Computer Software
6.5.1 Computer Usage
6.5.1.1 Physical Security of Computer Systems
6.5.1.2 Passwords
6.5.1.3 Viruses
6.5.1.4 Internet and E-mail Systems
6.5.1.5 The Laboratory's Intranet (LabLinks)
6.5.1.6 Software Policy
6.5.1.7 Computer System Backup, Data Restoration, and Data Archival
6.1.5.8 Remote Access to Computer Systems
6.5.1.9 Electronic Data

6.5.2 System and Software Verification
6.6 Change Control
6.7 Labware Cleaning
7 PURCHASING EQUIPMENT AND SUPPLIES
7.1 Procurement
7.2 Supplier Evaluation
8 ANALYTICAL METHODS
8.1 Scope of Testing
8.2 Analytical Test Methods
8.3 Client Supplied Methods
8.4 Method Validation
8.5 Procedural Deviation
9 INTERNAL QUALITY CONTROL CHECKS
9.1 Laboratory Quality Control Samples and Acceptance Criteria
9.1.1 Blanks
9.1.2 Surrogates
9.1.3 Matrix Spikes
9.1.4 Laboratory Control Samples
9.1.5 Duplicates and Matrix Spike Duplicates and Laboratory Control Sample Duplicates
9.1.6 Internal Standards
9.1.7 Serial Dilutions
9.1.8 Interelement Correction Standard
9.1.9 Second Source Check
9.2 Quality Control Sample Frequency and Corrective Action
9.3 Quality Control Charts
9.4 Measurement Uncertainty
10 ASSURING QUALITY OF TEST RESULTS
10.1 Data Management
10.2 Data Documentation
10.3 Data Calculations
10.4 Reporting Limits
10.5 Data Review
10.6 Data Qualification
10.7 Data Reporting
10.7.1 Reporting the Results
10.8 Data Storage, Seucirty, and Archival
11 AUDITS AND INSPECTIONS
11.1 Internal Quality Assurance Audits
11.2 Review of the Quality Assurance Program
11.3 Good Laboratory Practice Critical Phase Inspections
11.4 Client Audits
11.4.1 QA Department
11.4.2 CSRs
11.4.3 Laboratories
11.5 Agency Inspections
11.6 Proficiency Testing
12 CORRECTIVE AND PREVENTATIVE ACTION
12.1 Laboratory Investigation and Corrective Actions
12.2 Investigation Process

12.3 Client Feedback
12.4 Preventative Actions
13 SERVICE TO CLIENTS
13.1 Service to Clients
13.2 Review of Work Requests, Tenders, and Contracts
13.3 Timely Delivery
13.4 Subcontracting
13.5 Use of NELAP and A2LA Logo

Cover Page

Eurofins Document Reference	1-P-QM-GDL-9015377	**Revision**	14
Effective Date	Dec 31, 2015	**Status**	Effective
Historical/Local Document Number	DOD - Environmental Quality Policy Manual		
Local Document Level	Level 1		
Local Document Type	POL - Policy		
Local Document Category	ES - Environmental Sciences		

Prepared by	Barbara Reedy, Christiane Sweigart, and Kathryn Brungard
Reviewed and Approved by	Robert Strocko;Review;Wednesday, November 18, 2015 10:39:07 AM EST Duane Luckenbill;Review;Sunday, December 13, 2015 10:34:22 PM EST Dorothy Love;Approval;Thursday, December 17, 2015 3:54:23 PM EST

Title Page

Environmental Quality Policy Manual

Eurofins Lancaster Laboratories Environmental, LLC

2425 New Holland Pike
Lancaster, PA 17601
Phone: 717-656-2300
Fax: 717-656-2681

Reviewed and Approved by:
Vice-President/Technical Director
Microbiology Technical Director
Quality Assurance Director

(as documented on page 1)

Table of Contents

1. Introduction..... 9

1.1. Mission Statement..... 9
1.2. Quality Policy..... 9
1.3. Statement of Values..... 10
1.4. Sample Flow-Through Diagram..... 12
1.5. Certifications, Accreditations, and Registrations..... 13

2. Organization and Personnel..... 13

2.1. Company Overview and History..... 13
2.2. Organizational Structure..... 14
2.3. Management Responsibilities..... 14
2.4. Overview of the Quality Assurance Program..... 15
2.5 Quality Assurance Responsibilities..... 16
2.6. Communication of Quality Issues to Management..... 17
2.7. Personnel Qualifications and Responsibilities..... 17
2.8. Relationship of Functional Groups and the Quality Assurance Program..... 17
2.9. Balancing Laboratory Capacity and Workload..... 18
2.10. Identification of Approved Signatories..... 18
2.11. Personnel Training..... 18
2.12. Regulatory Training..... 20
2.13. Employee Safety..... 20
2.14. Client Services/Project Management Responsibilities..... 21
2.15. Confidentiality..... 21
2.16. Business Conduct..... 22
2.17. Operational Integrity..... 22

3. Buildings and Facilities..... 23

3.1. Facility..... 23
3.2. Security..... 24
3.3. Disaster Recovery..... 24
3.4. Environmental Monitoring..... 24
3.5. Water Systems..... 25
3.6. Housekeeping/Cleaning..... 25
3.7. Insect& Rodent Control..... 25
3.8. Emergency Power Supply..... 26
3.9. Facility Changes..... 26

4. Document Control..... 26

4.1. Hierarchy of Internal Operating Procedures..... 26

4.2. Document Approval, Issue, Control, and Maintenance..... 28
4.3. Client-Supplied Methods and Documentation..... 28
4.4. Laboratory Notebooks, Logbooks, and Forms..... 29
4.5. Control of External Documents..... 29

5. Sample Handling..... 30

5.1. Sample Collection..... 30
5.2. Sample Receipt and Entry..... 31
5.3. Sample Identification and Tracking..... 32
5.4. Sample Storage..... 32
5.5. Sample Return/Disposal..... 32
5.6. Legal Chain of Custody..... 33
5.7. Representativeness of Samples..... 33

6. Technical Requirements – Traceability of Measurements..... 33

6.1. Reagents and Solvents..... 33
6.2. Media..... 34
6.3. Calibration Standards..... 34
6.4. Equipment and Instrumentation..... 35
6.5. Computerized Systems and Computer Software..... 38
6.6. Change Control..... 40
6.7. Labware Cleaning..... 41

7. Purchasing Equipment and Supplies..... 41

7.1. Procurement..... 41
7.2. Supplier Evaluation..... 42

8. Analytical Methods..... 42

8.1. Scope of Testing..... 42
8.2. Analytical Test Methods..... 43
8.3. Client Supplied Methods..... 44
8.4. Method Validation..... 44
8.5. Procedural Deviations..... 45

9. Internal Quality Control Checks..... 45

9.1. Laboratory Quality Control Samples and Acceptance Criteria..... 45
9.2. Quality Control Sample Frequency and Corrective Action..... 47
9.3. Quality Control Charts..... 48
9.4. Measurement Uncertainty..... 48

10. Assuring Quality of Test Results..... 48

10.1. Data Management..... 48
10.2. Data Documentation..... 50
10.3. Data Calculations..... 51

10.4. Reporting Limits..... 51
10.5. Data Review..... 52
10.6. Data Qualification..... 52
10.7. Data Reporting..... 52
10.8. Data Storage, Security, and Archival..... 53

11. Audits and Inspections..... 54

11.1. Internal Quality Assurance Audits..... 54
11.2. Review of the Quality Assurance Program..... 54
11.3. Good Laboratory Practice Critical Phase Inspections..... 55
11.4. Client Audits..... 55
11.5. Agency Inspections..... 57
11.6. Proficiency Testing..... 57

12. Corrective and Preventive Action..... 58

12.1. Laboratory Investigations and Corrective Action..... 58
12.2. Investigation Processes..... 59
12.3. Client Feedback..... 60
12.4. Preventive Actions..... 60

13. Service to Clients..... 61

13.1. Service to Clients..... 61
13.2. Review of Work Requests, Tenders, and Contracts..... 62
13.3. Timely Delivery..... 62
13.4. Subcontracting..... 63
13.5. Use of NELAP and A2LA logo..... 63

Appendix A – Procedure Cross Reference List
Appendix B – Certifications, Accreditations, Registrations, and Contracts
Appendix C – Organizational Charts, Personnel to Sign Reports
Appendix D – Personnel Qualifications and Responsibilities
Appendix E – SOPs and Analytical Methods
Appendix F – Instrument and Equipment List
Appendix G – Preventive Maintenance Schedule
Appendix H – Calibration Schedules
Appendix I – NELAP Scope of Testing
Appendix J – Quality Control Types, Frequency, and Corrective Action
Appendix K – Microbiological Testing

Revision Log

Revision: 14	**Effective Date:** This version

Revision: 14	**Effective Date:** This version	
Section	**Justification**	**Changes**
Revision Log	Formatting requirement per 1-P-QM-QMA-9017356	Removed revision logs up to the previous version
Throughout document	Title change	Update QA Manager references to be QA Director
Throughout document	Clarification	General rewording for better clarity and flow of information
Section 1.3	Reflect current version	Revised mission statement
Section 1.4	Enhancement	Revised display of information
Section 2.4	Additional scope of document	Added service centers associated with Lancaster to the scope of this document
Section 2.6	Process change	Remove reference to quarterly reports as this information is communicated to management through different means
Section 2.11.1	Process change	Added “Note” regarding training for seasonal and temporary staff
Section 2.16	Unnecessary statement	Removed reference to Eurofins sister laboratories
Section 2.17	Clarification/Process change	Clarified that the Ethics Statement is signed annually Changed Ethics Committee to Ethics hotline service
Section 3.1	Changes to campus	Updated description of campus to reflect current state
Section 3.3	Clarification	Added IT systems to the areas addressed by disaster recovery
Section 3.4	Added information	Clarified actions taken if there are adverse environmental conditions in the facility
Section 4.2	Clarification	Added explanation for applying signatures electronically to document through the document control interface
Section 5.1	Added information	Revised to include information on the bottle lot checks
Sections 5.4 & 5.5	Enhancement	Added explanations of the bar code reading process used in sample tracking and the individual bottle code tracking
Sections 5.4, 6.1 & 6.3	Added information	Specified that samples and standards/reagents are stored separately.
Section 6.3	Updated requirement	Added information regarding the need for ISO Guide 34 and ISO 17025 approved materials.
Section 6.4.4	Clarification	Added notation for reporting noncompliant data when approved by the client and comments added to the report.
Section 6.5.1.2	Added information	Specified that passwords must adhere to the Eurofins Password Policy and must be “strong: passwords
Section 6.5.2	Enhancements	Added information on the software change request, periodic reviews and retirement documents. Generalized the explanation on validation plans.
Section 8.1	Reflects current process	Changed the listing of services to current offerings and updated the website link for certification
Section 10.1	Enhancement	Added Bottle orders and clarified to reflect current flow
Section 11.1	Clarification	Added ability of QA to stop work for critical internal audit issues
Section 11.2	Process change	Added electronic means of routing documents; removed quarterly report reference
Section 11.5	Unnecessary statement	Removed the need to stamp documents as confidential

Revision: 14		**Effective Date:** This version
Section 12.1	Clarification	Explanations added regarding actions for noncompliant QC data; removed quarterly report reference
Section 12.2	Updated process	Information on the ICAR process was revised to reflect the current practice using Jira
Section 12.3	Clarification	Added information regarding QA trend evaluation of client concerns and routing of the client satisfaction survey
Section 12.4	Process change	Revised to remove references to the Ethics Committee and add information on the Ethics Hotline service; removed quarterly report reference
Section 12.4	Enhancement	Added information about use of Project Cycle to proactively ensure meeting the needs of the client
Section 13.4	Process change	Removed reference to subcontract warranty statement and added Laboratory Analytical Services Subcontract form
Appendices A-F and J-I	Updated for current information	Updated to reflect current SOPs, personnel, methods, etc.

Revision: 13		**Effective Date:** Aug 8, 2014
Section	**Justification**	**Changes**
Revision Log	Formatting requirement per 1-P-QM-QMA-9017356	Removed revision logs up to the previous version
Throughout Document	Reflect re-identification of documents in EtQ	Replaced all prior Level 1, 2, 3, and 4 document numbers (analyses excluded) with EDR numbers
Title Page	Regulatory compliance	Added text for address, phone, reviewer/approver titles (previously listed on hardcopy covers and pre-EtQ versions)
Section 1	Updated training requirements	Removed requirement for all employees to read the appendices, they are available as resources; required for dept. 4052 only.
Section 1.2	Regulatory compliance	Inserted additional ISO17025 text at opening and closing of Quality Policy Statement
Section 2.1.1	New Section	Summarize processes to ensure business continuity and contingency plans
Section 2.2	Reflect current structure	Moved summation of technical director and QA manager to this section; changed employee responsible for daily operation from COB to VP. Throughout document, clarified management structure to include VP.
Section 2.6	Added process	Added ability for management and/or QA to issue a stop work notice.
Section 2.16	Regulatory compliance	Inserted additional ISO17025 text regarding ensuring impartiality, operation integrity, etc.
Section 3	Added building	Added building D
Section 4.2	Clarification	Noted that interim amendments to controlled procedures are not allowed.
Section 5.5	Added information	Noted that minimum sample retention period is 2 weeks form reporting
Section 6.4	Clarification	Standardized use or the terminology for equipment (supporting units) vs instruments (data producing units)

Revision: 13		**Effective Date:** Aug 8, 2014
Section 6.5.1.9	New section	Added to address passwords and audit trails for systems used to process electronic data
Section 6.5.2	Clarification	Clarified SDLC processes
Section 8.1	Added information	Added reference to laboratory website for all current accreditation records
Section 10.2	Added information	Added information regarding electronic data, signatures, and audit trails
Section 10.4	Regulatory compliance	Added DoD reporting requirements for DL, LOD, LOQ
Section 10.5	Clarification	Clarified process and intent of data review
Section 10.7	Updated process	Added process for identification of accreditation status Noted use of LlabWeb for secure data transfer
Section 12.1	Added process	Added ability for management and/or QA to issue a stop work notice.
Section 12.4	Clarification, new process	Clarified processes that address preventive action; changed “PPI” to “Lean”
Section 13.2	Clarification	Added detail on project evaluations
13.4	Added detail	Added information regarding the subcontractor warranty and the need to ensure subcontractor can meet accreditation requirements
Appendices A-J	Updated for current information	Updated to reflect current SOPs, personnel, methods, etc.

1. INTRODUCTION

This *Quality Policy Manual* is based upon Eurofins Lancaster Laboratories Environmental LLC’s (herein referred to as the laboratory) overall business and management philosophies, mission, and goals. This manual is written to present the policies employed by the laboratory as well as the support departments that serve the environmental laboratories and to comply with the requirements of the National Environmental Laboratory Accreditation Program, ISO 17025, and the Department of Defense (DoD). These policies define the “what” we do with emphasis on management’s responsibilities and commitment to quality. Governing SOPs are in place within the organization, to ensure the proper execution of this policy document (refer to Appendix A). This manual is required reading for laboratory personnel. The appendices are available resources to all personnel but are not required reading for all employees. The most recent and up-to-date *Quality Policy Manual* and all referenced documents are available to all laboratory personnel who work in or support the laboratory. The laboratory actively strives for continuous improvement of its quality systems to better serve our clients.

1.1 Mission Statement

The laboratory offers analytical and consulting services in the chemical and biological sciences with comprehensive expertise in environmental laboratory applications. The company mission statement describes the corporate philosophy:

At Eurofins Lancaster Laboratories, Environmental LLC we are people working together to serve the health and environmental needs of society through science and technology. We strive to be the recognized leader in all that we do.

Our mission is to provide independent laboratory services in the chemical and biological sciences with excellent quality and service. As a corporate community, we:

- Deliver quality by fully understanding and always meeting the requirements of those we serve.

- Live our values by relating to our clients, coworkers, shareholders, suppliers, and community in a fair and ethical manner.

- Manage our growth and financial resources so we can serve our clients well, provide a satisfactory return to shareholders, and maintain our meaningful and enriching workplace.

1.2 Quality Policy

The Executive Management Group recognizes quality as a key element of the laboratory's standard of service. The group supports the laboratory's commitment to quality as defined by NELAP, ISO 17025, DoD, and other regulatory agencies (i.e. states) through the strict adherence to the Quality Policy Statement. The Quality Assurance Director wrote the Quality Policy Statement, with final approval from the laboratory Vice-President. The policy cannot be revised without their approval.

The Quality Policy Statement gives employees clear requirements for the production of analytical data. Employees are trained on the components of the Quality Policy Statement during their first day of orientation. Each employee signs the statement upon hire as agreement to implement the policy in all aspects of their work. Employee agreement to any subsequent revisions of the statement is obtained by documented reading and understanding of an agreement to follow the Quality Manual, which contains the current version of the statement. The statement is as follows:

As an organization, all personnel are committed to high quality professional practice, testing and data, and service to our clients.

We strive to provide the highest quality data achievable by:

- Following all documentation requirements; describing clearly and accurately all activities performed; documenting "real time" as the task is carried out; understanding that it is never acceptable to "back date" entries and should additional information be required at a later date, the actual date and by whom the notation is made must be documented.

- Providing accountability and traceability for each sample analyzed through proper sample handling, labeling, preparation, instrument calibration/qualification, analysis, and reporting; establishing an audit trail that identifies date, time, analyst, instrument used, instrument conditions, quality control samples (where appropriate and/or required by the method), and associated standard material.

- Emphasizing a total quality management process and commitment to continuous improvement which provides accuracy, and strict compliance with agency regulations and client requirements, giving the highest degree of confidence; understanding that meeting the requirements of the next employee in the work flow process is just as important as meeting the needs of the external client.

- Providing thorough documentation and explanation to qualify reported data that may not meet all requirements and specifications, but is still of use to the client; understanding this occurs only after discussion with the client on the data limitations and acceptability of this approach.

- Responding immediately to indications of questionable data, out-of-specification occurrences, equipment malfunctions, and other types of laboratory problems, with investigation and applicable corrective action; documenting these activities completely, including the reasons for the decisions made.

- Providing a work environment that ensures accessibility to all levels of management and encourages questions and expression of concern on quality issues to management.

We each take personal responsibility to provide this quality product while meeting the company's high standards of integrity and ethics, understanding that improprieties, such as failure to conduct the required test, manipulation of test procedures or data, or inaccurate documentation will not be tolerated. Intentional misrepresentation of the activities performed is considered fraud and is grounds for termination.

I understand the expectations and commit to implementation of all applicable policies and procedures and to providing quality data.

1.3 Statement of Values

Eurofins Lancaster Laboratories Environmental is a team of people who work together to serve the health and environmental needs of society through science and technology.

At Eurofins Lancaster Laboratories Environmental, our mission is to provide independent laboratory services in the chemical and biological sciences with excellent quality and service. We fulfill our mission by incorporating our values into our work every day.

As a corporate community, we embrace our heritage of integrity and strive to live by the following principles:

- Fairness and honesty in all our relationships
- Mutual trust
- A respect for ourselves and others
- A sense of caring that leads us to act responsibly toward each other and society, now and in the future
- Loyalty to our clients and one another
- A spirit of open-mindedness as we deal with all
- Dedication to service
- Good stewardship of our resources
- A commitment to flexibility and continuous improvement

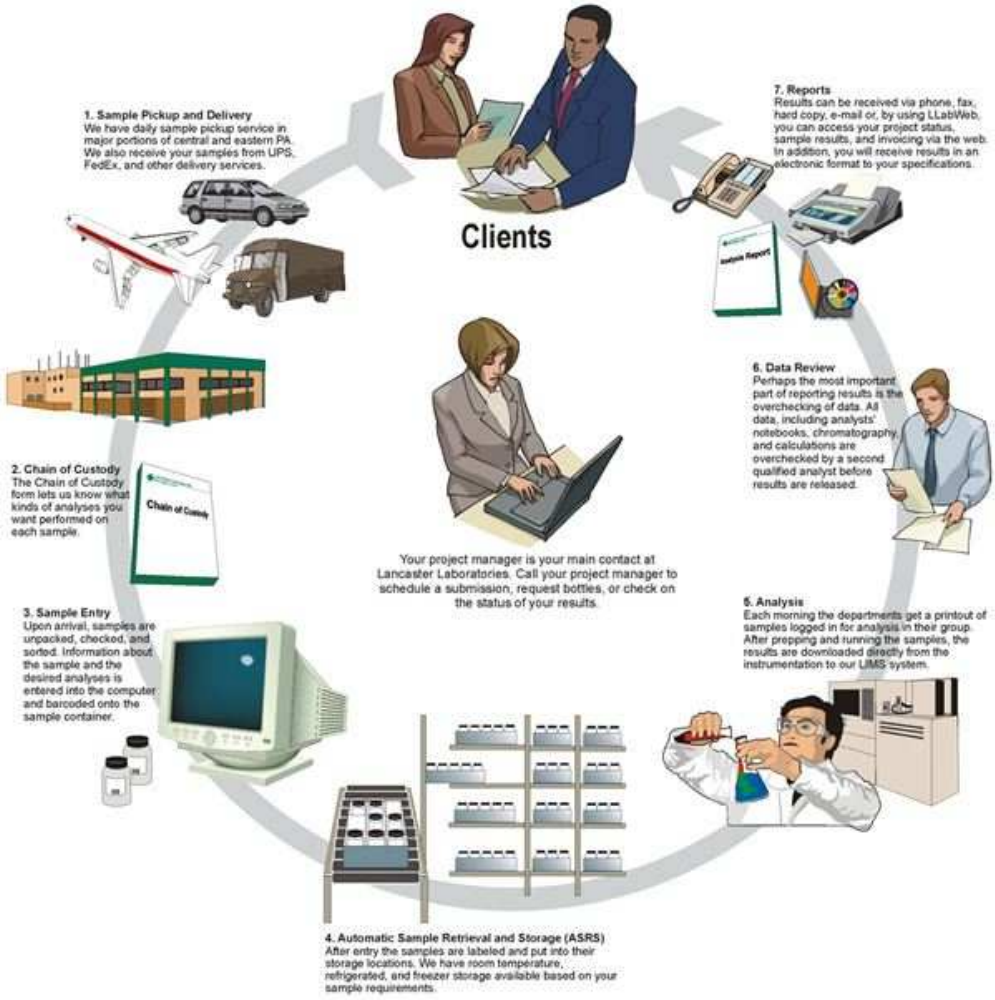
We are committed to:

- Delivering quality by fully understanding and always meeting the requirements of those we serve.
- Living our values by relating to our clients, coworkers, shareholders, suppliers and community in a fair and ethical manner.

- Managing our growth and financial resources so we can serve our clients well, provide a satisfactory return to shareholders and maintain our meaningful and enriching workplace.

At Eurofins Lancaster Laboratories Environmental, we each take personal responsibility to live these values in all of our dealings, knowing full well that our pledge may involve difficult choices, hard work and courage.

1.4 Sample Flow-Through Diagram



Certifications, Accreditations, and Registrations

Accreditation/Certification is the process by which an agency or organization evaluates and recognizes a laboratory as meeting certain predetermined qualifications and/or standards. It is the one generally accepted method by which a laboratory such as ours can demonstrate its capability of generating acceptable, professional, quality test results in those areas in which it claims competence. To this end, we have actively sought accreditation by organizations offering it in those areas relevant to our technical expertise. We strive to ensure that the facilities, equipment, procedures, records, and methods used by the laboratory in the testing of environmental samples are in compliance with the requirements of these standards.

Although organizations offering accreditation differ somewhat in the details of their programs, they generally evaluate laboratories in four basic areas: personnel (adequate staffing, education, training, and experience), physical facilities, instrumentation/equipment, and quality assurance program. This evaluation is performed by one or more of the following procedures: periodic on-site inspections of the laboratory by assessors experienced in technical operations, quality systems, and management; periodic analysis of proficiency test samples; and periodic updating of the laboratory's file to reflect changes in personnel, equipment, or services offered. Some states offer reciprocity with other state programs.

Appendix B lists accreditations and registrations held by the laboratory in support of environmental work. Current copies of all scopes of accreditation are available on the laboratory website and are kept on file in the Quality Assurance Department.

2.0 ORGANIZATION AND PERSONNEL

2.1 Company Overview and History

The laboratory was founded in 1961 by Dr. Earl Hess in response to a need for high quality technical services by the agricultural and industrial communities in southeastern Pennsylvania. Nourished in a culture of quality and caring about all those associated with the business, the corporation became an industry leader known for innovative business practices and people-friendly policies. The company was independently owned until the retirement of Dr. Hess in 1995. At that time, the laboratory was acquired by a publicly held company, Thermo TerraTech, Inc., a Thermo Electron company. Ownership changed in September 2000, when the laboratory was acquired by Goldner, Hawn, Johnson, and Morrison, Inc. (GHJ&M), a private equity investment firm. In August 2005, the laboratory was acquired by Fisher Scientific under their BioPharma Division. On November 9, 2006, Thermo Electron and Fisher Scientific merged to form Thermo Fisher Scientific. In April 2011, Thermo Fisher Scientific sold the laboratory to Eurofins Scientific. Effective July 1, 2013, the Pharmaceutical and Environmental Divisions were split into separate business entities and the company name became Eurofins Lancaster Laboratories Environmental, LLC. The laboratory continues to operate as an independent laboratory and is incorporated by the State of Delaware.

The laboratory provides a wide array of laboratory services to clients working in environmental industries. We strive to offer high quality technical services in the chemical and biological sciences with personal attention to client needs. These services include chemical analyses, microbiological testing, and analytical method development. We are, therefore, a technical service company and do not manufacturer or distribute goods. Our "product" is accurate and timely technical information and our continued existence depends on the quality of the services we offer and efficiency with which we deliver them.

2.1.1 Business Continuity and Contigency Plans

Various policies and practices are in place to address continuity of business and contingency plans to ensure continued operations or minimal disruption in operations should unplanned events (natural disasters, unexpected management changes, etc.) occur.

Section 2.2 of this document explains the identification of deputies for key management positions. Section 3.3 discusses the disaster recovery plan. Section 6.5 addresses the security and backup of our computer systems. Section 10.8 addresses handling of client records should the company have a change in ownership or go out of business.

2.2 Organizational Structure

The laboratory Vice-President/Technical Director, Duane Luckenbill, is responsible for the daily operations of the laboratory.

The Executive Management Group is defined as the Eurofins Environment Testing US Chairman of the Board and President and Eurofins Lancaster Laboratories Environmental, LLC Vice-President.

The management staff includes directors, managers and group leaders. Organizational charts are presented in Appendix C. A list of key personnel is also provided. The Vice-President and Quality Assurance (QA) Director have identified deputies for all key management personnel.

2.2.1 Technical Director

The Technical Director ensures that the laboratory's policies and objectives for quality of testing services are documented in this quality manual. The Technical Director must assure that the manual is communicated to, understood, and implemented by all personnel concerned.

2.2.2 Quality Assurance Director

The Quality Assurance Director ensures that the quality system is followed at all times. The QA Director reports directly to the Vice-President thus ensuring corrective actions to quality issues are taken promptly and are separate from business decisions. The QA Director has no direct supervisory responsibility for the generation of technical data to avoid any conflict of interest in administrating the QA program. The QA Director has the final authority to stop work that compromises our integrity or data quality. The situation must be investigated and appropriate corrective action must be put in place before the QA Director will authorize the resumption of work. The specific duties of the QA Director are communicated in job plan format.

2.3 Management Responsibilities

Laboratory management duties are outlined for supervisory personnel using a job plan format, which details each individual's responsibilities along with expected results. Typically, management duties include, but are not limited to:

- Personnel hiring and training
- Supervision of personnel
- Providing resources to ensure a work environment free from commercial, financial, and other undue pressures that may adversely affect the quality of their work
- Providing resources to ensure a safe work environment
- Directing daily work operations, including scheduling of work
- Ensuring compliance with the TNI Standards, ISO 17025, Department of Defense Quality Systems Manual, state agency programs, analytical methods, and client requirements.
- Assessing laboratory capacity and workload
- Resource allocation
- Ensuring quality of data produced

- Contributing to the continuous improvement of the laboratory operation

- Ensuring that corrective actions are carried out in an appropriate and agreed upon time-frame.

- Communicating problems and concerns to Senior and Executive Management to enlist a higher level of support for corrections and continuous improvements.

- Maintaining awareness of technical developments and regulatory requirements

2.4 Overview of the Quality Assurance Program

Quality Assurance (QA) is responsible for developing planned activities whose purpose is to provide assurance to all levels of management that a quality program is in place within the laboratory, and that it is functioning in an effective manner that is consistent with the requirements of NELAP, ISO 17025, DoD, and any other regulatory agencies (i.e. states) in which we hold accreditation. Although the laboratory is a wholly owned subsidiary of Eurofins Scientific, the Quality Assurance and Quality Systems operations described in this manual are specific to the Lancaster site and associated service centers.

The administration of the QA program is the responsibility of the QA Director in cooperation with all levels of management.

The QA program, as directed by executive management, was established to:

- Ensure accountability, accuracy, and traceability of all analytical data generated.

- Ensure that current regulatory, agency, and client requirements are being met.

- Ensure that operating procedures are in place to minimize the possible loss, damage, and tampering with data, in addition to ensuring that raw data is stored in a secured area and is maintained by designated archivists and/or system administrators.

- Ensure that curriculum vitae (CVs) and training records are maintained to document that staff members have the necessary education, training, and experience to perform their job responsibilities and functions.

- Ensure that regulatory training is provided to applicable employees on a routine and ongoing basis.

- Ensure that all procedures are available, controlled, and current.

- Ensure that documentation demonstrates that procedures are carried out in a compliant and effective manner.

- Ensure that all equipment and instrumentation is qualified, maintained, and calibrated, as appropriate, in accordance with written standard operating procedures.

- Ensure that all significant laboratory problems are investigated, evaluated for root cause and corrective action is put in place as documented

- Ensure that an internal audit program is in place to provide on-going monitoring and confirm that laboratory personnel are adhering to standard operating procedures and applicable regulations.

- Ensure that quality issues are brought to the attention of management in a timely manner.

2.5 Quality Assurance Responsibilities

The QA Director assigns tasks with input from the company Vice President. The primary responsibilities of QA include, but are not limited to the following:

- Oversee the laboratories' internal audit program which consists of various audit types and applies to all laboratory activities (technical and administrative).
- Review and approve standard operating procedures and analytical methods.
- Review and approve validation documentation.
- Review non-conforming quality control data
- Perform tracking and trending of quality measurements and report the status and effectiveness of the quality system to management.
- Approve investigation and corrective action reports (ICARs) and audit responses to ensure that they are completed in a timely manner, evaluated for root cause, that corrective actions are implemented as needed and to monitor corrective action for effectiveness.
- Host client and regulatory agencies during facility audits and follow-up to any cited deficiencies.
- Provide regulatory guidance to the laboratory and support areas.
- Monitor Good Laboratory Practice (GLP) regulatory activities.
- Communicate quality issues to management in a timely manner
- Provide and/or coordinate on-going regulatory training (e.g., GLP).
- Participate in the vendor and supplier approval process, including subcontractors.
- Review analytical data for compliance with our procedures.
- Prepare and review QA project plans (QAPPs) as required by EPA and client projects.
- Maintain and update this *Quality Policy Manual*.
- Maintenance of the Laboratory's accreditations, including but not limited to, administration of the proficiency test sample programs, both single and double blinds.
- Communicate (within 30 days) to the relevant state authorities when there are management or facility changes that impact the laboratory. Changes in the technical director must be communicated within 20 days.

2.6 Communication of Quality Issues to Management

The QA Department is responsible for preparing reports to Management to keep them apprised of outstanding quality issues. Reports to management foster communication, review, and refinement of QA activities to ensure that the QA program is adequate to meet regulatory and the laboratory's quality objectives. The following reports are used to communicate quality issues and include, but are not limited to:

- Internal, client, and agency audit reports and corrective action plans
- Proficiency test reports
- Investigation and corrective action reports
- Monthly quality status reports
- Plans for corrective action

Upon review of quality issues, management and/or QA may issue a stop work notice if an issue indicates the potential for a problem on a broader scale with an analysis. The investigation would need to be completed and the issue resolved before work could continue. The information is tracked through our Investigation and Corrective Action Report (ICAR) process.

2.7 Personnel Qualifications and Responsibilities

Full resumes and responsibilities of key personnel are provided in Appendix D.

Due to the number of analysts on staff, entry level chemists, technicians, and support personnel are not included in the resume section. However, all employees have job plans that define their responsibilities. Duties for these personnel typically include:

- Sample storage
- Sample preparations
- Performance of tests
- Calibration, operation, and maintenance of instruments
- Data entry
- Standard and reagent preparation
- Glassware preparation
- Data deliverables preparation

2.8 Relationship of Functional Groups and Quality Assurance Program

In addition to this *Quality Policy Manual*, aspects of the QA program are documented in a series of standard operating procedures that support the proper execution of this document. Technical operation procedures with required quality components are also in place. A list of the titles of relevant SOPs is provided in Appendix E. There are a variety of mechanisms used to communicate requirements and verify compliance with the QA program, including:

- Management requires that all employees read and be trained in the policies and SOPs that are pertinent to their jobs.

- Employee job plans define individual responsibilities. All job plans include QA aspects, and performance is reviewed annually.

- Laboratory audit findings are circulated to management and require a response and follow-up to items needing corrective action.

- Cross-functional meetings, including representatives from QA, Client Services, Marketing, management, and technical operations are held regularly to review specific projects and quality issues.

2.9 Balancing Laboratory Capacity and Workload

Evaluating laboratory capacity to perform specific projects is the responsibility of the Vice-President, laboratory directors and managers, and the Client Services director and manager. These responsibilities are documented in the individual job plans for these positions.

The laboratory facilities and staff size are very large compared to other laboratories serving the environmental industry. Many analysts are cross-trained to perform a variety of tests, and there is redundant equipment available in case of malfunctions. This minimizes the need to evaluate small and medium size projects against capacity available to complete them. Large projects are reviewed against capacity estimates before bids are submitted to ensure that the client's analysis schedule is met.

Regularly scheduled meetings are held with upper management, laboratory middle management, Client Services and QA personnel to review progress with current projects, as well as special requirements of new work scheduled for the laboratory.

Laboratory capacity and backlog is tracked on a continuous basis using information from the Laboratory Sample Information System (LIMS) including turnaround time, and work in-house.

2.10 Identification of Approved Signatories

All data is reviewed and verified prior to release to the client. Based on complexity or regulatory needs, some projects are designated for secondary (technical and/or QA) review of the Analysis Reports and/or data deliverables. Approved signatories for these secondary reviews are defined in the SOP on Data Entry, Verification, and Reporting. Directors, managers, group leaders, and other designated employees (such as QA, project managers, and senior technical staff) are designated to approve/release Analysis Reports. Request for approval of an employee to approve/release reports must be made through the QA Department. These authorized personnel are designated with an asterisk in the personnel list provided in Appendix C.

2.11 Personnel Training

The experience and training received by personnel is of great importance to our clients and regulatory agencies. Curricula Vitae (CVs) and on-going training documentation are available to demonstrate how personnel have been

prepared for the tasks they routinely perform. To ensure the highest quality of services at the laboratory, training programs and plans are developed to match skills with job functions. Accurate training documentation is the responsibility of both the employee and their supervisor. On a routine basis, the supervisor reviews and approves training documentation to verify that it is complete and current.

Training requirements can be met through education, prior job experience, internal and external training classes, on-the-job training, TRN training modules, procedure reading, or any combination thereof, to enable the person to perform assigned job functions and meet regulatory compliance.

Each analyst training to perform a new analysis is required to perform an initial demonstration of capability and meet the requirements for accuracy and precision before working independently on the test method. Typically, this is accomplished by the successful analysis of four known samples. However, there are certain tests performed that are not required by the mandated test method or regulation to perform the above procedure (i.e., EPA 1010, 9095). In this case, the analyst's documentation of proficiency is satisfied by the sign-off of having read, understood, and agreed to follow the SOP as written, on-the-job training and observation by a senior analyst.

Management personnel are responsible for planning ongoing professional growth and development activities for an employee through on-the–job training and/or internal and external training courses so an employee can maintain a current skill set to match job responsibilities.

An annual performance review based on job accountabilities, objective measures, and pre–defined standards is completed by management personnel for each employee. This assessment is documented and maintained. Input is obtained from other managerial personnel as needed.

2.11.1 New Hire Training

New employees are oriented as part of a year–long process that is designed to make the employee feel welcome and comfortable by defining our culture, traditions, philosophies, and work practices. During the orientation process an employee learns about personnel and safety policies and business strategies in addition to quality, ethics, and customer satisfaction expectations through a formal process administered by our Human Resources Department.

New employees are required to attend "core" technical orientation, as applicable, which can entail the participation in training module exercises, short session attendance, and/or other skill training specific to their assigned department or job function. Additional job-specific training required for an employee is based upon their assigned duties and is identified by their supervisor. Technical orientation occurs during the first few weeks of employment.

Note: Seasonal and temporary employees have reduced "core" training requirements based on the assigned tasks and as defined by QA, Safety, and the assigned department management.

The orientation process is designed to enable employees to initiate and take responsibility for their personal and professional career growth at the laboratory. The orientation process is conducted without regard to employee race, color, creed, national origin, sex, age, or disability in accordance with the laboratory's Employee Equal Opportunity (EEO) policy.

2.11.2 Ongoing Training

Refresher and ongoing training occurs through various means, which include but are not limited to, training in or independently review new/updated standard operating procedures and TRN training procedures; on-going regulatory training; in-house or off-site classes or seminars. The goal of this training is to ensure that employees

remain current with changes to laboratory systems and practices, as applicable to their job function. Retraining and re-qualification activities occur as directed by procedures or regulations. Employees are retrained if an issue or investigation warrants that retraining is a necessary corrective action. Management directs when employee re-training is required, and the extent of the re-training.

2.12 Regulatory Training

The QA Department is responsible for coordinating and conducting initial and ongoing regulatory training (i.e., GLP) for all applicable laboratory and support personnel. It is the responsibility of management within each department to ensure that personnel attend the required training sessions.

The choice of training format and topics covered for ongoing regulatory training is left to the discretion of QA and the trainer. All training sessions reinforce the concepts in the regulations as they are relevant to the laboratory.

Whenever possible, after training is completed, a demonstration of proficiency of the training topic is given. The demonstration of proficiency is generally in the form of a quiz although other demonstrations of proficiency are acceptable depending on the scope and content of the training. If necessary, training is presented and/or repeated one-on-one with individuals who do not demonstrate proficiency in the training topic. This is performed by QA in conjunction with applicable laboratory management personnel.

2.13 Employee Safety

The laboratory, being mindful of its responsibilities as an employer and active corporate citizen, has established the following objectives of its safety program:

- Provide a safe environment for its employees, visitors, and the community surrounding its place of business.
- Provide ongoing safety training for employees.
- Provide all necessary facilities and equipment to ensure the safety of its employees and to minimize all chemical exposure during the normal performance of their required tasks, and to take all necessary precautions to safeguard the surrounding environment.
- Provide periodic health physicals for employees.
- Foster and encourage safe operations and a proper safety attitude on the part of our employees through general operations and systems, training, and the *Chemical Hygiene Plan* (CHP).

The CHP addresses various aspects of our safety program in greater detail.

A Safety Committee works to enhance our overall safety program. The committee meets on a routine and ongoing basis and its specific responsibilities are detailed below:

- Review accident and incident reports. Make recommendations for methods of prevention to eliminate further accidents.
- Promote safety awareness and distribute safety information by various means (e.g., posters, videotapes, pamphlets, and books). Use internal communication channels to promote safety awareness.
- Enhance and recommend safety-training programs for all employees, as necessary.

- Maintain up-to-date information on employee concerns that are safety related. Offer input and information to the Chemical Hygiene Officer and/or Safety Officer, as needed.

2.134 Client Services/Project Management Responsibilities

Members of the laboratory Client Services/Project Management Group are responsible for organizing and managing client projects. Clients are assigned a project manager (a.k.a. "CSR") who serves as their primary contact at the laboratory. It is the project manager's responsibility to act as the client advocate by communicating client requirements to laboratory personnel and ensuring that clients provide complete information needed by the laboratory to meet those requirements. All client verbal communications are documented by the project manager in a controlled notebook. In addition to information management, Project Management responsibilities include:

- Coordinating and preparing proposals in conjunction with technical staff.
- Confirming certification status.
- Hosting client visits and audits.
- Coordinating and communicating turnaround time (TAT) requirements for high priority samples/projects.
- Answering common technical questions, facilitating problem resolution.
- Providing clients with sample status report or results (partial reports) prior to receipt of the final Analysis Reports (e.g., fax, e-mail, phone).
- Scheduling sample submissions, sample containers, and sample pick-up via the laboratory courier service.
- Informing the client of deviation from their contract.

2.15 Confidentiality

Strict confidentiality is maintained in all of our dealings with clients. Confidentiality agreements, therefore, are willingly provided.

All employees are required to protect company technical data, including client names and test results from disclosure to any third party. This policy, as described in the *Eurofins Lancaster Laboratories Employee Handbook*, is provided and presented to employees during their orientation period and whenever revisions are made.

Intellectual property associated with the testing that we perform under contract for a client is the property of the client.

In an attempt to ensure the confidentiality of our systems and procedures within our laboratory, it is our policy to restrict the distribution of our internal procedures to clients. Clients are permitted to review our procedures while on-site as part of an audit or visit. Based on this policy, we would request that any documents viewed would not be shared or made available to any third parties without the permission of the laboratory.

2.16 Business Conduct

Our business conduct policy applies to all operations of the company. All employees must avoid involvement in any activities that would diminish confidence in their competence, impartiality, judgment, or operational integrity. All employees must further avoid any relationship with other individuals or organizations that might impair, or even appear to impair, the proper performance of their company-related responsibilities. Employees must avoid any situation that might affect their independence of judgment with respect to any business dealings between the company and any other organization or individual. Any employee who believes that they have such a conflict, whether actual or potential, or who is aware of any conflict involving any other employee must report all pertinent details to the Vice-President or President of the company. The company's management vigorously enforces this policy and takes prompt and appropriate action, including termination, against any employee found to be in violation.

2.17 Operational Integrity

All employees review and sign the Employee Ethics Statement on their first day of employment and annually thereafter. Employees responsible for generating, handling, or reviewing laboratory data understand that the laboratory mission is to perform all work with the highest level of integrity. Under no circumstances are shortcuts or generating results to suit a client's purpose rather than good scientific practice considered acceptable. Any violation of the laboratory ethics policy results in a detailed investigation that could lead to termination.

All levels of management consider the following activities unacceptable:

- Knowingly recording inaccurate data.

- Fabrication of data without performing the work needed to generate the information. This includes creating any type of fictitious data or documentation.

- Time travel or adjusting clocks on computerized systems to make it appear that data was acquired at some time other than the actual time.

- Manipulation of data for the express purpose of passing system suitability or quality control criteria.

- Selective use of data generated, or not using data that was legitimately generated and has an impact on the outcome of the test.

- Executing significant deviations from approved test methods and procedures without prior approval from the laboratory management and/or the client.

If an issue does arise which could compromise data integrity, personnel are instructed to perform the following activities:

- Clearly document the situation and maintain all data generated. There is a big difference between poor judgment and fraud. Fraud usually involves intent to conceal an action taken. Therefore, the more documentation that is maintained, the less likely an action is considered fraudulent if further scrutinized.

- When out-of-specification results or quality type issues are detected, all supporting data and relative background information must be documented and presented for management review. Problem resolution and client contact, as applicable, must also be documented.

- Review any questionable situations and decisions with a supervisor.

- Bring a questionable or uncomfortable issue directly to the QA Director or a member of the QA Department as part of our QA open door policy.

- Utilize the company's anonymous Ethics hotline service. See Section 12.4 of this manual.

3 BUILDING AND FACILITIES

3.1 Facility

The laboratory is located at 2425 New Holland Pike, Lancaster PA. The facility consists of two campuses with multiple buildings located on the North and South sides of Route 23. The two campuses are connected by a pedestrian bridge that spans Route 23.

Building A resides on a commercial plot measuring 13.6 acres on the north side of Route 23. Building A is a three-story building of concrete and steel construction which houses both laboratory space and administrative offices. It is approximately 108,000 square feet and consists of approximately 47,000 square feet of laboratory space; 29,000 square feet of office space; and 32,000 square feet of storage, mechanical, and common areas. On this parcel, adjacent to Building A, sit two chemical storage buildings (Buildings I and L) with a total space of 2500 square feet. In addition, a 10,500 square foot storage building houses stability chambers (Building J). The bottles packing area, which includes preservation of bottles being sent to clients for sampling, is located in a separate 3100 square foot building (Building K). In addition, there are two other buildings (Buildings G and H) with a total square footage of 20,000 square feet that host recycling, storage, workshop and facilities maintenance areas.

The remaining buildings reside on a commercial plot measuring 35.7 acres on the south side of Route 23. These building are connected to the north campus buildings via a pedestrian walkway over the highway.

Building B is a three-story building of steel and concrete construction. It is approximately 56,000 square feet and consists of approximately 17,000 square feet of laboratory space; 14,000 square feet of office space; and 25,000 square feet of storage, mechanical, and common areas.

Building C resides between buildings B and D and consists of a three-story building of steel and concrete construction. It is approximately 47,000 square feet and consists of approximately 25,000 square feet of laboratory space; 6,900 square feet of office space; and 15,100 square feet of storage, mechanical, and common areas. The first floor houses the main lobby and visitor's entrance.

Building D is connected to building C. It is a 78,000 square foot, four-story building of steel and concrete construction and provides approximately 35,000 square feet of laboratory space, 19,000 square feet of office space, and 24,000 square feet of storage, mechanical, common area.

Two small support buildings (Buildings E and F) with a combined space of approximately 800 square feet are used for chemical and waste storage on the south campus.

The Lancaster campus also utilized an adjacent parcel for a technical training center. This space is approximately 6,500 square feet.

There is an automatic fire alarm and security system hooked up at the facility. This system is monitored offsite by Choice Security. The entire campus and all exterior doors are monitored by video surveillance.

This facility is serviced by public sewer. Drinking water comes from a private well while the facility sprinkler system is fed by the public water supply. The closest surface water is the Conestoga Creek.

3.2 Security

The laboratory is considered a secure facility. All outside doors except the main lobby entrance are locked during normal business hours to prevent unauthorized entry. An attendant monitors this entrance at all times.

During evenings, weekends, and holidays, all doors are locked and Security personnel are on site to prevent unauthorized entry into the building. Video cameras are utilized by Security personnel to monitor the facility grounds.

Every employee is issued a photo ID badge which also serves as a building access card. This badge must be worn at all times while on laboratory property so that employees are easily identified. Access to secured/designated areas within the building is limited to only applicable employees through the building security system. This system is administered by Security staff.

All visitors must register with the lobby attendant and are issued a visitor badge. A staff person must accompany visitors while in the facility. Additional visitor rules are outlined in the *Visitor Security and Safety Rules* pamphlet which is provided to all guests.

Building access cards are issued on a temporary basis to contractors or service technicians (e.g., electricians and plumbers) who need access to the building to work on a project. These cards provide the contractor with limited access during the normal workday and must be returned when the work is complete.

3.3 Disaster Recovery

A disaster recovery plan is in place to provide direction for situations where normal operations of the laboratory are not possible. In the event that the building or information technology (IT) systems would be severely challenged, a designated disaster recovery team, which includes Physical Services, Maintenance, Safety, Corporate Management, Public Relations, IT, QA and other applicable personnel depending on the scope of the disaster, would assemble at a designated area to assess the situation and formulate a plan.

The plan addresses, in general terms, how to approach the following issues: electrical failures, heating/air conditioning failures, fire/building evacuation, computer failures, hazardous material spills, injury to employees, pandemic flu, disruption of phone service, and stability chamber failures.

3.4 Environmental Monitoring

The air handling system for the main laboratory is specially designed to protect sensitive instruments from harmful vapors to ensure that samples are not contaminated. The Physical Services/Maintenance Group is responsible for maintaining the HVAC and exhaust hood systems. This is particularly important in our instrumentation rooms and computer center where a controlled environment, positive pressure system is maintained.

Most refrigerators, freezers, incubators, and ovens used for analysis are monitored by a computerized system equipped with stationary thermometer temperature probes linked to a master panel that is accessed through a computer. If a unit is outside of a predefined temperature range for a specified period of time, the system alarms. Units not on the computerized system must be monitored manually by recording thermometer temperature readings twice daily.

The laboratory is set up so that there is effective separation between neighboring areas in which there is potential for contamination. Laboratory storage blanks are also used to evaluate conditions under which samples for volatile

analysis are stored to monitor for cross-contamination potential. QA provides oversight of the environmental monitoring system.

QA and technical management, in consultation with facilities management as needed, evaluate any issues with environmental conditions that could have adverse effects on data to determine if alternative operational plans (moving testing to alternate laboratories, temporary shutdowns, etc.) need to be employed.

3.5 Water Systems

Well water and the public sewer system service the facility. The water system is monitored to meet the permit requirements of the Pennsylvania Department of Environmental Protection.

Reagent water is available to analysts for sample preparation (including dilution) and glassware cleaning. Two reverse-osmosis deionized water systems deliver highly purified water to a sealed fiberglass storage tank. From the storage tank the water is delivered to an ion-exchange-carbon filter system for further polishing. The water is also exposed to an in-line ultraviolet sterilization lamp before being circulated to taps throughout the laboratory.

Daily monitoring and preventive maintenance for the system is the responsibility of the Physical Services Department. Monthly and annual testing is performed as required by regulatory guidance. QA provides oversight of the water system monitoring. In addition, method blanks are tested with each batch (≤20) of samples.

3.6 Housekeeping/Cleaning

The laboratory is dedicated to providing a clean workplace. A third party professional cleaning service provides routine cleaning of “common areas” that include lavatories, drinking fountains, floors, and windows. Technical staff are responsible for the cleaning (or the contract of cleaning) of specific laboratory work areas.

Detergents used for cleaning contain no to very low levels of metals, pesticides/herbicides/ fungicides, or volatile solvents.

3.7 Insect & Rodent Control

Steps are taken to prevent, monitor, and control insect and rodent infestation. The coordination of this program is the responsibility of the Physical Services Department under the direction of QA. An outside service firm is contracted to perform routine and ongoing monitoring of the facility to ensure that preventive measures which are in place are effective and are working as intended.

No insect or rodent control chemical agents in a liquid or vapor form are applied or sprayed in any laboratory building, unless there is no other option, in which case department management must be contacted for approval.

3.8 Emergency Power Supply

The laboratory is located at the junction of two power grids that supply electrical service to the facility. If one of the power grids fails, we have the ability to work with the power company to have service switched to the other grid. Various types of diesel and natural gas generators are also available on a standby basis to supply power to selected areas of the laboratory in case of a power outage.

To reduce spikes and spurious line voltage changes to laboratory instruments that can affect results or damage electronic equipment, “conditional power” is fed to these sensitive instruments. All essential computer systems are

on uninterrupted power supply (UPS) which is a battery system that provides continuous conditional power for a limited time period in the event of a short power outage.

3.9 Facility Changes

Procedures are in place to manage change, ensure communication, and to minimize negative consequences through active participation of personnel involved in a facility change. The goal is to ensure that physical and environmental condition changes are adequately evaluated for impact and reduction of risk to quality, safety, health, employee, environment, property, analytical services, and business operations before and after the change is implemented.

4.0 DOCUMENT CONTROL

The administration of the document control system including tracking, filing, updating, and archiving of historical copies is the responsibility of the Office Services (OS) Department.

It is our policy to restrict the distribution of our internal procedures to clients and we discourage the distribution of company confidential documents outside of the facility. Clients are permitted to review our procedures while on-site as part of an audit or visit. Any documents that are distributed are only sent with the approval of QA.

The goals of the document control process are:

- Format documents according to consistent and defined standards
- Review and approve new documents
- Schedule review of existing documents
- Control of document versions and effective dates
- Review and approval of document changes
- Control document distribution and removal of obsolete documents
- Archive and protect obsolete documents

4.1 Hierarchy of Internal Operating Procedures

The hierarchy of controlled procedures at the laboratory is defined. These procedures and documentation are made available to promote consistency throughout the organization and to meet regulatory requirements. A list of relevant methods and procedures is located in Appendix E. The development of new procedures and the updating and reclassification of current procedures is an ongoing project.

4.1.1 Level 1 - Quality Policy Manual and Company Policies

The intent of these documents is to define “what” we do with emphasis on Executive and Management’s responsibility for quality.

The purpose of the *Quality Policy Manual* is to provide a framework to outline the quality systems at the laboratory. Organizational charts, list of SOPs, a list of equipment, instrumentation, and personnel resumes are included as attachments to this manual.

- Executive Management is responsible for ensuring that adequate personnel, resources, and support are available to carry out the requirements of this *Quality Policy Manual*.

- Management is responsible for ensuring that SOPs or other appropriate documents are written and available to personnel to define the practices and systems which support these policies.

- All employees are responsible for conducting business in a manner which is compliant with quality and company policies and associated SOPs or other appropriate documents. Review of these policies and procedures must be documented.

Additional company policies are written to support and expand upon this *Quality Policy Manual*. These policies contain more detailed information about a subject with approval signatures executed at the Executive and/or Management level.

4.1.2 Level 2 - Standard Operating Procedures

The intent of these standard operating procedures is to define "who, what, where, and when." These procedures provide specific information for a process or topic so that the requirements outlined in this *Quality Policy Manual* and company policies can be achieved. The review and approval of these SOPs is performed at the director/manager/group leader level, including QA review and signoff, and the responsibility of these SOPs lies with the area or person directing the operation.

SOPs can apply to site-wide operations, the entire company, across multiple departments, or a specific operating area.

4.1.3 Level 3 - Work Instructions (at a department level)

The intent of these procedures or documents is to define in greater detail the specific "how to". The level of detail in these documents must be sufficient so any appropriately trained person can perform the task accurately. Examples include, but are not limited to standard operating procedures (SOPs); maintenance and calibration procedures; and the laboratory analytical methods. Departmental level procedures/documents are reviewed and approved at the manager or group leader level including QA review and signoff.

4.1.4 Level 4 - Quality Records

The intent of these documents is to provide documented evidence to support our quality systems and operations. Examples include but are not limited to, data notebooks/logbooks, and preformatted data recording forms.

4.2 Document Approval, Issue, Control, and Maintenance

The document control process ensures that documents are approved and adequate for use. It ensures that documents are readily available to personnel and at locations where essential operations are performed.

Procedures are available in electronic form on the company's intranet site through our document management system. The Document Control Group maintains this system in a current and accurate state. These procedures can be printed from this system for reference by employees as the corresponding task is being performed. Prior to using a printed document, the employee must ensure that it is the current version.

Each procedure is uniquely identified and includes effective date, revision identification, and page numbering (total number of pages). All documents are searchable and uniquely identified in the document management system.

Controlled policies, procedures, and work instructions are reviewed and approved by appropriate individuals and are formally issued and administered through the Office Services Group. The review and approval signatures are applied as electronic signatures through the document control interface. Application of the signature is through secure log-in and password and can only be applied by those designated for the review or approval of the individual document.

Word versions of each procedure can be accessed within the document management system by designated personnel within the Document Control group. A PDF copy is maintained on a separate limited access server as a back up to the system.

Procedures undergo scheduled periodic review to ensure that they are accurate, current, and compliant. The frequency of review is either annual or biennial, depending on the procedure. QA is the final signature on procedures which gives QA the authority to implement the procedure; the exception is the Quality Assurance procedures for which the Vice President or his designee is the final signature. Upon the effective date of new or updated documents, all copies of obsolete documents are removed from service. The original historical copy of each outdated/obsolete procedure is clearly identified as a historical version and maintained in a permanent archive file separate from any current versions. (Note: OH EPA is required to review all revised documents applicable to its certification prior to the document being made effective).

Interim amendments to procedures are not allowed. Any needed changes require a revision to the document.

4.3 Client-Supplied Methods and Documentation

Client documentation to support environmental testing at the laboratory is maintained in a centralized area. This information is organized by client/project in the Client Services/Project Management Group. Client documentation includes the following information depending on project size and scope:

- Client supplied analyte lists
- Client supplied project plans
- Client contract quality manuals with specified limits, QC criteria, etc.
- Communication/correspondence records which relate to testing requirements, interpretation of results, or reporting formats

4.4 Laboratory Notebooks, Logbooks, and Forms

Procedures are in place to ensure that all data is traceable, authentic, complete, and retrievable. The following general requirements outline our system for the issuing, control, and archival of laboratory notebook and logbooks.

- The administration of notebooks and logbooks is controlled by the Office Services Group. They maintain a master index to uniquely number and identify each book distributed.
- Notebooks and logbooks can contain blank or preformatted pages.
- Notebooks and logbooks are bound, uniquely identified and have sequentially pre-numbered pages.

- If notebooks or logbooks contain preprinted laboratory form pages:
- A unique identification number is assigned to each form
- Forms are approved by appropriate management personnel before they are put into use
- Forms are reviewed on a routine basis to ensure they are still accurate and current
- Completed notebooks are returned to an archivist. Incomplete books are returned to Document Control:
- Two years from the issue date
- for employee specific notebooks – when the employee leaves the company
- for project specific notebooks – when the project for which it was used is complete
- In specific situations, records are bound to create books at the time of archival (e.g., temperature charts).
- At the time of archival any page(s) in the notebook or logbook that does not contain data documentation is crossed-out or a statement is written on the last page used to note that the book is complete to prevent data from being entered at a later date.
- Notebooks and logbooks identified as requiring permanent archival are assigned a designated qualifier.

4.5 Control of External Documents

Hard copy versions of external documents are controlled through the form system.

External documents such as copies of the 40 CFR and ASTM methods are stored exclusively in the QA Department. QA also keeps applicable agency documents on file, these include, but are not limited to, the TNI (The NELAC Institute) and ISO 17025 standards.

Environmental methods from the EPA or Standard Methods are available in the QA Department, but the technical areas also have copies that pertain to the tests that they perform. Any external document that is maintained in these areas must be inventoried and listed on a controlled form. Some methods are available on-line and are accessed through the Internet.

It is the laboratory's understanding that the need to control external documents is to ensure that the most current version of a method is referenced or appropriate manual is being used. Regulatory methods are used as references by the laboratory and testing is performed as per written SOPs that fall under our existing document control system and have scheduled reviews. The scheduled review of SOPs is used to ensure that the proper version of a method is referenced. While using the most current version of an analytical method is our typical practice, there are specific client needs and accreditation rules that require previous versions of a method to be used.

The technical areas are responsible for ensuring that all manufacturers' manuals are current and available to analysts. The vendor provides instrument manuals when new equipment is purchased or existing instruments are updated. These manuals are kept with the instruments to which they are associated.

5.0 SAMPLE HANDLING

5.1 Sample Collection

It is the responsibility of the client to send us representative and/or homogeneous and properly preserved samples of the system from which they are drawn. The laboratory assumes that all multiple sample containers with the same designator/description and bottle type contain a homogeneous, representative sample. We also assume that it is acceptable to deplete one container and move to the next, without implications unless otherwise indicated by the client.

The laboratory provides the appropriate sample containers, required preservative, chain-of-custody (COC) forms, shipping containers, labels, and custody seals. The laboratory also provides trip blanks and analyte-free water for field blanks. Preparation of methanol containers for field preservation of volatile soil samples is available.

Sample containers are purchased pre-cleaned by the supplier. For pre-preserved bottles, each lot of preservative is checked for contaminants before use. This also serves as a check on the associated containers. An annual bottle lot check is performed to evaluate the cleanliness of any containers not already covered by the preservative checks. The evaluation is to assess cleanliness to the laboratories' detection limits. These checks are processed through the LIMS as samples. Results are documented through the LIMS Analysis Report.

The laboratory provides instructions with all bottle orders that define how to sample, preserve, store, and ship the samples prior to their delivery at the laboratory. These instructions inform the client of the importance of proper sampling and advise them that non-compliant samples are rejected or reported with a qualifier.

If samples are collected by the laboratory personnel, applicable sampling methods are in place to perform the sampling operation.

As samples are analyzed at the laboratory, there are times when additional sample volume is necessary to complete testing or perform retesting. If this situation arises, "additional sample" is requested by the laboratory and/or submitted by a client to supplement current work being performed within our facility. Additional sample received is either assigned a new laboratory sample ID number and/or a comment noted on the final report to state that additional sample was received, depending on the situation. It is our goal to provide accurate traceability between sample submission and when testing is performed.

5.2 Sample Receipt and Entry

5.2.1 Sample Entry

Samples can be received at the laboratory 24 hours a day, 7 days a week, 365 days of the year. Receipt can occur in one of three ways:

- The laboratory courier services (i.e., Transportation Department)
- Personal delivery
- Commercial courier

All samples received for testing are delivered to the Sample Administration Department immediately upon arrival. This group is responsible for the unpacking and organizing of the samples. This process includes checking custody seals if present, paperwork agreement, signing the chain of custody, recording cooler temperatures, documenting the condition of containers, accounting for all sample bottles, and observing any safety hazards, and

reporting any problems to Client Services for communication to the client. This receipt process is documented in the LIMS.

5.2.2 Sample Entry

As soon as practical after sample receipt, all samples are entered into our LIMS. Samples awaiting log-in are stored in temporary holding areas, at appropriate storage conditions to maintain sample integrity. Samples scheduled for Volatile analysis are stored separately. If there is doubt about the suitability of items received or if items do not conform to the description provided or the testing required is not clear or specified, the client is contacted and the conversation documented.

At the time of entry, the LIMS assigns a unique laboratory sample number to each sample. This number is sequentially assigned and a label is generated and is attached to the sample container.

Samples are tracked to the minute upon arrival. This allows the client to see exactly how long it took the samples to pass through receipt, unpacking, and entry.

A sample acknowledgement is generated from the LIMS per sample entry group. Upon request, a copy of the Acknowledgement may be sent to the client on the day following sample log-in to confirm sample receipt and entry. Internally, appropriate personnel audit all applicable sample entry and client paperwork.

5.2.3 Sample Preservation Check

Support personnel check and document preservation of non-volatile liquid samples after the samples have been entered into the LIMS and before they are placed into storage. Any checks of volatile samples are performed and documented at the time of analysis.

5.2.4 Sample Rejection Policy

Any time a sample is received in a condition that does not meet the method, regulatory, or client requirements, the condition of the sample is clearly documented through the LIMS on a sample administration documentation log or sample problem form. This information is forwarded to the CSR and the client is contacted to discuss the best course of action. The client is given the option to resample or have the sample analyzed and reported with a comment.

5.3 Sample Identification and Tracking

A sample label is generated for each sample and, in addition to the assigned unique sample number, the following information is displayed on the label: client name, sample identification assigned by the client, sample collection information, bottle code ID, analyses requested, and any applicable notes to laboratory personnel. The label includes a barcode that is used to track this information about the sample/container and to trace each container's storage location.

To ensure accountability of results, the unique sample number assigned is used to identify the sample in all laboratory data documentation, including notebooks, instrument printouts, and final reports. The sample number is also used to identify additional containers of the sample that are created during sample preparation and analysis (e.g., subsamples, extracts, digests). Each container for a sample is tracked through the bottle code and an A.B.C… designator when there are multiple containers of the same type received. The link of the bottle code and sample number is used to identify which specific container was used for testing.

Routine sample tracking is documented using the Laboratory Sample Analysis Record (LSAR) which captures the date, time and analyst for each sample preparation and analysis. The information is compiled in the LIMS using electronic record tracking from the data upload and entry functions. This displays, per sample, on each Analysis Report.

5.4 Sample Storage

After sample entry, samples are placed in an assigned and identified storage location until needed for analysis. Room temperature, refrigerated, and frozen storage are available and samples are stored in accordance with regulatory, method, or client direction. The LIMS is used to assign storage locations, which assists in the orderly storage of samples. Sample storage locations are secured and monitored for accurate temperature control. Samples are stored separately from standards and reagents.

The central locked storage facility contains 3430 square feet of refrigerated space, including 2740 square feet equipped for automated sample retrieval. Samples are stored in the laboratory's automated storage and retrieval system (ASRS) or other assigned storage locations (separate volatiles areas) within the laboratory until completion of all analytical work.

When a sample is scheduled for analysis, the analyst requisitions it through the LIMS from the storage area. Barcode readers are used for LIMS documentation of the movement of the samples between storage and the laboratories. To maintain the integrity and security of the sample(s), the aliquot needed for analysis is removed and the sample(s) returned to storage as soon as possible

5.5 Sample Return/Disposal

Samples remain in the storage area following analysis until the testing results have been verified and the analysis report has been generated. On a regular basis, a list is generated from the LIMS that summarizes samples that can be removed from the storage area. At a minimum, water samples are held for 1 week and soil samples for 2 weeks after reporting before they would be eligible for disposal. Samples are either returned to the client or disposed of in accordance with local, state, and federal regulations. Removal of the containers from storage for permanent discard is also documented in the LIMS using the barcode reader.

Due to the variety of waste generated at the laboratory, several general categories of wastes and waste streams have been identified. Identification of waste occurs through information provided by the client, historical information, and/or analytical testing. The laboratory uses a sophisticated, computerized LIMS, which includes programming to assist in the identification of hazardous wastes at time of discard.

For reasons of environmental liability, client confidentiality, proprietary product formulation protection, etc., wastes generated by the laboratory are disposed of via incineration at EPA licensed facilities. The three exceptions include bulk neutralized acid waste, COD analysis waste, and lab pack waste containing mercury. None of these exceptions involve containers with client information.

5.6 Legal Chain of Custody

Samples being tested for litigation require locked storage and documentation of the time and personnel responsible when the sample was not in storage. This level of documentation is available upon client request and procedures to define these activities are in place and include the following:

- A chain-of-custody document is initiated for each bottle type submitted by the client.

- The chain of custody is signed each time the sample is stored, removed from storage, or changes hands.

- Clients requesting internal chain-of-custody documentation receive the completed forms after the analysis is complete.

5.7 Representativeness of Samples

Each analytical method provides specific procedures for ensuring that a representative aliquot of the sample is used for testing. These procedures include shaking water samples and mixing solid samples prior to removing an aliquot for testing. Analysts are also instructed in sampling techniques that prevent contamination of samples.

6 TECHNICAL REQUIREMENTS - TRACEABILITY OF MEASUREMENTS

6.1 Reagents and Solvents

The reliability of our analytical results can be directly affected by the quality of reagents used in the laboratory. Procedures are in place to address labeling, storage, and evaluation of these materials. Reagents and solvents include acids, bases, indicators, buffer solutions, colorimetric solutions (CS), test solutions (TS), and volumetric solutions (VS). The *Chemical Hygiene Plan* provides safety information in regard to the storage and handling of laboratory chemicals. All reagents are stored separately from samples.

Each analytical method includes a list of reagents needed to perform the test. Reagents/solvents are fully described, including chemical name, purity, and description of preparation. Where applicable, shelf life and storage conditions are also listed. The laboratory is responsible for checking that new supplies meet the method requirements. These checks are documented and maintained.

Departmental management ensures that an adequate inventory of reagents needed to perform testing is maintained. Reagents received at the laboratory funnel through the Shipping and Receiving Department and deliveries are verified and labeled with the date of receipt. Large volume reagents (e.g., solvents, acids) are stored in a building outside of the laboratory until needed for use.

In addition to the name and concentration of the reagent, all reagents are labeled with the manufacturer/vendor, storage conditions, the date opened, and an expiration or re-evaluation date. Before using any reagent, the analyst must ensure that the material was properly stored and labeled. If a reagent has passed its expiration date or shows signs of deterioration, the material is not to be used in the laboratory and must be discarded or segregated as expired. In some method development or research work, expired reagents may be used. These must be labeled as such or stored in a designated location.

If a re-evaluation date is reached before a reagent is completely consumed, the reagent will be inspected by physical observation for signs of degradation. Physical signs include, but are not limited to, color changes, clumping or other texture changes for solids and formation of precipitate in solutions. This evaluation is performed by an experienced chemist or microbiologist.

Subsequent reagent solutions or mixtures prepared at the laboratory are fully documented in a logbook and labeled to include: unique name, concentration, date prepared, name of analyst who prepared the reagent, storage conditions or reference to the logbook containing these details, and expiration/re-evaluation date. The information recorded allows these solutions to be traced to the original stock solution. The reference to the logbook is intended for use on containers that are too small to clearly document all of the information.

All reagent certificates and MSDSs are retained by the laboratory.

6.2 Media

Within the microbiology laboratory, procedures are in place to address preparation, labeling, storage, expiration, documentation, and quality/sterility evaluation requirements for these materials. These procedures are described in Appendix K.

6.3 Calibration Standards

Written calibration procedures are required, where applicable, for all instruments and equipment used in the laboratory. The source and accuracy of standards used for calibration purposes are integral to obtaining quality data. Requirements for calibration are provided in each analytical method including specifications for the standards used. Where available and practicable, calibration measurements made by the laboratory must be traceable to national standards of measurement (e.g., NIST). Certificates of Analysis (C of As) are maintained for each material, as applicable.

The laboratory's ISO 17025 and DoD accreditations require calibration materials to be certified and purchased from a reference material producer accredited to ISO Guide 34 and ISO 17025, when available. A list of accredited suppliers is maintained by QA. This is applicable to the tests under these scopes of accreditation and can be met through the stock standards used for calibration; a standard processed under the calibration such as an ICV or LCS; or comparison to a separate reference material at a frequency defined by at the test level (i.e. annually).

Standards are usually purchased from commercial supply houses either as neat compounds or as solutions with certified concentrations. Upon receipt at the laboratory, the material must be labeled with the date of receipt. The accuracy and quality of these purchased standards is documented on a C of A and these certificates are maintained on file in the laboratory. .

Most solutions and all neat materials require subsequent dilution to an appropriate working range. Records of all standard preparations include the dilution(s) made and a reference to the original and any intermediate mixtures. Solutions are labeled according to laboratory procedures and assigned unique names or code numbers that provide traceability to the original components.

All standards are stored separately from samples and in conditions as stipulated by the method or vendor (refrigerator, freezer, room temperature, etc.).

Each new preparation of standard is tested for integrity by comparison to standards from another source or previously prepared solutions. Standards are not used for sample analyses in the laboratory past their expiration date. In some method development or research work, expired standards may be used. These must be labeled as such or stored in a designated location.

6.4 Equipment and Instrumentation

The laboratory is equipped with all equipment and instrumentation required for testing the scope of work which it supports. All equipment and instrumentation is maintained in proper working order. A master list of our equipment and instruments is maintained by our accounting department and includes the date received and the condition at receipt (new v. used). Our major equipment and instrumentation capabilities are summarized in Appendix F. In addition, we have numerous other instruments including pH meters along with support equipment such as ovens, incubators, centrifuges, balances, etc.

6.4.1 General Requirements

- Equipment/instrumentation is assigned a unique designation. This unique number or system identification is used to track the equipment or instrument within data documentation.

- A maintenance logbook is established in conjunction with installation and is readily available to document all incidents and/or routine maintenance processes that pertain to the equipment or instrument as they occur. The corrective action taken, the date that the equipment/instrument is returned to service, and performance checks performed is documented.

- All test, measuring, and inspection of laboratory systems, equipment, and instrumentation used at the laboratory is routinely calibrated and maintained in accordance with applicable standard operating procedures.

- A member of the technical group, or designated individual, performs routinely scheduled maintenance and calibration of laboratory equipment and instruments as required by laboratory procedures. These activities are documented.

- If appropriate standards or expertise for calibration or maintenance are not available in-house, the operation is conducted by an outside service firm, with appropriate accreditation. Certificates or other data generated by the service firm are reviewed by applicable the laboratory personnel to verify acceptability. This information is maintained on file.

- All equipment or instruments taken out of service are tagged “DO NOT USE”. The following minimum information is documented on the tag:

- Date taken out of service

- Employee who took the equipment/instrument out of service

- Reason for tag-out

6.4.2 Standard Operating Procedures

Information regarding operation, maintenance, and calibration of equipment and instrumentation is found in the respective SOPs. The procedures include, where applicable, a routine schedule for preventive maintenance and calibration along with acceptance criteria and remedial action to be taken in the event of failure. These procedures are maintained in the document control system and reviewed on a regular basis to verify they remain current and accurate. Vendor supplied manuals are also available to provide additional information in regard to operation and maintenance.

6.4.3 Maintenance

- Instrument and equipment maintenance is performed as either a preventive or corrective operation.

- Preventive maintenance procedures and schedules are developed for each instrument or piece of equipment, where applicable. Preventive maintenance operations are performed by an analyst, equipment maintenance specialist, or contracted (manufacturer’s representative or service firm personnel). Documentation is maintained in the associated maintenance log for the procedure(s) performed as part of the preventive maintenance operation. It is the responsibility of departmental management to ensure that a preventive maintenance schedule is addressed by a procedure where appropriate and is followed.

- Corrective maintenance is performed by an analyst, equipment maintenance specialist, or contracted (manufacturer's representative or service firm personnel) in response to indications of equipment or instrument malfunctions. The unit must be clearly tagged as out of service. All corrective actions taken to bring the unit back into service are documented in the associated maintenance log. After repair, further notation is made in the log regarding the functional status. Calibration activities are performed, as applicable, and documented in the log before the unit is placed back into service.

- A supply of commonly needed replacement parts is maintained by the laboratory.

- A preventive maintenance schedule for major instruments is given in Appendix G. Maintenance of equipment used in microbiological testing is documented in Appendix K.

6.4.4 Calibration

- Calibration is the establishment of, under specified conditions, the relationship between the values/response indicated by a measuring instrument or system and the corresponding known/certified values associated with the standards used. Some types of calibrations are performed with a set frequency (e.g. daily) while others provide intermediate checks to ensure that the instrument response has not changed significantly.

- All measuring and testing instruments and equipment having an effect on the accuracy, precision, or validity of calibrations and tests are calibrated and/or verified on an on-going and routine basis. Methods for calibration of instruments and equipment vary widely with the nature of the device and the direction given by analytical procedures, departmental procedures, manufacturer recommendations, or regulatory requirements. Frequency of calibration can also depend on additional factors including ruggedness of the instrument or equipment and the frequency of use.

- Departmental management is responsible for developing or acquiring written calibration procedures for the types of instruments and equipment employed within their area, as applicable. Procedures address the following aspects: description of the calibration method, frequency/schedule for calibration, acceptance criteria, and corrective actions if failure occurs.

- Calibration information is recorded in a logbook that is associated with the instrument/equipment and/or a calibration certificate is maintained and/or data is generated and filed to document the activity.

- Calibration measurements are traceable to national standards of measurement (e.g., NIST) where available. Physical standards, such as NIST certified weights or thermometers are re-certified on a routine basis. Calibration certificates are maintained on file, where applicable, to indicate the traceability to national standards of measurement. These physical standards are used for no other purpose than calibration.

- Calibration failures are documented in the associated logbook and/or within the data generated from the instruments or equipment. Management personnel perform an evaluation and review of failures and assess any potential impact the failure might have on previously generated data. The laboratory utilizes "real-time" controls to ensure the accuracy of the data. These controls are used to assist in assessing the impact of the situation.

- After repair, adjustments, or relocation that could affect instrument response, calibration/verification activities are performed, as applicable, before the unit is returned to service.

- Analytical data is not reported from instrumentation or equipment with noncompliant calibration unless the client has agreed to receipt of the data and appropriate comments are applied to the final Analysis Report.

- A summary of the calibrations for most major instruments and equipment is given in Appendix H.

- Procedures for calibration of equipment used in microbiological testing are documented in Appendix K.

6.5 Computerized Systems and Computer Software

6.5.1 Computer Usage

The laboratory provides computer equipment for employees to use as a tool in performing their work. Computer equipment is the property of the laboratory and used in accordance with defined terms and conditions. Our goal is to provide standard hardware and software that meets the needs of the user. The majority of desktop PCs in use are standardized using cloning software.

6.5.1.1 Physical Security of Computer Systems

It is company policy to protect computer hardware, software and data documentation from misuse, theft, unauthorized access and environmental hazards. The corporate computer area and computer "Hot-Site" is locked and requires identification/building card access. All vendors, contractors, or other visitors must be escorted into this area. Controlled access of the laboratory buildings is outlined in Section 3.2.

6.5.1.2 Passwords

Passwords are important for the security of company data and resources. The laboratory's primary network operating system is Windows and each employee must have a user ID and password combination to access the system. Other computer systems also require a user ID password combination for access. The following procedures apply regardless of which system(s) is being utilized:

- Passwords must be created as strong passwords in accordance with Eurofins Password Policy and must be kept confidential.

- Users must log-out of a system when not in use to prevent unauthorized access. In addition, the network access will automatically timeout after a set period of inactivity, requiring a user to log-in to access the system.

- Forgotten passwords can only be reset by the IT Department or by an appropriate System Administrator.

- Network and LIMS passwords automatically expire every 90 days. The computer prompts a user to change the password when the expiration date nears.

6.5.1.3 Viruses

The laboratory centrally and continuously monitors the computer network for computer viruses. Employees are prohibited from using the company's computer equipment to propagate any virus. Anti-virus software is employed to detect viruses on the Windows network. A notification is sent when there is a particularly dangerous or virulent data destructive program that employees need to be aware of. However, employees are instructed to always be cautious and observant even if there are no current warnings. Employees must report any virus concerns to the anti-virus administrator or IT Management as soon as possible. Employees who share files between their home computer and the laboratory should install anti-virus software on their home computer. If an employee does not have such software, the laboratory can suggest various no-cost anti-virus software products.

6.5.1.4 Internet and E-mail Systems

The e-mail system is used primarily for the laboratory's business purposes. The *Eurofins Lancaster Laboratories'*

Employee Handbook provides additional information in regard to system usage. Employee access to the internet is restricted to those employees who have a business need for it. All employees have access to e-mail. Access to the internet is configured through a user's Windows network account. All internet and e-mail activity is subject to monitoring. All messages created, sent or received over the internet are company property and can be regarded as public information. E−mail and website filtering software is utilized.

6.5.1.5 The Laboratory's Intranet (LabLinks)

The Intranet is designed to be a useful tool for employees to acquire company information and to provide a company communication system. The *Eurofins Lancaster Laboratories' Employee Handbook* provides additional information in regard to usage.

6.5.1.6 Software Policy

- Copyright laws protect software, and the laboratory's intent is to abide by all software agreements.
- Software purchases must be formally requested and approved by management and/or validation personnel, as necessary.
- All software is used in accordance with applicable license agreements.
- Employees are not to install any software on computer(s) unless authorized by the IT Department.
- Software upgrades must occur in accordance with applicable change control procedures.
- Employees must not give software to outsiders (e.g., clients, contractors), unless approval is granted by management.
- Users must not make copies of any licensed software or related documentation without permission. Any user that illegally reproduces software is subject to civil and criminal penalties including fines and imprisonment.

6.5.1.7 Computer System Backup, Data Restoration, and Data Archival

Mission critical data is stored on several computers throughout the laboratory. These computers are connected through the local area network. Selected files on these computers are backed up using an enterprise-level backup software program. The objective of this backup is to have the ability to restore data after a total loss (e.g., theft, fire, natural disaster). Procedures are in place to perform data backups and restores.

6.1.5.8 Remote Access to Computer Systems

Employees are able to remotely connect to the laboratory computer systems through an encrypted (SSL) login. When logging in, users are authenticated with their Windows Active Directory account and password.

6.5.1.9 Electronic Data

Instrument software used for processing data must, when available, have password access and audit trails enabled. All data processed through the LIMS includes tracking features to document who and when data was entered and/or changed.

6.5.2 System and Software Verification

The laboratory LIMS is an in-house developed program. The design and updates to the system are written following typical Software Development Life Cycle (SDLC) processes for initial planning through testing and implementation. Before a new computer system/program or significant modification of an existing system/program is implemented in

our laboratory, it is necessary to generate a plan to specify the level of documentation required for the new or updated application. Developers, affected area management, and QA personnel review and approve the documentation.

The following are the typical documents that are compiled for these updates:

- System Change Request document – used for documenting/tracking changes in the programming
- Requirements documents – Describe the required system functionality and specifications
- Design documents – System overview, screen design, report layout, data description, system configuration, file structure and module design
- Testing documentation for system development/verification – Structural testing of the internal mechanisms and user testing of the installation and system qualification
- Periodic Review documents – periodic retesting of the programs is performed to ensure that the systems remain in a validated state.
- Retirement documents – used for documenting when a program is taken out of service
- Standard operating procedures and/or manuals

6.6 Change Control

Procedures are in place to define how to maintain facilities, processes, instrumentation, equipment, computerized systems, and computer software in a validated or controlled state through a plan of change control. Successful changes require a thorough evaluation and testing for potential consequences prior to implementation. Planning, authorizing, testing, and reviewing of proposed changes are documented throughout the change process. Changes are planned or could be made in response to an emergency situation. The following “general” elements apply to changes, as appropriate:

- Request to perform a change
- Evaluation of a change
- Authorization of a change request
- Preparation for an authorized change
- Execution and testing of the change
- Documentation of the change
- Approval of the change
- Change implementation and follow-up (Formal approval of the change is performed by designated responsible individuals and QA.)

6.7 Labware Cleaning

Dedicated washroom personnel support the laboratory operations in regard to labware preparation, washing, rinsing, and drying. Labware can include, but is not limited to glassware, plastic ware, utensils, and pipettes. Procedures are in place to outline the washing process for each type of labware. Most labware is cleaned using a Miele glass washing machine. Some labware is still washed by hand and either air-dried or dried in specifically designed ovens.

Most of the labware used in the laboratory is “common or non-dedicated” labware (common to a department), but some of the labware used in the laboratory may be identified as “dedicated” labware and exclusively used for certain analyses. Examples of dedicated labware include glassware used for high resolution mass spectrometer (HRMS) and low level mercury testing. This labware is isolated and cleaned only with “like” labware.

All glassware is class A and 100% visually inspected for breakage (e.g., cracks, chips), cleanliness, and dryness before being returned to the laboratory for use.

Generally, each test has controls in place to ensure that results are not adversely affected by unclean labware. These controls include blanks to detect positive interferences and recovery controls to detect negative interferences.

7 PURCHASING EQUIPMENT AND SUPPLIES

7.1 Procurement

It is the responsibility of management personnel within each department to ensure that the appropriate supplies are available and/or ordered with sufficient lead-time to perform analytical testing or to provide support to the testing areas. The individual technical departments have trained personnel who enter the supply order into the company’s requisition software system. The selection of these products is based on technical input at the analyst level and authorized by technical departmental management. The Purchasing Department maintains an ordering system in which purchase requisitions are managed. Common laboratory items (e.g., beakers, flasks, reagents) are ordered directly through the Purchasing Department. Purchase orders over a specified dollar amount require verification from the appropriate member(s) of the Executive Management Group before an order can be placed.

Upon receipt of an order, the Purchasing Department checks the order to ensure that all items were received as specified. Products that have specific storage requirements are taken to the technical area upon receipt. It is the technical area’s responsibility to ensure that the product is stored in the appropriate manner. Any checks on the quality of the materials received for use in a specific test are the responsibility of the laboratory using them. This is based upon the experience of the laboratory with the usability of the product. Generally, each test has controls in place to ensure that test results are not adversely affected by the materials.

Any problems encountered when using a material in the laboratory must be brought to the attention of the Purchasing Department and/or Quality Assurance, as applicable, to ensure that follow-up and corrective action occur.

7.2 Supplier Evaluation

Procedures are in place to evaluate vendors who supply us with: new equipment, instrumentation, computerized systems and computer software; commercially purchased glassware, including sample bottleware, reagents, chemicals, solvents, gases, media, and standards; and contracted and subcontracted services.

The laboratory strives to ensure that our suppliers continually improve their quality systems and we reserve the right to purchase from suppliers of our choice in order to best fulfill the needs of our clients and our business. When directed by a client to purchase from a specific supplier, we will do so. In this instance it is the client's responsibility to "qualify" the specified supplier. We attempt to purchase from businesses that we have an established purchase history or have previously acquired information regarding the supplier's quality programs.

The laboratory does not evaluate every supplier. Risk assessment is taken into consideration when making this decision. The risk assessment analysis includes system, material, services, and number of samples or operations the purchase may affect or support. Evaluations are not required for computer operating systems, utilities, toolsets, or systems software. They also are not required for any off-the-shelf configurable software package that has an extensive market performance history (e.g., Microsoft Word, Excel, Access).

Additional quality systems are also in place within the laboratory to further verify and support the materials used:

- C of A for every lot of purchased prepared microbiological media and for purchased chemicals, where available, are reviewed and maintained on file.

- For most chemical analyses a blank and a recovery check are routinely analyzed and serve as real time suitability testing of the reagent being used.

- Microbiological testing often employs positive and negative controls, which serve as real time control checks.

8 ANALYTICAL METHODS

8.1 Scope of Testing

Samples are analyzed in accordance with official published methods, standard methods, client-supplied methodology, or validated in-house methods. We recognize the importance of providing verifiable results and, therefore, use methods accepted and approved by a broad range of federal and state regulatory agencies. In order to meet the needs of our clients as well as regulatory agencies, the laboratory sometimes needs to support different versions of the same method (i.e. SW-846 8081A and 8081B). The laboratory can also assist in developing and validating analytical methods for specific products and matrices. All methods submitted for our review, as well as all analytical results, are considered confidential.

The laboratory performs a wide variety of environmental testing in support of the Safe Drinking Water Act (SDWA); Clean Water Act (CWA); Resource Conservation and Recovery Act (RCRA); Comprehensive Environmental Response, Compensation, and Liability Act (CERCLA/Superfund); and the Clean Air Act (CAA). Methods approved by ASTM are also used in testing. Potable water, wastewater, soil, sediment, sludge, oils, biota, tissue, soil gas, and air are among the matrices typically analyzed.
Our areas of expertise include:

Standard Services	Specialty Services
• Volatiles • Semivolatiles • Metals • Pesticides/PCBs/Herbicides • Petroleum Analysis • Waste Characterization • Non-potable Water Testing • Drinking Water • Soil and Surface Water Testing • Vapor and Air Analysis • Sediment and Tissue Testing • Method Development • Shale Oil & Gas Analysis •	• Low-Level Mercury • Dioxins & Furans • Hydrazines and NDMAs • Perchlorate • 1,4-Dioxane • Pharmaceutical Manufacturing Industry (PMI) Wastewater • EPA Method 25D • PCB Congeners • Explosives • Alkyl PAHs, Alkanes, Biomarkers • PFC (PFOA) • Organic Acids • Aldehydes •

A list of tests covered under the laboratory's NELAP accreditation can be found in Appendix I. All current certificates and scopes of accreditation are available on the laboratory's website at http://www.eurofinsus.com/environment-testing/laboratories/eurofins-lancaster-laboratories-environmental/resources/certifications/. A complete list of the tests routinely performed by the laboratory can be found in the *Schedule of Services*.

8.2 Analytical Test Methods

Each laboratory is required to establish and maintain analytical procedures for all the methods referenced in standard testing. The sources for these methods include the most recent versions of these compendia:

- Test Methods for Evaluating Solid Waste, SW-846
- Standard Methods for the Examination of Water and Waste
- Code of Federal Regulations, Chapter 40
- EPA 100 through 600 and 1600 series methods
- ASTM

The test methods used are re-written into a laboratory standard format, which provides consistency in content and allows the analysts to locate the information they need quickly. Procedures are in place to define the format, required approvals, and the control system for these method documents. Elements to address in SOPs are based on TNI and DoD required sections. The format requirements include, but are not limited to, the following:

- Uniquely assigned method number, which is used extensively for scheduling and documentation purposes.
- Reference to the original source of the method (e.g. SW-846)
- Scope
- Basic Principles
- Apparatus and Reagents
- Personnel Training and Qualifications
- Safety and Waste Disposal
- Detailed procedure (including any method modifications)
- Calculations
- QA/Quality Control
- Revision Log
- Approval signatures from technical management and QA personnel

Analytical methods are maintained as controlled documents to ensure that analysts are always working with the most current version and are reviewed periodically for accuracy.

8.3 Client Supplied Methods

Most of the client-supplied method requirements presented to us involve achieving specific quality control criteria, limits of quantitation (LOQ), and/or method detection limits (MDL) using standard EPA methods. These requirements are communicated to the appropriate technical groups prior to the project start up. Each technical group evaluates the scope of work and the requirements to ensure the criteria can be met using the standard EPA method. The data is monitored to ensure the criteria are met throughout the project. The CSR notifies the client if there is a more appropriate method available or if the client's criteria cannot be achieved on a certain sample matrix (i.e., due to matrix or dilutions).

Occasionally, we are asked to transfer a non-standardized method from a client into our lab or to develop a new method, when one is not available. In the case of a method transfer, we set up the client's method and perform some initial evaluation. After the initial evaluation, we may make recommendations on how to improve method performance. If the method appears to be adequate, we determine linearity, specificity, precision, accuracy, MDL, and LOQ by performing calibrations, analyzing method blanks, and carrying out method detection limit and quad studies.

In the case of method development, we work with the client and/or data user to determine the level of validation required ensuring that the method meets its intended purpose. In addition to the elements above, we also determine standard and sample stability and robustness depending on the scope of the project. Typically, a

standard operating procedure is written and submitted to the client with the results of the validation. These steps are completed prior to analysis of field samples. Data related to the setup of the method are archived.

8.4 Method Validation

Before new or revised analytical methods are authorized for routine use in the laboratory, validation data is required to demonstrate that the method as performed in our laboratory and analysts performing it are capable of meeting data quality objectives for precision and accuracy. A procedure is in place to outline this process.

Many methods published by USEPA include instructions for performing an initial demonstration of capability, which typically consist of determining the method detection limit and analyzing fortified samples in quadruplicate. This demonstration is performed and compared to acceptance limits for precision, accuracy, and detection limits, when available.

Methods that do not include specific validation requirements are validated by analyzing fortified samples or standard reference materials in replicate. The results of these analyses are used to assess accuracy and precision. Results of validation studies are documented and subject to review and approval by technical and QA management.

8.5 Procedural Deviation

Analysts are required to follow a documented method for all tests performed. Procedures are in place to ensure that deviations from analytical methods are documented, approved, and justified in an appropriate and consistent manner (Note: Deviation from the OH EPA approved SOPs is not permitted). We classify method deviations as either being a planned deviation or an unplanned deviation. In general, the following information is captured to document both types of situations:

- Description of the deviation
- Reason or justification for the deviation
- Impact the deviation had on the testing
- Signature/date of analyst performing the test
- Signature/date of Quality Assurance and Laboratory Management approving the deviation
- Signature/date of client approval, if necessary

Deviations to written procedures are documented in raw data records or through the ICAR (Investigation and Corrective Action Report) system. Both types of documentation require management and QA review and approval.

9 INTERNAL QUALITY CONTROL CHECKS

9.1 Laboratory Quality Control Samples and Acceptance Criteria

Quality control (QC) samples are analyzed with each batch of samples to demonstrate that all aspects of the analysis are in control within established limits of precision and accuracy. Management is responsible for ensuring that QC is analyzed as required by the referenced method. Each analytical SOP specifies (or cross-references

another procedure) the type of QC sample, frequency of analysis, acceptance criteria for QC sample results, and corrective action to be taken if QC sample results fall outside of the acceptable range.

QA staff, at the direction of the technical department, must program the LIMS with the acceptance criteria for each QC type (other than blanks). The acceptance criteria are based on statistically generated limits from historical laboratory data, on method defined limits, government agency recommendations, or on client/project specific limits.

These limits are used to flag samples that are out of specification.

The types of QC samples and the information each provides are discussed in the following paragraphs.

Quality control checks used for microbiological tests can be found in Appendix K.

9.1.1 Blanks

A blank is a designated sample designed to monitor for sample contamination during the analysis process. The blank consists of a clean matrix (i.e. reagent water, Ottawa sand, glass beads, Teflon chips) taken through the entire sample preparation and analysis process. The blank and field samples are treated with the same reagents, internal standards, and surrogate standards. Ideally, blanks demonstrate that no artifacts were introduced during the analysis process. The specific acceptance criteria for each test are usually based on the required reporting limit (MDL or LOQ).

9.1.2 Surrogates

Surrogates are organic compounds, which are chemically similar to the analytes of interest but are not naturally occurring in environmental samples. When required by the analytical method, surrogates are spiked into all the field and QC samples to monitor analytical efficiency by measuring recovery on an individual sample basis. The percent recovery is determined and compared to the acceptance criteria.

9.1.3 Matrix Spikes

A matrix spike sample is created by fortifying a second aliquot of a water or soil sample with some or all of the analytes of interest. Blanks are not used for matrix spike QC. The concentration added is known and compared to the amount recovered to determine percent recovery. Matrix spike recoveries provide information about the potential matrix effects on the data. Matrix effects can cause results to be outside of the acceptance criteria.

9.1.4 Laboratory Control Samples

Laboratory control samples (LCS) are samples of known composition that are analyzed with each batch of samples to demonstrate laboratory accuracy. Laboratory fortified blank (LFB) is another term used to describe a LCS. The samples are clean samples fortified with known concentrations. Percent recovery is calculated and compared to acceptance

9.1.5 Duplicates and Matrix Spike Duplicates and Laboratory Control Sample Duplicates

A duplicate is a second aliquot of a sample that is treated identically to the original to determine precision of the test. To compare the values for each analyte, the relative percent difference (RPD) is calculated by dividing the difference between the numbers by their average. Precision for analytes that are not typically found in environmental samples (i.e., organic contaminants) is determined by analyzing a pair of matrix spike duplicates, defined as two spiked samples and comparing the RPD for the spiked compounds. The acceptance criteria are described as a maximum for the RPD value.

9.1.6 Internal Standards

Internal standards are organic compounds, which are chemically similar to the analytes of interest but are not

naturally occurring in environmental samples. When required by the method, internal standards are added to every field and QC sample after extraction but prior to analysis. Comparison of the peak areas of the internal standards is used for quantitation of target analytes. Internal standard peak area and retention time also provide a check for changes in the instrument response. The acceptance criteria are stipulated in the analytical method.

9.1.7 Serial Dilutions

A serial dilution is the dilution of a sample with sufficiently high concentration by a factor of five to check for the influence of interferents. This QC check is performed for inorganics analyzed by ICP or ICP-MS. When corrected by the dilution factor, the diluted sample result must agree with the original sample within method specified limits.

9.1.8 Interelement Correction Standard

Analyzed to verify interelement and background correction factors. A solution containing both interfering and analyte elements of known concentration is analyzed at the beginning and end of each analytical run or a minimum of twice per 8 hours.

9.1.9 Second Source Check

A second source check is analyzed using either the LCS and/or an Initial Calibration Verification (ICV). The second source is a standard that is made from a solution or neat purchased from a different vendor than that used for the calibration standards. For some custom mixes, the same vendor but a different lot and preparation is used. This ensures that potential problems with a vendor supply would be evident in the analysis. Some tests use the continuing calibration verification standards as a second source from the initial calibration.

9.2 Quality Control Sample Frequency and Corrective Action

Each analytical method defines the frequency for the required QC samples and the corrective action required when a QC result fails to meet the acceptance criteria. A summary is provided in Appendix J.

The QC acceptance criteria are available to analysts in the laboratory. If the method reference requires the use of specific limits then the laboratory uses the published limits that are documented as part of the analytical method. Many methods require that each laboratory determine their own acceptance criteria based on statistical data obtained from performance of the method. In these cases, the limits are available to the analysts and are entered into the LIMS described below. Statistically determined acceptance criteria are subject to change as the laboratory recalculates its control limits. Due to their dynamic nature, acceptance criteria are not included in this manual.

The results of all quality control samples are entered into the LIMS in the same way as the results of client samples. The LIMS compares the individual values with the acceptance limits and identifies quality control sample results that are out of specification. If the results are not within the acceptance criteria, corrective action suitable to the situation must be taken. This includes, but is not limited to, checking calculations, examining other quality control analyzed with the same batch of samples, qualifying results with a comment stating the observed deviation, and reanalysis of the samples in the batch.

Each month, a summary of all QC entries (except blanks and surrogates) is generated from the LIMS. This summary is reviewed by QA staff and evaluated for changes in data that may indicate that an analysis is trending towards an out-of-control situation. The technical department is notified if a trend is observed.

The laboratory allows for marginal exceedances based on the number of analytes in the LCS. The exceedances are carefully monitored so that any systemic problems would be identified and corrective action taken. If the LCS is being reported based on the marginal exceedance allowance, a comment is added to the analytical report. Note: The use of marginal exceedance is not allowed for OH VAP work.

9.3 Quality Control Charts

The LIMS quality control system is used to report QC data to clients, to collect data for assessment of precision and accuracy statistical limits, and to generate control charts. Control charts are accessible to all employees through the LIMS interface. The system charts results from blanks, surrogates, matrix spike/matrix spike duplicates, duplicates, and laboratory control samples/laboratory control samples duplicate. These charts provide a graphical method for monitoring precision and bias over time. They can be used to detect quality problems by observation of patterns. QA staff uses the charts in conjunction with a LIMS generated monthly QC trend report to evaluate potential data trends.

9.4 Measurement Uncertainty

(ISO 17025) “All uncertainty components which are of importance in a given situation shall be taken into account using appropriate methods of analysis” (5.4.6.3). This means the laboratory must determine the uncertainty contribution of all steps in the testing process such as equipment, calibration, standards, reagents, preparation, cleanups, etc. Since, in most methods, the laboratory control sample (LCS) goes through the entire process of preparation to analysis; all factors that would contribute to uncertainty is evident through the LCS results. LCSs are performed with every batch of samples where appropriate for the method. Tests that do not have LCSs (i.e. TCLP; paint filter test), are evaluated on a case-by-case basis by taking into account the uncertainty of each of the steps taken to perform the test.

Measurement Uncertainty reports are generated by each technical department on an annual basis using a LIMS program and submitted to QA. Measurement Uncertainty is calculated as two times the standard deviation of the LCS recoveries for the group and date range of data points selected for all applicable methods. This is reported as a percentage. It is not necessary to apply or report the uncertainty value with sample results. When a client requests the measurement uncertainty it is applied by multiplying the determined analyte concentration by the uncertainty percentage.

10 ASSURING QUALITY OF TEST RESULTS

10.1 Data Management

At a minimum, data management is initiated when the laboratory receives the samples from the client. More often the process begins with client communication of their needs and requirements for a specific project and/or testing. When requested, bottle orders for the client’s sampling efforts are generated through the LIMS by the CSR. The CSRs are responsible for entering the information in the sample set up function of the LIMS. Upon receipt of the samples a unique tracking number for the sample group and the samples within the group is generated based on this information. At this point, the LIMS becomes an integral part of tracking the samples through laboratory operations. The flow of data from the time samples enter the laboratory until the data is reported is summarized in the following table:

Sample and Data Flow

Action	Personnel Involved
Bottle orders generated upon request • Bottles packed and shipped to the client under chain of custody documentation	Client Service Representative Bottles Preparation
Sample received at Lancaster Labs • Unpacked and reconciled against the client paper work or COC • Sample Entry Documentation log completed	Sample Registration

Action	Personnel Involved
Sample is entered into the LIMS • Lab ID number assigned • Analyses entered • Storage location assigned • Electronic record of sample number • Labels generated • Acknowledgement printed (record of samples received and analyses entered)	Sample Registration
Preservation checks performed Sample stored in assigned location (refrigerator, freezer, etc.) • Electronic record of sample #, bottle code, and location	Sample Registration
Acknowledgment sent to client (when requested)	Sample Registration
Samples requisitioned and removed from storage for analysis • Electronic requisition of sample number by bottle code • Necessary aliquot taken • Remaining sample returned to storage	Sample Registration Technical Personnel
Analysis is performed according to selected analytical method • Raw data recorded • Data Reviewed • Data uploaded to the LIMS from the instrument or manually entered by the analyst* (This is tracked by the unique sample number and batch number.)	Technical Personnel
LIMS performs calculations as programmed according to methods	Data Processing
Designated analyst or supervisor verifies raw data	Technical Personnel
Generation/release of reports (automated through LIMS)	Billing and Reporting Group
Data package deliverables are assembled, reviewed and released to client Electronic copy saved in the LIMS	Data Package Group
Electronic Data Deliverables (EDDs) are generated	EDD Group
Designated Data packages are overchecked by QA prior to release	QA
Hard copy of batch raw data is archived Electronic files are backed up and archived	Technical Personnel, Data Package Personnel, Office Services, IT

*Analyses requiring the analyst's interpretation may involve manual data reduction before entry into the LIMS.

10.2 Data Documentation

Analytical data generated in the laboratory is collected from the instruments or associated data system or is manually documented in bound notebooks. Analysts review data as it is generated to determine that the instruments/systems are performing within specifications. If any problems are observed during an analytical run or the testing process, corrective action is taken and documented.

Procedures are in place to ensure that all data is traceable, authentic, and complete. Electronic data records are maintained and tracked through the LIMS, requiring authorized, password protected user access. The following general requirements outline our system for notebook, logbook, and documentation recording:

- Observations, data, and calculations are recorded at the time they are made and are identifiable to the specific task.
- Entries must be legible, signed, and dated. The signature may be a wet or electronic signature.
- Errors are corrected in a manner that does not obliterate the original entry, initialed and dated, and coded with an explanatory identifier. Changes to electronic data are tracked through audit trail functions.
- Blank pages or substantial portions of pages which are left blank are crossed-out to eliminate the possibility of data entry at a later date.
- Notebook pages and instrument printouts are signed/dated to indicate second party data review; this may be a wet or electronic signature.
- At periodic intervals a supervisor or data reviewer checks equipment/instrument logbook entries and temperature recordings for completeness, legibility, and conformance to procedures.
- At a minimum, the following information is recorded as part of data documentation:
  - Date of analysis/operation
  - Signature/date of analyst performing test/operation
  - Identification of client sample(s) and material(s) analyzed
  - Materials, reagents, standards used to perform the testing/operation
  - Method used to perform testing/operation (including version number and/or effective date)
  - Equipment/instrumentation used to perform testing/operation
  - Calculations and how they were derived
  - Departures, planned or unplanned, from the analytical method
  - Signature/date of person reviewing data documentation
- For computer generated data, the following information is recorded:
  - Sample(s) analyzed/operations performed
  - Date of analysis/operation
  - Unique instrument identification

- Name/date of person operating the instrument
- Name/date of person reviewing data
- Any manual notations or interpretations made on instrument printouts are signed, dated, and reviewed

10.3 Data Calculations

Most instruments either include or are connected to a data system programmed to perform calculations to reduce the raw data to a reportable form. All calculations are maintained in the instrument manuals and/or as part of the analytical method.

In many cases, the data from the local instrument system are uploaded directly to the LIMS for review and reporting. This direct upload eliminates the need to retype data and an associated source of transcription errors from the analytical scheme.

Some instruments report data that require application of additional factors before the data is in final form. For example, an extract concentration may be reported by the instrumental data system, but additional dilution and preparation factors may be needed before the result represents the concentration of analyte in the sample. Analysts input these additional factors into the LIMS, where final calculations are performed.

Analysts manually enter collected data, such as titration data, into the LIMS, which is programmed to perform calculations for final reporting. Documentation of the programming for each calculation performed by the LIMS is maintained.

10.4 Reporting Limits

It is important to ascertain the limit of quantitation (LOQ) that can be achieved by a given method, particularly when the method is commonly used to determine trace levels of analyte. The Environmental Protection Agency has set forth one method for determining method detection limits (MDLs) from which LOQs can be extrapolated. This process is summarized in a laboratory procedure.

MDLs are verified or determined annually on each instrument and are the basis for the LOQ used in the default reporting format. Because MDLs change each time they are re-evaluated, they are not included in this manual, but are available in each laboratory and available to clients upon request.

The reporting limit used to determine whether a result is significant and reported as detectable is dependent upon agency and client requirements. A variety of formats are available and include use of the MDL, LOQ, method specified limits, and project specific limits. The MDL and LOQ for each analyte are programmed into the LIMS for reporting purposes.

Under the DoD program, the laboratory must establish a Detection Limit (DL) and Limit of Detection (LOD). As defined by the DoD program, the DL is the smallest analyte concentration that can be demonstrated to be different from zero or a blank concentration with 99% confidence. The laboratory determines the DL using the calculated value from the MDL Study. The DL can be derived from pooled MDL values obtained across instruments. The LOD is the smallest amount of a substance that must be present in a sample in order to be detected at the DL with 99% confidence. It is established by spiking a quality system matrix at a concentration of 2-4 times the DL. The LOD must be verified on a quarterly basis or with each batch of samples.

10.5 Data Review

Final review and verification of the data are performed by designated employees using the sample results, quality control information, method criteria and Project Notes entered into the LIMS. Data are initially evaluated by the analyst and then a second designated employee knowledgeable in the test, other than the employee responsible for performing the test, reviews the data. The reviews include checks for correct transcription, calculations, passing calibrations, compliant quality control results, holding time compliance, and project specific requirements. Any issues or errors identified during this stage are addressed, corrected, and reviewed with the responsible person.

After determining that all necessary requirements for valid data and for the project are met, the reviewer electronically approves the data by changing the LIMS status of the data from “complete” to “verified”. The LIMS is programmed with a list of approved reviewers for each test, and the system is password protected to ensure that only qualified individuals verify the data.

10.6 Data Qualification

Data qualifiers are used to provide additional information about the results reported. The most typical use for data qualifiers is for results that fall below the quantitation limit, in the region where it becomes more difficult to distinguish a positive result from the background instrument signal. The data systems used to generate and report results are programmed to flag values in this range as estimates.

Other qualifiers are applied to advise data users of any validation issues associated with the data. The laboratory makes every effort to meet all of the requirements for generation of data. Occasionally, generation of data that does not meet all the method requirements occurs due to sample matrix or other analytical problems. If the test cannot be repeated or reanalysis would not yield better quality data, qualified data is reported. Qualifiers can be in the form of comments on the analytical report or flags applied to the results.

10.7 Data Reporting

When all analyses are completed, reviewed and verified, a report is generated by the LIMS. The client receives a copy of the report containing the results of the analysis, associated QC data, and where necessary, explanatory comments to address non-conformances. To avoid ambiguity in interpreting results, a summary page that contains an explanation of all symbols and units used in reporting data is included with the Analysis Report submitted to clients. Some regulatory agencies also require the laboratory accreditation identification on the Analysis Reports. Where required, this information is added. The current list of agencies can be accessed in the LIMS. Copies of reports and associated supporting raw data are retained in our archives. The report contains the signature of the assigned client service representative who is the key contact for any questions concerning the results. Personnel authorized to review, sign, and release Analysis Reports are noted in the key personnel list provided in Appendix C.

The laboratory offers a variety of data reporting .levels and formats, from a basic report of sample and QC results only, to a comprehensive data package of QC/calibration information and raw data. The client and any agency involved direct the selection of report type. A summary of report formats and data packages types is provided in the laboratory *Schedule of Services*. Various electronic formats are also available formatted to client-specified file structure and sent via e-mail, direct upload, web-site access (LLabWeb), or common courier. LLabWeb is used for clients that require secure transfer of electronic data.

Client confidentiality of LLabWeb data is ensured by the use of a secured firewall internet environment coupled with the use of a user ID and password to gain login access to the system. User accounts are configured to only allow access to specific data associated with the user's business account number.

Amendments to a final report after issue are in the form of an additional document or data transfer and include a reference to the original report. When a completely new final report is required, it is uniquely identified and includes a reference to the original report it replaces.

10.7.1 Reporting the Results

Analytical reports are generated with a cover page that summarizes all samples in that group. This page lists the laboratory assigned sample number and the corresponding client description. The cover page identifies the laboratory contact person's name and phone number if there is a question about the report. Within this package, each page is uniquely identified and paginated. Analytical test results for methods listed on the laboratories' accreditation scope meet all requirements of NELAP accreditation and ISO 17025 unless noted otherwise. Ohio EPA VAP requires that a signed, notarized affidavit accompany each analytical report.

10.8 Data Storage, Seucirty, and Archival

The laboratory has documented procedures and instructions for the identification, collection, access, indexing, filing, storage, maintenance, and disposition of data records. Records are in the form of paper records, electronic data files, magnetic tape, and CD-ROMs.

All data records are maintained in a confidential manner in an environment to minimize deterioration or damage and to prevent loss. Some records are stored in off-site facilities, in such a way that they are readily retrievable. Retention time for records is in accordance with specific procedures or instructions. Prior to the destruction of data/records, and if requested by a client or agency, the laboratory will notify the client/agency that their data is scheduled for destruction so arrangements can be made to have the original data sent to the client.

If specified in client contract(s), archived records are transferred according to their instructions in the event of a change in laboratory ownership or if the laboratory goes out of business. If not specified by the client, the sale agreement must require that archived records be maintained as scheduled by the new owners. In the case of bankruptcy, appropriate regulatory and state legal requirements concerning laboratory records must be followed.

The laboratory maintains all documentation which is necessary for historical reconstruction of data:

- Analysis reports
- Data notebooks
- Data logbooks
- Instrument output
- Correspondence and client files
- Instrument and equipment logbooks
- QA records

- Corporate documents
- Electronic records

11 AUDITS AND INSPECTIONS

11.1 Internal Quality Assurance Audits

The QA Department, which is independent of laboratory activities, performs routine and on-going system, traceability, and observation audits to objectively review current systems, operations, and procedures as well as automated data integrity audits of electronic data records. The goal of the audits is to ensure that the quality system activities are effective and in compliance with regulatory programs, including NELAP. ISO 17025, DoD, and state agencies, as well as internal policies and procedures. Audits are documented and tracked in a QA database.

Audits are scheduled and conducted following a predefined schedule, based on criticality of operation and prior audit results, with the goal of evaluating systems and technologies across the operation. If warranted, additional audits are performed to follow up on promised corrective action or areas of concern.

Results of an audit are documented in a report format and distributed to applicable management personnel responsible for the area(s) under audit. Management is responsible to address all non-conformances found during an audit with root cause analysis and application of a corrective action plan.

Audit reports and responses are circulated to Management to communicate the outcome of the audit and the proposed plan(s) for corrective action, if warranted. If any of the audit findings cast doubt on the validity of the results, the clients must be notified within three business days of the investigation. Should an audit issue present a major concern regarding validity of laboratory methods, QA personnel can issue a stop work notice.

All records maintained as part of an audit are kept on file for three years.

On an annual basis, an audit of the QA Department is performed as directed by the laboratory's Executive Management. The auditors assigned to carry out this operation are qualified staff members independent of the QA Department.

The specific content and findings of internal audits are considered company confidential and are not shared with clients.

11.2 Review of the Quality Assurance Program

All levels of management are continually updated on the status of quality and compliance by circulation of pertinent documents. Management review is documented by signatures on the documents, electronic records of each person's review, along with any comments or request for additional follow-up. The types of documents circulated real-time include:

- Internal, client, and agency audit reports and responses
- Proficiency test results
- Investigation and corrective action reports
- Monthly QA status reports

Executive management reviews the elements of the total QA program on an annual basis to ensure its continuing suitability and effectiveness in meeting the stated objectives outlined in Section 2.4 of this manual. The evaluation entails review of reports to management, all audit findings, client complaints, laboratory investigations, staff adequacy and training, and projected growth in workload. Patterns or trends in any of these areas are reviewed as a means to continually improve the quality system. This review also includes an evaluation of any audit findings resulting from the audit of the QA Department. At the conclusion of this quality system review, executive management determines the need to introduce changes or improvements into the quality systems at the laboratory. The minutes from the meeting and any recommendations for improvement are documented and a copy is forwarded to the QA staff for review and follow-up.

11.3 Good Laboratory Practice Critical Phase Inspections

Any project that is subject to Good Laboratory Practice (GLP) regulations is audited by the QA Department, as required by the regulations, at intervals adequate to ensure the integrity of the study. Inspections of a GLP project include direct observation of analysts as they perform various phases of the study. Data documentation is reviewed as part of the inspection. The purpose of this type of audit is to ensure that there are no deviations from written methods, procedures, or study protocols.

Results of inspections are documented in a report format and distributed to applicable management personnel responsible for the area(s) under audit. Management is responsible to address all non-conformances found during an inspection. Inspection reports and responses are circulated to applicable laboratory management and an off-site study director, as applicable, to communicate the outcome of the inspection and the proposed plan(s) for corrective action, if warranted.

All records maintained as part of an inspection are kept on file.

11.4 Client Audits

Because clients place great importance on compliance with applicable regulations, data quality, and project requirements, they may audit our facility as assurance that their objectives are being met. QA, management staff, CSRs, and the analytical laboratories play a key role in these audits. Visits by clients can range anywhere from a tour (to verify laboratory facilities and instrumentation) to an intensive inspection of technical operations, procedures, regulatory compliance, and/or review of specific project(s).

- Audits are scheduled directly with the CSR or QA. The request to audit is communicated to all applicable laboratory departments.

- In accordance with our policy on client confidentiality, a client is permitted to review only data and results that apply to their work, or which have been approved by laboratory management.

- An escort (designated laboratory employee) remains with an auditor at all times.

Responsibilities are assigned to the following groups in regard to client audits:

11.4.1 QA Department

- Research previous audit reports and laboratory responses to past deficiencies.

- Follow-up with the applicable analytical laboratory areas to ensure action items were completed from the last audit, as necessary.
- Work with client to set audit agenda.
- Function as an escort during the audit
- Answer questions the auditor has in regard to laboratory and quality systems.
- Take notes of areas where corrective action or suggestions are recommended during the audit.
- Communicate audit issues to management at the completion of the audit.
- Respond to client audit reports.
- Ensure follow-up to cited items are addressed in a timely manner.

11.4.2 CSRs

- Gather and organize relevant information (e.g., client correspondence, analysis/project requests, copies of analytical data from archives).
- Be knowledgeable about client-specific project requirements and issues.
- Function as an escort during the audit.
- Communicate issues/problems to appropriate personnel.

11.4.3 Laboratories

- Gather and organize laboratory data and documentation in preparation for client review.
- Assure corrective action was implemented from past audit findings, if necessary.
- Be prepared to discuss project data/testing results during the audit.
- Be familiar with client-specific project requirements and be prepared to answer client questions.
- Be familiar with the location of routine laboratory information and equipment (e.g., SOPs, data notebooks, calibration data, etc.).
- Be prepared to answer specific technical questions in regards to laboratory procedures and instrumentation within the area.
- Functions as an audit escort within the department during the audit.
- Laboratory managers may function as an escort during the audit

11.5 Agency Inspections

It is laboratory policy to cooperate to the fullest extent and maintain cordial relations with all government agencies. The QA Department is assigned the responsibility of hosting and working with agency representatives. Their role includes, escorting the investigator(s); ensuring all questions are answered promptly and accurately; making note of all unresolved issues; informing management of the audit status and outcome; responding to the audit report and ensuring that appropriate corrective action is completed.

Inspections can be performed by investigators or auditors from the EPA, states, third-party accreditation bodies (i.e. A2LA, United States Department of Agriculture (USDA), or other regulatory agencies.

Government agencies have the right to investigate and inspect the laboratory during normal business hours and permission to inspect is granted by Executive Management.

Designated members of the QA Department are primary contacts for announced inspections. The QA Director is the primary contact for all unannounced agency inspections. If the QA Director is unavailable, Executive Management is notified, in addition to a member of the QA Department. The QA Director, or their designee, must obtain evidence of the investigator's authority either in the form of a letter or examination/explanation of credentials.

Inspections include the examination of records or the inspection of facilities. Investigators are usually concerned only with the records relating to their responsibilities. As a general rule, they are given copies of records and documents, if requested. The laboratory must have a record of all items provided to an investigator.

Investigators must be escorted through the laboratory. The laboratory is not obligated to show an investigator the following types of information: sales, financial or pricing information, or any personnel data other than training or qualification documentation. On a case-by-case basis, internal QA audit reports and investigation reports are made available for agency review. Any questions or concerns about a request made by an investigator in regard to recording devices or photographs must be reviewed with legal counsel.

The laboratory personnel are not permitted to sign affidavits. If an affidavit is presented during an inspection, all personnel are directed not to sign it, read it, nor listen to it being read. The only document that is acceptable to sign is an acknowledgement that an inspection report has been received. If there is any doubt as to what should be signed, legal counsel must be consulted.

11.6 Proficiency Testing

Many of the organizations that certify our laboratory to perform various analyses require proof of our competency. Laboratory performance is checked regularly by participation in a variety of proficiency testing (PT) programs. When available, blind samples are obtained from vendors that are accredited to provide PT samples under the TNI and/or ISO 17025 standards for all test and matrices routinely tested at the laboratory. In addition, some individual certification programs require analysis of specific sets of proficiency samples. The laboratory also chooses to participate in a double blind program.

Generally, the PT programs consist of samples or ampulated spiking solutions used to fortify laboratory samples. The laboratories analyze the samples in the same manner as a client sample and the data is sent to the agency or vendor for evaluation. After the study results are returned to the laboratory, any data falling outside the acceptance criteria is investigated, root cause is identified, and corrective action is implemented, if needed.

Results are circulated to management. No PT samples or portion of a PT sample are sent to another laboratory for analysis.

Double blind samples are submitted to the laboratories by the Client Services Department using a fictitious client name so that the analysts are not aware that the samples are PTs. The samples are submitted quarterly and include a cross-section of organic and inorganic tests. The acceptance criteria for these double blind samples are developed statistically using data from participating laboratories, providing a source of inter-laboratory comparison. Results are reviewed, investigated as needed, and circulated to management.

If a trend in PT failures is identified, additional blind samples are ordered for that specific test as corrective action.

Clients routinely submit blind and double blind samples to evaluate the laboratory's performance. If a report is issued to the laboratory, it is handled in the same manner as a scheduled PT study evaluation and follow-up.

12 CORRECTIVE AND PREVENTATIVE ACTION

12.1 Laboratory Investigation and Corrective Actions

Due to the technical nature of laboratory work and the broad scope of our QA program, a wide variety of laboratory issues can require investigation, root cause analysis, documentation, and corrective action. Prompt investigation and implementation of corrective action ensure that only data of known quality are reported and prevent the recurrence of errors. The following list provides "examples" of the type of issues that warrant investigation:

- Noncompliant QC results*
- Failed PT samples
- Reporting incorrect results
- Contamination issues
- Client technical complaints
- Procedural errors
- Missed holding times
- Systematic problems that compromise the accuracy or compliance of the data generated
- Problems with instrumentation and equipment which could compromise the data generated

These investigations must include the following:

- Identification of the problem
- Steps taken to investigate the problem
- Explanation of probable root cause(s) of the problem
- Steps taken to prevent future occurrence

- Determination of samples or systems affected by the problem

*Note: individual QC noncompliance does not require in depth investigation. Actions are taken as defined in the corresponding method and documented in the data. An adverse trend with noncompliance would be investigated.

Management is informed of problem situations. The QA staff track documentation, the status of the investigation activities, evaluates investigations for completeness and appropriateness, and monitors corrective action for follow-up/closure. Technical management and/or QA may issue a stop work notice if issues indicate the potential for problems on a broad scale or present a critical concern regarding the validity of the laboratory methods. The goal is to identify root cause, have the corrective action implemented promptly, and to the degree appropriate for the magnitude and risk of the problem. Tracking and trending of laboratory issues is performed by QA staff and reported to management on a monthly basis or immediately upon detection of a trend with potential for putting the laboratory or our clients at risk.

12.2 Investigation Process

All results from quality control (QC) samples are logged into the LIMS quality control system, which is programmed to alert analysts to unacceptable results. Analysts are required to review the results and determine the source of the problem. The source of the problem and proposed corrective action must be documented. Corrective action may include, but is not limited to, re-analysis, re-extraction or re-digestion, instrument maintenance, or re-calibration. If these actions do not yield compliant data within the required hold time, a Nonconformance Form is initiated to document actions and communication with the client. The original form is archived with the associated raw data. Nonconformance Forms are reviewed by the technical department's management, or designee. A copy of the form is reviewed by QA.

Missed holding times are investigated and documented using a Missed Holding Time form. The form includes documentation of the affected samples, reason the hold was missed and corrective actions taken, if applicable. Each form also has documented review and approval by the department manager, department director and the QA Director. Clients are informed of any problems involving holding time.

Other types of problems having potential impact on data quality or involve deviations to our processes are investigated and documented using an Investigation and Corrective Action Report (ICAR). This process was developed to ensure that laboratory problems are investigated, evaluated for root cause, corrective action is put into place to prevent recurrence, laboratory management review and QA approval occurs, and all steps are documented. These investigations are initiated and managed through a workflow interface (Jira). Any employee can initiate an ICAR through this system to document a laboratory problem. The investigation must be completed by designated members of management and approved/closed by QA. Each investigation has a unique tracking number assigned by Jira. Closed investigations are routed to the laboratory Vice-President, associated laboratory Director and the QA Director. Follow-up to ensure effective corrective action is managed by QA staff.

If a laboratory error is identified from the outcome of the investigation that impacts validity of client data, the client must be immediately notified in writing of the situation and corrected data provided as soon as possible. If the root cause of the problem has affected any other client sample results, all affected clients are notified immediately of the problem.

12.3 Client Feedback

The laboratory is in the business of providing high quality analytical testing services. The data that we supply to our clients must be technically complete, accurate, and compliant with applicable regulations. Complaints can be received via letter, phone call, e-mail, or face-to-face meeting.

When a complaint is received, it is our responsibility to determine, to the best of our ability, the extent of the issue and what data is in question. The person receiving the complaint documents this information and promptly forwards it to the appropriate management personnel where the work in question was performed. If a data reporting error is discovered, the final report and/or data must be regenerated with the correct value(s).

The CSR is responsible for entering client concerns into the LIMS and an automated summary report is sent to QA on a weekly basis for review. In some cases, an ICAR is initiated to address and document the situation. While an individual issue may not warrant a formal investigation, QA monitors these issues for potential trends and will issue an ICAR if a trend is evident.

On an annual basis, the laboratory sends a client satisfaction survey to all clients. The results of these surveys are compiled, routed to the laboratory Vice-President, technical and operations directors and the QA Director, and used to identify areas of improvement for the laboratory.

12.4 Preventative Actions

All employees are empowered and encouraged to use the concept of Preventive Action to avoid a problematic situation. The company supports, embraces and drives the process for continuous quality improvement by several means, such as: Ethics Hotline, the Suggestion Box (accessible to all employees on the company's Intranet 'LabLinks'), and training classes that include "Making Quality a Science" and Ethics. If an employee identifies a potential problem or an area of concern or it should be brought to the attention of his/her supervisor, Human Resources, QA Director or the Ethics Hotline.

The laboratory also utilizes a formal program to encourage preventive action through development of lean processes. The goal of this program is to optimize processes to ensure efficiency and operational improvements while maintaining compliance. The efficiency gains are inherently coupled with minimizing errors and rework. Teams of employees learn the tools and techniques to evaluate a process, identify potential sources of errors, delays or problems in an operation, determine system changes that will minimize these and work to implement the improvements. Each project includes thorough documentation of the evaluation, measurement, and implementation phases. The process is continually monitored to ensure that the anticipated results are sustained.

Employees are also encouraged to communicate to their supervisor any area(s) or operation(s) that they believe could be streamlined, make their job easier, would provide a quality improvement, or could provide a cost savings to the company.

Described below are some of the systems available to employees to assist with building quality and efficiency into their daily jobs. They stress a proactive approach/environment to problem solving and to review quality systems and operational efficiencies.

- "Making Quality a Science" is an introductory total quality management (TQM) course required for all employees to teach why quality is important and to explain the laboratory's quality philosophy and processes, and how to apply quality thinking and techniques on the job. Topics discussed include: communication, teamwork, serving the client, measurement, quality tools, and continuous process improvement. To foster continuous improvements of laboratory systems, process improvement teams are formed, as needed, if an employee needs help in solving a problem or addressing an issue. The goal of these groups is to have representation from various areas of the laboratory work together to look at a problem, evaluate the need for a temporary fix, brainstorm root

causes, plan process improvement, implement the process improvement, evaluate and follow-up to the corrective action.

- "Putting our Values to Work" (Ethics) is a seminar required for all employees to teach the laboratory's Statement of Values by examining how it translates to our everyday jobs and ethical decision making. Topics discussed include: Statement of Values, ethical paradigms, and ethical decision making. Mandatory ethics training refresher seminars are offered on an annual basis.

- The laboratory has contracted with an Ethics Hotline to provide an anonymous means of reporting ethics concerns or issues. The issue is forwarded by the service to the QA Director who will communicate internally with those who need to address the issue. All communication and actions are documented in a secure web interface managed by the hotline service company.

- The QA staff prepares monthly program status reports for management. The reports include a variety of metrics and graphs which are used to evaluate trends in laboratory performance across all quality and compliance areas. Management responds to any negative trends by developing a corrective action plan.

- The laboratory uses a Project Cycle process (further described in section 13.2) to proactively review and prepare for client projects in an effort to ensure full understanding by all laboratory staff of the client's needs and resolve any concerns in advance of receiving the work.

13 SERVICE TO CLIENTS

13.1 Service to Clients

We value our client relationships and support these partnerships through the following principles:

- Honesty and Fairness – Our corporate culture is founded on the principles of professionalism and high ethical standards in dealing with our clients. This may mean declining to provide the service requested (if we are convinced that to do so would be meaningless) or it may mean referring clients outside of our laboratory if we believe that another company can better meet their needs.

- Complete Service – We will give our clients full value on every service provided. We will provide detailed information on our methods, procedures, and QA programs if requested, and take a personal interest and initiative in helping solve our client's problems within the area of our professional expertise.

- Trustworthiness – All data and information developed for a client will be held confidential and not disclosed to a third party except on written request of the client. If information is subpoenaed, we must, by law, release it, but the client will be informed of the release.

- Commitment to Quality – We constantly strive to improve our service in quality, flexibility, and dependability, to keep our competitive edge. We will achieve this through: meeting the requirements of those we serve, staying apprised of regulatory and industry expectations, and providing prompt responses to client concerns.

- Basics of Superlative Service – Our focus is on our client's success. Through proactive collaborative communication, our leadership ensures we understand our client's expectations and strives to exceed them. We foster a service culture in our training, reward and recognition, and performance management process so each employee takes ownership to deliver superlative service to our clients. Feedback from clients, whether positive or negative, is an important part of our continuous improvement system. Ways in which feedback is gathered can include, but is not limited to, customer satisfaction surveys, client audits, and the customer complaint system, which is described within section 12.3.

We also view our fellow employees as our clients since they frequently receive the results of our labor. Meeting the requirements of the next employee in the workflow process is just as important as meeting the needs of an external client.

13.2 Review of Work Requests, Tenders, and Contracts

The laboratory places great importance on understanding and meeting client requirements for a project. We ensure, to the best of our ability, that client/project requirements are identified and communicated through the laboratory. Project evaluation can be achieved in various ways, including the review of analytical methods, protocols, business contracts, and quality project plans (QAPPs). The project review encompasses our Project Cycle process and individual topics to be evaluated for a project include, but are not limited to: scope of testing; required accreditations (i.e. individual state agencies, NELAP, DoD, and ISO 17025) held by the laboratory; appropriate and current testing methods; ability to meet project required reporting limits and QC (if applicable); inconsistencies clarified; and nonstandard work requests.

Project kick-off meetings can be arranged through the CSR or Business Development Group. These meetings allow the client and key technical personnel to discuss project issues and requirements prior to project initiation. Any differences between laboratory processes and the project requirements are discussed and addressed with the client and the laboratory staff before the project is accepted and samples arrive. Testing that cannot be performed at the laboratory may be subcontracted to another laboratory (see 13.4).

A key client contact, the CSR, is assigned to oversee the project. Communication between the client and laboratory staff is available and is coordinated through the CSR.

As a project continues, the CSRs provide continuous communication and status reports (if requested) about the project to the client. The CSR relays any project changes or modifications to the technical groups. If the client submits revised project documents (QAPPs, etc.) then the Project Cycle review process is repeated. The CSR also communicates any issues encountered by the technical laboratories back to the client and vice-versa.

13.3 Timely Delivery

Evaluating laboratory capacity and ability to perform specific projects is a joint responsibility between the Technical Director, Business Development, and the laboratory managers. We recognize that one of the most important aspects of the service we offer is turnaround time.

Many analysts are cross-trained to perform a variety of tests, and there is redundant equipment available in the laboratory area creating operation flexibility for routine work. Larger projects are reviewed against capacity estimates before bids are submitted to ensure that the client's schedule is met. Turnaround time is continually measured.

Regularly scheduled meetings are held with technical and support management, and project management personnel to review progress with current projects, as well as special requirements of new work scheduled for the laboratory.

Management receives a daily report of the status of all samples in the lab, including those with priority status or those that have exceeded a preset turnaround time. This enables the planning and organizing of the workload through efficient scheduling.

Any changes to the established timeline by the client or the laboratory must be communicated to the client or laboratory as soon as possible. Upon communication of changes, a new timeline is established and agreed upon by both parties. If a client requires a change in the scope of the project (e.g., number of samples submitted, change in analyses, revised protocol) the laboratory must be informed in writing and a new timeline and cost estimate is be provided.

13.4 Subcontracting

The laboratory may subcontract tests to other laboratories if the requested testing is not routinely performed in our laboratory. To a lesser extent, samples may need to be subcontracted to an overflow laboratory to ensure hold times and/or turn-around-times (TAT) are met.

Testing is only subcontracted with the client's knowledge and approval. The CSR must notify the client in writing when any of their requested analyses will be subcontracted to another lab. Client approval must be obtained in writing before samples are shipped.

Subcontract laboratories are selected based on their qualifications and accreditations. The subcontractor is requested to sign a Laboratory Analytical Services Subcontract. See form 9033100 to review details of the contract terms and information requested from the subcontract laboratory. If projects require a specific agency certification (i.e. individual state agencies, National Environmental Laboratory Accreditation Program (NELAP), Department of Defense (DoD) Environmental Laboratory Accreditation Program (ELAP), and ISO 17025), only an appropriately accredited laboratory is used. The client may also have a list of laboratories to be used for subcontracting. In these cases, the evaluation of the subcontract laboratory is made by the client.

Data obtained from subcontract laboratories is clearly marked as such when reported by the laboratory. The data are submitted to the client in the format obtained from the subcontractor.

13.5 Use of NELAP and A2LA Logo

It is not laboratory policy to use these logos on any company letterhead, including analytical reports.

End of document

Version history

Version	Approval	Revision information
14	31.DEC.2015	

F-2

PFAS Standard Operating Procedure (Redacted)

October 25, 2016

Analysis summary for Per- and Polyfluoroalkylsubstances at Eurofins Lancaster Laboratories Environmental (ELLE)

A. Extraction
   1. An aliquot of the water sample is spiked with isotopically labeled analogs of each of the native PFAS compounds for which an isotopically labeled analog is available. The sample is extracted using an SPE cartridge and target analytes are eluted with organic solvent. The solvent eluant is concentrated prior to instrumental analysis.
   2. A portion of the solid sample is spiked with isotopically labeled standards and is blended into a water/solvent dispersion and sonicated. The mixture is centrifuged and concentrated prior to instrumental analysis.

B. Analysis

   Extracts are analyzed by LC/MS/MS under a minimum 5 point calibration. The 5 point calibration standard solutions range from 0.2 ng/ml to 160 ng/ml depending on the specific target compound. Batch QC (MB, LCS, MS/MSD) are analyzed along with samples and continuing calibration standards.

C. Data

   Analysts are instructed to look for and include the branched chain isomers in the quantification of field samples for PFOA and PFOS (PFHxS as well). Calibration standards use the linear isomer of these compounds so that we have a known and predictable response as well as chromatographic retention time expectation. Then in field samples we are looking for a peak with the correct mass transitions at the retention time for the linear isomer. In addition, we then look for a peak or peaks that are eluting just prior to the linear peak, which would be the branched chain isomers. The areas of the branched and linear isomers are integrated together and quantified against the linear calibration standard to generate our final result.

   We analyze technical grade standards of PFOA, PFOS and PFHxS on each instrument with each initial calibration so that we have a qualitative indication of where the branched chain isomers are eluting relative to the linear isomer.

Charles J. Neslund
Technical Director, Environmental Sciences

Eurofins Lancaster Laboratories Environmental, LLC
2425 New Holland Pike
Lancaster, PA 17601
office 717-556-7231
mobile 717-799-0439
CharlesNeslund@eurofinsUS.com

PFAS Field Collection Considerations

Because PFASs can be found in a number of consumer products, several recommended practices that are listed below should be followed during the collection of samples to avoid potential cross contamination:

- Post-it Notes, markers/Sharpies®, waterproof field books, plastic clipboards should not be used (masonite or aluminum clipboards are recommended), binders, spiral hard cover notebooks, or glue materials should not be used at any time during sample handling, or field activity.
- All samples should be collected in high density polyethylene plastic (HDPE) bottles with an unlined cap that is Teflon™ free.
- The field personnel involved with sample collection and handling should avoid wearing new clothing (i.e., at least 6 washings with PFAS-free detergent since purchase). as well as Gore-Tex products and Tyvek suits and fabric softeners.
- Personnel collecting samples should not wear anti-stain and/or water resistant clothing or shoes immediately prior to or during sample collection. Rain gear made from polyurethane and wax-coated material is recommended.
- Personnel collecting and handling samples should wear nitrile gloves at all times while collecting and handling samples. Gloves should be changed frequently during sampling collection and handling.
- Sunblock/insect repellents used on site should consist of 100% natural ingredients and should be PFAS-free.
- Sample collectors should not use cosmetics, moisturizers, hand cream, or other related products.
- Many food and snack products are packaged in wrappers treated with PFASs. Therefore, hands should be thoroughly washed after handling fast food, carryout food, or snacks.
- No food or drink should be brought on site, with the exception of bottled water and hydration drinks.
- Blue Ice® should not be used to cool samples or be used in sample coolers.
- The use of decontamination soaps containing fluoro-surfactants such as Decon 90 must be avoided. Alconox® or Liquinox® is recommended.

Eurofins Lancaster
Laboratories Environmental, LLC

2425 New Holland Pike
Lancaster, PA 17601

T | 717-656-2300
F | 717-656-2681
www.LancasterLabsEnv.com

We provide plastic (polyethylene) bottle ware to our clients and qualify all sample bottles to ensure they are PFAS-free. The lids on the bottles do not have Teflon™ lining. We have specially cleaned and tested PFAS-free deionized water that we use for all aspects of the PFAS extraction and analysis. We provide this water to clients for their field and equipment blanks. We have a separate lab space for the extraction and analysis of samples for PFAS analysis as well as the cleaning of glassware.

Eurofins Lancaster
Laboratories Environmental, LLC

2425 New Holland Pike
Lancaster, PA 17601

T | 717-656-2300
F | 717-656-2681
www.LancasterLabsEnv.com

Eurofins Document Reference	1-P-QM-WI -9039651	**Revision**	1
Effective Date	Oct 7, 2016	**Status**	Effective
Historical/Local Document Number	10954, 14091 (1-P-QM-WI-9012802)		
Local Document Level	Level 3		
Local Document Type	TEST - Testing Document		
Local Document Category	ANALYSIS-ES - Analysis-Environmental Science		

Prepared by	Timothy Trees
Reviewed and Approved by	Christine Ratcliff;Review;Monday, October 3, 2016 6:48:48 PM EDT Dorothy Love;Approval;Tuesday, October 4, 2016 8:02:47 AM EDT

eurofins	Always check on-line for validity.	Level: **Work Instruction**
Document number: **T-PFAS-WI14355**	**Polyfluorinated Alkyl Substances (PFASs) in Aqueous Samples by Method 537 Revision 1.1 Modified Using LC/MS/MS**	
Old Reference: **1-P-QM-WI-9039651 (1-P-QM-WI-9012802)**	**REDACTED FOR PROPRIETARY CONTENT**	
Version: **6**	Document users: **6_EUUSLA_PFAS_Analyst, 6_EUUSLA_PFAS_Data_Reviewers, 6_EUUSLA_PFAS_Sample_Prep**	Organisation level: **5-Sub-BU**
Approved by: **UKA4** Effective Date **01-MAR-2018**		Responsible: **5_EUUSLA_PFAS_Manager**

LIMS ID

14091, 14343, 14344, 14434, 14465, 14473

This documentation has been prepared by Eurofins Lancaster Laboratories Environmental LLC and its affiliates ("Eurofins"), solely for their own use. The user of this document agrees by its acceptance to return it to Eurofins upon request and not to reproduce, copy, lend, or otherwise disclose its contents, directly or indirectly, and not to use if for any other purpose other than that for which it was specifically provided. The user also agrees that where consultants or other outside parties are involved in the evaluation process, access to these documents shall not be given to said parties unless those parties also specifically agree to these conditions.

THIS DOCUMENT CONTAINS VALUABLE CONFIDENTIAL AND PROPRIETARY INFORMATION. DISCLOSURE, USE OR REPRODUCTION OF THESE MATERIALS WITHOUT THE WRITTEN AUTHORIZATION OF EUROFINS IS STRICTLY PROHIBITED. THIS UNPUBLISHED WORK BY EUROFINS IS PROTECTED BY STATE AND FEDERAL LAW OF THE UNITED STATES.

Revision Log
Reference Cross
Reference Scope
Basic Principles
Reference Modifications
Interferences
Precaution to Minimize Method Interference
Safety Precautions and Waste Handling
Personnel Training and Qualifications
Sample Collection, Preservation, and Handling
Apparatus and Equipment
Reagents and Standards
Calibration
Procedure
Calculations
Statistical Information/Method Performance
Quality Assurance/Quality Control

Revision Log

PROPRIETARY CONTENT

Reference

1. US EPA Method 537 — Determination of Selected Perfluorinated Alkyl Acids in Drinking Water by Solid Phase Extraction and Liquid Chromatography/Tandem Mass Spectrometry (LCMSMS), Version 1.1, Modified, September 2009.

2. Determination of Selected Perfluorinated Alkyl Acids in Drinking Water by Solid Phase Extraction and Liquid Chromatography/Tandem Mass Spectrometry (LCMSMS), EPA 537 Version 1.1, Modified, September 2009. Department of Defense Quality System Manual Version 5.1, Table B-15.

3. Standard Test Method for Determination of Perfluorinated Compounds in Soil by Liquid Chromatography Tandem Mass Spectrometry (LC/MS/MS), ASTM Method D7968, 2014.

4. ISO 25101:2009(E) Water quality — Determination of perfluorooctanesulfonate (PFOS) and perfluorooctanoate (PFOA) — Method for unfiltered samples using solid phase extraction and liquid chromatography/mass spectrometry. March 2009.

5. Method for Trace Level Analysis of C8, C9, C10, C11, and C13 Perfluorocarbon Carboxylic Acids in Water. Karen Risha, John Flaherty, Roice Wille, Warren Buck, Francesco Morandi, and Tsuguhide Isemura. Anal. Chem. 2005, 77, 1503-1508.

6. *Chemical Hygiene Plan*, current version.

Cross Reference

Document	Document Title
T-PEST-WI9847	Common Equations Used During Chromatographic Analyses
T-PFAS-WI13881	Standards Management in the PFAS Laboratory

Scope

This method is applicable for the determination of selected per- and polyfluorinated alkyl substances (PFAS) in aqueous samples to include non-potable waters and non-regulatory potable water when directed by the client. The compounds analyzed in this method are listed in the table below. The most current MDLs and LOQs are listed in the LIMS.

Analyte	Acronym	CAS#
Perfluorobutanesulfonic acid	PFBS	375-73-5
Perfluorodecanoic acid	PFDA	335-76-2
Perfluorododecanoic acid	PFDoDA	307-55-1
Perfluoroheptanoic acid	PFHpA	375-85-9
Perfluorohexanesulfonic acid	PFHxS	355-46-4
Perfluorohexanoic acid	PFHxA	307-24-4
Perfluorononanoic acid	PFNA	375-95-1
Perfluorooctanesulfonic acid	PFOS	1763-23-1
Perfluorooctanoic acid	PFOA	335-67-1
Perfluorotetradecanoic acid	PFTeDA	376-06-7
Perfluorotridecanoic acid	PFTrDA	72629-94-8
Perfluoroundecanoic acid	PFUnDA	2058-94-8
Perfluoro-n-butanoic acid	PFBA	375-22-4
Perfluoro-n-pentanoic acid	PFPeA	2706-90-3
8:2 - Fluorotelomersulfonate	8:2FTS	39108-34-4
N-methylperfluoro-1-octanesulfonamidoacetic acid	NMeFOSAA	2355-31-9
N-ethylperfluoro-1-octanesulfonamidoacetic acid Perfluoroundecanoic acid	NEtFOSAA	2991-50-6
4:2-Fluorotelomersulfonate	4:2-FTS	757124-72-4
Perfluoropentanesulfonate	PFPeS	2706-94-4
6:2-Fluorotelomersulfonate	6:2-FTS	27619-97-2

Perfluoroheptanesulfonate	PFHpS	375-92-8
Perfluorononanesulfonate	PFNS	474511-07-4
Perfluorodecanesulfonate	PFDS	335-77-3
10:2-Fluorotelomersulfonate	10:2-FTS	120226-60-0
Perfluorododecanesulfonate	PFDoDS	79780-39-5
Perfluorohexadecanoic acid	PFHxDA	67905-19-5
Perfluorooctadecanoic acid	PFODA	16517-17-6
Perfluorooctanesulfonamide	PFOSA	754-91-6
2-(N-methylperfluoro-1-octanesulfonamido)-ethanol	NMePFOSAE	24448-09-7
N-methylperfluoro-1-octanesulfonamide	NMePFOSA	31506-32-8
2-(N-ethylperfluoro-1-octanesulfonamido)-ethanol	NEtPFOSAE	1691-99-2
N-ethylperfluoro-1-octanesulfonamide	NEtPFOSA	4151-50-2

Basic Principles

A 250-mL aqueous sample is fortified with isotopically-labeled extraction standards and is passed through a solid phase extraction (SPE) cartridge to extract the analytes. The compounds are eluted from the solid phase with a combination of solvents. The extract is concentrated to ~400-500ul with nitrogen in a heated water bath, and then reconstituted to 1ml with methanol. Isotopically-labeled injection internal standards are added to the sample extract and it is analyzed by LC/MS/MS operated in negative electrospray ionization (ESI) mode for detection and quantification of the analytes. Quantitative analysis is performed using isotope dilution.

Reference Modifications

EPA Method 537 is written specifically for the analysis of drinking water samples. The following modifications to the method have been made to accommodate all aqueous samples.

1. A labeled isotopic analog is spiked into samples for all compounds where an isotopic analog is commercially available. These isotopic compounds are referred to as extraction standards. For those compounds, an isotope dilution calibration model is used. Where labeled isotopes are not available, an internal standard calibration model using the extraction standards is used.
2. Prior to instrumental analysis, separate but similar isotopic analogs are added to the sample extract prior to instrumental analysis. Using an internal standard calibration model these injection standards are used to calculate recoveries of the extraction standards..
3. Field reagent blanks are not processed as listed in EPA 537 Version 1.1 section 8.3

4. Trizma is not used for waters except in the cases where the water comes from a chlorinated water source.
5. Branched isomers of PFOS, PFHxS, NetFOSAA and NMeFOSAA are not included in the calibration curves.
6. Peak asymmetry factors are not calculated.
7. MRL confirmation is not performed.
8. Spike concentrations are not rotated between low, medium and high levels.
9. SPE is used for sample preparation. Cartridge types and elution profiles differ from EPA 537 Version 1.1

MDL studies and IDOCs have been performed to validate method performance.

Interferences

Compounds which have similar structures to the compounds of interest and similar molecular weights would potentially interfere. Method interferences may be caused by contaminants in solvents, reagents (including reagent water), sample bottles and caps, and other sample processing hardware that lead to discrete artifacts and/or elevated baselines in the chromatograms. The analytes in this method can also be found in many common laboratory supplies and equipment, such as PTFE (polytetrafluoroethylene) products, LC solvent lines, methanol, aluminum foil, etc. A laboratory blank is performed with each batch of samples to demonstrate that the extraction system is free of contaminants.

Precaution to Minimize Method Interference

PROPRIETARY CONTENT

Safety Precautions and Waste Handling

See *Chemical Hygiene Plan* for general information regarding employee safety, waste management, and pollution prevention.

The toxicity or carcinogenicity of each reagent used in this method has not been precisely defined. PFOA has been described as “likely to be carcinogenic to humans”. Each chemical should be treated as a potential health hazard and exposure to these chemicals should be minimized.

Exposure to these chemicals must be reduced to the lowest possible level by whatever means available, such as fume hoods, lab coats, safety glasses, and gloves. Gloves, lab coats, and safety glasses should be worn when preparing standards and handling samples. Avoid inhaling solvents and chemicals and getting them on the skin. Wear gloves when handling neat materials. When working with acids and bases, take care not to come in contact and to wipe any spills. Always add acid to water when preparing reagents containing concentrated acids.

All laboratory waste is accumulated, managed, and disposed of in accordance with all Federal, State, and local laws and regulations. All solvent waste and extracts are collected in approved solvent waste containers in the laboratory and subsequently emptied by personnel trained in hazardous waste disposal into the lab-wide disposal facility. HPLC vials are disposed of in the lab container for waste vials, and subsequently lab packed. Any solid waste material (disposable pipettes and broken glassware, etc.) may be disposed of in the normal solid waste collection containers.

Personnel Training and Qualifications

All personnel performing this procedure must have documentation of reading, understanding, and agreeing to follow the current version of this SOP and an annual documented Demonstration of Capability (DOC).

Initially, each chemist performing the extraction must work with an experienced employee for a period of time until they can independently perform the extraction. Also, several batches of sample extractions must be performed under the direct observation of another experienced chemist to assure the trainee is capable of independent preparation. During the training period, the new chemist may also learn the operation of the LC/MS/MS, calibration techniques, data processing and review, and maintenance procedures. Proficiency is measured through a documented Initial Demonstration of Capability (IDOC)

The IDOC and DOC consist of four laboratory control samples (or alternatively, one blind sample for the DOC) that is carried through all steps of the extraction and meets the defined acceptance criteria. The criteria include the calculation of mean accuracy and standard deviation.

Sample Collection, Preservation, and Handling

A. Sample Collection

The samples are collected in 250 mL polyethylene bottles containing 1.25 grams of Trizma, resulting in a Trizma concentration in the sample of 5 g/L. Trizma functions as a free chlorine scavenger; therefore, any chlorinated water supplies require the preservative. Water samples from non-chlorinated water sources would not necessarily require the Trizma preservative. Keep the sample sealed from time of collection until extraction.

NOTE: PFAS contamination during sampling can occur from a number of common sources, such as food packaging and certain foods and beverages. Proper hand washing and wearing nitrile gloves will aid in minimizing this type of accidental contamination of the samples.

B. Sample Storage and Shipment

1. Samples must be chilled during shipment and must not exceed 10°C during the first 48 hours after collection. Sample temperature must be confirmed to be at or below 10°C when the samples are received at the laboratory.

2. Samples stored in the lab must be held at a temperature of 0° to 6°C, not frozen, until extraction.

3. Water samples must be extracted within 14 days. Extracts must be analyzed

within 28 days after extraction. Extracts are stored at room temperature.

Apparatus and Equipment

PROPRIETARY CONTENT – LC/MS/MS that functions in ESI negative ION mode.

Reagents and Standards

All solvents, acids, and bases are stored in glass bottles in flammable proof cabinets or pressure resistant steel drums. Solvents, acids, and bases are stored at ambient temperature for up to 1 year. All non-solvents are stored according to manufacturer's storage conditions.

A. Reagents:

PROPRIETARY CONTENT

B. Standards: See SOP T-PFAS-WI13881

Calibration

A. Initial Calibration

1. A minimum of five calibration standards are required. In general, Cal1, Cal2, Cal3, Cal4, Cal5, Cal6, and Cal 7 are included in the initial calibration. S/N ratio must be ≥ 10:1 for all ions used for quantification.

2. Initially an MDL standard is analyzed to ensure all compounds can be detected at the MDL level. Following the MDL standard, the Cal1-Cal7calibration standards are analyzed. If compounds are not detected in the MDL standard, the source of the problem must be determined and the MDL standard reanalyzed.

3. Analyze a Cal3 level standard that contains linear and branch chained isomers of PFOA, PFOS and PFHxS. The analysis of this standard is used to demonstrate where the branch chained isomers elute and not included in the calibration curve. This will assist the chemist in identifying and properly integrating these compounds in samples.

4. Fit the curve with a linear through zero or linear with a concentration weighing factor of 1/x or quadratic regression with a concentration weighing factor $1/x^2$.

5. Isotopically labeled compounds are not available for PFPeS, PFHpS, PFNS, PFDS, PFDoS, 10:2-FTS, PFTrA, PFHxDA, and PFODA. See below for referenced extraction standards.

Compound	Extraction standard
PFPeS	13C3-PFBS
PFHpS	13C3-PFHxS
PFNS	13C8-PFOS
PFDS	13C8-PFOS
PFDoS	13C8-PFOS
10:2-FTS	13C2-8:2-FTS
PFTrDA	13C2-PFDoDA
PFHxDA	13C2-PFTeDA
PFODA	13C2-PFTeDA

6. Initial calibration acceptance criteria

When each calibration point, except the lowest point (Cal1), is calculated back against the curve, the back calculated concentration should be within ±70-130% of its true value. The lowest calibration point (Cal1) should calculate to be within ±50-150% of its true value. The R^2 value for each calibration curve must be ≥ 0.99 for each analyte.

DoD QSM5.1 criteria:

a. All calibration points must be within ±70-130% of their true values.

b. The %RSD of the response factors for all analytes must be < 20%.

If the criteria are not met, the source of the problem must be determined and corrected. Situations may exist where the initial calibration can be used. In those cases, the data will be reported with a qualifying comment.

7. Initial Calibration Verification (ICV)

A check standard prepared from a second source (ICV) is injected to confirm the validity of the calibration curve/standard. The calculated amount for each analyte should be ± 30% of the true value.

B. Continuing calibration

1. Once the calibration curve has been established, the continuing accuracy must be verified by analysis of a continuing calibration verification (CCV) standard every ten samples and at the end of the analysis sequence.

a. The CCV run after the initial calibration must be at the CAL3 level.

b. Subsequent CCV standards should alternate between the low, mid and high levels of the calibration curve.

DoD QSM5.1 criteria: The CCV standards must alternate between the CAL1-CAL3 levels. All analyte concentrations must be within ±30% of their true values.

2. Acceptance criteria

a. The calculated amount for each compound (native and extraction standard) in the CCV standard must be within ±30% of the true value. Samples that are not bracketed by acceptable CCV analyses must be reanalyzed. The exception to this would be if the CCV recoveries are high, indicating increased sensitivity, and there are no positive detections in the associated samples, the data may be reported with a qualifying comment.

DoD QSM5.1 criteria: If acceptance criteria are not met, immediately analyze two additional consecutive CCVs. If both pass acceptance criteria, samples may be reported without reanalysis. If either fail, or two consecutive CCVs cannot be run, repeat CCV and reanalyze all samples since last successful CCV.

b. The absolute areas of the injection internal standards should be within 50-150% of the average areas measured during the initial calibration.

Procedure

A. Sample Preparation - **all samples**

1. Mark the outside of each sample container with pen ("Sharpie") to record the level of the sample in the container prior to extraction.

2. If required, add 1.25 grams of Trizma to a 250 ml HDPE bottle for the laboratory reagent blank (LRB) and the lab fortified blank (LFB). Fill each bottle with 250 ml of Milli-Q water. Record 250 ml as the volume for the batch QC samples on the batchlog.

3. If sample has dissolved and/or settleable solid content; i.e., is cloudy or has a layer of sediment/solids at the bottom of the bottle, an aliquot should be taken from the original bottle and diluted with reagent water in order to minimize difficulty passing through the SPE sorbent bed. If unsure whether or not less-than-full sample volume should be used for SPE extraction, consult a supervisor.

a. Determine aliquot to be used for extraction (50ml; 100ml).

b. Label a clean 250ml HDPE bottle with associated ELLE sample number.

c. Label appropriate number of 50ml centrifuge tubes.

d. Shake/invert sample bottle to thoroughly mix the sample before pouring aliquot(s).

e. Pour sample from original bottle into centrifuge tubes. Cap tubes and centrifuge for 5 minutes at full speed (one full cycle).

f. On a calibrated, top-loading balance, place labeled empty 250ml PP wide-mouthed bottle.

g. Decant centrifuged sample aliquot(s) from centrifuge tube(s) to the 250ml bottle until desired volume (weight in grams) is reached. 100g = 100ml; 50g = 50ml, etc.

h. Add Milli-Q water to the bottle until a weight of 250g (total of 250ml) is reached.

i. Shake/invert several times to mix thoroughly.

j. Record the aliquot taken from the original bottle (50ml; 100ml) as the sample volume.

B. Solid Phase Extraction (SPE) - **all samples**

PROPRIETARY CONTENT

15. Reconstitute to 1.0 ml with 100% methanol. Vortex to mix. Centrifuge all 15 ml collection tubes at for 5 minute at full speed (~4100 rpm).

16. Place each empty sample bottle on the top-loading balance and tare.

17. Fill each tared sample container to the mark placed on the bottle prior to extraction with DI water.

18. Record the weight as the sample volume on the batchlog.

19. Transfer 400 µL of the final extract to labeled auto-sampler vials. Add 20 ul of labeled internal standard spike and cap and vortex the auto-sampler vial. Samples are now ready for analysis.

20. Cap the centrifuge tube. Store the remaining centrifuged extracts at room temperature for dilution or reinjection if needed.

C. **Extract Treatment for DoD samples:**

PROPRIETARY CONTENT

D. Serial Dilution Sample Prep

PROPRIETARY CONTENT

E. LC/MS/MS Analysis

1. Mass Calibration and Tuning

a. At instrument set up and installation and after the performance of major maintenance, calibrate the mass scale of the MS with calibration compounds and procedures described by the manufacturer. The entire mass range must be calibrated.

b. When masses fall outside of the ±0.5 amu of the true value, the instrument must be retuned using PPG according to the manufacturer's specifications. Mass assignments of the tuning standard must be within 0.5 amu of the true value. Refer to the instrument manufacturer's instructions for tuning and conditions. These values are stored in the tune file for future reference.

2. The mass spectral acquisition rate must include a minimum of 10 spectra scans across each chromatographic peak.

3. Acquisition method: See attachment 1

4. DoD QSM5.1 criteria for Instrument Sensitivity Check (ISC) and Instrument Blanks

a. Prior to sample analysis and at least every 12 hours, an instrument sensitivity check (ISC) must be performed. The CAL1 standard will be analyzed. All analyte concentrations must be within +/- 30 of their true values. If the criteria is not met, correct problem and rerun ISC. If problem persists, repeat the ICAL. No samples can be analyzed until the ISC meets acceptance criteria.

b. Instrument blanks need to be analyzed immediately following the highest standard analyzed and daily or at the start of a sequence. The concentration of all analytes must be ≤ 1/2 the LOQ. If acceptance criteria are not met the calibration must be performed using a lower concentration standard for the high standard until the criteria are met.

5. Load sample vials containing standards, quality control samples, and sample extracts into autosampler tray. Allow the instrument adequate time to equilibrate to ensure the mass spec and LC have reached operating conditions (approximately 5 minutes) before the first injection. Analyze several solvent blanks clean the instrument prior to sample acquisition.

6. After the initial calibration, inject a solvent blank, followed by the ICV, L/B standard, closing Cal 3 level CCV , CCV, extraction batch QC, and samples. Bracket each set of ten samples with a CCV standard, alternating between the Cal3, Cal4, and Cal5 levels.

Note: For DoD QSM5.1: CCVs will range from the CAL1 to the CAL3 level standard.

7. After injections are completed, check all CCV recoveries and absolute areas to make sure they are within method control limits. See Calibration section B.2 for acceptance criteria. Process each chromatogram and closely evaluate all integrations, baseline anomalies, and retention time differences.

If manual integrations are performed, they must be documented and a reason given for the change in integrations. The manual integrations are documented during data processing and all original integrations are reported at the end of the sample PDF file with the reason for manual integration clearly listed.

8. Quantitate results for the extraction blank.

a. Non-DoD criteria: No target analytes at or above the reporting limit may be found in the extraction blank for acceptable batch results. If a target analyte is detected in the extraction blank but not detected in the sample, the data is reported. If a target analyte is detected in the method blank at a concentration greater than the reporting limit and also in the sample, the sample must be reextracted. If the target analyte in the sample is detected at a concentration greater than 10 times the amount detected in the method blank, the data is reported.

b. DoD QSM5.1 criteria: No target analytes detected > 1/2 the LOQ or > 1/10 the regulatory limit, whichever is greater. If criteria is exceeded, reextract all samples with positive detections associated with the method blank.

9. Calculate the recoveries of spiked analytes for the LCS, matrix spike and matrix spike duplicate (MS/MSD) by comparing concentrations observed to the true values. The advisory QC acceptance limits for LCS and MS/MSD recovery are 70 to 130% for each analyte. The advisory QC acceptance limit for the relative percent difference (%RPD) between LCS/LCSD and MS/MSD is ≤30%. If LCS and/or LCSD recoveries are acceptable, proceed to sample quantitation. If the LCS recoveries are unacceptable, the samples associated with the LCS may need to be reanalyzed. If LCS recoveries are above the advisory QC acceptance limits, and there are no positive detections in the sample, the data may be reported. If MS/MSD recoveries are outside QC acceptance criteria, the associated data will be flagged or noted in the comments section of the report.

10. Isotopically labeled extraction standards are added to all samples, extraction blank, LCS/LCSD, and MS/MSD prior to extraction.

a. Non-DoD criteria: The recovery of the extraction standards should be within QC acceptance criteria. If the extraction standard recovery(ies) is(are) outside the QC limit(s), consult a supervisor to determine the appropriate course of action based on batch and sample results.

b. DoD QSM5.1 criteria: All extraction standard recoveries must be within 50% to 150% of the true value. If recoveries are outside the acceptance criteria, samples must be reextracted.

11. Isotopically labeled injection standards are added to each QC and field sample extract prior to analysis.

a. Non-DoD criteria: The absolute areas of the injection standards should be within 50-150% of the average areas measured during the initial calibration. If the internal standards are recovered outside 50-150%, consult a supervisor to determine the appropriate course of action based on batch and sample results.

b. DoD QSM5.1 criteria: Peak areas must be within -50% to +50% of the area measured in the ICAL midpoint standard. On days when an ICAL is not performed, the peak areas must be within -50% to +50% of the peak area measured in the daily initial CCV. If injection internal standards fall outside the acceptance window, analyze a second aliquot of the extract. If none remains, reanalyze the first aliquot.

12. Compare the retention times of all of the analytes, surrogates and internals standards. The relative retention times should not vary by more than 0.2 retention time units.

13. The MDL standard and the linear/branch chain standard are used when assessing the correctness of the computer generated peak integrations. For results that have responses at or near the MDL, the analysts will calculate 1/2 of the area ratio of that compound in the MDL standard. If the area ratio for the compound in the sample exceeds that 1/2 the area ratio from the MDL standard, the peak is reported as a positive detection.

14. If the calculated concentation exceeds the calibration range of the system, dilute the extract with MeOH and add the appropriate amount of extraction standard to match the original concentration. Add 10 ul of injection internal standard and analyze the dilution.

Dilution Example 1/10: Mix 0.877 mL of MEOH with 0.100 mL of sample extract and 0.0225 mL of labeled extraction standard. Vortex to mix. Using an auto-pipette, transfer 200 uL of the mixed solution into a labeled auto-sampler vial containing a plastic insert. Using a syringe, add 10 uL of labeled injection std to the 200 uL aliquot. Cap and vortex thoroughly to mix.

.

Calculations

A. Peak Area Ratio

$$\text{Peak Area Ratio} = \frac{\text{Analyte Response}}{\text{Labeled Analyte Response}}$$

B. Analyte Concentration using linear through zero curves (MQ Data processing system)

Concentration = (area ratio ÷ slope) x Dilution Factor x Internal Standard concentration

Where: internal standard concentration = 1 ng/ml

C. Sample Concentration (used only for aqueous samples using the MultiQuant data processing system on the AB Sciex LC/MS/MS)

Sample concentration (ng/l) = Calc conc x (Sample volume ÷ Sample weight) x DF

D. See *T-PEST-WI9847* for additional calculations used to evaluate the calibrations and quality control samples.

Statistical Information/Method Performance

The method is evaluated through both initial and ongoing Demonstrations of Capability (IDOC and DOC). The IDOC includes performance of quad studies, MDL studies, and when available, acceptable scores obtained in Performance Testing (PT) studies. Annual MDL studies are performed as are annual analyst DOCs.

Quality Assurance/Quality Control

For each batch of samples extracted, a method blank, an LCS/LCSD (Milli Q water spiked with all compounds to be determined carried through the entire procedure), and an MS must be extracted. If an MSD is submitted then an LCSD would not be extracted. A batch is defined as the samples to be extracted on any given day, but not to exceed 20 field samples. If more than 20 samples are prepared in a day, an additional batch must be prepared. If any client, state, or agency has more stringent QC or batching requirements, these must be followed instead. Statistical control limits must be calculated for recoveries of LCS and MS when sufficient data points have been collected

---

T-PEST-WI9847 Common Equations Used During Chromatographic Analyses T-PFAS-WI13881 Standards Management in the PFAS Laboratory

---

Attachment:
Attachment 1
Attachment 2

End of document

Version history

Version	Approval	Revision information
4	03.JAN.2018	
5	24.FEB.2018	
6	01.MAR.2018	

ATTACHMENT G

TRC - Laboratory Quality Manual

Geotechnical Services
Quality Management Plan

Revision No.: 02
Effective Date: June 22, 2016

Business Confidential Controlled Document

Uncontrolled if printed. See TRCNet to confirm the current revision.

Geotechnical Services
Quality Management Plan

Confidential and Proprietary Information Notice

This Quality Management Plan and its contents are confidential and proprietary business information of TRC Companies, Inc. (TRC) and may not be reproduced, copied or transmitted in any media whatsoever without the express written consent of TRC. TRC provides copies to clients as documentation of the TRC Geotechnical Services quality management program. TRC reserves the right to recall, reissue, or revise this plan and its contents as the need arises.

Revision No.: 02
Effective Date: June 22, 2016

© 2016 TRC Companies, Inc. All rights reserved.

MANAGEMENT REVIEW AND AUTHORIZATION

We, the undersigned, acknowledge and confirm that the quality processes and procedures described in this Quality Management Plan are in alignment with the quality assurance requirements applicable to the professional services we provide to our clients and reflect current procedures within our respective Practices.

6/20/2016

X Mark E Lynch

Mark E. Lynch
Infrastructure Sector Director
Signed by: pmullins@trcsolutions.com

6/20/2016

X Douglas W. Massih

Douglas W. Massih
Deputy Infrastructure Sector Director
Signed by: Massih, Douglas W.

6/20/2016

X

Charles E. Malson
Practice Leader, Geotechnical Drilling Services

6/20/2016

X Petro W Kazaniwsky

Petro W Kazaniwsky, P.E.
Principal Engineer, Geotechnical Engineering-East
Signed by: Kazaniwsky, Petro W.

6/20/2016

X Charles E Malson

Charles E. Malson
Practice Leader, Geotechnical Engineering-East
Signed by: Malson, Charles E.

6/20/2016

X Petro W Kazaniwsky

Petro W. Kazaniwsky, P.E.
Quality Leader, Geotechnical Engineering-East
Signed by: Kazaniwsky, Petro W.

6/21/2016

X Kent M. Screechfield

Kent M. Screechfield
Practice Leader, Geotechnical Engineering-...
Signed by: Screechfield, Kent

6/20/2016

X Scott M. Leck

Scott M. Leck, P.E., G.E.
Quality Leader, Geotechnical Engineering-...
Signed by: Leck, Scott

X Amelia Speth

Amelia Speth
National Quality Director, TRC
Signed by: Speth, Amelia

ABSTRACT

This Quality Management Plan (QMP) provides an overview of the quality program that is applies to TRC's Infrastructure Sector, Geotechnical Services projects. Although the details and emphases of project-specific quality procedures vary based on the applicable requirements and specifications, each project incorporates the core concepts of this quality program. Characteristics of the quality program described in this QMP include:

- Quality begins with the individual and all employees take responsibility for the quality of their individual work.
- The quality program is intended to align with the substantive elements of ISO 9001. *(QMP Section 1)*
- Prior to commencing work, the scope and quality-related requirements of the project should be understood by all parties involved and processes and controls should be in place so that professional services and/or work products meet the project-specific requirements. *(QMP Section 3)*
- Peer review, proportionate to risk and appropriate to the project, should be consistently implemented. *(QMP Section 5.2)*
- Project planning and management procedures include establishing budgets sufficient to complete the specified scope of work and corresponding quality checks, confirming that project team capabilities and expertise are in line with the job requirements, and establishing provisions for scheduling and reviews. *(QMP Section 5.3)*
- Subcontractors are selected on their ability to perform the service required, meet applicable safety, quality, and financial standards, and are willing to accept and comply with applicable quality standards. *(QMP Section 5.4)*
- Consistent with TRC's commitment to continuous improvement, each Practice should perform annual quality program self-assessments and employees are encouraged to bring opportunities for improvement to the attention of their Supervisor. *(QMP Sections 7 and 9)*

While this QMP introduces quality-related protocols and procedures, detailed descriptions of many of the task-specific procedures are described in Practice-specific procedure manuals.

HISTORY OF REVISIONS

Rev.	Date	Purpose
02	06/22/16	Revisions limited to TRC organization description updates, key personnel changes, editorial changes for clarification purposes, hyperlinks to TRCNet, and TRC Quality Management Policy update (Appendix A).
01	03/12/12	Routine quality program review and update. This Quality Management Plan (QMP) merges previous regional-specific QMPs into a single document.
00	06/23/08	*Western Region Infrastructure Sector Quality Management Plan*. QMP was specifically designed to describe the Western Region Infrastructure Sector quality program and procedures.
00	11/19/07	FY2007 TRC Revised its Quality Management Systems. Initial *Eastern Region Infrastructure Geotechnical Practice Quality Management Plan* specifically designed to describe the Eastern Region Infrastructure Geotechnical Practice quality program and procedures.

TABLE OF CONTENTS

Page

1.0 Quality Management Policy 1
1.1 Applicability and Communication of the Quality Management Program 1
2.0 Operational and Quality Objectives 2
3.0 Compliance Requirements 2
3.1 Identification of Applicable Requirements 2
3.2 Integrating Applicable Requirements into Projects 3
4.0 Risk Assessment – Risk Management 4
4.1 Process to Identify and Manage Risks on Proposal Opportunities 4
4.2 Process to Identify and Manage Risks on Individual Projects 4
5.0 Operational Controls 5
5.1 Organization and Responsibilities 5
5.1.1 Corporate Organization and Responsibilities 6
5.1.2 Infrastructure Sector Organization and Responsibilities 7
5.1.3 Geotechnical Practices Organization and Responsibilities 8
5.1.4 Project Organization and Responsibilities 9
5.2 Peer Review 11
5.2.1 How to Integrate Peer Review into Activities 12
5.2.2 Selection of Peer Reviewers 12
5.2.3 Reviewing Interim and Final Work Products 12
5.2.4 Peer Review Comments and Records 13
5.3 Project Planning and Management 13
5.4 Controls for Purchasing Materials and Subcontracting Services 13
5.4.1 Evaluation of Subconsultants/Subcontractors 13
5.4.2 Subconsultant/Subcontractor Management 14
5.5 Recordkeeping and Document Control Procedures 14
5.5.1 Document Control Procedures 14
5.5.2 Records Retention 14
5.6 Internal Communication 14
5.7 Communication with the Client and Technical Interfaces 15
5.8 Project-Specific Quality Assurance Programs 15
5.9 Construction Support Services 15
5.10 Geotechnical Drilling Quality Procedures 15
5.11 Measurement and Test Equipment Controls 16
5.12 Tests, Measurements, and Analyses 16
5.13 Field and Laboratory Data Acquisition, Review, and Validation Procedures 16
5.14 Identification and Traceability 17
5.15 Custody, Handling, Storage, and Shipping of Samples 17

5.16 Software Control 17
5.17 Corrective and Preventive Actions 17
5.18 Workplace Safety 18
5.19 Review and Update of Operational Controls 18
6.0 Training and Qualifications 18
6.1 Employee Training Requirements 18
6.2 Training Records 19
7.0 Self-Assessment 19
8.0 Management Review 20
8.1 Management Review of the TRC Quality Management System 20
8.2 Revisions to the TRC Quality Management System and Policy 20
9.0 Continuous Improvement 20

Appendix A: TRC Quality Management Policy
Appendix B: Comparison of Published Quality Standards and the Geotechnical Services Quality Management Plan

LIST OF FIGURES

Page

Figure 5-1. TRC Organizational Model 6
Figure 5-2. Infrastructure Sector Organizational Model 8

LIST OF TABLES

Page

Table B-1. Comparison of ISO 9001-2015 and the Geotechnical Services QMP B-2

1.0 QUALITY MANAGEMENT POLICY

TRC's QM Policy establishes a framework that promotes Operational Excellence and emphasizes Continuous Improvement.

TRC's Quality Management (QM) Policy highlights our dedication to consistently delivering services that meet applicable requirements and our clients' expectations. Each of TRC's operational sectors and business units (i.e., Energy, Environmental, Infrastructure, and Pipeline Services) is responsible for designing and implementing QM programs that are both appropriate for the services provided by their Practices and consistent with the TRC QM Policy. The Infrastructure Sector, Geotechnical Services (i.e., Geotechnical Drilling Services [GDR] and Geotechnical Engineering [GEI] Practices) QM program is described in this Quality Management Plan (QMP) and is in alignment with TRC's QM Policy and the core elements of ISO 9001 (i.e., the international quality management systems standard).

TRC's QM Policy (Appendix A) is consistent with the TRC Mission, Vision, and Value Statement on Quality and confirms our commitment to meeting client expectations and applicable requirements.

TRC
Results you can rely on

OUR MISSION
We understand our clients' goals and embrace them as our own, applying creativity, experience, integrity and dedication to deliver superior solutions to the world's energy, environmental and infrastructure challenges.

OUR VISION
We will solve the challenges of making Earth a better place to live – community by community and project by project.

OUR VALUES
Safety – *We create a working environment that promotes safe performance.*
Quality – *We always strive for excellence in the services we provide and in the results we produce for our clients.*
Integrity – *We are committed to the highest ethical standards.*
Creativity – *We believe in looking at challenges and opportunities from new angles and in exercising our curiosity.*
Teamwork – *We work together to succeed.*
Accountability – *We take responsibility for all of our decisions and actions.*
Passion – *We deliver superior results because we care deeply about what we do.*

1.1 Applicability and Communication of the Quality Management Program

This QMP applies to employees, projects, and operations within the geotechnical services practices (i.e., GDR and GEI Practices). Although the details and emphases of project-specific quality practices and procedures vary based on the applicable requirements and specifications, GDR/GEI project include the core concepts of the program described in this QMP, unless otherwise specified by the client in contract documents.

This QMP is readily accessible to all TRC employees on the TRCNet.

2.0 OPERATIONAL AND QUALITY OBJECTIVES

The following are quality objectives that apply to TRC's operations:

- Consistently achieve applicable requirements for each product and service offering.
- Maintain an ethical business organization.
- Provide timely responses in our service to clients and each other.
- Promote a customer-focused business culture aimed at client satisfaction.
- Encourage teamwork and enhance coordination with key personnel and task leaders to facilitate efficient, timely and quality deliverables.
- Make accuracy and precision of work products a priority.
- Establish and maintain appropriate schedules.

Goals and objectives describe what the QM Program is striving to accomplish. As with any QM Program, the underlying objective is to promote achievement of high quality services and work products.

3.0 COMPLIANCE REQUIREMENTS

With the aim of enhancing customer satisfaction, identify project-related requirements and seek to achieve them.

A common definition of a "quality service" is one which "conforms to specifications" or "meets objectives." With quality as a company value, TRC recognizes the importance of: 1) confirming that applicable parties understand the scope of work, applicable requirements, and/or quality specifications before commencing work; and 2) establishing appropriate processes and controls to guide project work such that it fulfills those requirements.

Note: "Requirements" can include:

- Customer requirements (e.g., contractual agreements, project scope of work).
- Federal, state, and local laws and regulatory requirements.
- Industry standards.
- TRC policies, procedures, and expectations.

3.1 Identification of Applicable Requirements

The Geotechnical Services QM program is intended to align with guidance provided in prominent regulatory and industry standards and programs, as appropriate and applicable, including the International Organization for Standardization, *Quality Management Systems – Requirements* (ISO 9001:2015). Table B-1 (Appendix B) compares this QMP with ISO 9001:2015.

In addition, requirements stated in the following documents are incorporated into projects and technical services provided by the GDR and GEI Practices, as applicable to the contractual scope of work:

- Client contracts and Master Services Agreements.
- Local or State professional engineering standards and regulations.
- State and local laws, codes, and/or specifications.
- FHWA and Department of Transportation codes and specifications.
- Client codes and specifications.
- Applicable ASTM and AASHTO standard specifications.
- ASTM, Standard Practice for Agencies Engaged in the Testing and/or Inspection of Soil and Rock as Used in Engineering Design and Construction (ASTM D 3740).
- AASHTO Materials Reference Laboratory (AMRL), Soils Laboratory Certification program based on AASHTO Standard Practice R18-10 (2015), conducted by National Institute for Standards and Technology (NIST).
- Cement and Concrete Reference Laboratory (CCRL) Certification program based on ASTM C 1077, conducted by National Institute for Standards and Technology (NIST).
- American Concrete Institute (ACI) Certification programs for concrete laboratory testing technicians and field sampling and testing technicians.
- United States Army Corps of Engineers (USACOE) Laboratory Testing Certification Program.
- State Environmental Departments' requirements for certification of drillers and requirements for performing test borings and installation of wells.
- New Jersey Department of Transportation (NJDOT) and Federal Aviation Administration (FAA) Soils Laboratory Certification Programs.
- Pennsylvania Department of Transportation (PennDot): Publication 222 - Subsurface Boring, Sampling and Testing.
- Pennsylvania Department of Transportation (PennDot): Publication 213 – *Work Zone Traffic Control Guidelines*.
- Foundation Q.A. Certification Program of Pile Dynamic Pile Tester.
- Federal Motor Carrier Safety Regulations, Title 49 Parts 4O, 382, 383, and 39O-399, Commercial Driver License (CDL) Class A or Class B.
- State Licenses, as applicable.
- Nuclear Regulatory Commission (NRC), 10 CFR 30 Licensing Requirements for Nuclear Devices.
- GDR Drilling Safety Manual**.**

3.2 Integrating Applicable Requirements into Projects

It is the responsibility of the Project Manager, as appropriate and applicable, to:

- Identify the requirements associated with the services to be provided.

- Confirm that the implemented procedures and controls are consistent with achievement of the requirements, as reasonable, necessary and appropriate.
- Modify, as needed, the procedures and controls to reflect changes made to the requirements.

The peer review process (detailed in Section 5.2 of this QMP) assists in evaluating whether appropriate procedures and controls are integrated into each project such that applicable requirements are met.

4.0 RISK ASSESSMENT – RISK MANAGEMENT

Risk-based thinking is used to focus TRC's quality program on practical and cost-effective controls (as opposed to "over engineering" or creating unnecessary paperwork) that are needed to prevent errors or other quality failures and to support Project Managers and technical leads in managing risk specific to each project.

Identify, evaluate and manage the risks related to successful performance of project assignments.

4.1 Process to Identify and Manage Risks on Proposal Opportunities

Prior to investing company resources into an opportunity for a major project (as defined in the *Project Cycle Manual / Project Manager's Guide to the Project Cycle*) costs and certain risks are evaluated using a Go/No-Go process. For such an analysis, the Project Manager scores a series of risk-based evaluation questions each on a scale of 1-10, identifies applicable risk factors, and evaluates the opportunity along with the assessment results. Results of the Go/No-Go decision process are recorded in Vision. More information on the Go/No-Go process is in the *Project Manager's Guide to the Project Cycle*..

Internal reviews of routine proposal opportunities focus on requirements and capabilities to confirm that TRC can fulfill contractual obligations associated with project performance. This includes, but is not limited to, comparison of TRC experience and capabilities to the specified scope of work, review of the required staff credentials (e.g., PE, PG) and evaluation of personnel and equipment resource availability.

4.2 Process to Identify and Manage Risks on Individual Projects

Based on the relative level of risk, an appropriate, reasonable and necessary level of oversight, analysis and review is applied to each project. In addition, the risk factors associated with a project can dictate what quality controls are applied to the project.

At the discretion of the Project Manager and commensurate with the complexity, size and scope of work, a risk management review may be performed during the proposal stage and again at the time of project initiation. When such an evaluation is performed, the following are example questions that may be considered.

1. Has the Project Manager previously managed a project of this category, size, and complexity?
2. Has project safety been evaluated and can we perform the project safely?
3. Has the Project Manager or TRC worked for this client previously?

4. Is the scope of work clearly defined, understood, and documented and has it been discussed with the client before beginning the work?
5. Is the budget estimate sufficient based on realistic expectations and past experience?
6. Are contract-specified billing rates acceptable?
7. Is the schedule well defined and sufficient?
8. Has the client clearly defined a point-of-contact for the project?
9. Is the client's representative experienced in dealing with similar projects and with consultants?
10. Will the project be assigned an adequate team and will that team continue with the project until completion?
11. Is the project staff experienced with the procedures and equipment required for this project?
12. Has the project team recently performed projects similar to this one?
13. Will work be performed by one Practice or multiple Practices and/or by personnel from one office or multiple offices, and is the communication/coordination process understood by the project team?
14. Are there subcontractors on the team and is the communication/coordination process understood by the project team?
15. Are there external factors that potentially add risk for TRC (e.g., legal, stakeholders)?
16. Are there other potential risks associated with this work (e.g., performance penalties impacting TRC for not meeting schedules)?

Based on the answers to these, the project may be considered a higher risk or a more complex/sensitive project. The Project Manager may consult with their Practice Leader and/or other senior personnel to assist in this evaluation and in designing approaches that may be used to manage any areas of elevated risk identified during the evaluation.

5.0 OPERATIONAL CONTROLS

Processes and procedures are in place to mitigate risk, consistently deliver contract-compliant work products, and communicate quality-related roles and responsibilities.

This section describes quality-related organizational roles and responsibilities and the baseline operational controls used to produce quality services and work products on projects.

Consistent with the risk and complexity of each project, these controls provide a framework for meeting client expectations regarding the schedule, budget, and in accordance with applicable requirements.

5.1 Organization and Responsibilities

TRC personnel and other resources are organized into operational business sectors or units and groups providing shared corporate services. The Sectors and Business Units are is organized into Practices (e.g.,

Geotechnical Engineering, Geotechnical Drilling Services) and employees are assigned to project teams. An overview of the company's organizational model is in Figure 5-1.

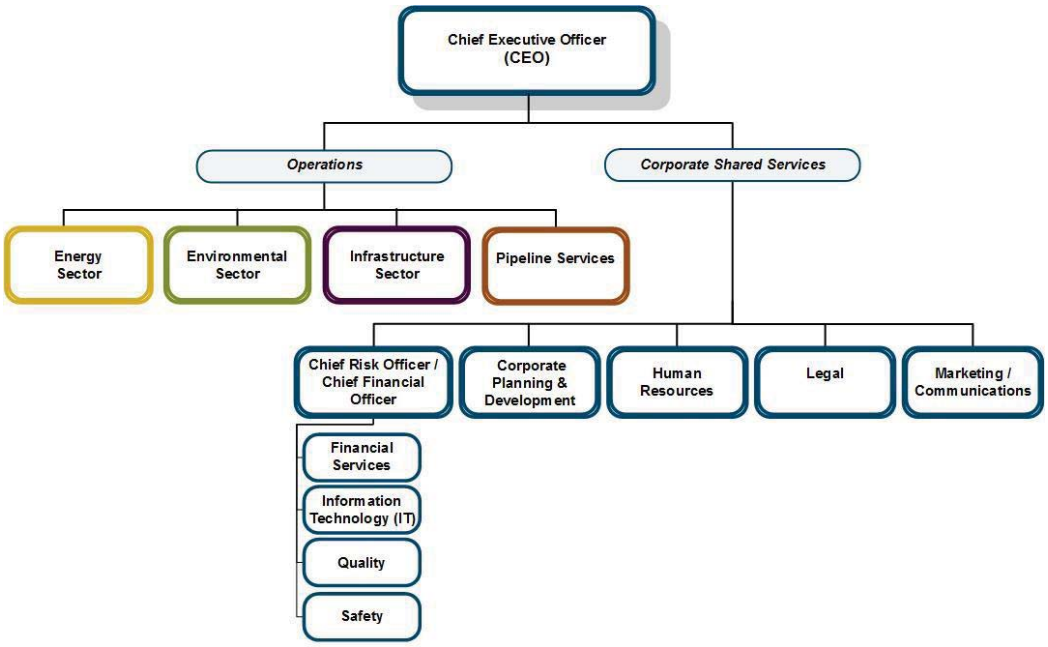


Figure 5-1. TRC Organizational Model

5.1.1 Corporate Organization and Responsibilities

With respect to quality, TRC's executive management is responsible for:

- Establishing quality management expectations and publishing a quality management policy.
- Appointing a National Quality Director.
- Establishing a Quality Council.
- Conducting quality management system reviews.
- Supporting Operational Sectors, Business Units, Practices and Corporate Services groups in developing tools which promote their quality programs.

TRC's corporate organization is designed to meet these policy requirements within a structure that promotes efficiency, cooperation, and accountability.

Chief Executive Officer

TRC's Chief Executive Officer (CEO) reports to the Board of Directors of TRC Companies, Inc. The CEO is ultimately responsible for overall company management, the direction of corporate affairs, and is accountable to the Board of Directors for corporate performance.

Chief Risk Officer/Chief Financial Officer

TRC's Chief Risk Officer/Chief Financial Officer (CRO/CFO) is accountable to the CEO and the Board of Directors and is responsible for overseeing programs and implementing practices with respect to risk management that directly impact the strategic direction of the Company. In addition to the Company's

accounting and financial services, information technology, and corporate safety functions, the corporate quality function reports to the CRO/CFO.

General Counsel and Ethics Officer

TRC's General Counsel also serves as TRC's Ethics Officer and as a resource to the CEO and the entire company in managing legal aspects of our business.

Quality Council

The TRC Quality Council is a collaborative forum to promote the communication of quality and continuous improvement across the entire company. The Quality Council includes representatives from each of the Operational Sectors and Business Units and select Corporate Shared Services groups. A written charter guides its operation with the chairperson role rotating between the Sector Directors and meeting minutes posted to TRCNet making them accessible to all employees. Responsibilities of the Quality Council include an annual review of the company's QM policy, sponsorship of annual goals for continuous improvement, and fostering dialog and sharing of best practices.

National Quality Director

TRC's National Quality Director reports to the CRO and is charged with coordinating the establishment of quality-related expectations, serving as a company resource on quality, and working with the Operational Sectors, Business Units, and Corporate Shared Services groups to:

- Assess operational quality and performance needs.
- Facilitate development and monitoring of annual quality goals and metrics.
- Lead or consult on continuous improvement initiatives that enhance TRC's ability to consistently produce quality services and work products.

Sector Directors

TRC's business model includes the following Sectors / Business Units led by Sector Directors who report to TRC's CEO and are charged with performance excellence and profitable growth. Their responsibilities include business performance oversight, strategic support and vision with respect to the various service offerings, business development, and general management support. This includes managing the fiscal resources for the sector, staff management, allocating resources to implement their respective QMPs, and communicating corporate information to the appropriate staff via their designated management team.

5.1.2 Infrastructure Sector Organization and Responsibilities

TRC's Infrastructure Sector (Figure 5-2) includes six Practices.

Infrastructure Sector Director

Responsibilities of the Infrastructure Sector Director, as described above, include confirming that adequate resources are available to implement Practice-specific QM programs.

Deputy Director

The Infrastructure Deputy Director works closely with the Sector Director on all matters related to the operations, management, and business performance of the Infrastructure Sector. This includes providing

the Sector, specifically Practice Leaders, with engineering, technical, business development, quality management, and operational direction and leadership.

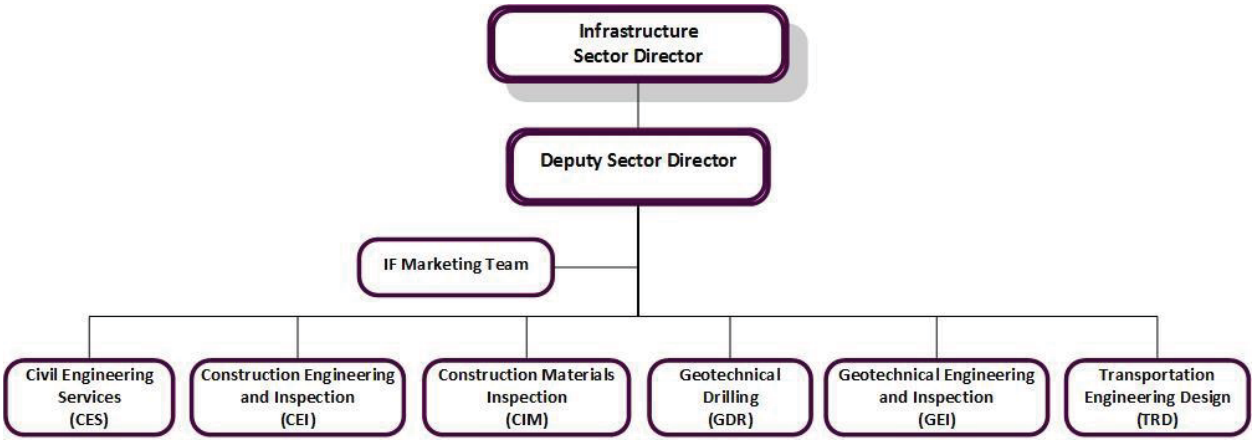


Figure 5-2. Infrastructure Sector Organizational Model

5.1.3 Geotechnical Practices Organization and Responsibilities

The following sections describe the principal quality-related roles and responsibilities of individuals within the geotechnical services (i.e., GDR and GEI) Practices.

Practice Leaders

The GDR and GEI Practice Leaders report to the Deputy Sector Director and are responsible for day-to-day operational management of the Practice and for providing performance oversight for projects conducted within the Practice (including technical, engineering, and financial performance). Quality program-related responsibilities include, but are not limited to the following:

- Appointing a Practice-specific quality leader.
- Providing clear communication to managers, and other key staff regarding their roles and responsibilities regarding the quality program and quality assurance (QA) and quality control (QC) procedures.
- Authorizing resources to implement the quality program across their Practice, as described in this QMP.

Practice Quality Leaders

The GEI/GDR Practice Quality Leaders report directly to their respective Practice Leader on all activities or issues related to the quality program, have a direct line of communication to TRC's National Quality Director and are responsible for providing leadership and support to the Practice-specific quality program, as described in this QMP.

The Practice Quality Leaders work closely with Project Managers, the Practice Leaders, Deputy Director, and other staff to confirm that quality assurance (QA) and quality control (QC) procedures within the Practice-specific program meet necessary guidance (i.e., client contracts, industry standards, construction regulations, as applicable) and anticipated client expectations. The Practice Quality Lead responsibilities include the following:

- Serving as a resource to GDR/GEI employees on quality-related issues and/or questions.
- Communicating and providing training on QMP features to GDR/GEI personnel.
- Promoting compliance with QMP requirements through leadership and guidance.
- Performing annual Practice-specific quality program self-assessments or quality audits focused on continuous improvement and an evaluation of the effectiveness of and compliance with this QMP and Practice-specific quality procedures.
- Participating in, or conducting corrective actions and follow-up, as necessary and appropriate.
- Providing Practice-wide communications to promote quality-related topics.
- Steering the process of developing annual quality goals and monitoring progress.
- Reporting to management on the status of the quality program and results of the annual quality program self-assessments and/or quality audits.
- Participating in biannual reviews and updates (as applicable) of the Geotechnical Services QMP.

5.1.4 Project Organization and Responsibilities

The Geotechnical Practices provide various services to a diverse group of private-sector and government clients. Some projects are interdisciplinary in nature and require contributions from persons with different yet complimentary skills and experience. In addition, ensuring that appropriate staff assignments are made on each project (i.e., accommodating all project-specific requirements) will generally require the services of various specialty services can also require assigning employees from multiple offices to a single project. TRC's organizational model provides for this flexibility. TRC employees may be committed to a single project full-time or may participate in several projects that require their skills simultaneously.

Project Managers

The Project Manager, in cooperation with and with support from their Practice Leaders and the Sector Director, has primary responsibility for a project within TRC. The Project Manager is key to leading the quality process. This responsibility may include:

- Upholding project quality through adequate planning and timely communication.
- Determination of project-specific personnel qualifications, expertise, and training requirements (including any client-specific safety training). Based on the project's needs, coordinating the selection of staff for each project assignment with the respective Office and Practice Leaders, as needed.
- Distribution of project instructions, as necessary, reasonable and appropriate to communicate logistical (e.g., budget, schedule) and technical (e.g., codes, standards, scope of work, SOPs) information to the project team. This may take the form of a detailed proposal, a compilation of

the project initiation spreadsheet, a detailed scope of work or work plan, a document written specifically as project instructions, or another format chosen by the Project Manager.

- Defining, documenting, and implementing applicable QA and QC elements or activities and confirming that necessary procedures are approved, issued and followed.
- Designating specific peer review roles, responsibilities, and schedules or frequencies.
- Reviewing contractual requirements (e.g., explicit scope of work, invoicing process) and developing tools to support the achievement of the requirements.
- Managing the technical and administrative/contractual aspects and schedule of the project, including client and regulatory agency communication, as required. Being available to the project team for action or direction on any issues requiring additional management or technical support.
- Monitoring and reviewing the project financial status in terms of budget management and work completed as compared to the objectives, requirements, and other contract specifications.
- Managing subcontractor services and confirming that the established scope of services is fulfilled.
- As appropriate, confirming that requirements for permits, insurance certificates, and any other work clearance or authorizations are understood and incorporated into the project schedule, and that compliance is achieved.
- Communicating project budget and invoicing information to TRC's Financial Services Group through the Vision system and submitting supporting documentation, as appropriate.
- Communicating with the client representative regarding material (i.e., significant) aspects of the project, including expenditures, progress, any problems and recommended solutions, and issues that require decisions on technical matters.
- Keeping appropriate TRC personnel informed on material matters related to the project.
- Providing the final review and approval of project deliverables or work product outputs prior to issue to verify adequate quality of the product or service.

Peer Reviewers

Responsibilities of peer reviews, as reasonable, necessary, and appropriate to the project, include:

- Working with the Project Manager to understand the client's expectations, needs, constraints, priorities, and TRC's contractual commitments.
- Being available to the Project Manager and other project team members to provide additional technical guidance or insight.
- Staying informed of changes in project direction resulting from evolving client needs and/or TRC constraints.
- Reviewing all interim and final work products.
- Pursuing all concerns to satisfactory resolution.

For commercial construction-related services, the respective Project Manager assigns one or more individuals the role of Peer Reviewer for the field-related activity, depending on the project scope and requirements. The GEI/GDR Quality Leader, or another qualified geotechnical engineer, will also act as the designated peer reviewer during the preparation of formal correspondence or other written documentation.

For drilling services (GDR), which in the majority of the cases are subcontracted services, the client's representative oversees the actual operations to ensure that the project and applicable quality requirements are met. Review of proposals and contracts is performed by the GDR Practice Leader. In the event that the drilling services are performed in conjunction with TRC geotechnical engineering services, a designated project engineer will act as an independent peer reviewer of the drilling activity.

Concrete and soil laboratory services follow the requirements of the relevant laboratory certification programs and the test results including those of proficiency sample testing are reviewed in accordance with the relevant certification requirements. Peer reviews of written procedures and personnel testing data are conducted in accordance with the relevant laboratory certification requirements. When laboratory testing is performed for geotechnical engineering or commercial construction services (GEI), the respective Project Manager (or appointed staff member) also will act as a peer reviewer.

Field, Laboratory, Office Technical, and Engineering Personnel

Field, laboratory and other technical personnel including engineering staff of the GDR/GEI Practices can be the single most important link to the implementation of a quality program. As the "first line of defense" in the collection and processing of project data, it is crucial that field, laboratory, engineering and other technical support staff know what they're doing, and have the proper tools and training to do it consistently correctly. Regardless of their staff level and project involvement, it is important that the field, laboratory, and office engineering and technical personnel are aware of their job responsibilities, the project or quality program requirements associated with each assignment, and have the right tools and training to do it right, all the time.

5.2 Peer Review

One of the cornerstones of TRC's quality program is the Peer Review process. Peer review (sometimes referred to as Project QA Review or QC Check) is strategically integrated into the overall QA process for proposals and projects. Each document and/or deliverable that provides technical information or a professional opinion on which the client will base geotechnical engineering or business decisions should undergo a peer review prior to its release/publication. The amount of peer review and the type of peer reviewer(s) engaged may vary depending on project/proposal circumstances. The benefits gained from effective peer review include the following:

- Increased value is provided to the client from expertise (e.g., knowledge, experience) of senior staff and technical area specialists effectively applied to each deliverable.
- Work products are checked (i.e., QC check) to confirm adequacy and technical merit.
- Errors in outgoing work products (e.g., documents, drawings, invoices) are reduced.

5.2.1 How to Integrate Peer Review into Activities

Peer review should be integrated into the overall project schedule. The types and frequency of peer review should be proportionate to the size, complexity and risk of each activity or task on the project. Throughout the effort, the Peer Reviewer serves as a resource of pertinent information and expertise, a sounding board for exploring alternatives, and an informed yet independent reviewer.

5.2.2 Selection of Peer Reviewers

The Project Manager is responsible for the selection of appropriate peer reviewers. A summary of the qualifications of Peer Reviewers includes:

- Capable of understanding the technical aspects of the project (or the aspects associated with assigned review responsibilities) and to comprehending the client's expectations.
- Independent of the work activity to be reviewed (i.e., the individual is not responsible for conducting the work, preparing the geotechnical report or attachments, supervising the task).
- Quality conscious, thorough, dependable, assertive, and constructive in raising and resolving potential issues.
- Certified or registered in the applicable subject area when required by regulations or industry standards (e.g., Registered Professional Engineer for engineering design projects), when applicable.

In the case of smaller projects, the Project Manager may assume the role of peer reviewer as long as the project reports and other deliverables are generated by employees other than the Project Manager. When multiple peer reviewers are designated, roles and responsibilities for each should be defined based on the project's scope of work and the qualifications of the reviewers.

5.2.3 Reviewing Interim and Final Work Products

The qualified peer reviewer is responsible for reviewing interim and final work products including proposals, reports, engineering design drawings and specifications, technical memoranda, or other deliverables produced in accordance with the contract and client directives. Practice-specific peer review procedures should be utilized, as applicable and appropriate. However, at a minimum, peer reviews should consider the following aspects of quality:

- Does the work product meet the project objectives and goals?
- Is the work product technically defensible?
- Were applicable codes, standards, and contractual requirements complied with?
- Was the work conducted in accordance with applicable local, state, and federal regulations, codes or standards?
- Was good professional judgment used in executing the work?
- Are conclusions and recommendations complete and supported by the work performed?
- Does the project meet the applicable TRC and GEI/GDR Practice technical standards and policies?

- Are written documents adequate in terms of general clarity, spelling, grammar, correct insertion of references to outside source materials, and accurate cross-references within the document?

5.2.4 Peer Review Comments and Records

It is important that all peer review comments be resolved. For review comments to be considered "resolved" it does not necessarily require that a change be made based on the review comments; however, every peer review comment is put forth and appropriately considered and/or discussed prior to determining the appropriate actions that will be taken. Project files should include records demonstrating the implementation of Peer Review. Refer to Practice-specific peer review procedures for specific requirements on peer review documentation.

5.3 Project Planning and Management

Integral to the success of TRC projects and internal operations is adequate activity design and planning. Project planning begins during the proposal and/or cost estimating phase of each project. Procedures that should be addressed in all design and planning efforts, such as project planning and management, include:

- Establishing and reviewing all objectives, requirements, and specifications.
- Determining the appropriate methods for translating the objectives, requirements, and specifications into an approach.
- Establishing project budgets that are sufficient to complete the specified scope of work and all corresponding quality checks, controls, or quality-related activities.
- A process to ensure that project team skills and expertise meet the project requirements.
- Establishing adequate provisions for scheduling and cost controls.
- Establishing provisions for reviews, verifications, and approvals.

Project management consists of technical and administrative activities to define and communicate the objectives, requirements, and specifications to the project team. Project Managers guide the team such that products or services provided by the geotechnical projects meet client expectations and technical, regulatory, or other requirements. Project Managers monitor work progress such that products and services are delivered within the established schedule. The *Project Cycle Manual / Project Manager's Guide to the Project Cycle* guides Project Managers through the process of managing project financial records. Typical project management activities were previously discussed in Section 5.1.4 of this QMP.

5.4 Controls for Purchasing Materials and Subcontracting Services

Recognizing that procured material and services need to conform to the applicable quality-related specifications or requirements, standard TRC procurement policies and procedures are followed. Procurement documentation is consistent with the process and procedures described in the *Purchase Order Training* slides found on TRCNet.

5.4.1 Evaluation of Subconsultants/Subcontractors

TRC's GDR/GEI Practice selects subconsultants (or subcontractors) on their ability to perform the services required and any specific quality control and other requirements related to their services. TRC uses subconsultants who have the necessary technical expertise required for the project, meet applicable

safety, quality, and financial standards, are willing to accept and implement The TRC project-specific Quality Control Plan for the Contract (when applicable), and requires a competitive fee for performance of the services.

5.4.2 Subconsultant/Subcontractor Management

It is the responsibility of the Project Manager to monitor the quality of services provided by any subconsultant or subcontractor and confirm that the services meet the project requirements. This may require seeking peer review in other offices or even outside sources as generally using a subconsultant signifies the services being provided are not within TRC's core competencies at that specific location or in general.

5.5 Recordkeeping and Document Control Procedures

5.5.1 Document Control Procedures

TRC quality program documents (including this QMP) are required to be quality assured and controlled. Document control procedures are used to guide the quality program documents and their subsequent revisions to be reviewed, approved, and distributed to appropriate individuals and organizations. Persons qualified to evaluate their accuracy and completeness are designated to perform the preparation, review, and approval of quality program documents.

Projects, even those that do not include formal document control requirements, should incorporate the following guidelines, as appropriate:

- Prior to distribution, a Peer Reviewer performs a review of work products that provide technical information or a professional opinion in which the client will base geotechnical or business decisions.
- Dates are printed on documents transmitted to clients and agencies.
- Vendor and client documents that may affect the quality of GDR/GEI work products and services (e.g., specifications, equipment plans and manuals, client instructions, drawings) are reviewed prior to release for use within the project team.
- Project filing systems are maintained by the GDR/GEI offices for the maintenance and retention of CAD drawings, reports prepared by computer word processing, and any other project deliverables.

5.5.2 Records Retention

The GDR/GEI Practices abide by TRC's *Records Storage Policy*, as it applies to project records and TRC quality program records.

5.6 Internal Communication

Maintaining open lines of communication across the project team and between TRC and the client is a key element of a successful project. The Project Manager takes a leadership role in initiating and encouraging communication throughout the project. Communication across the project team will include both verbal (Skype conference calls, face-to-face meetings, etc.) and written (e-mail, project work plan) methods, as appropriate.

5.7 Communication with the Client and Technical Interfaces

Project Managers (or designee) communicate with clients on matters which affect the cost, schedule, and quality of a project. TRC will keep the client representative informed on communications with subcontractors, other consultants, regulatory agencies, utility companies, etc., as appropriate. Where an issue with an outside organization might be sensitive or of a special nature, the Project Manager will discuss the issue with the client's representative before distributing written communications or making commitments on behalf of the client.

5.8 Project-Specific Quality Assurance Programs

Integrating the flexibility to design custom quality assurance (QA) programs (i.e., varying combinations of QA and QC elements) on projects is a feature of our quality program. By designing custom programs, the emphasis is on integrating adequate QC measures and QA features and avoiding excessive controls that can occur with a "one-size-fits-all approach. It is the responsibility of the Project Manager to determine and incorporate adequate and appropriate QA elements and QC procedures on each project.

In addition, the geotechnical soils and concrete testing laboratories have location-specific Laboratory Quality Systems Manuals (QSMs) that consist of the laboratory test procedures and related quality practices and procedures. The QSMs are consistent with AASHTO R 18-10 (2015), *Standard Recommended Practice for Establishing and Implementing a Quality Management System for Construction Materials Testing Laboratories*, American Association of State Highway and Transportation Officials.

5.9 Construction Support Services

The GEI/GDR Practice provides a variety of construction support services, primarily in the role of Geotechnical Engineering Inspectors. When the construction support services are limited to serving as the inspection agency, GEI Practice personnel are responsible only for QA, QC, and if required, third-party verifications. The client or their representative is responsible for construction management activities.

TRC's GEI/GDR Practice also provides construction-related geotechnical engineering services. The role of the engineer may include reviewing of the progress schedule and on-site inspection activities with respect to the contractual documents, plans, shop drawings, and specifications. In addition, the engineer or his representative may be asked to evaluate the contractor's suggested modifications to the plans and specifications and report proposed modifications to the design engineer for approval. The specific requirements for field engineering and the results are reviewed by the Project Manager, or by a peer reviewer.

5.10 Geotechnical Drilling Quality Procedures

Drilling, sampling and testing activities of the GDR practice are performed in accordance with procedures referenced in the contract, applicable ASTM or AASHTO standards, or applicable DOT standards. The Project Manager, upon review of applicable documents relating to a project, instructs the field staff as to the applicable standards for the drilling activities. Typically, the client's field representative provides field oversight and QA to assure that the work is performed in accordance with the applicable standards. Field personnel are responsible for maintaining the proper documentation, which upon completion of the

work is checked by a designated GDR peer reviewer. In the case of a combined GEI/GDR project, a designated GEI engineer acts as the peer reviewer.

5.11 Measurement and Test Equipment Controls

Area-specific procedures are developed to provide guidelines for measuring and test (e.g., field or laboratory) equipment related to selection of the appropriate equipment (e.g., the proper range, type and accuracy for the test being performed), controlling the equipment, calibration, and adjustments to be performed at stated frequencies to maintain accuracy within tolerance limits. The appropriate application of these procedures is determined on a project-specific basis and/or in accordance with AASHTO Standard Practice R 18-10, as it applies to soils and concrete laboratories.

The respective Project Manager should:

- Maintain an inventory of test equipment and assign a unique identifier to each piece of test equipment which will provide traceability between the equipment, calibration records, and measurement data generated by the piece of equipment.
- Maintain a logbook (or equivalent) that documents the history and status of test equipment (e.g., dates of the last and next calibration, radioactive leak tests, and repairs needed).

Test (field and laboratory) equipment calibrations are performed in accordance with SOPs and/or manufacturers' maintenance plans either by GEI/GDR Practice personnel or by an approved subcontractor, and are documented and traceable to appropriate standards or the manufacturer's recommendations.

Test equipment will be removed from service when its accuracy is suspect. If test equipment is found to be out of calibration repeatedly, it should be repaired or replaced and the impact on test results should be documented.

5.12 Tests, Measurements, and Analyses

TRC's quality program recognizes the importance of planning testing and analysis programs such that data of known and acceptable quality are produced. Testing will be performed according to written and approved test procedures based on recognized standards and methods (e.g., AASHTO, ASTM, ACI), as applicable. Standardized forms should be established for field and laboratory testing in accordance with applicable testing standards, as applicable. The qualifications of inspection, testing, or analysis personnel should be documented to show compliance with the requirements of applicable codes and standards. The test procedures should describe the required prerequisites, test instrumentation, methods to be employed, characteristics to be tested, applicable calibration and QC procedures and acceptance limits, required environmental conditions, and documentation and record keeping procedures. These procedures will be approved by the client, if required. TRC senior technical personnel should oversee the performance of all testing programs and evaluate test results. Subcontracted testing laboratories, if required, should be perform testing in accordance with project requirements.

5.13 Field and Laboratory Data Acquisition, Review, and Validation Procedures

Data acquisition, review and validation, and any analysis procedures, if required, are developed at the beginning of the specific project (as applicable to the scope of work) and the process is documented in the

project, field or laboratory QA Plan, work plan, or other detailed scope of work documents. For field investigation or construction monitoring projects, TRC may specify data quality objectives to evaluate through the course of the project. TRC soils and concrete testing laboratories participate in a proficiency sampling program, as appropriate and applicable to the relevant certification programs.

5.14 Identification and Traceability

At the start of each project, the Project Manager identifies the project requirements regarding identification and traceability and establishes the appropriate procedures. Examples of project requirements for identification and traceability include:

- Sample identification and traceability requirements for soil, rock or concrete obtained in the field (i.e., at the project site).
- Naming and revision tracking of engineering drawings and reports and supporting calculations or data modeling.
- Laboratory measurement data traceability including unique identification of how and when the data were generated, procedures to ensure data integrity during data processing and reporting, and naming conventions used to report the data.

5.15 Custody, Handling, Storage, and Shipping of Samples

TRC's GEI/GDR Practices understand and implement chain-of-custody procedures for soil, rock, or concrete samples, when applicable to the project. Handling and storage of the samples should adhere to applicable standards. Disposal of samples should be in accordance with contractual requirements. Authorization for sample disposition commonly made by the Project Manager.

Project-specific quality procedures should specify the appropriate record keeping requirements and handling, storage, shipping, and maintenance methods. Procedures appropriate to safeguard sample integrity and to minimize deterioration of equipment and materials should be established for each applicable project. Nuclear density gauge equipment should be stored and transported in strict compliance with NRC or agreement and State regulatory requirements.

5.16 Software Control

TRC maintains a corporate software policy that specifies the use of licensed copies only of commercial software. TRC's IT Policy is on TRCNet.

5.17 Corrective and Preventive Actions

TRC's quality management program encourages prevention of items, services, or activities that do not conform to specified quality requirements. However, when instances of non-conformance occur, prompt and effective corrective actions to remedy the situation are essential.

When a nonconformance (e.g., error or omission) has been identified, the goal is to correct the problem and prevent future occurrences whenever practical. Remedies can include, but are not limited to, repeating or reworking the item, and using the nonconforming item or work product when it can be determined and documented that the error/non-conformance will result in no adverse effect on the quality of the project.

With client satisfaction a primary goal of this quality program, TRC strives to conduct an immediate investigation to determine the nature of client concerns about work-product quality. Commonly, the issue turns out to be that of miscommunication or an unintentional misunderstanding of the project scope of work and objectives. Resolution typically involves further discussion with the client to gain a full understanding of their perceptions and expectations. Corrective action begins promptly for each deficiency.

5.18 Workplace Safety

TRC has an *Environmental Health and Safety Policy*. A copy of this policy is made available to all TRC employees on the TRCNet in the document titled: *TRC Health and Safety Management System*. Implementation of this policy demonstrates that the company, including the GDR/GEI Practices, is committed to providing a safe and healthy workplace for its employees and is committed to adherence to all client-specified health and safety requirements. Safety Coordinators oversee TRC's Health and Safety Program under the direction of TRC's National Safety Director.

5.19 Review and Update of Operational Controls

As part of the self-assessment process, the GEI/GDR Quality Leaders review the operational controls with respect to how effective current practice is in managing the quality of products and services, accuracy of the QMP descriptions, and how complete the QMP is in identifying the key operational controls. Changes may also be identified by any employee who then communicates the need with their Practice Quality Leader. As appropriate, revisions to documentation or to standard procedures are initiated by the GEI/GDR Practice Quality Leaders following concurrence by the GEI/GDR Practice Leaders and/or TRC's National Quality Director (if the change involves significant revision to the QMP).

6.0 TRAINING AND QUALIFICATIONS

Maintaining a staff of qualified individuals is vital to TRC's ability to consistently provide services that meet our client's needs and requirements. Therefore, the goals of our personnel training and qualifications process include the following:

Employees should have an awareness of how their activities, compentence, training and experience can directly impact the quality of deliverables and services provided to TRC clients.

- Standardizing procedures to assist with exercising appropriate care in the recruitment, hiring, and initial orientation of personnel.
- An expectation that Project Managers will identify staff assignments that require specific training or certification (or equivalent) to demonstrate qualifications and will confirm that these requirements are met, when applicable.
- Documentation of completed training sessions and/or staff qualifications, as necessary.

6.1 Employee Training Requirements

TRC personnel participate in company-sponsored quality assurance training (e.g., general awareness training on their sector-specific QMP or other quality program procedures) and business ethics and integrity training (e.g., based on the policy document titled: *TRC Corporate Code of Business Conduct*

and Ethics). A large portion of staff training is accomplished through informal, on-the-job training and mentoring by more experienced co-workers. As determined by the Practice Leaders and based on requirements associated with individual job assignments, employees may also be given opportunities to receive safety (e.g., safe driving guidelines, laboratory safety) and/or professional subject matter (e.g., project management, NICET, ACI). Laboratory staff competency evaluations, as applicable, are performed to meet the requirements of AASHTO Standard Practice R 18-10.

6.2 Training Records

Records of personnel educational qualifications and professional certifications are maintained in the corporate human resources files. Employee in-house training records can be maintained within the TRC Academy system or by the employee's own personnel records. Any training records subject to AASHTO Standard Practice R 18-10 should be contained within the appropriate laboratory QSM manual.

7.0 SELF-ASSESSMENT

Self-assessements are focused on continuous improvement and evaluating the suitability and effectiveness of the quality program.

TRC is committed to operating within a culture of continuous improvement. A quality assessment and audit program is a fundamental tool used to identify, evaluate, and prioritize opportunities for improvement to services provided to our clients and improvements to internal processes.

Self-assessments (and subsequent continuous improvement plans) are used to:

- Identify the strengths and weaknesses of a program, process, or specific work area.
- Encourage the dissemination of strengthening activities or procedures into other areas of the Practice or company.
- Implement corrective actions to minimize weaknesses, areas with notable risk of quality problems or deficiencies in the services provided.
- Evaluate compliance with and the effectiveness of the quality management program.
- Identify potential continuous improvement initiatives.

Annual self-assessments should be conducted by and within each Practice to evaluate compliance with its quality program documents (including its QMP) and the effectiveness of the quality program. The GEI Practice soils and concrete laboratories subject to AASHTO Standard Practice R 18-10 are internally audited in accordance with the requirements of AASHTO Standard Practice R 18-10. Results and observations of the self-assessment should be compiled into a report by the Practice Quality Leader, discussed with the Practice Leader(s), and submitted to the National Quality Director (or designee) for inclusion in company-wide quality management reviews.

8.0 MANAGEMENT REVIEW

8.1 Management Review of the TRC Quality Management System

TRC's National Quality Director (or designee) conducts reviews of the company's QM system to evaluate program effectiveness and verify compliance with the TRC QM Policy. The following general four-step process will be used to guide management reviews.

TRC's Management Team is actively engaged and committed to fostering performance excellence.

1. Review TRC QM Policy and quality program objectives.
2. Compile and review available quality program-related self-assessment and performance data.
3. Evaluate the information gathered in steps 1 and 2.
4. Prepare a summary report suitable for submittal to the TRC Executive Management Team.

8.2 Revisions to the TRC Quality Management System and Policy

Based on the results of the management review process drive revisions to the TRC's QM Policy and/or quality management system, as appropriate.

9.0 CONTINUOUS IMPROVEMENT

TRC's continuous improvement culture is focused on striving to enhance customer satisfaction through performance excellence.

TRC strives to provide excellence to its clients; thus, continually improving the processes, procedures, and capabilities of each organization and project team member is an underlying company theme. The following activities provide awareness of opportunities for improvement:

- TRC employees are encouraged to watch for continuous improvement opportunities while performing their routine job assignments.
- Practice Quality Leaders use the results of their quality program self-assessments to recommend areas for improvement.
- When Sector and Practice management teams develop performance improvement objectives, the corresponding implementation plan identifies potential operational changes.
- Feedback received from outside organizations (e.g., clients, third-party accreditation programs, regulatory agencies) provide external perspectives on TRC performance and their expectations.
- The annual management review performed by the National Quality Director evaluates the effectiveness of the quality management system.

After evaluating each nominated opportunity for improvement, those deemed to provide sufficient value or return on the investment proceed to an implementation plan.

APPENDIX A: TRC QUALITY MANAGEMENT POLICY

TRC
Results you can rely on

Quality Management Policy

At TRC we strive for excellence in the services we provide and in the solutions we produce for our clients. We are committed to meeting or exceeding our client expectations and delivering superior solutions. Key tenets of our Quality Management Policy include the following:

- We understand our clients' goals and embrace them as our own.
- We actively seek and respond to our clients' feedback regarding the quality of our services
- Our leadership champions and participates in quality activities and reviews.
- Quality performance goals and objectives are periodically reviewed for effectiveness.
- A tiered quality organization is dedicated to the attainment of quality objectives across all levels of TRC's organization.
- A culture of performance excellence is fostered through the development of staff competencies, empowerment, and personal accountability for quality workmanship.
- Every employee is responsible for compliance with quality requirements in each work activity.
- Project teams are equipped with sufficient skill sets, training, resources, and job tools, as appropriate for each project.
- Risks to quality of services are assessed and managed as appropriate for each project.
- Self-assessments, peer reviews, independent internal audits, and external audits are conducted in accordance with the complexity and risk of each activity.
- Contractors, suppliers, and partners are accountable for compliance with TRC's quality requirements, policy, and procedures.
- Quality performance is regularly monitored and corrective actions are managed to prevent adverse operational impacts and recurrence.
- Employees at all levels in the organization participate in a program of continual improvement.

Authorization		Rev. 8
Approval X Thomas W Bennet Jr Digitally signed by Thomas W Bennet Jr DN: cn=Thomas W Bennet Jr, o=TRC Companies Inc, ou=ZEX, email=tbennet@trcsolutions.com, c=US Date: 2016.05.09 13:25:28 -04'00' Thomas W. Bennet, Jr. SVP, Chief Risk Officer	***Content Management*** X Speth, Amelia Digitally signed by Speth, Amelia DN: dc=local, dc=ROOT, dc=EMPLOYEES, ou=User Accounts, ou=West Palm Beach, cn=Speth, Amelia, email=ASpeth@trcsolutions.com Date: 2016.05.24 14:33:37 -04'00' Amelia Speth National Quality Director	***Effective Date*** May 9, 2016 ***Review and Reissue Date*** May 8, 2018

APPENDIX B

COMPARISON OF PUBLISHED QUALITY STANDARDS AND THE GEOTECHNICAL SERVICES QUALITY MANAGEMENT PLAN

Table B-1. Comparison of ISO 9001:2015 and the Geotechnical Services QMP[1]

ISO 9001:2015		QMP, Revision 02	
Context of the Organization			
Understanding the Organization and its Context	4.1	1.0 1.1 5.1	Quality Management Policy Applicability and Communication of the Quality Management Program Organization and Responsibilities
Needs and Expectations of Interested Parties	4.2	3.0 3.1	Compliance Requirements Identification of Applicable Requirements
Scope of Quality Management System	4.3	1.1	Applicability and Communication of the Quality Management Program
Quality Management System Processes	4.4	4.0 5.0 7.0 8.0	Risk Assessment – Risk Management Operational Controls Self-Assessment Management Review
Leadership			
Leadership and Commitment	5.1	1.0	Quality Management Policy
Policy	5.2	1.0 App. A	Quality Management Policy TRC Quality Management Policy
Organization, Roles, Responsibilities, and Authorities	5.3	5.1	Organization and Responsibilities
Planning			
Actions to Address Risks and Opportunities	6.1	4.0 4.1 4.2	Risk Assessment – Risk Management Process to Identify and Manage Risks on Proposal Opportunities Process to Identify and Manage Risks on Individual Projects
Quality Objectives and Planning to Achieve Them	6.2	2.0 5.8	Operational and Quality Objectives Project-Specific Quality Assurance Programs
Planning of Changes	6.3	5.19	Review and Update of Operational Controls
Support			
Resources	7.1	5.1.1 5.1.2 5.1.3 5.1.4 5.2.1 5.2.2 5.3 5.14	Corporate Organization and Responsibilities Infrastructure Sector Organization and Responsibilities Geotechnical Practices Organization and Responsibilities Project Organization and Responsibilities How to Integrate Peer Review into Activities Selection of Peer Reviewers Project Planning and Management Identification and Traceability
Competence	7.2	5.1.4 5.2.2 5.3 6.0 6.1 6.2	Project Organization and Responsibilities – Project Managers Selection of Peer Reviewers Project Planning and Management Training and Qualifications Employee Training Requirements Training Records

ISO 9001:2015		QMP, Revision 02	
Awareness	7.3	5.3 5.8 6.1	Project Planning and Management Project-Specific Quality Assurance Programs Employee Training Requirements – Quality
Communication	7.4	5.6 5.7	Internal Communication Communication with Client and Technical Interfaces
Documented Information	7.5	5.2.4 5.5	Peer Review Comments and Records Recordkeeping and Document Control Procedures
Operation			
Operational Planning and Control	8.1	5.1.2 5.3 5.12	Infrastructure Sector Organization and Responsibilities Geotechnical Practices Organization and Responsibilities Project Planning and Management
Requirements for Products and Services	8.2	5.1.4 5.3 5.7	Project Organization and Responsibilities – Project Managers Project Planning and Management Communication with the Client and Technical Interfaces
Design and Development of Products and Services	8.3	5.1.4 5.2 5.3	Project Organization and Responsibilities – Project Manager Peer Review Project Planning and Management
Control of Externally Provided Processes, Products and Services	8.4	5.4	Controls for Purchasing Materials and Subcontracting Services
Production and Service Provision	8.5	5.3 5.8	Project Planning and Management Project-Specific Quality Assurance Plans
Release of Products and Services	8.6	5.1.4 5.2.3	Project Organization and Responsibilities – Project Managers Reviewing Interim and Final Work Products
Control of Nonconforming Outputs	8.7	5.17	Corrective and Preventive Actions
Performance Evaluation			
Monitoring, Measurement, Analysis, and Evaluation	9.1	5.2 5.13 5.19 7.0	Peer Review Field and Laboratory Data Acquisition, Review, and Validation Procedures Review and Update of Operational Controls Self-Assessment
Internal Audit	9.2	7.0	Self-Assessment
Management Review	9.3	7.0 8.0 8.2	Self-Assessment Management Review Revisions to TRC Quality Management System and Policy
Improvement			
General	10.1	9.0	Continuous Improvement
Nonconformity and Corrective Action	10.2	5.17	Corrective and Preventive Actions
Continual Improvement	10.3	9.0	Continuous Improvement

[1] International Organization for Standardization, ISO 9001:2015, *Quality Management Systems – Requirements*, ISO 9001, fifth edition, 2015-09-15.

TRC

AASHTO ACCREDITED

CERTIFICATE OF ACCREDITATION

AMERICAN ASSOCIATION OF STATE HIGHWAY AND TRANSPORTATION OFFICIALS
AASHTO

TRC Engineers, Inc.

in

Mount Laurel, New Jersey, USA

has demonstrated proficiency for the testing of construction materials and has conformed to the requirements established in AASHTO R 18 and the AASHTO Accreditation policies established by the AASHTO Committee on Materials and Pavements.

The scope of accreditation can be viewed on the Directory of AASHTO Accredited Laboratories (aashtoresource.org).

Bud Wright,
AASHTO Executive Director

Moe Jamshidi,
AASHTO COMP Chair

This certificate was generated on 07/27/2018 at 9:39 AM Eastern Time. Please confirm the current accreditation status of this laboratory at aashtoresource.org/aap/accreditation-directory

Scope of AASHTO Accreditation for:

TRC Engineers, Inc.

in Mount Laurel, New Jersey, USA

Quality Management System

Standard:	Accredited Since:
R18 Establishing and Implementing a Quality System for Construction Materials Testing Laboratories	07/29/2009

This certificate was generated on 07/27/2018 at 9:39 AM Eastern Time. Please confirm the current accreditation status of this laboratory at aashtoresource.org/aap/accreditation-directory

Scope of AASHTO Accreditation for:

TRC Engineers, Inc.

in Mount Laurel, New Jersey, USA

Soil

Standard:		Accredited Since:
R58	Dry Preparation of Disturbed Soil and Soil Aggregate Samples for Test	07/29/2009
T88	Particle Size Analysis of Soils by Hydrometer	07/29/2009
T89	Determining the Liquid Limit of Soils (Atterberg Limits)	07/29/2009
T90	Plastic Limit of Soils (Atterberg Limits)	07/29/2009
T99	The Moisture-Density Relations of Soils Using a 5.5 lb [2.5 kg] Rammer and a 12 in. [305 mm] Drop	07/29/2009
T100	Specific Gravity of Soils	07/29/2009
T180	Moisture-Density Relations of Soils Using a 10 lb [4.54 kg] Rammer and an 18 in. [457 mm] Drop	07/29/2009
T193	The California Bearing Ratio	07/29/2009
T208	Unconfined Compressive Strength of Cohesive Soil	07/29/2009
T216	One-Dimensional Consolidation Properties of Soils Using Incremental Loading	07/29/2009
T236	Direct Shear Test of Soils Under Consolidated Drained Conditions	07/29/2009
T265	Laboratory Determination of Moisture Content of Soils	07/29/2009
T267	Determination of Organic Content in Soils by Loss on Ignition	10/07/2015
T296	Unconsolidated, Undrained Compressive Strength of Cohesive Soils in Triaxial Compression	07/29/2009
T297	Consolidated-Undrained Triaxial Compression Test on Cohesive Soils	07/29/2009
D421	Dry Preparation of Disturbed Soil and Soil Aggregate Samples for Test	07/29/2009
D422	Particle Size Analysis of Soils by Hydrometer	07/29/2009
D698	The Moisture-Density Relations of Soils Using a 5.5 lb [2.5 kg] Rammer and a 12 in. [305 mm] Drop	07/29/2009
D854	Specific Gravity of Soils	07/29/2009
D1140	Amount of Material in Soils Finer than the No. 200 (75-µm) Sieve	02/14/2018
D1557	Moisture-Density Relations of Soils Using a 10 lb [4.54 kg] Rammer and an 18 in. [457 mm] Drop	07/29/2009
D1883	The California Bearing Ratio	07/29/2009
D2166	Unconfined Compressive Strength of Cohesive Soil	07/29/2009

This certificate was generated on 07/27/2018 at 9:39 AM Eastern Time. Please confirm the current accreditation status of this laboratory at aashtoresource.org/aap/accreditation-directory

Scope of AASHTO Accreditation for:

TRC Engineers, Inc.

in Mount Laurel, New Jersey, USA

Soil (Continued)

Standard:	Accredited Since:
D2216 Laboratory Determination of Moisture Content of Soils	07/29/2009
D2435 One-Dimensional Consolidation Properties of Soils Using Incremental Loading	07/29/2009
D2850 Unconsolidated, Undrained Compressive Strength of Cohesive Soils in Triaxial Compression	07/29/2009
D2974 Determination of Organic Content in Soils by Loss on Ignition	10/07/2015
D3080 Direct Shear Test of Soils Under Consolidated Drained Conditions	07/29/2009
D4318 Determining the Liquid Limit of Soils (Atterberg Limits)	07/29/2009
D4318 Plastic Limit of Soils (Atterberg Limits)	07/29/2009
D4543 Preparing Rock Core as Cylindrical Test Specimens and Verifying Conformance to Dimensional and Shape Tolerances	02/14/2018
D4644 Slake Durability of Shales and Weak Rocks	10/07/2015
D4767 Consolidated-Undrained Triaxial Compression Test on Cohesive Soils	07/29/2009
D5084 Hydraulic Conductivity of Saturated Porous Materials Using a Flexible Wall Permeameter	07/29/2009
D5731 Point Load Strength Index of Rock	10/07/2015
D6913 Particle-Size Distribution (Gradation) of Soils Using Sieve Analysis	02/14/2018
D7012 Compressive Strength of Rock Core Specimens (Method C)	02/14/2018
D7928 Particle-Size Distribution (Gradation) of Fine-Grained Soils Using the Sedimentation (Hydrometer) Analysis	02/14/2018

This certificate was generated on 07/27/2018 at 9:39 AM Eastern Time. Please confirm the current accreditation status of this laboratory at aashtoresource.org/aap/accreditation-directory

ATTACHMENT H

Acronyms and Abbreviations

List of Acronyms and Abbreviations Used in Tables, Figures and Attachments
Delaware Sand & Gravel Superfund Site
New Castle County, Delaware

Acronym/Abbreviation	Definition
% CH_4	Percent Methane
% LEL	Percent Lower Explosive Limit
% R	Percent Recovery
% O_2	Percent Oxygen
° C	degrees Celsius
ACL	Army Creek Landfill
AoA	Area of Attainment
ASAP	as soon as possible
AWC	Artesian Water Company
BCEE	bis(2-chloroethyl)ether
BOD	Biochemical Oxygen Demand
CO_2	Carbon Dioxide
CO	Carbon Monoxide
CRDL	Contract Required Detection Limit
CRQL	Contract Required Quantification Limit
CVAA	Cold Vapor Atomic Absorption
d-Fe/Mn	Dissolved Iron/Manganese
DDA	Drum Disposal Area
DI	De-ionized
DS&G	Delaware Sand and Gravel
ELFExS	Enhanced Low-Flow Extraction System
Fe	Iron
FSWP	Feasibility Study Work Plan
ft	feet
ft-bgs	feet below ground surface
ft-bmp	feet below measuring point
ft-btoc	feet below top of casing
gal	gallons
gal/ft	gallons per foot
GC/MS	Gas Chromatograph/Mass Spectrometer
GMW	Gas Monitoring Well
gpm	gallons per minute
H_2S	CO_2
HAL	Health Advisory Level
HASP	Health and Safety Plan
HPDE	High Density Polyethylene
ICP MS	Inductively Coupled Plasma Mass Spectroscopy
IDL	Instrument Detection Limit
in	inch
LCS	Laboratory Control Sample
LDPE	Low-Density Polyethylene
LFExS	Low-Flow Extraction System
LFG	Landfill Gas

List of Acronyms and Abbreviations Used in Tables, Figures and Attachments
Delaware Sand & Gravel Superfund Site
New Castle County, Delaware

Acronym/Abbreviation	Definition
LEL	Lower Explosive Limit
ll	low level
LS	Lower Sand
MCAWW	Methods for Chemical Analysis of Water and Wastes
MCL	Maximum Contaminant Limit
MDL	Method Detection Limit
min	minute
mg/l	milligram per liter
ml	milliliter
ml/min	milliliter per minute
Mn	manganese
MPCU	Middle Potomac Confining Unit
MS	Matrix Spike
mS/cm	millisiemens per centimeter
MSD	Matrix Spike Duplicate
MSL	Mean Sea Level
mV	Millivolt
MW	Monitoring Well
NA	Not Applicable
NCC	New Castle County
ng/l	nanograms per liter
NR	No Record
NTU	Nephelometric Turbidity Unit
PCBs	Polychlorinated Biphenyls
PDIWP	Pre-Design Investigation Work Plan
PFAs	Per-and poly-fluoroalkyl substances
PID	Photo-ionization Detector
ppm	parts per million
PVC	Polyvinyl Chloride
PZ	Piezometer
QA	Quality Assurance
QA/QC	Quality Assurance / Quality Control
QC	Quality Control
QL	Quantitation Limit
RCRA	Resource Conservation and Recovery Act
RPD	Relative Percent Difference
RSLs	Regional Screening Levels
SAP	Sampling and Analysis Plan
SIM	Selected Ion Monitoring
S/m	Siemens per meter
SMCL	Secondary Maximum Contaminant Limit
SOP	Standard Operating Procedures
ss-PRGs	Site-Specific Preliminary Remediation Goals

List of Acronyms and Abbreviations Used in Tables, Figures and Attachments
Delaware Sand & Gravel Superfund Site
New Castle County, Delaware

Acronym/Abbreviation	Definition
std	Standard Unit (pH)
S.U.	Standard Unit (pH)
SVOCs	Semivolatile Organic Compounds
TAL	Target Analyte List
TCL	Target Compound List
TICs	Tentatively Identified Compounds
TSS	Total Suspended Solids
TTO	Total Toxic Organics
TZ	Transition Zone
TZExS	Transition Zone Extraction System
ug/l	Microgram Per Liter
UPA	Upper Potomac Aquifer
UPCU	Upper Potomac Confining Unit
UPCUTZ	Upper Potomac Confining Unit Transition Zone
UPDC	Upper Potomac Dividing Clay
US	Upper Sand
USEPA	United States Environmental Protection Agency
VAP	Vertical Aquifer Profiling
VOCs	Volatile Organic Compounds
WMA	Waste Management Area

ATTACHMENT I

Standard Operating Procedures for Drilling and Groundwater Sampling

TP-1.2-12
Rev. 7

Record of Revision

Section	Description
All	Editorial changes throughout
7.2	Added electric water level measuring tape and well development instrumentation to field equipment list
8.1	1st bullet - included initiating fees 4th bullet - added instruction to obtain a copy of all well reports submitted by the contractor for the project file
8.2.1	3rd bullet - included statement excluding riveted, screwed or glued joints from use
8.2.2	Added more detail for use of centralizers
8.2.4	1st bullet - added statement that specific spacing requirements may be required by state regulations 3rd bullet - added statement to include that end sections of casing may also be designed to screw together 5th bullet, new - includes cleaning requirements
8.3.7	Added provisions for approval from the client/landowner as to the location and storage of drums
8.3.8	Added statement requiring decontamination of geophysical logging equipment
8.4.2	Added statement defining best practice regarding cement and cement grout in a borehole
8.4.5	Added consideration of state regulations in regard to placement of filter packs
8.4.8	Added consideration of state regulations for monitoring well labeling requirements
8.6	Added provisions for repair of torn asphalt during site cleanup
8.7 and Exhibit C	Changed Procedure Alteration Checklist to Field Change Request
8.8	Added permanent before landmarks

TABLE OF CONTENTS

Page No.

1. INTRODUCTION 1
1.1 Purpose 1
2. APPLICABILITY 1
3. DEFINITIONS 1
3.1 Monitoring Wells 1
3.2 Bentonite 1
3.3 Drive Casing 1
3.4 Grout 1
3.5 Well Screen 1
3.6 Monitoring Interval 1
5. DISCUSSION 2
6. RESPONSIBILITIES 2
6.1 Hydrogeologist/Field Engineer 2
6.2 Task Leader 2
6.3 Project Manager 3
7. EQUIPMENT 3
7.1 Drilling Equipment 3
7.2 Field Engineering Equipment and Logs 3
7.3 Health and Safety Equipment 4
8. PROCEDURES 5
8.1 Regulatory Considerations 5
8.2 Materials 5
8.2.1 Well Screen 5
8.2.2 Centralizers (Optional) 6
8.2.3 Riser Pipe 6
8.2.4 Steel Casing 6
8.2.5 Filter pack 7
8.2.6 Cement/Bentonite Grout 7
8.2.7 Bentonite Grout 7
8.2.8 Volclay 8
8.2.9 Pure Gold 8
8.2.10 Bentonite Pellets/Chips 8
8.2.11 Concrete 8
8.3 Well Borehole Drilling 8
8.3.1 General Precautions 8

TABLE OF CONTENTS (Cont.)

Page No.

8.3.2 Documentation 9
8.3.3 Initial Equipment Evaluation and Inspection 9
8.3.4 Decontamination of the Drill Rig and Drill Equipment 10
8.3.5 Setting up the Drill Rig 10
8.3.6 Borehole Size and Drilling Methods 10
8.3.7 Capture of Drill Cuttings and Groundwater 11
8.3.8 Verifying the Completed Borehole 11
8.4 Well Installation 11
8.4.1 General Requirements 11
8.4.2 Evaluation of the Borehole 12
8.4.3 Assembly of Well Screen and Riser 12
8.4.4 Setting the Well Screen and Riser String 12
8.4.5 Placement of the Filter Pack 12
8.4.6 Placement of the Bentonite Seal 13
8.4.7 Grouting the Annular Seal 13
8.4.8 Placement of Protective Well Monument, Concrete Pad/Seal 13
8.5 Well Development and Acceptance 14
8.5.1 Pumps and Accessories for Well Development 14
8.5.1.1 Submersible Pumps 14
8.5.1.2 Bladder Pumps 14
8.5.1.3 Jet Pumps 14
8.5.1.4 Bailers 15
8.5.1.5 Compressed Air 15
8.5.1.6 Bottled Nitrogen 15
8.5.2 Well Development 15
8.5.3 Well Recovery Test 16
8.5.4 Well Acceptance 16
8.6 Site Cleanup 16
8.7 Field Change Request 16
8.8 Well Location 17

LIST OF FIGURES

8-1 Typical Monitoring Well Installation
8-2 Protective Monument Details

1. INTRODUCTION

1.1 Purpose

This document establishes uniform procedures for the selection of materials and installation of monitoring wells in unconsolidated and consolidated deposits.

2. APPLICABILITY

This technical procedure is applicable to all Golder Associates Inc. (Golder) personnel involved with the design and installation of groundwater monitoring wells.

3. DEFINITIONS

3.1 Monitoring Wells

A monitoring well is a well completed within a specific zone of interest, of sufficient diameter and construction to allow sampling and/or pump testing.

3.2 Bentonite

Bentonite is an expanding sodium bentonite clay used to seal the annular space between the production casing and the wall of the borehole. Calcium bentonite may be more appropriate in calcium-rich environments due to reduced cation exchange potential.

3.3 Drive Casing

Drive casing is a pipe used to line a borehole to prohibit caving and/or prevent direct flow from the formation into the borehole. Hollow stem augers may be considered as analogous in function to drive casing for the purposes of this Technical Procedure.

3.4 Grout

Grout is a cement and/or bentonite mixture, originally fluid enough to be pumped through tremie pipes, used to seal casing within a borehole. Specifications are given in Section 8.2 below.

3.5 Well Screen

A well screen is a manufactured wire-wrapped or slotted pipe which allows the flow of water from the formation into the well.

3.6 Monitoring Interval

The monitoring interval is the only zone in which groundwater can enter the well.

4. REFERENCES

4.1 ASTM D 1785 - Threaded, Flush Couplings on PVC Pipe.

4.2 Golder Associates Inc. Technical Procedure TP-1.2-1, "Rock Core Drilling and Sampling."

4.3 Golder Associates Inc. Technical Procedure TP-1.2-5, "Drilling, Sampling, and Logging of Soils."

4.4 Golder Associates Inc. Technical Procedure TP-1.4.6, "Water Level Measurement."

4.6 Federal Regulations and Guidance

- CFR 264/265 Subpart F
Resource Conservation and Recovery Act (RCRA) Ground-Water Monitoring

- Comprehensive Environmental Response, Compensation and Liability Act (CERCLA)

- Technical Enforcement Guidance Document (OSWER-9950.1)

5. DISCUSSION

General discussions are included in Section 8.0 below.

6. RESPONSIBILITIES

6.1 Hydrogeologist/Field Engineer

Hydrogeologist/Field engineers are responsible for well installation in compliance with this procedure.

6.2 Task Leader

Task leaders are responsible for:

- Direct supervision of personnel drilling boreholes and installing monitoring wells;

- Assurance that equipment and materials are available to permit accomplishment of the task;

- Review and approval of daily work reports; and

- The completion of drilling operations and well installations to the satisfaction of Golder Associates' standards of operation and the clients requirements.

On small projects, the Task Leader's responsibilities may be assumed by Project Manager.

6.3 Project Manager

The Project Manager shall be responsible for:

- Selecting location of the boreholes at the site and determining depth and the various design details of the monitoring well completion;
- Selection and contracting of services of drilling subcontractors;
- Scheduling;
- Providing guidelines or specific work instructions for technical requirements beyond the scope of the applicable technical procedures; and
- The completion of drilling operations and well installations to the satisfaction of Golder's standards of operation and the clients' requirements.

7. EQUIPMENT

7.1 Drilling Equipment

The following equipment may be used on most well drilling and installation jobs:

- A suitable drill rig (usually auger, cable tool or rotary) with all equipment and accessories required for completing the job. The drill rig should be examined to determine the general condition, how well it has been maintained, and the general cleanliness of the drill rig and equipment. The drilling equipment should be relatively free of oils and grease;
- A steam cleaner for cleaning drilling equipment between holes with associated wash rinse solutions, storage tanks, brushes, and other equipment as necessary to capture and contain decontamination solutions;
- Additional equipment that may be required for drilling such as water trucks, drive casing, booster compressors, mud pumps, additional support vehicles and equipment, etc.; and
- A grout pump, mixer, and suitable clean tremie pipe or line.

7.2 Field Engineering Equipment and Logs

The following equipment is recommended for documenting the construction of groundwater monitoring wells:

- Clipboard;

- Indelible ink pens, pencils, and felt tip markers;
- Engineering pocket tape measure with 0.01 foot increments;
- Weighted engineering tape for sounding well material depth; the length of tape should be suitable for total anticipated well depth (check the accuracy of the weighted tape);
- Electric water level measuring tape suitable for total anticipated depth of borehole;
- Bottles or suitable containers for collecting samples of well construction materials;
- Sample containers for stratigraphic samples;
- Sample containers, cooler, etc. for chemical laboratory samples;
- History of Hole forms (Exhibit A);
- Borehole Log forms (See TP-1.2-5);
- Monitoring Well Construction Forms (Exhibit B);
- Field Change Request (Exhibit C); and
- Well development instrumentation, typically pH and conductivity meters and thermometer.

7.3 Health and Safety Equipment

- Hard hat;
- Steel toed rubber boots;
- Suitable clothing-long sleeve shirts, long pants, etc.;
- Tyvek, other protective clothing, and respiratory protection devices as required by applicable site health and safety plans;
- Protective eyewear;
- OVA, HNU or other monitoring equipment as required by applicable health and safety plans; and
- Hearing protection.

8. PROCEDURES

8.1 Regulatory Considerations

In most states, monitoring well installation is specifically regulated by state laws and guidelines that pertain to the drilling, installation and abandonment of monitoring wells. STATE REGULATIONS MUST BE REVIEWED BEFORE DESIGNING, DRILLING, AND INSTALLING MONITORING WELLS. At a minimum, the following actions must be taken:

- Initiate start cards or other documents and fees that may be required to drill and install monitoring wells;

- Ensure that monitoring wells will be drilled and installed by a contractor that is licensed and meets all requirements for conducting operations in that particular state;

- Initiate utility location process and document the location of all utilities before drilling. Check local codes for safety equipment (i.e., traffic codes, fire codes, etc.) required around drill rig during operation within city limits. Check that additional permits such as excavation or cultural reviews are not required; and

- Ensure that all follow up well installation reports required by regulatory agencies are completed by the drilling contractor and submitted (obtain a copy of all well reports submitted by contractor for project files).

8.2 Materials

8.2.1 Well Screen

The well screen shall normally consists of machine slotted, schedule 40 or 80 (ASTM-D1785) PVC pipe with flush joint threads. The use of other materials such as stainless steel may be required for certain situations where PVC may be reactive or inhibit the collection of representative groundwater samples; use of alternative materials shall be specified by the Project Manager or site-specific work plans. The well screen is typically between five to twenty feet in length.

Minimum requirements are identified below:

- A flush threaded PVC bottom plug shall be installed securely in order to withstand all installation and development pressures without becoming damaged or dislodged.

- As a general rule, screen length should be the minimum necessary to achieve the objective for monitoring. The lengths of screens shall be determined by the Project Manager based on the site hydrogeology. The screened interval will usually be 10 to 20 feet in length (taking into account any annual fluctuations in the water table) if the well is designed to intercept the top of the water table. Wells completed at depth below the top of the water table will usually be completed with 5 to 10 feet of screen.

- All flush joint threads shall be sealed with the supplied manufactured o-ring seals; teflon tape shall only be applied to the male threaded ends if the pipe is not equipped with o-rings. Riveted, screwed or glued joints shall not be permitted.

- The slot size will typically be 0.010 or 0.020-inches. The slot size shall be specified by the Project Manager and be determined relative to the formation particle size and the filter pack grain size.

- The screens shall have been factory cleaned to remove all oils, greases, solvents, waxes, etc. used in the manufacturing process and shall be individually wrapped and sealed or collectively stored and sealed in boxes from the factory.

8.2.2 Centralizers (Optional)

If required by the Project Manger or project work plans, friction or bolt-on style PVC or stainless steel centralizers shall be used. Deeper monitoring wells (i.e. >50' depth) will usually benefit from centralizers if the well is installed in a borehole several times the diameter of the PVC. Centralizers are not required for installing monitoring wells through auger flights.

8.2.3 Riser Pipe

The riser pipe shall consist of schedule 40 or schedule 80 (ASTM D1785) PVC pipe with flush joint threads; the interval between joints shall be five to twenty feet. Minimum requirements are itemized below:

- A PVC slip cap shall be provided for the top of the riser.

- All flush joint threads will be sealed with the supplied manufactured o-ring seals; teflon tape shall only be applied to the male threaded ends if the pipe is not equipped with o-rings.

- Riveted or glued joints shall not be permitted

- The riser pipe shall have been factory cleaned to remove all oils, greases, solvents, waxes, etc. used in the manufacturing process and shall be individually wrapped and sealed or collectively stored and sealed in boxes from the factory.

8.2.4 Steel Casing

In the event that a well requires permanent steel casing for hole stability or contamination seclusion, the following minimum requirements shall apply:

- The minimum inside diameter of steel casing shall be 4 inches in diameter larger than the nominal diameter of the well screen and riser (specific spacing requirements may be required by state regulations).

- The minimum wall thickness of steel casing shall be 0.125 inches; thicker casing may be required by state regulations or good engineering practice.

- The ends of sections of casing shall be threaded or beveled for welding or shall be designed to screw together.

- The casing should be in good condition and not out of round.

- The steel casing should be steam cleaned before use.

8.2.5 Filter pack

The filter pack shall consist of dry, uniform, well rounded and spherical grains of silica sand. The size of the sand grains will be selected by the Golder Hydrogeologist/Field Engineer based on the water-bearing aquifer formation characteristics and the size of the screen slots. The sand shall be packaged in sacks with polyethylene liners and clearly labeled as to manufacturer and mesh size of sand.

8.2.6 Cement/Bentonite Grout

Cement/Bentonite grout shall consist of a mixture of Portland Cement with 3-5% bentonite by weight added to help expand the cement on setting, thus providing a tighter seal. No other additives to the cement shall be permitted. Cement shall be Portland Cement Type I or Type II meeting ASTM C 150. The use of Hi Early Type III Cement is prohibited. Bentonite shall be powdered sodium bentonite furnished in sacks without additives; calcium bentonite may be used in certain calcium-rich environments. Water used during the installation operation for mixing cement/Bentonite grout shall be obtained from a potable source of known chemical quality designated by the Golder Hydrogeologist/Field Engineer.

Cement shall be mixed with water in the proportions of five to six gallons of water per 94-lb sack of cement. Three to five pounds of bentonite powder shall be added to the mix for each sack of cement used. The grout shall be thoroughly mixed with a paddle type mechanical mixer or by circulating the mix through a pump until all lumps are removed. Grout which is lumpy shall be rejected.

8.2.7 Bentonite Grout

Bentonite grout shall be made from a bentonite powder and/or granules for use below the water table. The bentonite grout shall have a specific gravity of 2.5, a dry bulk density of 55 lb/ft^3, and a pH of 9 to 10.5. Bentonite grout, when mixed, will have a thick batter-like consistency. Bentonite grout shall be mixed and used according to manufacturer's specifications and recommendations. Bentonite shall be powdered or granular (8-20 mesh) sodium bentonite furnished in sacks without additives. Water used during installation operations for mixing bentonite grout shall be obtained from a potable source of known chemical quality identified by the Golder Hydrogeologist/Field Engineer. The grout shall be thoroughly mixed with a paddle type mechanical mixed or by circulating the mix through a pump until all lumps are removed. Grout which is lumpy shall be rejected.

8.2.8 Volclay

Volclay is a trade name bentonite product that is modified with an initiator to increase the solids and set strength of the grout. The grout remains pumpable up to 2 hours but should be placed as soon after mixing is completed as possible. It sets up after 8-24 hours with final cure 24-72 hours after placement. The normal mix is 2.1 pounds of Volclay to 1 gallon of water. 14.3 pounds of Volclay mixed with 6.7 gallons of water yields 1 cubic foot of grout. A 50-pound bag of Volclay with the 2-pound bag of initiator mixed with about 24 gallons of water makes up about 3.5 cubic feet of grout. Because of potential desiccation and shrinkage, volclay shall only be used for sealing within water-saturated environments.

8.2.9 Pure Gold

Pure Gold is a trade name bentonite product that is an organic-free high-solids bentonite clay grout that has been tested and is certified to be free of contaminants. A 50-pound bag of Pure Gold grout is mixed with 14 gallons of water to yield approximately 2.2 cubic feet of grout. The grout should have density of 10.2 lbs/gal. Because of potential desiccation and shrinkage, pure gold shall only be used for sealing within water-saturated environments.

8.2.10 Bentonite Pellets/Chips

The two to five feet seal above the filter sand usually consists of bentonite pellets. Bentonite pellets will usually be a nominal 1/4- or 3/8-in. diameter round, or cylindrical pellets or chips consisting of untreated sodium bentonite, packaged in plastic buckets or plastic lined sacks. Each sack or bucket shall be clearly labeled as to the pellet size. The dry bulk density shall be at least 80 lbs/ft^3. The diameter of the pellets or chips shall be less than one-half the width of the annular space into which they are to be placed.

8.2.11 Concrete

Concrete, when used in monitoring well construction, shall be composed of either premixed, bagged concrete, or a mix of six sacks of cement per cubic yard. Neither additives nor borehole cuttings shall be mixed with the concrete. The concrete pad is not usually steel reinforced. Concrete shall be mixed in accordance with manufacturer's specifications; water must be from an approved source with known chemical quality. The construction of the concrete monument pad/surface seal is shown on Figure 8-2.

8.3 Well Borehole Drilling

8.3.1 General Precautions

All water used for mixing cement or bentonite grout or used down hole during drilling must be chemically characterized, particularly for the analytes of interest at the site. The Project Manager or Task Leader must approve the water source before use. All bentonite and cement must be pure. Additives either contained in these materials or to be added to the mixture must be approved in writing by the Golder Project Manager. All well screens and well casings must be factory cleaned and sealed or decontaminated prior to use. The seal must not be opened until use. The casing should not be allowed to touch the ground while installing. All down hole

drilling equipment must be decontaminated and cleaned prior to use on a borehole or before being demobilized from the site as noted in Section 8.3.4. below. The decontamination includes steam cleaning with a non-phosphate detergent followed by rinsing with approved tap water. The tap water rinse must be thorough to remove all detergent and grit. All wash solution will be captured and tested prior to disposal. The appropriate state agency will be consulted with the test results for determination of the proper mode of disposal of decontamination fluids.

8.3.2 Documentation

The Hydrogeologist/Field Engineer shall be responsible for ensuring that the drilling contractor completes and submits any required start cards, and, following completion of the well, any well records required by state agencies. All such records shall be reviewed and approved by the Golder Project Manager prior to submittal and copies of these documents shall be retained for the project files.

The drilling contractor shall be responsible for maintaining a daily log that shall be forwarded to the Hydrogeologist/Field Engineer on a daily basis. This log shall be a continuous, chronological log for each borehole/well and should include the following information: Date, well number, drillers name and company, depth and drilling characteristics of each stratigraphic unit drilled, the depth and thickness of each stratigraphic and water bearing zone encountered, any moist zones encountered, the time work starts and stops each day (and the hours spent at each task), time and durations of all shutdowns, depths of collected samples, method of drilling and any problems encountered, size of drill bit used, size, amount and type of any temporary or permanent casing used, amount of water yielded or lost during drilling, type and amount of any drilling additives used, and the type and amount of any lubricants, if any, used on the drilling equipment and the types and quantities of any well construction materials used.

The Golder Hydrogeologist/Field Engineer shall be responsible for documenting the drilling operations, sample collection and construction of the monitoring well. All significant drilling events during the installation of each monitoring well shall be documented daily on History of Hole forms (Exhibit A). The documentation shall include the time that the events occurred; particular emphasis shall be placed on documenting the production hours and hours delayed in production, with appropriate explanations. Notes on the weather conditions and drilling personnel shall also be recorded. During completion, all backfilled materials and depths of well screens will be sounded with a measuring tape with attached weight on the sounding end. Well completion details shall be recorded on the Monitoring Well Installation Log (Exhibit B) by the Golder Hydrogeologist/Field Engineer. All information presented on the Monitoring Well Installation Log shall be recorded, or N/A for Not Available or Not Applicable entered as appropriate.

8.3.3 Initial Equipment Evaluation and Inspection

As soon as possible after the drill rig arrives on site, and certainly prior to beginning the drilling operation or decontamination of the drill rig, inspect and evaluate the drilling equipment supplied by the drilling contractor. The equipment should appear to be in good working condition, be maintained properly, and be the proper equipment specified for the job. Examine the rig for any oil or other fluid leaks that may be hard to detect after the drill rig has been decontaminated. If leaks are detected, discuss them with the contractor and arrange for them to be repaired or arrange for other means of containing the leaks around the well site. Inspect all

equipment to be used by the contractor; this is the time to have arrangements made to get missing equipment to the site.

8.3.4 Decontamination of the Drill Rig and Drill Equipment

The drilling rig and all equipment in contact with soil materials or borehole fluids shall be steam cleaned (high pressure at 80 psi and high temperature at +180° F is acceptable) between each borehole. The contractor shall normally supply the equipment necessary for this decontamination. Fittings shall be carefully greased and fluids carefully added to the drill rig before cleaning. Decontamination shall be conducted at an approved clean location outside the immediate drilling site. Any hazardous materials removed from the equipment must be contained to prevent the spread or release of such materials. Tools and sampling equipment (e.g., split spoon sampler, submersible pumps) to be used for soil sampling or aquifer testing shall generally be decontaminated by steam-cleaning. However, at the Project Manger or Task Leader's direction, the following additional decontamination method may be required: clean by wiping or scrubbing off visible particulate matter; wash with a laboratory-grade non-phosphate detergent (e.g., Alconox), rinse with clean water and methanol (technical grade or better); and final rinse with distilled deionized water. The detergent, methanol (where necessary), and distilled water will usually be provided by Golder. All other decontamination equipment (e.g., brushes, clean water, buckets, steam cleaner) shall normally be provided by the contractor.

8.3.5 Setting up the Drill Rig

Very fragile surfaces such as a sod yard should be protected using sheets of plywood to protect the land surface. Cut a hole in one sheet of plywood the size of the borehole for placing directly over the hole and place other sheets as needed around the site. Account for the prevalent wind direction if the rig will require a preferred orientation for extra cooling. Plan for the direction the dust or cuttings will be blown and the direction that will be downwind of the borehole if it is likely to emit volatile organic vapors. Decide which direction the water produced during the drilling operations will flow. Drill rig set up shall be planned with the contractor. The drill rig shall be set up by the contractor using levels to insure that the borehole is as plumb as possible. The verticality of the kelly or mast should be determined in two directions: side to side and fore and aft. Ensure that the drill rig is adequately blocked below the jacks so that the rig is stable and minimum damage occurs to the land surface.

8.3.6 Borehole Size and Drilling Methods

The borehole size will be partly determined by the anticipated drilling requirements to reach the anticipated depth, i.e., will temporary casing have to be driven with the possibility of telescoping casing if refusal is encountered. The borehole size will also be determined by the completion size of the final well production casing and by the state or other regulatory requirements governing the size of the annular space around production and surface casing. The drill hole size and drilling methods will be determined by the Project Manager; methods may be selected from those provided in TP-1.2-5, "Drilling Sampling, and Logging of Soils", or TP-1.2-1, "Rock Core Drilling and Sampling".

8.3.7 Capture of Drill Cuttings and Groundwater

The soil or rock cuttings produced during the drilling operations and any groundwater produced by drilling below the water table may have to be captured if contaminants are anticipated or the site does not allow for the disposal of clean drill cuttings around the drill site. Unless otherwise directed by the Project Manger, the drill cuttings and groundwater should be captured in 55-gallon drums. The borehole number and depths of the cuttings contained in each drum should be noted in permanent paint on the side of the drum and on the lid. The soils and water can then be tested for the presence of the targeted contaminants. If the contaminants are detected, then the drummed cuttings and water must be disposed of in a proper manner as directed by state or other regulatory agencies. It is usually preferred to have the contractor move all the 55 gallon drums to one or two drum storage locations that has been approved by the client/landowner.

8.3.8 Verifying the Completed Borehole

Once the borehole has been completed, verify the final depth and determine how clean the borehole is by lowering and retrieving a weighted engineering tape to the total depth of the well. Lower a water level measuring probe down the borehole and record the water level in the borehole following completion of the borehole in compliance with procedure TP-1.4-6, "Water Level Measurement". If the borehole is to stand for any period of time before completion of the well, reprobe the borehole to determine the total depth and the water level within the borehole just prior to completion of the well. It is generally a poor practice to allow a borehole to stand open very long if there is the possibility of part of the borehole caving. At the Project Manager's direction, the stratigraphic sequence encountered in the borehole may be verified by geophysical logging. A variety of probes are available for stratigraphic, hydrogeologic and borehole geometry determinations. Geophysical logging equipment will require the same diligence in decontamination to prevent the potential introduction of contaminants into the borehole or well.

8.4 Well Installation

8.4.1 General Requirements

A properly completed well should exhibit the following characteristics:

- The only portion of the completed well which will have well screen and a filterpack should be adjacent to the particular single water-bearing zone to be monitored.

- All other sections, except within 5 feet of land surface, should be sealed with a bentonite or cement/bentonite type grout. The liquid slurry grouts will be placed by pumping the grout through a tremie pipe to the appropriate locations, or, if bentonite pellets are used, the pellets will be properly rehydrated and compacted in place.

- The uppermost 5 feet or more (or to below frost line) of the borehole annulus will be filled with a cement grout for a surface seal, stability in freezing weather, and for supporting protective surface monuments.

See Figure 8-1 for a typical well installation.

8.4.2 Evaluation of the Borehole

Compare the final completed borehole and the stratigraphic log of the samples or cuttings to the anticipated design for the monitoring well. Compare the strata to what was anticipated and verify that the depth is adequate. If the desired monitored interval is above the bottom of the borehole, the borehole must be backfilled with a low permeability grout. The grout shall be tremied slightly below the final depth of the well to the bottom of the borehole. The auger flights or steel casing shall be pulled or hammered out of the hole in five foot increments. This procedure should be repeated until the bottom of the drive casing or auger flights and top of the grout are equivalent to the bottom elevation of the desired monitoring interval. The grout should be allowed to set prior to placing the gravel/sand pack and well casing/screen to establish a solid foundation. If bentonite grout does not harden adequately to permit a firm foundation for supporting the well, then a cement/bentonite grout should be used and allowed to harden. **Bentonite and/or cement grout should never be allowed to harden while the drive casing is in contact.** Care must be taken with cement and cement grouts. Cement can alter the pH of the groundwater that it comes in contact with. The best practice is to try and keep cement from being in direct contact with the sandpack of the monitored interval.

8.4.3 Assembly of Well Screen and Riser

The well screen including the bottom plug shall be clean and free of contamination immediately prior to assembly. Take precautions to ensure that grease, oil, or other contaminants do not contact the well screen. All workers handling the well screen shall wear a new pair of cotton or synthetic gloves. The male threaded part of each joint shall be wrapped with teflon tape if not equipped with O-rings. Joints shall be tightened by hand; however, if necessary, clean pipe or chain wrenches may be utilized if care is exercised not to cut or damage the PVC casing. The well screen and riser shall be inserted into a well hole which is at least partially filled with water.

8.4.4 Setting the Well Screen and Riser String

The well screen shall be lowered to the predetermined level and held in position by suspending the string of riser pipe or if the string tends to float, by manipulating the hydraulic ram. On deep holes where the weight of the string riser pipe is significantly greater than the flotation force, care shall be taken to keep the riser pipe plumb. The riser shall extend above the ground at least three feet, and may be trimmed to the proper length after the grout is in place. If the plumbness of the riser is especially critical or the well is extremely deep, a probe or "dummy" should be lowered down the inside of the riser to verify that the riser is not kinked. The screen and riser string should be suspended from the surface as much as possible during completion, rather than sitting in the borehole. At the Project Manager's direction or if required by project work plans, centralizers shall be placed a minimum of 20-40 feet apart throughout the riser interval.

8.4.5 Placement of the Filter Pack

The volume of sand required to fill the annular space between the well screen and the wall of the borehole shall be computed and carefully measured. The sand pack shall typically extend three to five feet above the uppermost row of slots in the well screen except where limited separation between aquifers occurs (check state regulations). The well screen shall be centered

in the well hole and temporary casing by suspending the well screen and riser string and/or using centralizers. At the Project Manager's direction or if required by project work plans, centralizers shall be placed at a minimum of 10-20 feet apart through the screen interval. While holding the riser pipe with the drill rig, the temporary casing or hollow stem augers shall be carefully withdrawn such that the lowermost point on the casing is exactly at the top of the sand packed portion of the well. This may be accomplished in increments; however, after each increment, a weighted engineering tape shall be inserted and lowered to ascertain that the sand pack has not been raised during casing withdrawal operation. If necessary the sand pack shall be tamped back into place.

8.4.6 Placement of the Bentonite Seal

A volume of pellets or chips to create a seal 3 to 5 feet long at a minimum should be computed and measured out for introduction into the annular space. The bentonite pellets or chips may be placed by either tremieing the pellets to the required interval or hand pouring the pellets into the annular space at the top of the well. Tremieing of the pellets is usually done on deeper completions or if specifically required in the well completion specifications. **Extreme care must be exercised and the pellets or chips slowly introduced into the annular space to prevent bridging above the level to be completed**. **In addition, the bottom of the casing must be maintained above the uppermost level of the pellets to avoid locking the well casing**. If the bentonite seal is being constructed above the water level in the borehole, exactly five gallons of water per a 5-gallon bucket of pellets or bag of bentonite chips should be slowly poured into the annular space to rehydrate the bentonite. A weighted seal tamper should be lowered down the annulus and used to tamp the pellets or chips into a cohesive mass of clay.

8.4.7 Grouting the Annular Seal

A sample of the final grout shall be collected in a suitable container just prior to ordering the grout injection. The volume of grout, either bentonite or cement/bentonite, required to completely fill the annular space between the seal and the ground surface shall be prepared in the proportions specified in Section 8.2. The volume shall include a quantity to compensate for losses. All hoses, tubes pipes, water swivels, drill rods, or other passageways through which the grout will be pumped shall have an inside diameter of at least 0.5 inches.

The grout shall be injected via a side discharge tremie pipe with its opening temporarily set immediately above the bentonite seal. The grout shall be pumped into the tremie pipe continuously until it flows out of the annulus at the surface. Any temporary casing if used or auger flights shall be removed immediately and in advance of the time when the grout begins to set. Casing removal and injection may proceed concurrently provided the top of the column of grout is always at least twenty feet above the bottom of the casing and provided injection is not interrupted. If casing removal does not commence until grout injection is completed, then additional grout shall be periodically poured into the annular space so as to maintain a continuous column of grout up to the ground surface. The well shall not be disturbed for 48 hours to permit the grout to gain sufficient strength.

8.4.8 Placement of Protective Well Monument, Concrete Pad/Seal

A well protector as shown in Figures 8-1 and 8-2 shall be emplaced before the grout has set. The well protector shall be positioned and maintained in a plumb position with temporary

braces as required. An eight inch clearance between the top of the riser and the well protector shall be maintained for the sampler. Grout which has overflowed the well hole shall be carefully removed so as to prevent the formation of horizontal projections (mushrooming) which may be subject to frost heave. A 1/4 inch diameter hole shall be drilled in the well protector 6 inches above the ground surface to permit water to drain out of the annular space. Coarse sand and/or pea gravel shall be placed in the annular space at least 6 inches above the hole to prevent insects from entering through the drilled hole. A 4-inch thick concrete pad shall be constructed around the protective monument. The pad should be approximately 2 to 3 feet in radius and should slope slightly away from the monument on all sides. Three protective "bumper" posts may be installed evenly spaced around the well at a 3 to 5 foot radius. Each post should be flagged and/or painted for improved visibility. If the monument is to be painted, the monument shall be painted before placing it in the grout.

A permanent, weatherproof, well identifier shall be affixed to the outside of the protective well monument (check state regulations for monitoring well labeling requirements).

8.5 Well Development and Acceptance

This section covers the development of a newly constructed well and the measurement of the well characteristics.

8.5.1 Pumps and Accessories for Well Development

All wells shall be developed to produce representative formation water. This section describes the approved pumps and accessories to be used in development of monitoring wells. All pumps and other devices used in well development shall be clean and free of oil, grease, solvents, or any other foreign contaminants.

8.5.1.1 Submersible Pumps

Submersible pumps shall include electric motor powered centrifugal or positive displacement type pumps which are operated under submergence. If a submersible pump is utilized for well development, it shall be of a type and capacity such that it can pump water from the well continuously for a period of at least five minutes without shutting off. Backpressure or other methods may be utilized to accomplish the desired rate of pumping. The pump shall be capable of being turned on and off instantaneously to create surging in the well. The pump shall not be fitted with a backflow check valve.

8.5.1.2 Bladder Pumps

A bladder or diaphragm pump shall operate by compressed air cycling to inflate and deflate a diaphragm which creates a pumping action. Bladder pumps approved for well development shall be capable of continuously pumping at least 3 gpm when installed in the well.

8.5.1.3 Jet Pumps

Jet pumps use the Venturi principle to create subatmospheric pressure to allow the pump to be used below a depth at which suction alone would not normally lift the water. Jet pumps

approved for well development shall be capable of pumping at least 3 gpm continuously when installed in the well.

8.5.1.4 Bailers

Bailers shall not be used for well development except after an approved submersible bladder, jet or suction pump has been installed in the well or compressed air or bottled nitrogen has been used, and/or the rate of well recovery is so slow that these methods are ineffective. Small diameter wells may also require bailing for development due to the limited access of the casing diameter.

8.5.1.5 Compressed Air

Compressed air supplied by an engine-driven compressor equipped with an approved oil filter and trap may be used provided the source of compressed air is capable of evacuating 50 percent of the column of water from the well once every minute.

8.5.1.6 Bottled Nitrogen

Bottled nitrogen may be used provided a regulator is employed and the system is capable of evacuating 50 percent of the column water from the well once every minute.

8.5.2 Well Development

Well development should begin after the monitoring well is completely installed and prior to water sampling. Development should be continued until representative water, free of the drilling fluids, cuttings, or other materials introduced during well construction is obtained. Representative water is assumed to have been obtained when pH, temperature, and specific conductivity readings stabilize and the water is visually clear of suspended solids. The minimum duration of well development should vary in accordance with the method used to develop the well. For example, surging and pumping the well may provide a stable, sediment free sample in a matter of minutes; whereas, bailing the well may require several hours of continuous effort to obtain a clear sample. The duration of well development and the pH, temperature, and specific conductivity readings should be recorded on the well completion diagram by the Golder Hydrogeologist/Field Engineer.

Purged groundwater shall be captured and contained in 55 gallon steel drums or suitable tank(s), if a reasonable potential exists for the groundwater to contain hazardous substances as directed by the Project Manager or as specified in the project work documents.

If required, each drum or tank containing captured purge water shall be properly labeled with a weather proof label as to the contents, the well(s) from which the contained purge water originated and the date on which the contents were generated. Storage of the drums or tanks shall be as specified in the project work documents or as directed by the Project Manager.

Captured and contained purge water shall be characterized for discharge, treatment and/or disposal. Characterization of the captured and contained purge water should be specified in the project work documents or by the Project Manager, but could rely on the analytical results of groundwater samples associated with each drum or tank, or could involve direct sampling and analyses of the contained water.

The requirements and options available for discharge, treatment and/or disposal are dependent upon many variables such as chemical consistency, local and state regulations and location of site. Discharge, treatment and/or disposal of captured and contained purge water must be in accordance with local, state and Federal regulations and shall be specified in the project work documents.

8.5.3 Well Recovery Test

A well recovery test shall be performed immediately after development. Readings shall be taken at appropriate intervals (usually one minute or less) until the well has recovered to within 90% of its static water level. The extremely long recovery periods of some wells may prohibit carrying this test out to it's completion; the decision to terminate shall be noted and justified on the Well Installation Log.

8.5.4 Well Acceptance

A well will be accepted by the Golder Hydrogeologist/Field Engineer when development has been completed in accordance with these specifications, and the required drilling contractor's documentation has been furnished to Golder. Once a well has been approved, the contractor shall be relieved of any further responsibility for the performance, maintenance, or testing of that well.

The final completed well shall be probed using a weighted engineering tape to determine that the well is open to total depth. A 10-foot rigid cylinder 1 standard size smaller than nominal well casing size shall be lowered to the total depth of the well and retrieved to verify straightness of the well for tool access.

8.6 Site Cleanup

The Staging area, decontamination area, each well site and any other areas used during the well installation program shall be cleaned up by the contractor and inspected by the Golder Hydrogeologist. All trash should be removed; the 55-gallon drums of cuttings shall be organized into a temporary storage area and any concrete or asphalt that has been covered with mud shall be washed off. Cut or torn asphalt areas will be patched by the contractor using a cold patch material. Try to restore the area as much as possible to the condition before the well installation program.

8.7 Field Change Request

Variation from established procedure requirements may be necessary due to unique circumstances encountered on individual projects. All variations from established procedures shall be documented on Field Change Request (Exhibit C) and reviewed by the Project Manager and the QA Manager.

The Project Manager may authorize individual Hydrogeologist/Field Engineers to initiate necessary variations. If possible, the request for variation shall be reviewed by the Project Manager and the QA Manager prior to implementation. If prior review is not possible, the

variation may be implemented immediately at the direction of the Hydrogeologist/Field Engineer, provided that the Project Manager is notified of the variation within 24 hours of the implementation, and the Field Change Request is forwarded to the Project Manager and QA Manager within 2 working days of implementation. If the variation is unacceptable to either reviewer, the activity shall be reperformed or action shall be taken as indicated in the Comments section of the reviewed Field Change Request. All completed Field Change Request shall be maintained in project records.

8.8 Well Location

The level of accuracy required for horizontal well location and elevation shall be specified by the Project Manager or applicable project plans. At a minimum, well locations shall be approximated with tape and compass from known permanent landmarks. Elevation of the riser casing shall be approximated. A licensed surveyor, if required, shall maintain 3rd order precision and accuracy. The surveyed point for the well riser shall be the highest point on the riser casing with the PVC slip cap removed.

Figure 8-1

Figure 8-2

Exhibit A

Exhibit B

Exhibit C

December 2012

TECHNICAL GUIDANCE

GAI Soil Description System

Document Number: TG-1.2-6 GAI Soil Description-Rev 1

TECHNICAL GUIDANCE

Golder Associates

Record of Issue

Revision	Description	Issue Date	Approved by
GAL A	GAL Working Draft	March 15, 2011	DEB
GAL B	GAL Draft for Review	April 11, 2011	DEB
GAL 0	GAL Approved for Issue	September 20, 2011	DEB
GAL 1	GAL Edits to address feedback comments including from GAI	Draft completed December 2012. To be finalized and issued in 2013	DEB
GAI 1	GAI Approved for Issue	December 31, 2012	MS

Table of Contents

1.0 ABBREVIATIONS 1

2.0 INTRODUCTION 2

2.1 Definitions 2

2.2 Classification vs. Description 2

2.3 Soil Classification Systems 3

2.4 Soil Constituents 4

2.4.1 Cobbles and Boulders 4

2.4.2 Organic Constituents 4

2.4.3 Coarse-grained Constituents 4

2.4.4 Fine-grained Constituents 4

2.5 Major Soil Groups 4

2.5.1 Organic Soils 5

2.5.2 Coarse-grained Soils 6

2.5.3 Fine-grained Soils 6

2.5.4 Note on Behavior vs. Soil Groups 6

3.0 ORDER OF SOIL DESCRIPTION 7

4.0 CLASSIFICATION AND SOIL NAME 9

4.1 Dual Symbol 9

4.2 Borderline Symbol 9

5.0 DESCRIPTION OF CONSTITUENTS 13

5.1 Coarse-grained Constituents 13

5.1.1 Particle Size 13

5.1.2 Particle Size Distribution 13

5.1.3 Particle Shape and Angularity 15

5.2 Fine-grained Constituents 16

5.2.1 Atterberg Limits Testing 16

5.2.2 Field Identification Tests 17

6.0 SECONDARY AND MINOR CONSTITUENTS 21

7.0 GENERAL CHARACTERISTICS 23

7.1 Color 23

7.2 Weathering 23

7.3 Structure 23

7.3.1 Zoning 24

7.3.2 Fissures 24

7.3.3 Cementation 25

7.3.4 Other Defects 25

7.4 Sensitivity 25

7.5 Contamination 26

7.6 Additional Observations 26

7.6.1 Geological Formation/Regional Name 26

7.6.2 Definitions of General Geological Descriptions 26

7.6.3 Cobbles and Boulders 27

8.0 BEHAVIOR BASED PARAMETERS 29

8.1 Cohesive vs. Non-cohesive 29

8.2 Compactness/Consistency 29

8.2.1 Non-cohesive Soils – Compactness 29

8.2.2 Cohesive Soils – Consistency 31

8.3 Field Moisture Condition/Water Content 32

8.3.1 Non-cohesive Soils – Field Moisture Condition 32

8.3.2 Cohesive Soils – Water Content 32

9.0 EXAMPLES OF SOIL DESCRIPTIONS 33

9.1 Example Field Borehole Log 33

9.2 Example Soil Classifications Based on Laboratory Test Results 33

9.3 Example Record of Borehole 34

10.0 DESCRIPTION OF ORGANIC SOILS 35

11.0 POTENTIALLY CONTAMINATED SOILS 37

11.1 Health and Safety 37

11.2 General 37

11.3 Indicators of Potential for Contamination 37
11.4 Odors 39
12.0 FROZEN SOILS 40
12.1 Definitions 40
12.2 General Guidance 40
12.2.1 Step 1 – Description of the Soil 40
12.2.2 Step 2 – Description of the Frozen Soil 40
12.2.2.1 Non-visible Ice 40
12.2.2.2 Visible Ice 41
12.2.3 Step 3 – Description of Substantial Ice Strata 43
12.3 Further Considerations 43
12.3.1 Excess Ice Determination 43
12.3.2 Pore Fluid Salinity 43
13.0 UNUSUAL OR DIFFICULT MATERIALS 44
13.1 Fill and Refuse/Waste 44
13.2 Rock Fill 46
13.3 Tailings 46
13.4 Topsoil 46
13.5 Pavement Materials/Pavement Structure 47
13.6 Is it Soil or is it Rock? 49
13.7 Duricrusts 51
14.0 REFERENCES 53

APPENDICES

APPENDIX A
Example Grain Size Distributions for USCS Coarse-Grained Soil Groups

APPENDIX B
Example Descriptions for Varved/Laminated Soils

APPENDIX C
Example Soil Classifications and Descriptions

APPENDIX D
Example Field Borehole Log (to be done),

Field Borehole Log Templates, and

Field “Cheat Sheets”

APPENDIX E
Example Soil Classifications based on Laboratory Test Results

APPENDIX F
Example Record of Borehole Sheet (to be done) and

Strata Symbols

APPENDIX G
"Method of Soil Classification,"

"Abbreviations and Terms used on Record of Boreholes and Test Pits," and

"List of Symbols" Sheets

TABLES

Table 2-1: Soil Classification Systems 3
Table 2-2: Example Organic Soil Classifications 6
Table 3-1: Order of Soil Description 7
Table 4-1: Summary of Soil Group Symbols and Group Names 11
Table 5-1: Identification of Particle Size 13
Table 5-2: Visual Identification of Particle Size Distribution 14
Table 5-3: Identification of Angularity and Particle Shape 15
Table 6-1: Modifiers for Secondary and Minor Constituents 22
Table 7-1: Zoning Terms and Descriptions 24
Table 7-2: Identification of Particle Size 27
Table 8-1: Compactness of Non-cohesive Soils 30
Table 8-2: Consistency of Cohesive Soils 31
Table 8-3: Field Moisture Condition 32
Table 8-4: Water Content 32
Table 10-1: Naming of Organic Soils 35
Table 10-2: Assessment of Degree of Decomposition or Humification 36
Table 11-1: Examples of Indicators of Potential Contamination 38
Table 11-2: Examples of Odors 39
Table 12-1: Summary of Frozen Soil and Ice Strata Descriptions 42
Table 13-1: Soil vs. Rock 50
Table 13-2: Typical Duricrusts 52

FIGURES

Figure 1: Soil Groups Flowchart 5
Figure 2: USCS Group Symbol and Group Name Flowchart 10
Figure 3: Plasticity Index vs. Liquid Limit Chart 17
Figure 4: Range of Percentages (Source: American Geological Institute) 21

1.0 ABBREVIATIONS

Abbreviations	Description
AASHTO	American Association of State Highway and Transportation Officials
ASTM	American Standard Test Methods
BPT	Becker Penetration Test
C_C	Coefficient of Curvature ($\frac{(D_{30})^2}{D_{10} \times D_{60}}$)
C_U	Coefficient of Uniformity ($\frac{D_{60}}{D_{10}}$)
CPT	Cone Penetration Test
CFEM	Canadian Foundation Engineering Manual
D_{10}	10% of the soil particles by mass are finer than this particle size
D_{30}	30% of the soil particles by mass are finer than this particle size
D_{60}	60% of the soil particles by mass are finer than this particle size
DCPT	Dynamic Cone Penetration Test
LL	Liquid Limit (ASTM D 4318)
N	SPT 'N' in accordance with ASTM D 1586, uncorrected for overburden pressure effects
NAVFAC	Naval Facilities Engineering Command, Soil Mechanics and Foundations & Earth Structures Design Manuals, 7.01 and 7.02 (now contained in UFC)
NP	Non-plastic
PI	Plasticity Index (ASTM D 4318)
PL	Plastic Limit (ASTM D 4318)
SPT	Standard Penetration Test
S_t	Sensitivity ($s_{u(undisturbed)}/s_{u(remolded)}$)
UCS	Unconfined Compressive Strength
UFC	Unified Facilities Criteria – current location of US Department of Defence engineering design manuals; supersedes references to originating agencies (e.g., NAVFAC)
USCS	Unified Soil Classification System (as used in ASTM D2487, ASTM D2488, and AS1726)
w	Water Content

2.0 INTRODUCTION

This Technical Guidance presents the system to be used for all soil classification and descriptions performed by Golder Associates Inc. (GAI) in the United States unless project specific approval is given by the Project Director and the Division Manager/Office Manager. Situations that may require the use of an alternate guidance include:

- Work performed in or for a jurisdiction in which a different guidance is the accepted standard practice.
- Work where an alternate guidance is required as part of the contract or as directed by the client.
- Work performed as part of a project led by another Golder company, and in which a different guidance is being used.

This GAI Technical Guidance is based on the Golder Associates Ltd. (GAL) Canadian Technical Procedure, GAL Soil Description, Rev. 1. Differences between the GAI and GAL guidance are as follows:

- The GAI guidance eliminates the use of intermediate plasticity clay (CI) as a Group Symbol. The GAI guidance instead uses CL, but a description of the plasticity (low, medium, or high) should be included in the description (see Figure 3).
- The GAI guidance provides an example field boring log to use as a template for field soil logging activities (see Appendix D).
- The GAI guidance incorporates Imperial units.
- The GAI guidance uses United States English spellings and other standard punctuation conventions.

This soil description system is generally consistent with the concepts presented in ASTM D2487, D2488, AS 12726, and the Canadian Foundation Engineering Manual (CFEM, 2006), with some differences intended to improve consistency with laboratory testing performed to ASTM standards, to align with other Golder companies, or to address perceived inconsistencies within the CFEM.

This guidance supersedes the guidance presented in Appendix A of the 1993 GAL Field Procedures Manual and TP-1.2-6 of the 1996 GAI Field Identification of Soils. It is noted that this guidance is intended for use in the field as well as for soil classification and description as presented on Record of Boreholes and in reports.

2.1 Definitions

Soil – CFEM (2006) defines soil as *"that portion of the earth's crust that is fragmentary, or such that some individual particles of a dried sample may be readily separated by agitation in water."*

Rock – CFEM (2006) defines rock as *"a natural aggregate of minerals that cannot be readily broken by hand and that will not disintegrate on a first wetting and drying cycle."*

2.2 Classification vs. Description

In this Technical Guidance, the following differentiation between soil classification and soil description is used:

- Soil classification involves grouping soil into categories based on selected index parameters. Classification can be achieved through visual/tactile methods or laboratory testing.

- Soil classification alone (regardless of classification system) does not capture all the relevant engineering information.
- Soil classification is a component of a soil description.
- Soil description captures additional important information such as other constituents, behavior, compactness/consistency, field moisture condition, color, structure, etc.

Classification of soils enables the user to quickly assess some of the physical characteristics of a soil material, but other components of the soil description are essential for a complete understanding of the engineering attributes of a particular material in a specific application. The most important elements of the soil description will vary depending on the application in question.

Soil **classification** is a component of a complete soil **description**. The soil description system described in this Technical Guidance includes classification (Group Symbol and Group Name) and descriptive terms.

2.3 Soil Classification Systems

The various standards associations around the world use several soil classification systems. They are broadly divided into two groupings as shown in Table 2-1.

Table 2-1: Soil Classification Systems

Method of Soil Classification	Used or Advocated by:
Unified Soil Classification System (USCS), which classifies based on particle size/distribution and plasticity	■ Standard Test Method for Classification of Soils for Engineering Purposes – ASTM D2487 ■ Standard Practice for Description and Identification of Soils (Visual-Manual Procedure) – ASTM D2488 ■ Australian Standard for Geotechnical Site Investigations – AS1726 ■ AASHTO Soil Classification System
Classification based on bulk behavior (cohesive or non-cohesive) plus particle size distribution	■ British Standard "Description of Soils and Rocks" – BS5930-1999 ■ British Standard "Geotechnical investigation and testing – Identification and classification of soil" – BS EN ISO 14688-1 2002 and BS EN ISO 14688-2 2004

The method of soil classification used in this Technical Guidance is based on a modified Unified Soil Classification System. Soil behavior and those parameters related to behavior are included in other components of the soil description. The soil classification system used in the superseded Appendix A of the 1993 GAL Field Procedures Manual is a behavior-based classification system, similar to that used in the British Standard BS5930-1999.

CFEM does not provide a classification system. Rather, it presents concepts which can be incorporated into a description system.

2.4 Soil Constituents

Soil constituents are the various particles that comprise the overall bulk soil mass. The constituents are categorized into cobbles/boulders, organic, coarse-grained and fine-grained soils.

2.4.1 Cobbles and Boulders

Cobbles are particles between 75 mm and 300 mm (3 inches and 12 inches) in size. Boulders are particles greater than 300 mm (12 inches) in size. The sizes are the maximum dimension in any one direction.

The presence of cobbles and boulders in a soil is extremely important. While cobbles and boulders may sometimes be considered as a coarse-grained constituent, in this Technical Guidance they will be discussed as a separate component in a soil mass. Cobbles and boulders are not included in soil classifications. Their presence is included in the description. See Section 7.6.3 for the description of cobbles and boulders.

2.4.2 Organic Constituents

These share the common property that they are NOT of mineral origin, but rather comprise plant matter in various stages of decomposition. They are classified on the basis of the type and degree of decomposition of the plant (organic) matter.

2.4.3 Coarse-grained Constituents

Sands and gravels comprise coarse-grained constituents. These share the common property that they are of mineral origin and the individual particles are larger than 0.075 mm (75 µm or No. 200 sieve size), typically large enough to be visible to the naked eye. They are classified on the basis of their particle size and particle size distribution.

While cobbles and boulders may sometimes be considered as a coarse-grained constituent, in this Technical Guidance they will be discussed as a separate component. In a soil mass, cobbles and boulders are not included in soil classifications. Their presence is included in the description. See Section 7.6.3 for the description of cobbles and boulders.

2.4.4 Fine-grained Constituents

Silt and clay comprise fine-grained constituents. These share the common property that they are of mineral origin and the individual particles are smaller than 0.075 mm (75 µm or No. 200 sieve size), typically not large enough to be visible to the naked eye. **The distinction between silt and clay is classified on the basis of their plasticity; they are NOT classified on the basis of their particle size.** This is a key distinction, as there are some texts or procedures (including CFEM, 2006) that differentiate silt from clay on the basis of particle size.

2.5 Major Soil Groups

Soil Groups are defined based on the proportions of each of the constituents, as shown in the following flowchart.

As noted previously, cobbles and boulders are discussed separately from these soil groups.

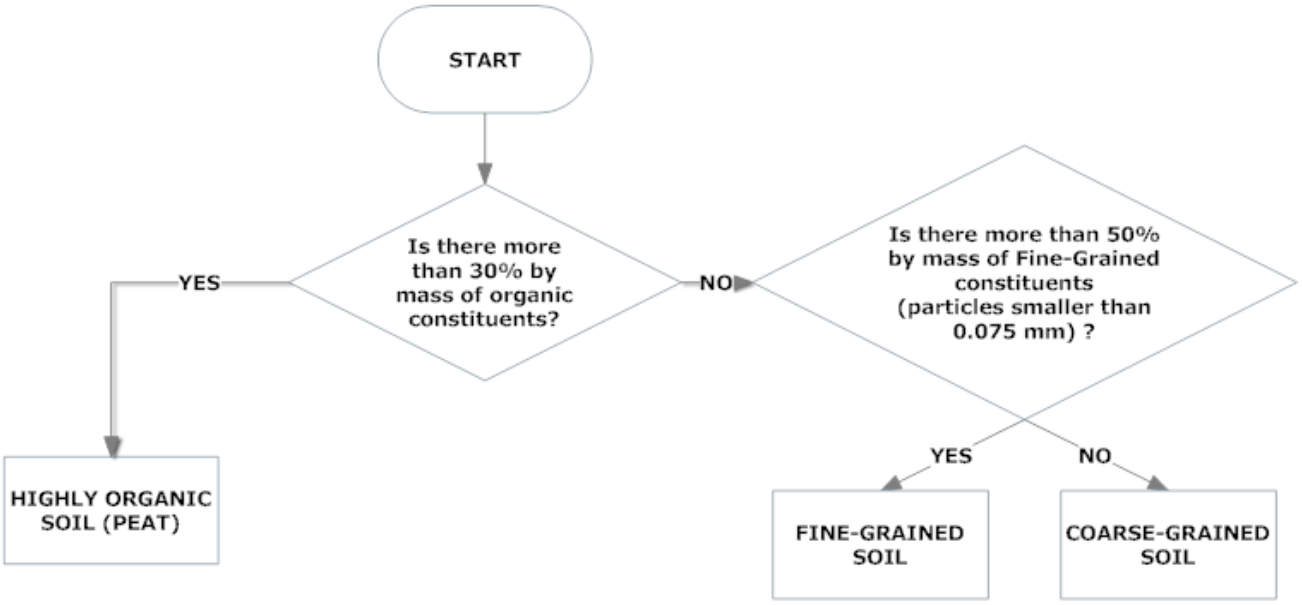


Figure 1: Soil Groups Flowchart

2.5.1 Organic Soils

A material containing at least 30% by mass of plant matter in various stages of decomposition and has a fibrous to amorphous texture, a dark-brown to black color, and an organic odor is considered a highly organic soil and should be classified as PEAT (PT) (NAVFAC DM7.01). See Section 10.0.

Materials containing between 5% and 30% by mass of organic matter are considered organic soils but are classified following the guidance for inorganic soils, with the organic component noted in the Group Symbol and Group Name, as follows:

- Soils having >50% of material smaller than 0.075 mm and an organic content >5% by mass and with Plasticity Index (PI) and Liquid Limit (LL) that plot below the A-Line on the Plasticity Chart will be classified as either (OH) clayey ORGANIC SILT or (OL) ORGANIC SILT.
- Soil having >50% of material smaller than 0.075 mm and an organic content >5% by mass and with PI and LL that plot above the A-Line on the Plasticity Chart will be classified according to the Group Symbol and Group Name for inorganic soils with the descriptor “organic” added as a prefix, such as (CL) organic SILTY CLAY or (CH) organic CLAY.
- Soils having <50% of material smaller than 0.075 mm and an organic content >5% by mass will be classified according to the Group Symbol and Group Name for inorganic soils with the descriptor “organic” added as a prefix, such as (SM) organic SILTY SAND.

Materials containing <5% by mass of organic matter are classified using the guidelines for inorganic soils with the organic component noted in the description as a minor constituent, e.g., (SM) SILTY SAND, trace organics.

Examples of the above are summarized as shown in Table 2-2.

Table 2-2: Example Organic Soil Classifications

% of Organic Constituents (by mass)	% of Material <75 µm (passing No. 200 sieve)	Atterberg Limits	Example Group Symbol and Group Name
>30%	-	-	(PT) Fibrous PEAT
5% to 30%	>50%	PI and LL plot below A-Line	(OL) ORGANIC SILT
	>50%	PI and LL plot above A-Line	(CL) organic SILTY CLAY
	<50%	-	(SM) organic SILTY SAND
<5%	-	-	(SM) SILTY SAND, trace organics

2.5.2 Coarse-grained Soils

These soils consist predominantly of individual particles that are large enough to be visible to the naked eye (i.e., larger than 0.075 mm). They include gravel and sand. They are classified primarily based on particle size and particle distribution.

2.5.3 Fine-grained Soils

These soils consist predominantly of individual particles that are not visible to the naked eye (i.e., smaller than 0.075 mm), and are referred to as silt and clay. They are classified and described on the basis of their plasticity.

2.5.4 Note on Behavior vs. Soil Groups

The bulk behavior of a soil is not necessarily related to a soil Group Symbol or Group Name. Some coarse-grained soils may behave as cohesive and some fine-grained soils may behave as non-cohesive. In practical terms, cohesive soils are those that remain intact when rolled between the fingers (i.e., stick together when wet). Cohesive soils are also known as plastic soils.

Soil behavior is further discussed in later portions of the Technical Guidance.

3.0 ORDER OF SOIL DESCRIPTION

Table 3-1: Order of Soil Description

Item/Parameter	
1) FILL, REFUSE/WASTE, ROCK FILL, TAILINGS, TOPSOIL (if applicable).	(Unusual/Difficult Materials, see Section 13.0)
2A) USCS Group Symbol – (BLOCK LETTERS).	(Group Symbol) and Group Name,
2B) Soil Group Name; e.g., CLAYEY SILT - with the Group Name in BLOCK LETTERS.	
3) Description of Primary Constituents; Particle size, grading (for sands and gravels containing <12% fines) and shape (optional) for coarse-grained soils; Plasticity (Low, Medium, or High) for fine-grained soils.	Constituents (Based on particle size and plasticity)**;**
4) Description of Secondary Constituents; including modifiers, with an estimate of their percentage (optional), and giving their particle size for coarse-grained secondary constituents or plasticity for fine-grained secondary constituents.	
5) Other minor constituents including modifiers.	
6) Color.	General characteristics**;**
7) Weathering (if applicable).	
8) Structure: e.g., zoning, fissuring, cementation, lamination, stratification, discrete layers or features (if applicable).	
9) Sensitivity (if applicable).	
10) Contamination, staining, and odor (if applicable).	
11) Additional observations: origin or geological notes (e.g., TILL, ALLUVIUM, LOESS) or presence of cobbles/boulders, deleterious materials (e.g., with pieces of brick, asphalt, and concrete) or mineralogy (e.g., calcareous, micaceous) (if applicable).	
12) Behavior (non-cohesive or cohesive).	Material bulk behavior characteristics.
13) Field Moisture Condition (non-cohesive soils). Water Content (cohesive soils).	
14) Compactness (non-cohesive soils). Consistency (cohesive soils).	

The descriptions of the Constituents, the General Characteristics, and the Material Bulk Behavior Characteristics are separated by semi-colons **(;)**: e.g., (GP) sandy GRAVEL, some non-plastic fines**;** light brown, hydrocarbon odor**;** non-cohesive, dry, dense.

For the unusual/difficult materials of engineering and construction significance that have their own unique strata symbol (i.e., fill, refuse/waste, rock fill, tailings, topsoil), the identifying term for the origin is stated first, at the beginning of the description (see Section 13.0). It is capitalized and separated from the rest of the description by a hyphen **(–)**: e.g., FILL **–** (GP) sandy GRAVEL, some non-plastic fines; light brown, hydrocarbon odor, with pieces of brick and topsoil; non-cohesive, dry, dense.

For the other unusual/difficult materials that do not have a unique strata symbol (see Section 13.0), the identifying term is included in the General Characteristics section of the description of the material (specifically in the Additional Observations).

The order does not necessarily infer the relative importance of each item/parameter. The order reflects a systematic way of describing materials and an order compatible with reporting formats.

The level of detail of information included in descriptions will vary with the materials encountered and may vary with the needs of the project. Examples of comprehensive and acceptably abbreviated descriptions are discussed in Section 9.0.

However, it must be noted that the abbreviation or omission of information from the descriptions may limit the usefulness of the soil descriptions for subsequent use, so it may only be done with approval by the Senior Technical Reviewer.

4.0 CLASSIFICATION AND SOIL NAME

The USCS Group Symbols are assigned using the flow chart shown on Figure 2 and in Table 4-1. All proportions are by mass. Note that the portions of cobbles and/or boulders are excluded prior to using this flowchart (see Section 7.6.3).

Also note that this Technical Guidance incorporates the use of some soil names which differ from some other USCS based systems. Examples include the use of the names CLAYEY SILT and SILTY CLAY for consistency with previous Golder practice.

The use of Dual Symbols and Borderline Symbols is as shown on Figure 2 and is described in more detail below.

4.1 Dual Symbol

A dual symbol is two symbols separated by a hyphen, for example, GP-GM, SW-SC, CL-ML. For non-cohesive soils, the dual symbols must be used when the soil has between 5% and 12% fines (i.e., to identify transitional material between “clean” and “dirty” sand or gravel).

e.g., (SW-SC) SAND, some plastic fines; (GP-GM) GRAVEL, some non-plastic fines.

For cohesive soils, the dual symbol must be used when the liquid limit and plasticity index values plot in the CL-ML area of the plasticity chart (as shown on Figure 3).

e.g., (CL-ML) SILTY CLAY to CLAYEY SILT.

4.2 Borderline Symbol

A borderline symbol is two symbols separated by a slash, for example, GM/SM, CL/ML. A borderline symbol may be used to indicate that the soil has been identified as having properties that are on the transition between similar materials. In addition, a borderline symbol should be used to indicate a range of similar soil types within a stratum. Where a borderline symbol is used to describe a single sample, the classification uses the Group Name based on the predominant material only.

e.g., single sample: (GM/SM) SILTY GRAVEL; or (CL/CH) SILTY CLAY,…

For multiple samples within a stratum, the classification uses the Group Name based on both materials.

e.g., range of soil types: (GM/SM) SILTY GRAVEL to SILTY SAND,…

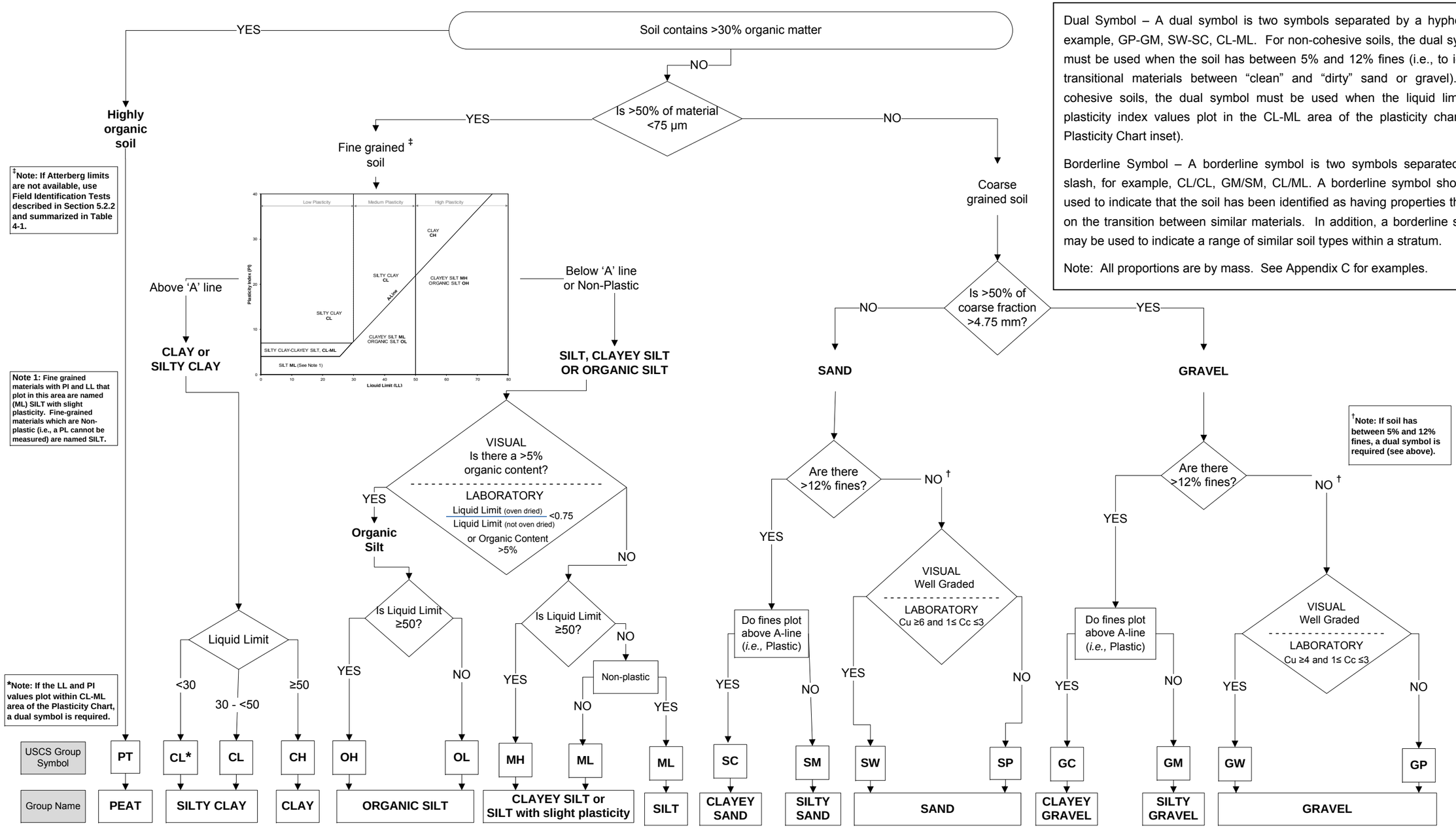


Figure 2: USCS Group Symbol and Group Name Flowchart

Dual Symbol – A dual symbol is two symbols separated by a hyphen, for example, GP-GM, SW-SC, CL-ML. For non-cohesive soils, the dual symbols must be used when the soil has between 5% and 12% fines (i.e., to identify transitional materials between "clean" and "dirty" sand or gravel). For cohesive soils, the dual symbol must be used when the liquid limit and plasticity index values plot in the CL-ML area of the plasticity chart (see Plasticity Chart inset).

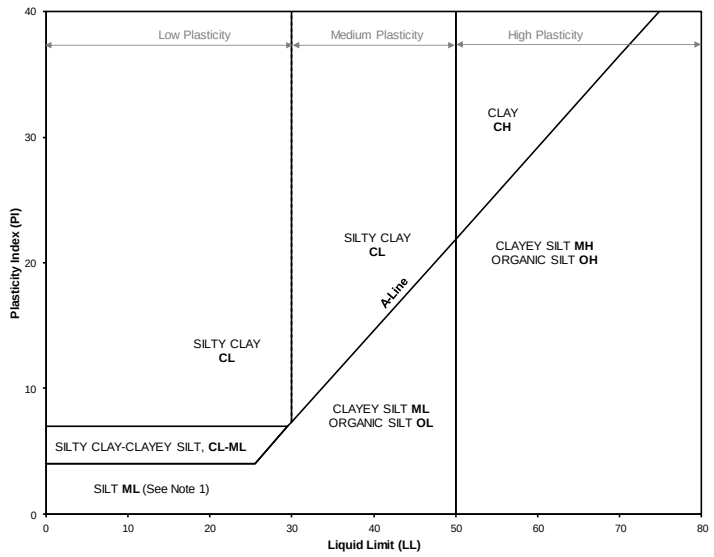
Borderline Symbol – A borderline symbol is two symbols separated by a slash, for example, CL/CL, GM/SM, CL/ML. A borderline symbol should be used to indicate that the soil has been identified as having properties that are on the transition between similar materials. In addition, a borderline symbol may be used to indicate a range of similar soil types within a stratum.

Note: All proportions are by mass. See Appendix C for examples.

Table 4-1: Summary of Soil Group Symbols and Group Names

Organic or Inorganic	Soil Group	Type of Soil		Gradation or Plasticity	$Cu = \frac{D_{60}}{D_{10}}$	$Cc = \frac{(D_{30})^2}{D_{10} x D_{60}}$	Organic Content	USCS Group Symbol	Group Name
INORGANIC (Organic Content <30% by mass)	COARSE-GRAINED SOILS (>50% by mass is larger than 0.075 mm)	GRAVELS (>50% by mass of coarse fraction is larger than 4.75 mm)	Gravels with <12% * fines (by mass)	Poorly Graded	<4	≤1 or ≥3	<30%	GP	GRAVEL
				Well Graded	≥4	1 to 3		GW	GRAVEL
			Gravels with >12% fines (by mass)	Below A-Line	N/A			GM	SILTY GRAVEL
				Above A-Line	N/A			GC	CLAYEY GRAVEL
		SANDS (>50% by mass of coarse fraction is smaller than 4.75 mm)	Sands with <12% * fines (by mass)	Poorly Graded	<6	≤1 or ≥3		SP	SAND
				Well Graded	≥6	1 to 3		SW	SAND
			Sands with >12% fines (by mass)	Below A-Line	N/A			SM	SILTY SAND
				Above A-Line	N/A			SC	CLAYEY SAND

Organic or Inorganic	Soil Group	Type of Soil	Laboratory Tests	Field Indicators (See Section 5.2.2)					Organic Content	USCS Group Symbol	Primary Name
				Dilatancy	Dry Strength	Shine Test	Thread Diameter	Toughness (of 3 mm thread)			
INORGANIC (Organic Content <30% by mass)	FINE-GRAINED SOILS (≥50% by mass is smaller than 0.075 mm)	SILTS (Non-Plastic or PI and LL plot below A-Line on Plasticity Chart below)	Liquid Limit <50	Rapid	None	None	>6 mm	N/A (cannot roll 3 mm thread)	<5%	ML	SILT
				Slow	None to Low	Dull	3mm to 6 mm	None to low	<5%	ML	CLAYEY SILT
				Slow to very slow	Low to medium	Dull to slight	3mm to 6 mm	Low	5% to 30%	OL	ORGANIC SILT
			Liquid Limit ≥50	Slow to very slow	Low to medium	Slight	3mm to 6 mm	Low to medium	<5%	MH	CLAYEY SILT
				None	Medium to high	Dull to slight	1 mm to 3 mm	Medium to high	5% to 30%	OH	ORGANIC SILT
		CLAYS (PI and LL plot above A-Line on Plasticity Chart below)	Liquid Limit <30	None	Low to medium	Slight to shiny	~ 3 mm	Low to medium	0% to 30% **(see Note 2 below)**	CL	SILTY CLAY
			Liquid Limit 30 to <50	None	Medium to high	Slight to shiny	1 mm to 3 mm	Medium		CL	SILTY CLAY
			Liquid Limit ≥50	None	High	Shiny	<1 mm	High		CH	CLAY
HIGHLY ORGANIC SOILS (Organic Content >30% by mass)		Peat and mineral soil mixtures							30% to 75%	PT	SILTY PEAT, SANDY PEAT
		Predominantly peat, may contain some mineral soil, fibrous, or amorphous peat							75% to 100%		PEAT



Note 1 – Fine-grained materials with PI and LL that plot in this area are named (ML) SILT with slight plasticity. Fine-grained materials which are Non-plastic (i.e., a PL cannot be measured) are named SILT.
Note 2 – For soils with <5% organic content, include the descriptor "trace organics." For soils with between 5% and 30% organic content include the prefix "organic" before the Primary name.

* Dual Symbol – A dual symbol is two symbols separated by a hyphen, for example, GP-GM, SW-SC and CL-ML. For non-cohesive soils, the dual symbols must be used when the soil has between 5% and 12% fines (i.e., to identify transitional material between "clean" and "dirty" sand or gravel). For cohesive soils, the dual symbol must be used when the liquid limit and plasticity index values plot in the CL-ML area of the plasticity chart (see Plasticity Chart at left).

Borderline Symbol – A borderline symbol is two symbols separated by a slash, for example, GM/SM, CL/ML. A borderline symbol should be used to indicate that the soil has been identified as having properties that are on the transition between similar materials. In addition, a borderline symbol may be used to indicate a range of similar soil types within a stratum.

The soil Group Name corresponding to the USCS symbol is **CAPITALIZED**. Other adjectives denoting secondary constituents are **lower cased**. The description for unusual/difficult materials (see Section 13.0) is also CAPITALIZED and placed at the beginning of the description, **before** the Group Symbol.

CORRECT	INCORRECT
sandy SILTY CLAY (SILTY CLAY is the primary soil name, sandy denotes a secondary component)	SANDY SILTY CLAY, sandy silty CLAY
sandy GRAVEL (GRAVEL is the primary soil name, sandy denotes a secondary component)	SANDY GRAVEL
gravelly CLAYEY SAND (CLAYEY SAND is the primary soil name, gravelly denotes a secondary component)	GRAVELLY CLAYEY SAND, gravelly clayey SAND
FILL – (GP) sandy GRAVEL; brown, with pieces of brick and topsoil; …	(GP) sandy GRAVEL; brown, with pieces of brick and topsoil (FILL); …

5.0 DESCRIPTION OF CONSTITUENTS

The description of constituents follows the same basic principles and uses the same terminology, whether the constituent is Primary, Secondary, or Minor. Differences between the description of Primary, Secondary, and Minor constituents include the order of the description. For this section, the intent is to gain an understanding of the correct methods and terminology for identifying constituents for each soil group.

5.1 Coarse-grained Constituents

5.1.1 Particle Size

Coarse-grained constituents are identified in accordance with the terminology in Table 5-1. Identification is carried out initially using visual methods in the field and may be confirmed using a particle size distribution test(s) in the laboratory.

Table 5-1: Identification of Particle Size

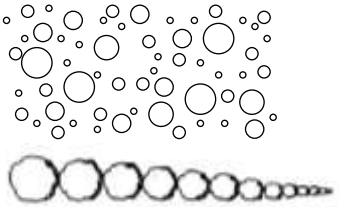
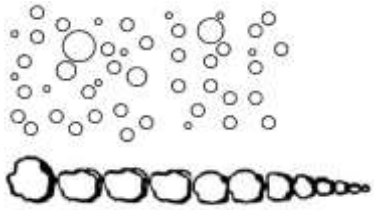
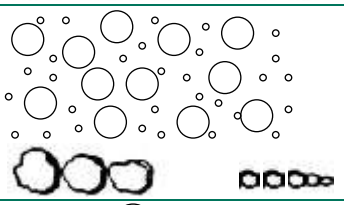
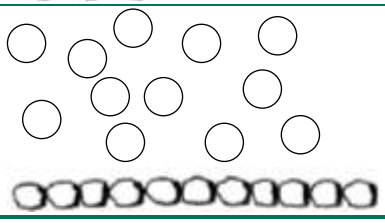
Soil Constituent	Particle Size Descriptor	Millimeters	Inches (US Std. Sieve Size)
GRAVEL	Coarse Fine	19 – 75 4.75 – 19	0.75 – 3 (4) – 0.75
SAND	Coarse Medium Fine	2.00 – 4.75 0.425 – 2.00 0.075 – 0.425 (75 µm – 425 µm)	(10) – (4) (40) – (10) (200) – (40)

5.1.2 Particle Size Distribution

The assessment of particle size distribution of sand and gravel is initially carried out using visual-manual methods in the field, and may be confirmed using a particle size distribution test(s) in the laboratory (ASTM D422). When visually assessing the distribution of particle sizes by mass, it should be noted that larger particles weigh more relative to the same volume of smaller particles. Although a well graded soil has a wide range of different sized particles, it will have fewer large particles than small.

For sand and gravel sized constituents, the distribution of sizes by mass is assessed in accordance with Table 5-2. Particle size distribution is not applicable to cobbles and boulders; for these materials, describe the specific size range, where possible. Example grain size distribution curves for coarse-grained materials (along with the accompanying Group Symbol) are provided in Appendix A (Ministry of Environment, 1982).

Table 5-2: Visual Identification of Particle Size Distribution

Particle Size Distribution Descriptor	Sub-category	Description	Visual Example
Well Graded		Even distribution of particle sizes	
Poorly Graded		Uneven distribution of particle sizes	
	Gap Graded	Intermediate particle sizes absent	
	Uniformly Graded	Primarily one particle size	

Examples of the correct use of these terms and the accompanying range of particle sizes are:

- ***Fine- to coarse-grained***, well graded (Note: this could also be poorly graded if there is an uneven distribution of particle sizes across the range)
- ***Fine-grained***, uniformly graded
- ***Fine- to medium-grained***, well graded (even though this is missing coarse-grained sizes, it may be well graded across the fine to medium range, which is an example of why both the particle size AND the size distribution are described)
- ***Fine- and coarse-*grained**, gap graded (may also simply be described as poorly graded, as gap graded is a subset of poorly graded)

Note that when laboratory particle size distribution test results are available, well graded materials are defined by the coefficient of uniformity (Cu) and the coefficient of curvature (Cc) as shown on Figure 2 and in Table 4-1.

"Well Graded" is geologically equivalent to "Poorly Sorted," and "Poorly Graded" is geologically equivalent to "Well Sorted." However, the terms "Poorly Sorted" or "Well Sorted" should not be used in the soil descriptions.

5.1.3 Particle Shape and Angularity

The shape and angularity of coarse-grained constituents is assessed in accordance with Table 5-3. These terms may be omitted at the discretion of the Senior Technical Reviewer. The assessment of particle shape and angularity is carried out using visual methods in the field and is generally not confirmed using a laboratory test method. The terms can be combined when appropriate (e.g., angular, flat). The shape terms flat or elongated are typically only used for gravel. Gravels can be flat and elongated if both criteria are met.

Table 5-3: Identification of Angularity and Particle Shape

	Term	Description	Visual Example
Angularity	**Rounded**	Smoothly curved sides No edges Smooth or polished surfaces	
	Subrounded	Plane sides Well-rounded edges Partially polished surfaces	
	Sub-angular	Plane Sides Partially rounded edges Unpolished surfaces	
	Angular	Plane sides Sharp edges Unpolished surfaces	
Shape (typically only applicable to gravel)	**Flat**	Width to thickness ratio >3	
	Elongated	Length to width ratio >3	

5.2 Fine-grained Constituents

The fine-grained constituents of a soil are identified based on assessment of their plasticity. When a soil is in a "plastic state" (PL<w<LL), it means the soil can be molded and will retain the molded shape upon drying. When the soil becomes either too wet (w>LL) or too dry (w<PL), it can no longer be molded and has passed out of the plastic state and into a liquid or solid state, respectively. A plastic soil has the ability to remain in the plastic state over a range of water content. A non-plastic soil may only exhibit plastic behavior over a very narrow range of water content. A fine-grained soil is identified by assessing the range of water content over which it can behave in a plastic state; this range will vary considerably for different fine-grained materials.

Therefore, the important distinctions in the assessment of the plastic behavior of a fine-grained soil are the water content boundaries within which plastic behavior occurs. These boundaries are known as the Atterberg Limits and are defined as follows:

- Plastic Limit (PL): the water content of a soil at the boundary between the semi-solid and plastic states. This limit is reached when the soil starts to crumble when rolled into thin (3 mm diameter) threads. If the water content is decreased below this limit, it cannot be molded.
- Liquid Limit (LL): the water content of a soil at the boundary between the plastic and liquid states. If the water content is increased above this limit, the soil becomes a thick viscous slurry.
- Plasticity Index (PI): the range of water content over which a soil behaves plastically, the "plastic range." Numerically, it is the difference between the Liquid Limit and the Plastic Limit.

The plasticity is largely influenced by the mineralogy of the fine-grained constituents of the soil, rather than grain-size of the individual particles and, on this basis, the identification is through a series of behavioral tests rather than assessment of particle size distribution. The behavioral tests for plasticity include laboratory and field identification tests with the common theme that they are carried out to assess how plastic the soil is; that is, to assess the range of water content over which the soil exhibits plastic behavior. The laboratory tests (i.e., Atterberg limits) and field identification behavioral tests used for identification of fine-grained constituents are described herein.

5.2.1 Atterberg Limits Testing

Atterberg limits testing is carried out in the laboratory according to *ASTM D4318 Liquid Limit, Plastic Limit and Plasticity Index of Soils*, or equivalent approved laboratory testing methods. Detailed discussion of the laboratory testing method is outside the scope of this Technical Guidance; however, the process generally involves removing any particles larger than fine sand (any particles sized larger than the 425 µm [No. 40] sieve) from the bulk soil sample and testing the remaining soil. The Atterberg limits testing is carried out to determine the Liquid Limit (LL), Plastic Limit (PL), and Plasticity Index (PI). While ASTM D4318 suggests that the LL, PL, and PI be reported to the nearest whole number, omitting the percent (%) designation, for some clay soils it may be necessary to plot the results to one decimal place to properly differentiate between immediately adjacent soil classifications. It is noted, however, that this degree of refinement is not recommended for most soils and where results are plotting near the transition between different soil types, the use of a Borderline Symbol (see Section 4.2) may be more appropriate.

The Liquid Limit and Plasticity Index are then plotted on a Plasticity Index versus Liquid Limit Chart (also referred to as a Plasticity Chart, a Casagrande Chart, or an A-chart). The chart was originally derived by Casagrande and has since been modified by various authors. This Technical Guidance has adopted the version shown on Figure 3, consistent with Casagrande (1947) and CFEM (2006). The location where the test results plot on the graph indicates whether the fine-grained constituent is SILTY CLAY, CLAY, CLAYEY SILT or ORGANIC SILT (depending on organic content), or SILT with slight plasticity.

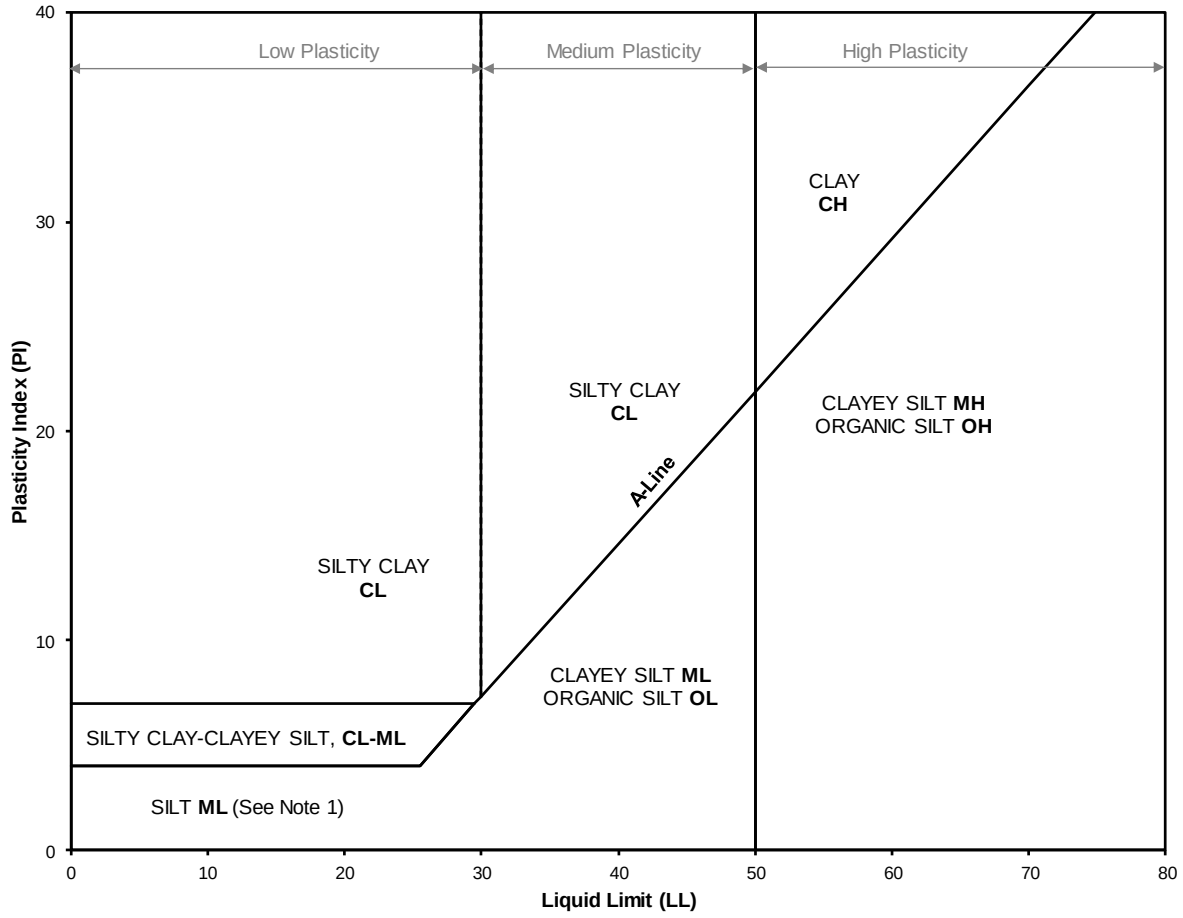


Figure 3: Plasticity Index vs. Liquid Limit Chart

Note 1 – Fine-grained materials with PI and LL that plot in this area are named (ML) SILT with slight plasticity. Fine-grained materials which are non-plastic (i.e., when a PL cannot be measured, a PI cannot be calculated, laboratory testing will typically report PL = NP or not testable) are named (ML) SILT.

5.2.2 Field Identification Tests

The plasticity of the fine-grained constituents can be identified in the field using a series of tactile identification tests. The tests are mainly applicable to the fine-grained portion of the soil and must be performed over a wide range of water content. Sand and gravel in the sample will affect the results and, if these particles are present in

significant quantity and unable to be removed, their effect should be considered when assessing the results. The presence of sand and/or gravel is captured when describing the bulk behavior of the materials (Section 8.0).

Some or all of the following tests may be carried out in the field depending on how quickly an adequate level of confidence can be reached in identifying the soil (i.e., some soils may be clearly identified using just one or two tests, while others may be more difficult to assess and require the entire suite of tests). The tactile test results are assessed to estimate the constituents and rate the plasticity in accordance with Table 4-1. The various test methods are summarized as follows:

- Dilatancy:

Mold a small part of soil to the consistency of putty, adding water as needed. Place the soil in the open palm of one hand and shake horizontally, striking vigorously against the other hand several times. Observe the appearance of the soil after shaking (e.g., look for presence of water on the surface) and then squeeze slightly by cupping the hand. The reaction is the speed with which water appears while shaking and then disappears while squeezing, and is rated as “rapid,” “slow,” or “none.”

- Dry Strength:

Mold a sample of soil to the consistency of putty, adding water as needed into a ball about 12 mm (1/2 inch) in diameter. Allow to dry completely using sun or air drying (or place in warm area). Once the sample is completely dried, test its dry strength by pressing between the fingers. The strength is rated as:

- High dry strength – sample cannot be damaged or crushed at all or only very slightly.
- Medium dry strength – sample can be crushed to powder with great or considerable pressure.
- Low dry strength – sample can be readily crushed to powder with gentle pressure.

■ Shine Test:

A sample of cohesive soil is cut with a knife blade, or a smooth object such as your fingernail is used to stroke the sample and create a smooth surface. The surface created on the sample is observed closely under direct light. Soils with high plasticity typically have a shiny appearance, and soils with low plasticity have a dull appearance.

Shininess may be rated as follows:

- Shiny
- Slight to shiny
- Slight
- Dull to slight
- Dull
- None

Do not mistake the shininess of soils that contain mica for the shininess created by the colloidal content of clays. It is important to avoid mistaking the appearance of free water on the sample for shininess.

■ Thread Diameter (Plasticity):

Roll a moist ball of soil to the smallest thread possible without crumbling. Measure/estimate the diameter of the thread.

- Non-plastic: Thread diameter is >6 mm (1/4 inch) or a thread cannot be rolled at any water content
- Low: Can roll a thread between 3 mm and 6 mm (1/8 inch and 1/4 inch) diameter
- Medium: Can roll a thread between 1 mm and 3 mm (1/16 inch and 1/8 inch) diameter
- High: Can roll a thread less than 1 mm (1/16 inch) diameter

- Toughness:

Mold a soil sample to the consistency of putty, adding water or drying as needed. Roll out the sample on a flat surface or between the hands to form a 3 mm diameter thread. Refold and continue to roll out the sample into a thread until the process reduces the sample water content to the point that the thread just crumbles at this diameter. This indicates that the sample is at the plastic limit. After the thread crumbles, lump all the pieces together and knead until the lumped sample crumbles. Note the toughness of the material during kneading. The toughness is rated as:

- Low toughness – slight pressure is required to roll the thread near the plastic limit. The thread and the lump are weak and soft.
- Medium toughness – medium pressure is required to roll the thread to near the plastic limit. The thread and the lump have medium stiffness.
- High toughness – considerable pressure is required to roll the thread to near the plastic limit. The thread and the lump have high stiffness.

These tests may also be used to estimate the plasticity of fines in a coarse-grained soil.

6.0 SECONDARY AND MINOR CONSTITUENTS

Guides for visual estimation of proportions are presented on Figure 4.

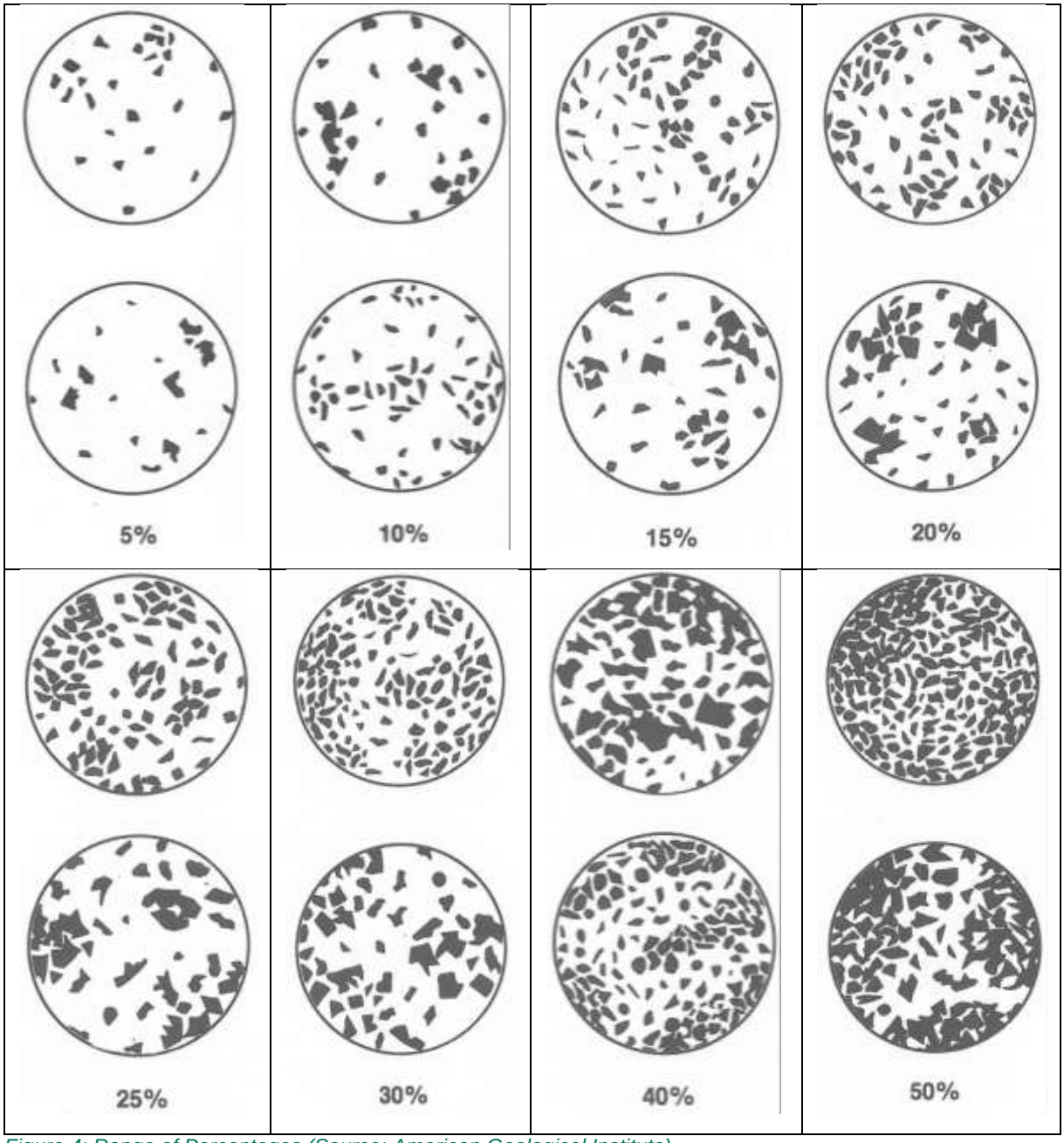


Figure 4: Range of Percentages (Source: American Geological Institute)

The modifiers shown in Table 6-1 are used to describe the proportions of secondary and minor constituents after assigning the soil Group Symbol and Group Name in accordance with Figure 2 and/or Table 4-1. Group Names (i.e., SILTY CLAY or CLAYEY SAND) are not assigned using the modifiers below.

Table 6-1: Modifiers for Secondary and Minor Constituents

Constituent	% (by mass)	Modifier
Minor	≤5	Use “trace” or omit.
	>5 to 12	Use “some.”
Secondary	>12 to 35	Prefix primary soil name with “gravelly, sandy, SILTY or CLAYEY” as applicable. Note that the terms SILTY or CLAYEY are prescribed using Table 4-1.
	>35	Use “and” to combine major constituents (i.e., SAND and GRAVEL, SAND and SILT, SILTY CLAY and SAND).

When describing a coarse-grained soil that contains less than 12% fines, use “trace” or “some” to describe the estimated or measured proportion of fines, and describe the plasticity of the fines only if possible (i.e., “some medium plasticity fines” or “trace fines”). Note the requirements for use of Dual Symbols as described on Figure 2 and in Section 4.1.

Since fine-grained soils are classified based on plasticity, **it is not appropriate to estimate the proportion of silt in a material classified as CLAY, or the proportion of clay in a material classified as SILT**. Materials classified as SILTY CLAY and CLAYEY SILT are classified that way based on plasticity. **SILT and CLAY are not differentiated by particle size in this system.** A laboratory hydrometer test is used if the particle size distribution of the fines must be established.

When “and” is used, it is usually preferable to place the description of the particle sizes ahead of the constituent name (i.e., fine to coarse SAND and fine GRAVEL).

CORRECT	INCORRECT
CLAY, some fine to coarse sand, trace fine gravel.	CLAY, some silt, some fine to coarse sand, trace fine gravel. (See note 1)
SILTY CLAY and fine SAND, some gravel.	CLAY and sandy SILT, some gravel. (See note 2)
fine to coarse SAND and fine GRAVEL, some low plasticity fines.	fine to coarse SAND and fine GRAVEL, some silt, trace clay. (See note 2)
sandy CLAYEY SILT, trace gravel.	clayey SANDY SILT, trace gravel.
SILTY GRAVEL, fine to coarse, some fine to coarse sand.	SILTY GRAVEL, fine to coarse, some fine to coarse sand, trace clay. (See note 3)
CLAYEY SAND, fine to medium, trace fine gravel.	CLAYEY SAND, fine to medium; some silt, trace fine gravel. (See note 3)

Notes:
1) As fine-grained materials are classified and named based on plasticity, fine-grained constituents cannot be a minor constituent of a fine-grained soil.
2) Fines are classified and described by plasticity, so cannot be differentiated in this manner.
3) The fine fraction of a coarse-grained material is classified as either SILTY or CLAYEY based on the aggregate plasticity of the fines.

7.0 GENERAL CHARACTERISTICS

The following general characteristics are important information that should be included in the overall description of soils, where applicable.

7.1 Color

Color may be an important indicator of soil properties or subsurface conditions. Organic soils are often indicated both by color and odor. In temperate climates where chemical weathering and oxidization is not rapid, the change of soils from brown tones to grey is an indicator of the historical groundwater level. The color of the fresh soil sample or exposure should be described. If the color represents a dry condition, this factor should be noted.

In most cases, as appropriate, only the primary color modified by a single adjective needs to be used (e.g., grey, dark grey, oxidized red-brown). The use of the suffix "-ish" may be used if appropriate (e.g., reddish-brown). However, where color is a significant factor in assessing the derivation/properties of a soil, color should be formally determined and described in accordance with the Munsell Soil Color Charts (Munsell, 2009), which is a classification system developed with the United States Department of Agriculture (USDA) Natural Resources Conservation Service specifically for field classification of geological materials.

If the sample contains layers or zones of varying color, this should be included in the description of each individual sample, although not necessarily repeated in the summary description of the overall deposit.

Soil color should be described as "mottled" if there are localized spots, streaks, or splotches of one or more different colors or shades within a soil mass of another predominant color.

7.2 Weathering

A hardened crust can be present at the surface of clay deposits as a result of desiccation, frost action, and weathering in general. Within the weathered crust, the water content can be reduced and the undrained shear strengths (of the intact, non-fissured portions) can be much higher than those measured in the unweathered and intact clay underlying the crust. The presence of this portion of the deposit is relevant to downstream engineering and, if present, its thickness and/or transition to underlying unweathered material should be noted.

7.3 Structure

Structure is the term applied to features or discontinuities within the overall soil mass. Structure is often an indication of the method of deposition or geologic history of the soil. Most importantly, the structure may greatly change the characteristics of the soil mass from that indicated by individual samples and may govern the engineering behavior of a soil.

Soil structure must be noted in the field as the evident structure in soil samples is often lost during transportation. Soil structures greater in size than that visible in borehole samples can be more evident in test pit investigations, in construction excavations, and in tunnels; where evident, the soil structure, size, orientation, and dip should be noted. The following terms are used in describing soil structure. Note that some terms can only be assessed when logging test pits, excavation faces, or natural exposures, and are not appropriate for borehole logging.

7.3.1 Zoning

Table 7-1: Zoning Terms and Descriptions

Term	Description
Heterogeneous	Soil mass is of non-uniform, variable composition or structure.
Homogeneous	Soil mass is of uniform composition or structure.
Repetitive Structures	
Laminated	Closely spaced, alternating layers of differing soils and/or differing colors or shades of soils of similar gradation, usually arranged in a regular pattern. The following terms may be used: Thinly laminated – mean spacing less than 6 mm (1/4 inch) Thickly laminated – spacings of 6 mm to 20 mm (1/4 inch to 3/4 inch)
Stratified, Layered, or Bedded	Differing soils or visible variations in soil constituents or color arranged in layers, generally but not necessarily parallel to one another. The following terms may be used, depending on bed spacings: Very thinly bedded – 20 mm to 60 mm (3/4 inch to 2-1/4 inches) Thinly bedded – 60 mm to 200 mm (2-1/4 inches to 8 inches) Medium bedded – 200 mm to 600 mm (8 inches to 2 feet) Thickly bedded – 600 mm to 2 m (2 feet to 6 feet) Very thickly bedded – over 2 m (6 feet)
Varved	A laminated soil consisting of two distinct soils (usually clay and silt) occurring in a regularly repeating pattern resulting from seasonal variations in sediment load in a lacustrine environment.
Discrete Layers or Features	
Lens	An inclusion of a different soil type within surrounding soils, which thins out laterally (horizontally) and may not be continuous over any significant distance. Typically identified by test pits or correlation between boreholes.
Parting	Paper thin separation of one soil type by another. Usually applied to fine-grained soils.
Pocket	A different soil type of very limited thickness or lateral extent (a small lens).
Seam	A soil layer of considerable extent but with a thickness of less than about 10 mm (1/2 inch).
Layer	A different soil type with a thickness between 12 mm and 300 mm (1/2 inch to 1 foot)
Stratum	A different soil type with a thickness greater than 300 mm (1 foot)

Examples of a varved and laminated soil are shown in Appendix B. For these soils, the individual laminae should be described separately, if possible:

e.g., (CH and ML) CLAY and SILT; grey and light brown, varved; w>PL, cohesive, soft.

e.g., (CL, CH, ML and SM) SILTY CLAY, CLAY, SILT, and SILTY SAND; grey and brown thinly laminated; w>PL, cohesive, soft.

7.3.2 Fissures

Fissured: Generally applied to dried, overconsolidated, or weathered fine-grained soils (silts and clays) containing cracks or physical discontinuities that can be vertical, horizontal, or inclined. Fissuring is indicated when otherwise strong soil breaks along definite or pre-existing fracture planes. The degree of fissuring may be described as:

- Highly fissured – fissures spaced 10 mm (1/2 inch) or closer over most of the interval described.
- Moderately fissured – two or more fissures occur per 150 mm (6 inches) length of sample or exposure.
- Slightly fissured – only random, infrequent, and narrow fissures (average distance between fissures consistently greater than 75 mm [3 inches]).

Friable, Blocky, Platy: Otherwise cohesive soil breaks into small (friable), larger (blocky), or thin, plate like (platy) fragments with little effort.

Slickensided: Polished or striated surfaces resulting from movement of a material block relative to the adjacent block(s); often an indication of an existing failure or slip surface. If continuous slickensided shear zones are found, an estimate should be made of their angle in relation to the horizontal plane.

7.3.3 Cementation

Soils or defects within soils may be held together by cementing materials, often chemical precipitates or deposits within the overall soil mass.

If the cementing agent allows fracturing by hand of the particle aggregations when saturated, it is described as weakly cemented.

If the cementing agent prevents fracturing of the saturated aggregations, it is described using the guidance for rock.

The nature of the cementing agent should be identified if possible from its appearance, strength, reaction to acid, etc. The degree of cementation may be described as weak or strong. Since calcium carbonate is the most common cementing agent, a note of its presence on the basis of the reaction with dilute hydrochloric acid (HCl) is important. The intensity of the HCl reaction should be described as non-reactive (NR), weak reaction (WR), or strong reaction (SR).

7.3.4 Other Defects

Other defects to be noted may include:

- Roots
- Root holes or tubes (hollow)
- Tube casts (solid material filling old tube)
- Surface cracks

Defects may be re-cemented and therefore may be stronger than the parent soil. The approximate dimensions and spacing of defects should be noted.

7.4 Sensitivity

Sensitivity (S_t) can be an important characteristic of fine-grained soils. It is defined as the ratio of undisturbed, or intact, undrained shear strength to remolded undrained shear strength (i.e., $S_t = s_{u(und)}/s_{u(rem)}$) and can be determined by laboratory testing or in the field by the vane test. Classes of sensitivity may be defined as follows (CFEM, 2006):

Low sensitivity	S_t <2
Medium sensitivity	2 < S_t <4
Sensitive	4< S_t <8
Extra-sensitive	8< S_t < 16
Quick clay	S_t >16

An indication of the sensitivity should be included in the description when a soil is noted to have a large reduction in strength upon remolding. The most sensitive types of clay, often termed “quick” clay, are commonly found in eastern North America in particular around the St. Lawrence River lowlands.

If applicable, the natural water content relative to the liquid limit (i.e., w>LL) may also be used as an indicator of sensitivity.

7.5 Contamination

If hazardous materials are suspected to be present, immediately contact and inform the Field Supervisor, Health & Safety Officer, or Project Manager, as appropriate health and safety procedures must be established and utilized (see Section 11.1).

Description of soils obtained from a subsurface investigation carried out for environmental purposes should be in accordance with the classification described in this Technical Guidance. Where downstream engineering is/may be required, such as for follow-up site remediation, the subsurface investigation should be carried out consistent with geotechnical/foundation investigation methods/guidelines. The indicators of contamination should be described in as much detail as possible, as noted in Section 11.0.

7.6 Additional Observations

7.6.1 Geological Formation/Regional Name

Exercise caution before adding the geological name to field logs. If there is doubt or inadequate information to determine the actual geologic or regional name, do not specify; “possibly” may be added to the designation, if appropriate.

In many situations it is preferable to show the geological name on the Record of Borehole rather than the Field Log.

When known, the geological name of the deposit or the recognized regional name of the soil may be applied to the soil strata or units at the end of the General Characteristics section of the soil description, **capitalized and in brackets.** This includes the use of the term (TILL).

Soils should not be described using slang or other inappropriate terms such as “hardpan,” “gumbo,” or “blue clay.”

7.6.2 Definitions of General Geological Descriptions

- Fill: Any material placed by mankind. Fill can be comprised of a variety of materials (see Section 13.1).
- Lacustrine deposits: Soils deposited in lakes.
- Alluvial soil: Any soil deposited by running water. Such soils usually contain a variety of materials, including fine particles of silt and clay and larger particles of sand, rounded gravel, and cobble sizes.

- Colluvial soil: A non-stratified mixture of silt, clay, and angular sand, gravel, cobble, and boulder size material accumulated at the foot of a slope or on the slope itself; deposited chiefly under the influence of gravity.
- Till: A non-stratified random mixture of clay, silt, sand, gravel, cobbles, and boulders deposited by glaciers. Alternating layers of clayey till and till containing boulders are possible.
- Outwash: A stratified alluvial soil transported and deposited by a glacial meltwater stream.
- Loess: A uniform Aeolian (wind) deposit of silty material having an open structure and relatively high cohesion due to a clay matrix or cementation by calcareous material at grain contacts. A characteristic of loess deposits is that they display nearly vertical slopes.
- Marl: A calcium carbonate or lime-rich mud that contains variable amounts of clays and silt. Marl occurs as loose, earthy deposits consisting chiefly of an intimate mixture of clay and calcium carbonate, formed under freshwater conditions containing 35-65% clay and 65-35% carbonate.
- Residual soils: Soils that have formed in place due to decomposition of rock. Shales form residual clays. Limestones typically form clay soils. Granitic rocks typically form silty sand with angular sand grains.

7.6.3 Cobbles and Boulders

The sizes of cobbles and boulders are defined in Table 7-2. Rock fill of cobble/boulder sizes are described as suggested in Section 13.0.

Table 7-2: Identification of Particle Size

Constituent	Particle Size	
	Millimeters	Inches (US Std. Sieve Size)
Boulders	>300	>12
Cobbles	75 – 300	3 – 12

Soils are typically classified without considering the proportion by mass of cobbles and boulders, so that the soil between cobbles/boulders is described in the same way as if the cobbles/boulders were not present.

However, the presence of cobbles and boulders within a soil deposit may be very significant, particularly from the perspective of construction. Consequently, their presence **must** be noted. For instances where cobbles and boulders are not confirmed by coring or sampling, their inferred presence should be noted from auger grinding or split-spoon refusal.

As a standard guideline, cobbles and boulders or “nests” of cobbles and boulders, if encountered in boreholes, test pits, or exposures should be individually noted and recorded on the appropriate logging sheet. Vague, unquantified descriptions such as “occasional,” “some,” and “numerous” should not be used on field records.

The dimensions of the individual cobbles and boulders or thickness of cobble and boulder “nests” should be measured and specifically reported. Where cobbles and boulders are too numerous to report every occurrence,

a range of sizes and an estimate of their percentage relative to the bulk soil mass may be reported. Where it is not possible to measure the diameter of cobbles and boulders, such as in a borehole, their presence should be noted by depth or elevation.

Cobbles and boulders are not assigned a USCS Group Symbol or Group Name. In situations where cobbles and boulders are sporadically encountered within a soil matrix, the classification of the soil matrix constitutes the basis of the description with the presence of cobbles and boulders noted:

> e.g., (CL) SILTY CLAY, some sand, trace gravel; brown, with cobbles and boulders, (TILL); cohesive, w<PL, stiff. Cobbles and/or Boulders inferred from auger resistance between depths of about 8 m and 9 m.

In situations where cobbles and boulders comprise the predominant portion of a layer or stratum (i.e., in a cobble or boulder "nest"), the description will begin with the identifying term "Cobbles/Boulders." The description will then be comprised of the general characteristics of the particles including color and sizes (where test pitting or coring is carried out and the particles are visible or core samples are obtained) or indicate only the range of depths over which the cobbles and boulders are inferred from auger resistance or split-spoon refusal (where test pitting or coring is not carried and/or where samples are not obtained).

> e.g., COBBLES/BOULDERS – light brown to dark grey, with particle sizes ranging from about 95 mm to 400 mm.

> e.g., COBBLES/BOULDERS – inferred from auger resistance between depths of about 8.5 m and 9.1 m.

In some situations where the composition of a deposit is variable and includes a mixture of cobbles and boulders within a soil matrix in similar proportions, the constituents of the deposit could be described separately:

> e.g., **MIXTURE OF SOIL** and **COBBLES/BOULDERS** (estimate % if appropriate)
>
> SOIL is: (SM) gravelly SILTY SAND, trace clay; brown; non-cohesive, moist, compact.
>
> COBBLES/BOULDERS are: Subrounded granite up to 600 mm in size.

The proportions of the cobbles/boulders are only estimated when large exposures (test pits, dozer trenches, natural or pre-existing cuts or exposures) allow reasonable estimation. Cobble/boulder proportions cannot be estimated from boreholes, but their presence and inferred size should be noted as appropriate.

CORRECT	INCORRECT
(SM) SAND, fine to coarse, and GRAVEL, fine to coarse; brown with cobbles (~20% by volume);	coarse BOULDERS and fine COBBLES
Mixture of SOIL and COBBLES/BOULDERS (~50% by volume) SOIL is: (SM) gravelly SILTY SAND, fine to coarse; grey brown; non-cohesive, moist, dense. COBBLES/BOULDERS are: subrounded, up to 600 mm in size.	gravelly SILTY SAND and small BOULDERS
500 mm diameter BOULDER at 1.6 m to 2.1 m (for a test pit or core sample).	contains BOULDERS
COBBLES AND/OR BOULDERS inferred from auger resistance between depths of about 8 m and 9 m.	Numerous COBBLES and/or BOULDERS

8.0 BEHAVIOR BASED PARAMETERS

8.1 Cohesive vs. Non-cohesive

In practical terms, cohesive soils are those that remain intact when rolled between the fingers (i.e., stick together when wet). The word cohesive also usually describes a soil that has an undrained shear strength.

Non-cohesive (cohesionless or non-plastic) soils may include both coarse-grained and/or fine-grained constituents, and are soils that fall apart when not confined.

The behavior will be obvious for some materials. For example, coarse-grained soils with less than 12% fines (GP, GW, SP, and SW) will always be non-cohesive, and high plasticity clay (CH) containing less than 12% coarse particles will always be cohesive. The behavior of materials containing a mixture of constituents is often not implicit in the classification.

Regardless of whether a material's behavior is implicit or not, for clarity, the field logs and Records of Borehole shall identify and include in the description whether the soil mass (after removing cobbles and boulders) is cohesive or non-cohesive.

8.2 Compactness/Consistency

8.2.1 Non-cohesive Soils – Compactness

The compactness of non-cohesive (cohesionless or non-plastic) soils is described in qualitative terms based on the results of field penetration tests. Most commonly, the compactness is evaluated based on the 'N'-values recorded in the Standard Penetration Test (SPT) carried out as part of borehole investigations, in accordance with the typical SPT 'N' ranges provided in Table 8-1 (after Terzaghi and Peck, 1967). The compactness can also be evaluated using other in situ penetration test methods (e.g., Cone Penetration Test [CPT], Dynamic Cone Penetration Test [DCPT], or Becker Penetration Test [BPT]), provided the penetration data are converted to equivalent SPT 'N'-values.

In test pit investigations, the compactness may be estimated based on resistance to shoveling or based on the resistance to penetration by a steel bar, in accordance with the descriptions in Table 8-1. In these instances, the test pit log should state the following: "The compactness condition has been inferred by subjective field identification methods only. SPT 'N'-values not obtained."

In boreholes, in the absence of in situ penetration test data, a description of the compactness of non-cohesive soils should not be provided. However, a description could be proposed on field logs based on the observations of resistance to drilling penetration (e.g., "inferred to be loose to very loose based on ease of auger advance" or "inferred to be dense to very dense based on slow drilling rate/auger grinding").

It should be understood that the "compactness" descriptions are defined based on the relative resistance of the soil to penetration, which is a function of a number of conditions, including the relative density of the soil, the soil particle size relative to the penetration equipment, and the overburden stress conditions. The SPT 'N'-values in Table 8-1 have not been corrected for the effects of overburden pressure. Consequently, an increase in the degree of apparent "compactness" based on penetration resistance can be expected due to increasing overburden pressure within a soil of uniform composition and relative density. Furthermore, the ranges of SPT 'N'-values in Table 8-1 are most applicable to SPTs carried out in sands. Lower 'N'-values, and therefore a lower apparent "compactness," can be expected from SPTs carried out in silts and sand/silt mixtures, and higher

'N'-values, and a corresponding higher apparent "compactness," can be expected from SPTs carried out in gravels and sand/gravel mixtures, assuming similar relative densities and overburden pressures. Cobbles and coarse gravel, in particular, can result in high 'N'-values that are not representative of the compactness condition of the surrounding soil mass; the presence of such particles (observed or inferred) should always be noted on the field logs and affected 'N'-values should not be used to describe the compactness condition.

The SPT 'N'-values in Table 8-1 can be assumed to be N_{60} values (i.e., they are representative of tests conducted such that the average energy transferred by the hammer to the split-spoon sampler is about 60% of the theoretical free-fall energy of the hammer). Modern automatic-trip hammers can deliver significantly higher energy than older systems that use donut hammers or safety hammers operated using a rope and cathead. Therefore, it is important to make note of the type of hammer system used for each investigation and record the hammer mass (typically 63.5 kg or 140 pounds), to allow proper interpretation of the field data.

Soil disturbance to a depth greater than 0.15 m (6 inches) below the bottom of the borehole can result in low SPT 'N'-values that are not representative of the compactness of the surrounding soil mass (e.g., heaving of sands inside of drill casing due to water pressure imbalance). The drilling method should be selected to minimize the risk of this occurring (e.g., mud rotary or wash rotary methods). In addition, the Field Representative should ensure that soundings of the depth to the bottom of the borehole are carried out inside of the drill casing prior to SPT to check if heaving has occurred, when appropriate, and to record this information on the field borehole logs. This can also be achieved practically by observing the stick-up of the sampling rods.

Table 8-1: Compactness of Non-cohesive Soils

Description	SPT 'N' (blows/foot)	Field Identification of Soil Exposures
Very Loose	0 – 4	Easily penetrated with shovel handle.
Loose	4 – 10	Easily penetrated with 12 mm (1/2 inch) rebar pushed by hand. Easily excavated with hand shovel.
Compact	10 – 30	Easily penetrated with 12 mm (1/2 inch) rebar driven with 2.3 kg (5 pound) hammer. Difficult to excavate with hand shovel.
Dense	30 – 50	Penetrated to a maximum 300 mm (12 inch) depth with driven 12 mm (1/2 inch) rebar. Must be loosened with pick to excavate.
Very Dense	>50	Penetrated to a maximum 50 mm (2 inch) depth with driven 12 mm (1/2 inch) rebar. Very difficult to excavate even with pick.

Notes:
1) SPT 'N'-value in accordance with ASTM D 1586, uncorrected for overburden pressure effects or energy transfer. Different hammer weights, sampler size, and sampler fall will result in a different correlation of blow counts to compactness descriptions.
2) Definition of compactness descriptions based on SPT 'N'-values ranges from Terzaghi and Peck (1967) – can be assumed to correspond to typical average N_{60} values (i.e., they are representative of tests where the average energy transferred by the hammer to the split-spoon sampler is about 60% of the theoretical free-fall energy of the hammer).
3) Coarse gravel and cobbles, in particular, can result in high 'N'-values that are not representative of the compactness condition and the corresponding 'N'-values should not be used to describe the compactness condition.
4) Heaving sands will result in low SPT 'N'-values not representative of in situ conditions. Where such conditions occur, they should be noted on the field borehole logs. Proper drilling techniques (including maintaining the casing full of water or the use of a heavy drilling fluid to stabilize the borehole – i.e., mud) could be adopted to minimize this occurrence.

8.2.2 Cohesive Soils – Consistency

For cohesive (plastic) soils, the consistency of the soil may be described qualitatively based on field identification methods, measured SPT 'N'-values, or the behavior of the material. Consistency descriptions based on measured shear strengths such as those from the in situ field vane test or undrained laboratory shear strength tests take precedence over field identification methods or 'N'-value correlations. SPT 'N'-value correlations provide only a guideline to estimating the consistency of soils. The undrained shear strength should be measured by the in situ vane shear test during drilling for very soft to stiff cohesive soils. Other in situ tests such as the CPT may be used to infer the consistency of cohesive soils (in these instances the established correlations between tip resistance and undrained shear strength should be applied and the descriptive term selected based on the inferred shear strength). The pocket penetrometer test may be used to estimate the unconfined compressive strength, and therefore the consistency, of the soil. However, caution should be exercised as the unconfined compressive strength assessed by the pocket penetrometer may be overestimated by as much as 50%.

The terms used to describe the consistency of cohesive soils and their definition based on undrained shear strength, unconfined compressive strength and field measurements are provided in Table 8-2.

Table 8-2: Consistency of Cohesive Soils

Description	Field Identification	Undrained Shear Strength (See Note 1)	Unconfined Compressive Strength	SPT 'N' (blows/foot) (See Notes 2 & 3)
Very Soft	Extrudes between fingers when squeezed	<12 kPa <0.12 tsf	<25 kPa 0.25 tsf	0 – 2
Soft	Molded by light finger pressure	12 to 25 kPa 0.12 to 0.25 tsf	25 to 50 kPa 0.25 to 0.5 tsf	2 – 4
Firm	Molded by strong finger pressure	25 to 50 kPa 0.25 to 0.5 tsf	50 to 100 kPa 0.5 to 1 tsf	4 – 8
Stiff	Indented by thumb	50 to 100 kPa 0.5 to 1 tsf	100 to 200 kPa 1 to 2 tsf	8 – 15
Very Stiff	Indented by thumbnail	100 to 200 kPa 1 to 2 tsf	200 to 400 kPa 2 to 4 tsf	15 – 30
Hard	Difficult to indent with thumbnail	>200 kPa >2 tsf	>400 kPa >4 tsf	>30

Notes:
1) Undrained Shear Strength equals one-half the Unconfined Compressive Strength (UCS).
2) SPT 'N'-value in accordance with ASTM D 1586, uncorrected for overburden pressure effects or energy transfer. Different hammer weights, sampler size, and sampler fall will result in a different correlation of blow counts to consistency descriptions.
3) 'N'-values should be considered ONLY an approximate guide to consistency; for sensitive clays, the 'N'-value approximation for consistency terms does not apply.

It is stressed that only cohesive (plastic) soils should be described in terms of consistency. Fine-grained soils that are non-cohesive (such as SILT, rock flour, or non-plastic organic soils) are described using the terms given in Table 8-1.

As a standard guideline, consistency should only be assessed by in situ tests or tests on "fresh" samples or exposures. Consistency should not be assessed on or reported for material that has been wetted or allowed to

dry, freeze, or thaw. Neither density nor consistency should be reported for naturally frozen soil; such material should be recorded as “frozen.”

For investigations where the consistency of soils is estimated from the field identification methods only, the log should state the following: “The consistency has been inferred by subjective field identification methods only.”

8.3 Field Moisture Condition/Water Content

The field moisture condition of non-cohesive materials and the water content (w) of cohesive materials are to be included as part of the description. The term “water content” is used to be consistent with ASTM D2216 “Standard Test Methods for Laboratory Determination of Water (Moisture) Content of Soil and Rock by Mass.” The term “water content” may be known in other systems as the “moisture content.”

8.3.1 Non-cohesive Soils – Field Moisture Condition

The following terms are used to describe the field moisture condition of non-cohesive soils.

Table 8-3: Field Moisture Condition

Term	Abbreviation	Description
Dry	D	Little or no moisture visible or felt; soil grains flow freely through fingers.
Moist	M	Soils are darker than in the dry condition and may feel cool; soil grains tend to cohere.
Wet	W	As moist, but with free water forming on hands when handled.

Other terms such as “saturated,” “damp,” and “humid” are not clearly defined or cannot be determined in the field and should not be used.

When the purpose of an investigation is to characterize a potential borrow material/source for earthworks construction, the in-place water content may also be compared to the optimum water content as determined by the laboratory Proctor density test (i.e., “dry of optimum,” “at about optimum,” or “wet of optimum”) provided that the Field Representative has suitable experience estimating optimum water content for the soil being described.

8.3.2 Cohesive Soils – Water Content

The water content (w) of cohesive soils is described relative to the Plastic Limit (PL) using the following terms.

Table 8-4: Water Content

Term	Description
w<PL	Material is drier than the plastic limit (cannot be rolled to a thread of 4 mm).
w~PL	Material is close to (i.e., within approximately 1.5% of) the plastic limit (can be rolled to a thread of between 2 mm and 4 mm).
w>PL	Material is wetter than the plastic limit (can be rolled to a thread of less than 2 mm).

9.0 EXAMPLES OF SOIL DESCRIPTIONS

Example soil descriptions (both comprehensive and acceptably abbreviated), using a comprehensive list of constituents and minor components, general characteristics and bulk behavior characteristics, together with abbreviated formats, are presented in Appendix C. The example descriptions are provided for various soils based on the results of laboratory grain size distributions and Atterberg limits tests, and include material bulk behavior characteristics based on field Standard Penetration Test (SPT) 'N'-values and/or in situ vane shear strength test results and field moisture condition or water content.

For each example, a "Comprehensive" description, which includes and describes the details of the primary, secondary, and minor constituents, general characteristics, and bulk behavior characteristics, is provided along with an acceptably "Abbreviated" description.

All efforts should be made to include as much information as possible on the field borehole log and to develop a comprehensive description of the soil in the field and based on the subsequent laboratory sample review. The choice of using the full "Comprehensive" description or the "Abbreviated" description on the final Record of Borehole will depend on the nature of the project and the requirements of the downstream engineering and is at the discretion of the Project Manager.

9.1 Example Field Borehole Log

Soil descriptions in the field are based on visual identification of the particle size distribution for coarse-grained soils and field classification methods (i.e., tactile tests) for fine-grained constituents, as described in Section 5.0. In addition, the type/method and results of any in situ tests, groundwater condition(s), method(s) of borehole advancement and equipment types(s) used, and other pertinent site observations, are to be clearly noted/described on the field borehole logs. Of particular relevance to the SPT is noting the type of drive hammer, whether "Automatic" or "Cathead," and the make or manufacturer in the case of an automatic hammer, as the 'N'-values are dependent on the type of hammer used. An example Field Borehole Log is presented in Appendix D.

It is noted that the term "Borehole Log" only applies to the document prepared in the field. The formal document included in the report, that incorporates the results of an independent examination of the soil samples in the laboratory as well as any laboratory testing, and has undergone a separate review, is termed a "Record of Borehole" (see Section 9.3).

Also presented in Appendix D are field "Cheat Sheets" and a blank field boring log. The field cheat sheets provide guidance on the field logging of soil samples/strata, including tactile test methods, a flow chart for soil classification, and a description of general characteristics and bulk behavior characteristics. Example soil descriptions are also provided, including a guide as to the order of presentation of the soil constituents and characteristics. The blank field boring log provides a template for recording field logging and a quick reference guide to soil classification and descriptions.

9.2 Example Soil Classifications Based on Laboratory Test Results

Example soil classifications based on laboratory test results, comprised of grain size distributions and Atterberg limits, of actual soil samples obtained from boreholes, are presented in Appendix E. Based on these test results, soil classifications are presented in the respective figures for each soil sample. Where known, the origin of the soil has also been noted in the description if considered pertinent to the overall characterization of the deposit.

Recognizing that soil deposits can be comprised of highly variable strata, including lenses, layers, varves, and laminations, the soil classification of individual samples and ultimately the classification given to the deposit/stratum will be influenced by the procedure by which the laboratory tests are carried out, whether on the soil mass or on individual layers/varves. Example soil descriptions are presented in Appendix B, assuming that individual portions representative of the layer/varve were tested.

9.3 Example Record of Borehole

An example of a Record of Borehole is presented in Appendix F. The soil descriptions are based on the classification obtained from the laboratory tests for grain size distribution and Atterberg limits. In addition, the general characteristics and material bulk behavior characteristics based on the field descriptions contained in the Field Borehole Log, as well as on examination of the soil samples in the laboratory, have been taken into consideration. Additional laboratory test results are also included in the Record of Borehole as appropriate, such as the water content, unit weight, and an indication of the type of other laboratory tests carried out. The strata plot is based on the soil symbols in the gINT chart, included in Appendix F.

Standard sheets to be included in each geotechnical report providing guidance on the classification of the strata depicted on the Record of Borehole or Record of Test Pit sheets and a description and meaning of the various abbreviations used throughout the text of the report are presented in Appendix G. These sheets are titled “Method of Soil Classification,” “Abbreviations and Terms used on Record of Boreholes and Test Pits,” and “List of Symbols.”

10.0 DESCRIPTION OF ORGANIC SOILS

Highly organic soils (PEAT) are composed of at least 30% organic constituents and typically exhibit:

- A high capacity to hold water; water content generally ranging from 300% to 1500%.
- Shrinkage on air drying.
- Relatively low density.
- Feel spongy.
- Dark grey, brown, or black when highly decomposed, but may be yellow if less decomposed.
- A characteristic odor of decay, which is due to H_2S gas released during the decay.

Organic soils are mineral soils that contain between 5% and 30% organic constituents.

As shown in Table 10-1, highly organic soils and organic soils are named and classified on the basis of organic content (after UFC 3-220-10N, 2005; formerly NAVFAC DM7.01, 1986).

Table 10-1: Naming of Organic Soils

Category	Group Symbol and Name	Organic Content (% by mass)	Distinguishing Characteristics for Visual Identification
Highly Organic Soils	(PT) Fibrous PEAT	75% to 100%	Light weight, spongy, and often elastic at natural water content. Plant structure easily identifiable. Shrinks considerably upon air drying. Much water (colorless or turbid) squeezes from sample.
	(PT) Amorphous PEAT	75% to 100%	Light weight, spongy, but not usually elastic at natural water content. Plant structure visibly altered to unidentifiable. Shrinks considerably on air drying. Much turbid water squeezes from sample.
	(PT) SILTY PEAT	30% to 75%	Relatively light weight, spongy. Thread usually weak and spongy near plastic limit. Shrinks on air drying; medium dry strength. Usually can squeeze water from sample readily. Low dilatancy.
	(PT) SANDY PEAT	30% to 75%	Sand fraction visible. Thread weak and friable near plastic limit, shrinks on air drying, low dry strength. Usually can squeeze water from sample readily, high dilatancy, “gritty.”
Organic Soils	(OH) Clayey ORGANIC SILT	5% to 30%	Often has strong H_2S odor. Thread may be tough depending on clay fraction. Medium dry strength, low dilatancy.
	(OL) ORGANIC SILT	5% to 30%	Threads weak and friable near plastic limit, or threads may not be rolled. Low dry strength; medium to high dilatancy.

The various stages of plant decomposition are used to classify peat and range from completely fibrous (with a recognizable cellular structure) to completely amorphous peat (completely decomposed materials where fibers are absent), with various stages of decomposition defined between the extremes. The distinction between fibrous and amorphous peat is usually sufficient for geotechnical purposes. Index measures for classifying peat into one of these categories are described in Table 10-2. When necessary, distinction regarding the degree of decomposition of PEAT into decomposition groups H1 to H10 can be made using field indicators or laboratory testing as shown in Table 10-2, but is not considered a mandatory component of most soil descriptions.

The extent of details provided in the description of the organics may vary depending on the thickness of the organic materials and the importance of the organic material to the downstream engineering.

Table 10-2: Assessment of Degree of Decomposition or Humification

Identification	Laboratory Classification (ASTM D4427)	Field Guidelines (ASTM D 5715)		
		Degree of Decomposition	Nature of Material Extruded on Squeezing	Nature of Plant Structure in Residue
Fibrous	Fibric (Peat with greater than 67% fibers)	H1	Clear, colorless water; no organic solids squeezed out	Unaltered, fibrous, undecomposed
		H2	Yellowish water; no organic solids squeezed out	Almost unaltered, fibrous
		H3	Brown, turbid water; no organic solids squeezed out	Easily identifiable
Amorphous	Hemic (Peat with between 33% and 67% fibers)	H4	Dark brown, turbid water; no organic solids squeezed out	Visibly altered but identifiable
		H5	Turbid water and some organic solids squeezed out	Recognizable but vague, difficult to identify
		H6	Turbid water; 1/3 of sample squeezed out	Indistinct, pasty
	Sapric (Peat with less than 33% fibers)	H7	Very turbid water; 1/2 of sample squeezed out	Faintly recognizable; few remains identifiable, mostly amorphous
		H8	Thick and pasty; 2/3 of sample squeezed out	Very indistinct
		H9	No free water; nearly all of sample squeezed out	No identifiable remains
		H10	No free water; all of sample squeezed out	Completely amorphous

Note (1): This table is based on the von Post system which describes the extent of decomposition (humification) of peat on a scale of H1-H10; these rankings are included herewith for completeness, but the Technical Guidance does not require an H ranking to be routinely reported.

11.0 POTENTIALLY CONTAMINATED SOILS

11.1 Health and Safety

The guidelines or methods presented in this section do not address all health and safety issues that may arise due to the presence of hazardous materials within the soil or pore fluids. If hazardous materials are suspected to be present, immediately contact and inform the Field Supervisor, Health & Safety Officer, or Project Manager, as appropriate health and safety procedures must be established and utilized.

11.2 General

Where borehole logging is completed for environmental purposes, the soil should be accurately described in accordance with the soil classification system described in this Technical Guidance. If the gross contamination precludes an accurate description, the description will be completed to the extent practicable with any limitations to the description clearly noted. For investigations using direct push or continuous core sampling (i.e., where SPT 'N'-values are not available), the consistency behavioral characteristic of a fine-grained soil should be estimated using the field identification methods described in Table 8-2.

Where information that is required on a geotechnical engineering log is omitted, the Record of Borehole should include a statement such as "Record of Borehole not prepared for geotechnical engineering purposes."

When necessary to describe the soil using the texture classification adopted by the USDA, it will be necessary that laboratory testing of the grain size distribution also include the "Hydrometer" test to provide a numerical value (%) of the content of clay and silt size materials. In these cases, the textual classification should be included following the description provided in accordance with this Technical Guidance.

11.3 Indicators of Potential for Contamination

If indicators of contamination are present, describe them in as much detail as possible, as these descriptions may assist in selecting samples for laboratory analysis, in the location of potentially contaminated zones at other testing locations, or during future excavations. From an environmental perspective, zones of particular interest include shallow soils where surficial contaminant releases may have occurred, high permeability lenses that act as preferential pathways for contaminant migration, and the water table where petroleum hydrocarbons could accumulate and transition from coarse- to fine-grained soils that could act as a barrier to downwards migration of contaminants. Specific indicators of the presence of contamination may include the following:

- An unusual or distinctive color (e.g., black, dark brown, red).
- Staining or residues.
- Diesel, oil, tar, or other non-aqueous products present in the pore spaces (may present staining or greasiness on gloves or sample containers).
- A sheen on free water, if free water is present.
- The results of headspace screening performed with an appropriate field instrument (e.g., combustible gas detector, photoionization detector).
- Distinctive odor.

- Flakes, fragments, or pieces of foreign substances or debris such as metal, paint, brick, or wood waste. A distinction should be made between natural wood debris (e.g., twigs and branches) and man-made wood waste (e.g., wood chips and dimensional lumber). Refer to Section 13.1 for additional detail regarding the description of fill materials.

Examples of some of these indicators are presented in Table 11-1.

Table 11-1: Examples of Indicators of Potential Contamination

Item	Description	Visual Example
Clinker/Slag	Slag is a partially vitreous by-product of smelting ore. Usually presents as gravel size to cobble size particles with characteristic pores. Associated with metals contamination.	
Wood Waste	Man-made wood products, dimensional lumber, wood chips, hogfuel. May be associated with wood preservatives including petroleum hydrocarbons. Degradation associated with production of tannins and lignins, low oxygen conditions, non-chlorinated phenols. May present as brown to black.	
Paint Staining	Waste paint can infiltrate soil and present as staining and/or solidified nuggets.	
Asbestos	Asbestos is a naturally occurring mineral that is incorporated into many common products found in demolition debris (e.g., corrugated board, floor tiles, pipes, etc.). Characterized by the presence of fibers.	

If such indicators are present, include (where appropriate) the following detail:

- Relative proportion, quantity, and size of foreign substances or debris.
- Consistency of foreign substance (e.g., viscous, dry, powdery).
- Particular characteristics (e.g., description of an odor). See Table 11-2.

11.4 Odors

Note that the odor of soil samples should only be recorded if incidentally noticed during the course of typical soil sampling activities. **Field personnel shall not deliberately smell field samples**. As odors are subjective, the description of any incidental odors that are noted should be kept simple. For instance, "hydrocarbon odor" would be appropriate for petrol, diesel, or kerosene. A second description may be included in brackets on the field log (i.e., not to be included in the final Record of Borehole) if considered valuable (e.g., mothball odor, rotten egg odor). The odor description should appear on the field log sheet in the "Additional Observations" column. Other examples of odors are presented in Table 11-2.

Table 11-2: Examples of Odors

Odor	May Indicate
Hydrocarbon	BTEX or TPH
Tar	PAH or phenolics
Solvent	Organic solvents
Sweet	Volatile halogenated organics
Pool Water	Chlorine or chlorinated compounds
Rotten Egg, Gas	Sulphides or sulphates
Bleach	Chlorine
Putrescible, Garbage, Decomposing	Methane, bacteria, nutrients

12.0 FROZEN SOILS

The degree of description appropriate for frozen soils depends on the project needs. The following should be applied in all investigations in permafrost areas. Simply describing the soil as frozen may be adequate in areas where the soils are seasonally frozen.

The general guidance for the description of frozen soils is based on visual inspection and on ASTM 4083 and NRCC-7576; reference to these documents is recommended. This guidance is applicable for relatively undisturbed and frozen soils. Considerations are provided for simple laboratory testing to augment the visual inspection.

12.1 Definitions

Permafrost: The thermal condition under which soil and/or bedrock exist at a temperature of 0°C or below for a period of two or more consecutive years. Note that ice may or may not be present in permafrost zones.

Frozen soil: Soil with a temperature below 0°C, where part or all of the pore fluid is in the solid phase (i.e., ice).

12.2 General Guidance

The guidance comprises three steps:

Step 1 – description of the soil

Step 2 – description of the frozen soil

Step 3 – description of substantial ice strata

Field logs shall be prepared such that two stratigraphic records are presented side by side: soil and frozen soil/ice. Reporting records shall be produced to present this information in a similar format.

Temperature greatly influences properties and behavior of frozen soils, and temperature measurement should be included as part of the field logs.

12.2.1 Step 1 – Description of the Soil

This step is to be performed once the soils have thawed; reference to the appropriate sections in this document shall be made.

12.2.2 Step 2 – Description of the Frozen Soil

Two groups of frozen soil, based on visual observation by the unaided eye, are considered: non-visible ice and visible ice. Refer to Table 12-1 for a summary.

12.2.2.1 Non-visible Ice

Frozen soils with non-visible ice (Group Symbol: N) are subdivided between friable (poorly-bonded) material and well-bonded material. The use of the following subdivisions is to be made only if proper inspection is made.

Friable (poorly-bonded) Material

- Nf – Segregated ice is not visible to the unaided eye; therefore, the degree of saturation is low.

Well-bonded Material

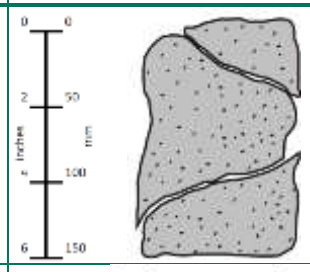
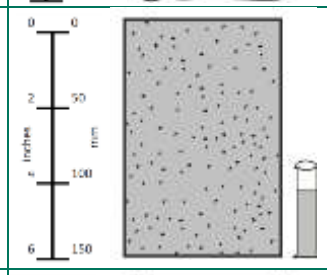
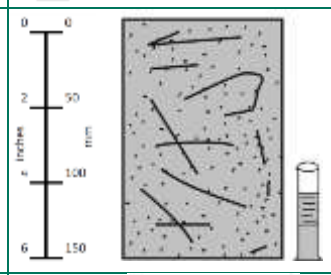
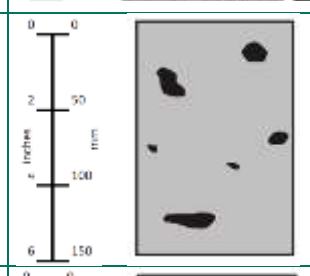
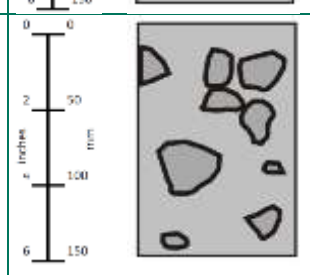
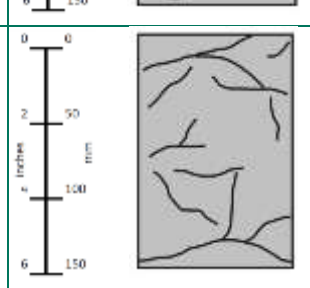
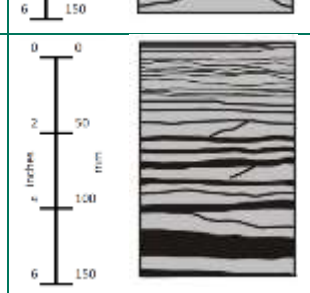
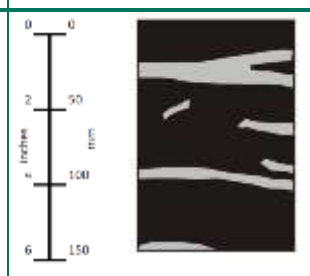
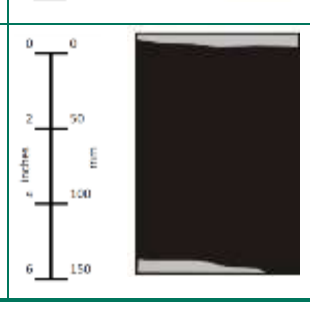
- N_b – The ice in the frozen soil cements the material into a solid, hard mass. Segregated ice is not visible to the unaided eye.
- N_{bn} – If segregated ice is not visible with the use of a magnifying glass, this indicates that no excess ice is present.
- N_{be} – If uniformly distributed excess ice is present (i.e., visible to the unaided eye). Refer to Section 12.3.1 to determine the presence of excess ice.

12.2.2.2 Visible Ice

Frozen soils with visible ice (Group Symbol: V) are subdivided as described below. The use of a subdivision is to be made only if proper inspection is made. Refer to Table 12-1 for a summary. Note that these soils have segregated ice visible to the unaided eye, but have individual ice layers and/or masses that are less than 25 mm in thickness.

- V_x – Frozen soil with visible ice with individual ice crystals or inclusions.
- Vc – Frozen soil with visible ice occurring as coatings on particles.
- V_r – Frozen soil with visible ice with random or irregularly oriented formations.
- V_s – Frozen soil interspersed with stratified or distinctly oriented visible ice formations.
- V_u – Frozen soil with uniformly distributed visible ice.

Table 12-1: Summary of Frozen Soil and Ice Strata Descriptions

MATERIAL	DESCRIPTION	GROUP SYMBOL	SUBGROUP			FIELD IDENTIFICATION
			DESCRIPTION	SYMBOL		
FROZEN SOIL	Non-visible Ice	N	Friable (poorly-bonded)	N_f		Identify by visual examination. To determine presence of excess ice, use magnifying glass and laboratory testing (refer to Section 12.3.1). Estimate degree of ice saturation for unsaturated soils: medium or low. Note presence of crystals or ice coatings around larger particles. LEGEND: Soil - ☐ Ice - ⬚ or ■
			Well-bonded No excess ice -	N_b	N_{bn} 	
			Well-bonded Excess ice	N_b	N_{be} 	
	Visible Ice (by unaided eye)	V	Individual ice crystals or inclusions	V_x		For the ice phase, record the following as applicable: ■ Location ■ Orientation ■ Thickness ■ Length ■ Spacing ■ Hardness ■ Structure ■ Color ■ Shape ■ Size ■ Pattern Estimate volume of visible segregated ice present as a percent of total sample volume. LEGEND: Soil - ☐ Ice - ⬚ or ■
			Ice coatings on particles	V_c		
			Random or irregularly oriented ice formations	V_r		
			Stratified or distinctly oriented ice formations	V_s		
			Uniformly distributed ice	V_u		
ICE STRATA	Ice strata are greater than 25 mm (1 inch) in thickness	ICE	Ice with soil inclusions	ICE + Soil Type		Designate material as ICE (note special forms of ice, e.g., hoarfrost) and use descriptive terms as follows, usually one item from each group, as applicable: 1. Hardness (of mass, not individual crystals): HARD, SOFT. 2. Color (examples): COLORLESS, GREY, BLUE. 3. Structure (avoid confusion with surface scratches or frost coating on ice): CLEAR, CLOUDY, POROUS, CANDLED, GRANULAR, STRATIFIED. 4. Admixtures (examples): CONTAINS, FEW THIN, SILT INCLUSIONS. LEGEND: Soil - ☐ Ice - ⬚ or ■
			Ice without soil inclusions	ICE		

12.2.3 Step 3 – Description of Substantial Ice Strata

Ice strata that are greater than 25 mm (1 inch) in thickness are denoted by the symbol: ICE. Ice is subdivided as described below:

- Ice stratum containing soil inclusions is denoted by: ICE + Soil Type.
- Ice stratum containing no soil inclusions is denoted by: ICE.

12.3 Further Considerations

12.3.1 Excess Ice Determination

The following guidance may be used with frozen fine-grained soils to determine the excess ice content; excess ice in coarse-grained soils is typically visually identified as ice strata. A representative sample, similar in size to that used for a standard water content determination is subjected to the following steps:

- The soil sample is thawed and melt water is captured; mass of water is measured and recorded (M_{mw}).
- The standard water content determination is made on the soil sample (ω).
- The total water content of the sample is calculated by adding the mass of water from the standard water content to the mass of melt water and dividing by the mass of soil (ω_t).
- The ice content is then calculated: $\omega_i = \omega_t - \omega$

Ice contents greater than 10% are considered indicative of excess ice.

12.3.2 Pore Fluid Salinity

The salinity of the pore fluid collected from samples after thawing should be determined. A blood salinity meter is commonly used in the field for rapid determination of pore fluid salinity.

13.0 UNUSUAL OR DIFFICULT MATERIALS

Unusual or difficult materials of engineering and construction significance should be described in a manner consistent with the USCS classifications, wherever possible. For materials where this is not possible (such as rock fill and refuse/waste), guidance on appropriate descriptions is provided below.

Some of the materials described in the following sections have their own unique symbols used in stratigraphic plots and on Record of Borehole sheets, as presented in Appendix F. For these materials, the identifying term for the origin should be capitalized and included at the beginning of the description, as indicated in Section 3.0. Typical examples of these types of materials may consist of: natural but re-worked or placed soils and soils mixed with obvious manufactured (brick, concrete, glass, metal) constituents (FILL); landfilled deleterious materials (REFUSE/WASTE); broken rock (ROCK FILL); crushed materials and sediments from mining or quarrying operations (TAILINGS); and soil containing organic matter (TOPSOIL).

Other unusual materials described in this section include roadway and processed granular materials (PAVEMENT STRUCTURE); intermediate or transitional materials (Mixtures of Soil and Rock), or highly weathered materials (RESIDUAL SOIL); and chemically altered layers within a soil profile (Duricrusts). These materials do not have a unique symbol(s) and the identifying term for their origin should be capitalized and included in the General Characteristics section of the description of the material (specifically in the Additional Observations).

13.1 Fill and Refuse/Waste

Fill is defined as any material that was placed by mankind and can comprise:

- Reworked natural site materials
- Reworked natural materials from elsewhere (which may be geologically different)
- Rip-rap and Rock Fill (for embankment construction or erosion protection)
- Demolition debris or other man-made materials
- Refuse or landfill waste
- Granular base and subbase materials (e.g., pavement structures, under floor slabs, etc.)
- Combinations of the above

Some characteristics that may be indicative of FILL include:

- Presence of foreign objects such as glass, plastic, slag, etc.
- Buried organic matter
- Unusually variable range of colors
- Very distinct changes in soil profile
- Large variability in shear strength, consistency, or compactness
- Stratigraphy that is inconsistent with regional geology

- "Cloddiness" of clay soil indicating previous disturbance by excavation

It is essential that the thickness and composition of FILL, where present, is recorded accurately. The fact that soils have been deposited on a site by other than natural mechanisms may have significant geotechnical and environmental implications. The material should be identified clearly on the field log as FILL, and then described in sufficient detail to give the reader of the log a good understanding of the material composition. The description should include information on the following aspects if applicable (this list is not exhaustive):

- Inferred origin of the material (as notes on field log)
- Presence and size of large particles or objects such as rock fill, concrete, masonry, old cars, etc.
- Presence of voids or collapsible hollow objects
- Chemical waste and dangerous or hazardous substances
- Organic matter, with a note on the degree of decomposition
- Odorous smell
- Striking color tints
- Any dates readable on buried papers, etc.
- Signs of heat or combustion underground, e.g., steam emerging from borehole
- Structure and variability

In situations where FILL is comprised predominantly of soil, the classification of the soil matrix constitutes the basis of the description with the presence of deleterious materials noted, as applicable:

> e.g., FILL – (SW) SAND, some gravel; brown, with pieces of brick, wood, plastic, and glass; non-cohesive, moist, compact.

In situations where fill is comprised predominantly of refuse and/or waste, it is not possible to classify the constituents according to the USCS Group Symbol and Group Name. As such, the description will begin with the identifying term "REFUSE/WASTE" and be comprised of the general characteristics of waste materials, including color and odor, where applicable:

> e.g., REFUSE/WASTE – black to dark grey, garbage/decomposing odor, with wood chips, plastic bags, paper, cloth and wire, trace to some fine sand.

Where the composition is highly variable and includes a mixture of soil, rock, and deleterious materials in similar proportions, the constituents of the fill could be described separately:

e.g., FILL – MIXTURE of SOIL (≈60%) and REFUSE (≈40%)

SOIL is: (CH) CLAY, some fine to coarse sand, trace fine gravel, grey brown; w>PL, firm.

REFUSE/WASTE includes topsoil, branches, several plastic bags, concrete slabs up to 1 m in size, galvanized iron sheets, glass bottles.

13.2 Rock Fill

Rock fill is not assigned a Group Symbol or Group Name. Where rock fill is inferred by instances of auger grinding or split-spoon refusal, then its presence should be described in the same manner as cobbles and boulders (see Section 7.6.3). Alternatively, its presence should be confirmed by test pitting or coring, if it is not exposed.

The description will begin with the identifying term “ROCK FILL” and be comprised of the general characteristics of the fill material, including color and sizes (where test pitting or coring is carried out and the fill is visible or core samples are obtained) or indicate only the range of depths over which the rock fill is inferred from auger resistance or split-spoon refusal (where test pitting or coring is not carried out and/or where samples are not obtained).

e.g., ROCK FILL – dark grey to pink, with particle sizes ranging from about 300 mm to 900 mm.

e.g., ROCK FILL – inferred from auger resistance between depths of about 1.2 m and 3.1 m.

13.3 Tailings

Tailings are a waste product of mineral extraction and are the materials remaining after the process of separating the valuable fraction from the uneconomic fraction of an ore. Tailings are distinct from overburden or waste rock, which are the materials displaced during mining without being processed.

The extraction of minerals from ore can be done two ways: placer mining, which uses water and gravity to extract the valuable minerals; or hard rock mining, which typically uses combinations of crushing and/or grinding of rock, and/or the addition of chemicals or heat. Tailings particles associated with placer mining are typically rounded and comprised of alluvial deposits (sand and gravel sizes). Tailings particles associated with hard rock mining are typically angular and range in size from a few microns to several millimeters.

Tailings are to be classified and described using the procedures presented in this Technical Guidance, but the description must begin with the identifying term “TAILINGS” such that it is clear that they are not naturally occurring. Note: the physio-chemical behavior of tailings may vary significantly when compared to naturally occurring soils.

e.g., TAILINGS – (ML) SILT, trace to some fine sand; light brown; non-cohesive, moist, loose.

13.4 Topsoil

The thickness of topsoil (A horizon) must be accurately measured and recorded at each soil investigation location. The topsoil or A horizon includes any organic (duff) material that is resting on top of the mineral soil

surface. This material includes leafy, decomposed, and/or organic matter (including peat materials). It is typically sufficient to describe only the primary soil constituent, but the description must begin with the identifying term "TOPSOIL."

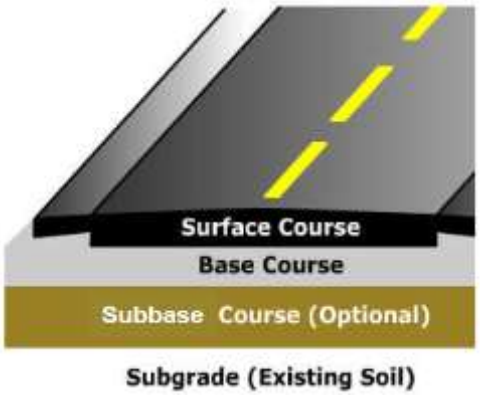
e.g., TOPSOIL – (SM) SILTY SAND; brown; moist.

For more detailed investigations, texture (using percentage of sand, silt, clay in the texture triangle), structure (aggregations of soil particles), consistency (soil strength), and any additional features that are observed (mottles, coarse fragments, roots, porosity, effervescence, etc.) are also identified. For additional information, consult Agriculture Canada (1982) or SCWG (1998).

13.5 Pavement Materials/Pavement Structure

Pavement base and subbase materials are a specific type of fill. They may be encountered during pavement investigations or other types of geotechnical investigations. This section is not intended to capture all the information which may be required for specific pavement investigations. Rather, it provides general information which must be considered when pavements are penetrated during the investigations.

Pavements usually contain some or all of the following layers:



Surface Course or Wearing Surface – Sprayed bituminous seal, asphalt or concrete

Base – with a percentage of crushed rock, usually 20 mm or 40 mm (3/4 inch or 1 1/2 inches) nominal size

Subbase – generally of lower quality than the Base, may not require crushed rock, should have good drainage properties

Subgrade – either natural soil/rock or compacted FILL

It is important to clearly identify the extent and nature of pavement materials where present. The question often arises, "Are the existing pavements suitable for future use?" or "Can we stabilize/recycle the existing pavements?" In some cases Golder is asked to comment on these issues after the completion of fieldwork, despite the fact that our field investigation may not have been planned to specifically address the issue of pavement composition. Hence these layers should always be logged carefully in the field.

Where possible, the boundaries between these layers should be noted on the field logs. The observed structure should also be sketched.

The following notes indicate some important issues to be addressed when logging pavement materials.

1) Where present, the type of wearing surface should be described, including the stone size, and the layer thickness noted to the nearest 5 mm (1/4 inch). Wearing surfaces may be either a surface treatment (sprayed bituminous seal) or asphalt (also known as asphaltic concrete or hotmix). Surface treatments or sprayed seals are constructed by applying a layer of bitumen to the surface followed by the placement of

graded aggregates into the bitumen and then compacting. Such seals are usually between 10 mm and 30 mm (1/2 inch to 1 1/4 inches) thick (they may incorporate more than one layer) and typically produce a rough wearing surface. Hotmix asphalt is produced at an offsite plant, where asphalt cement or bitumen is mixed with carefully selected aggregates, brought to the site by trucks and placed by a paving machine in a layer of relatively constant thickness (generally 25 mm to 70 mm [1 inch to 2 3/4 inches]) over the prepared granular base. A number of layers of hotmix asphalt can be placed one on top of the other. The total hotmix asphalt thickness may range from 25 mm to over 200 mm (1 inch to over 8 inches).

The granular base and subbase fill layers should be described as a soil and the layer thicknesses should be noted to the nearest 10 mm (1/2 inch). It is useful to describe the rock type (e.g., basalt, quartzite, dolomite, etc.) and to include descriptions of whether the particles are rounded or angular and whether the gravel appears well graded or poorly graded.

e.g., (GW) sandy GRAVEL, fine to coarse, well graded, angular, trace non-plastic fines; brown (PAVEMENT STRUCTURE); non-cohesive, dry, dense.

As a minimum, the materials should be described as PAVEMENT MATERIAL or EXISTING PAVEMENT.

2) If any of the materials present high resistance or refusal to drilling or excavation, this must be noted on the field log.

3) Where it is possible to distinguish between the granular base and subbase layers, the layer name should be included in the General Characteristics section of the description of the material (specifically in the Additional Observations).

e.g., (SW) SAND, fine to coarse, well graded, sub-angular, trace gravel, trace non-plastic fines; light brown (PAVEMENT STRUCTURE, Subbase); non-cohesive, moist, compact.

4) Stabilization (the addition of cement, lime, or other materials to granular base or subbase materials) is often used to increase the strength and hence the design life of pavements. The presence of stabilized materials must be noted because:

- The design life and potential failure mechanisms are different for unstabilized (unbound) and stabilized (bound) pavements. Our assessment of pavement condition or “failure” causes may be significantly influenced by the presence of stabilized materials.
- A stabilized pavement may be significantly more difficult to excavate or rip. If we do not record the presence of stabilized materials, we may be liable for a contractor’s claim for construction difficulties.

5) Some pavements incorporate geosynthetic materials between the pavements layers described above, typically placed above the subgrade or beneath a bituminous seal. The presence of any such materials within the pavement should be noted.

6) Old asphalt layers, such as bitumen impregnated macadam (coarse gravel and cobbles) layers, should be identified as they may have a significant impact on the performance of pavement overlays.

13.6 Is it Soil or is it Rock?

Materials that appear to be intermediate between soil and rock in terms of strength, weathering, or because of mixed composition, are often poorly described. Table 13-1 shows that the strength ranges for very stiff and hard soils overlap with the strength range for extremely weak rock (shown by the shaded section).

Examples of difficult or borderline materials may include:

- Mixtures of large rock fragments (>75 mm) and soil components, which may be formed by weathering along joints in a rock mass, alluvial processes, colluvial processes, or partial cementation, such as in duricrusts (calcrete, silcrete, ferricrete).
- Completely weathered rocks (All rock material is decomposed and/or disintegrated to soil. The original mass structure is still largely intact.)
- Highly weathered rocks (More than half of the rock material is decomposed and/or disintegrated to a soil. Fresh or discolored rock is present either as a discontinuous framework or as corestones.)
- Extremely weak rock, which is defined by ISRM (1981) as having Uniaxial Compressive Strengths of 0.25 MPa to 1.0 MPa (35 to 150 psi).

Residual Soil is material formed by disintegration of underlying parent rock or partially indurated (cemented or hardened) material. Residual soil may have a wide range of shear strength/compressive strength. Residual soil should be classified and described according to the primary soil constituents.

> e.g., (CL) sandy SILTY CLAY; brown-grey fissured, friable, with cobble size quartzite pieces (RESIDUAL SOIL); cohesive, w<PL, hard.

Table 13-1: Soil vs. Rock

Material Strength Range	Description	Field Identification	Undrained Shear Strength	Unconfined/Uniaxial Compressive Strength
SOIL	Very Soft	Extrudes between fingers when squeezed	<12 kPa <0.12 tsf	<25 kPa 0.25 tsf
	Soft	Molded by light finger pressure	12 to 25 kPa 0.12 to 0.25 tsf	25 to 50 kPa 0.25 to 0.5 tsf
	Firm	Molded by strong finger pressure	25 to 50 kPa 0.25 to 0.5 tsf	50 to 100 kPa 0.5 to 1 tsf
	Stiff	Indented by thumb	50 to 100 kPa 0.5 to 1 tsf	100 to 200 kPa 1 to 2 tsf
	Very Stiff	Indented by thumbnail	100 to 200 kPa 1 to 2 tsf	200 to 400 kPa 2 to 4 tsf
	Hard	Difficult to indent with thumbnail	>200 kPa >2 tsf	>400 kPa >4 tsf
ROCK	Extremely Weak	Indented by thumbnail		0.25 to 1 MPa 35 to 150 psi
	Very Weak	Crumbles under firm blows with point of geological hammer, can be peeled by a pocket knife		1 to 5 MPa 150 to 725 psi
	Weak	Can be peeled by a pocket knife with difficulty, shallow indentations made by firm blow with point of geological hammer		5 to 25 MPa 725 to 3,500 psi
	Medium Strong	Cannot be scraped or peeled with a pocket knife, specimen can be fractured with single firm blow of geological hammer		25 to 50 MPa 3,500 to 7,500 psi
	Strong	Specimen requires more than one blow of geological hammer to fracture it		50 to 100 MPa 7,500 to 15,000 psi
	Very Strong	Specimen requires many blows of geological hammer to fracture it		100 to 250 MPa 15,000 to 35,000 psi
	Extremely Strong	Specimen can only be chipped with geological hammer		>250 MPa >35,000 psi

Materials having an Unconfined Compressive Strength (UCS) of less than about 0.5 MPa (5 tsf); an undrained shear strength of less than about 250 kPa [2.5 tsf]) and non-cohesive materials should be described using the terms provided in this Technical Guidance.

A clear description of such materials must be provided because many civil contracts and specifications have different rates for construction activities involving SOIL and ROCK. For example, contractors and engineers preparing specifications may depend on our Records of Boreholes and/or Records of Test Pits to assess how much of each material type is to be excavated or drilled using a certain method. If the description of a material is

limited to “Completely Weathered Shale,” the assumption may be that this material will require rock excavation methods, whereas the material may actually be a soil which is significantly easier to excavate.

The type of equipment (where appropriate) that was used to carry out the investigation must be accurately recorded to allow us to make our observations, such as:

- Hitachi EX300 hydraulic excavator with 1.2 m wide toothed bucket
- Trailer mounted Gemco 210B rotary drilling rig with solid augers and a V-bit
- Caterpillar D9 bulldozer

As our business is to provide engineering advice, we should describe materials primarily in terms of their engineering properties, followed by an assessment of their geological origin. A material that can be remolded by hand and is likely to have the engineering properties of soil should be described as a **soil** with observations of structure, ***and*** an interpretation regarding origin (i.e., completely weathered rock). The presence of obvious rock structure does not define a material as rock in engineering terms.

The least ambiguous method to describe mixed materials is to use a simple main heading such as:

“Mixture of Boulders and Soil,” or “Rock Fragments in a Soil Matrix.” The remainder of the description should describe each of the components (i.e., soil and rock) separately, and give an assessment of their proportions.

A typical description may be:

> **MIXTURE of SOIL (≈60%) and ROCK (≈40%)**
>
> SOIL is: (CH) sandy CLAY; pale brown; w<PL, cohesive, hard.
>
> ROCK is: QUARTZITE present as subrounded cobbles and boulders up to about 600 mm in size; pale brown; medium strong.

13.7 Duricrusts

Duricrusts are formed by precipitation of previously dissolved chemicals, which form an altered layer within a soil profile. Duricrusts are generally variable, and may range from gravel sized nodules to relatively continuous layers of uniformly strong rock materials. Some examples of typical duricrusts are presented in Table 13-2.

Table 13-2: Typical Duricrusts

Duricrust Name	Description
Calcrete	Calcareous material consisting mostly of calcite (calcium carbonate). Calcretes are also referred to as caliche, kunkar, and travertine.
Ferricrete	A material produced by secondary deposition of previously dissolved iron oxides. Ferricretes include lateritic soil, laterites, ferruginous breccias, and ferruginous sandstones. Also known as ironstone.
Laterite	A red or yellow soil containing numerous gravel size particles enriched in iron and/or alumina. These particles may be soft or hard, but do harden on exposure to the atmosphere.
Silcrete	Siliceous material, usually of high to very high substance strength. Crystalline structure similar to quartzite.

14.0 REFERENCES

AASHTO (1993), American Association of State Highway and Transportation Officials, AASHTO Guide for Design of Pavement Structures, 1993.

Agriculture Canada (1982), The Canadian Soil Information System (CanSIS), Manual for Describing Soils in the Field 1982 (Revised). Research Branch - Agriculture Canada, Ottawa, Ontario, LRRI Contribution No. 82-52.

Alaska Guide to Description and Classification of Peat and Organic Soil (March 2007).

AS1726 (1993), Australian Standard for Geotechnical Site Investigations.

ASTM D2487, Standard Test Method for Classification of Soils for Engineering Purposes.

ASTM D2488, Standard Practice for Description and Identification of Soils (Visual-Manual Procedure).

ASTM D4083, Standard Practice for Description of Frozen Soils (Visual-Manual Procedure).

ASTM D4427, Standard Classification of Peat Samples by Laboratory Testing.

ASTM D5715, Standard Test Method for Estimating the Degree of Humification of Peat and Other Organic Soils (Visual/Manual Method).

BS EN ISO 14688-1 2002 and BS EN ISO 14688-2, (2004), British Standard “Geotechnical investigation and testing - Identification and classification of soil”.

BS5930-1999, British Standard “Description of Soils and Rocks”.

Burwash and Wiesner (1984), “Classification of Peats for Geotechnical Engineering Purposes,” Proceedings: Cold Regions Engineering Specialty Conference, April 4-6, 1984, Canadian Society for Civil Engineering, Montreal, Quebec, Canada.

Casagrande, A. (1947), “Classification and Identification of Soils,” Proceedings of the American Society of Civil Engineering, June 1947.

CFEM (2006), Canadian Foundation Engineering Manual. 4th Edition. Canadian Geotechnical Society. BiTech Publishing.

ISRM (1981), International Society for Rock Mechanics ISRM, 1981. Rock characterization, testing and monitoring. In: Brown, E.T. (Ed.), ISRM Suggested Methods. Pergamon Press, Oxford, p. 211.

Korpijaakko, E. O., and Woolnough, D. F. (1977), “Peatland Survey and Inventory”, Muskeg and the Northern Environment in Canada, University of Toronto Press.

Ministry of Environment, 1982. Manual of Policy, Procedures and Guidelines for Private Sewage Disposal Systems. O.Reg. 374/81, pg 1 of 14 to page 8 of 14).

Munsell (2009), Munsell Soil Color Book, 2009, Revised edition. Produced by Munsell Color: Grand Rapids, MI, USA.

NAVFAC DM7.01 and 7.02 (September 1986), Soil Mechanics Design Manual, Naval Facilities Engineering Command, United Facilities Criteria (UFC) Technical Publications.

NRCC-7576, Guide to a Field Description of Permafrost for Engineering Purposes, National Research Council Canada (NRCC) document NRCC-7576.

Oxford Dictionary, Earth Sciences.

SCWG (1998), The Canadian System of Soil Classification. Soil Classification Working Group, Agriculture and Agri-Food Canada Publication 1646 (Revised).

Terzaghi and Peck (1967), Soil mechanics in engineering practice.

Unified Facilities Criteria UFC 3-220-ION, 2005. Soil Mechanics. US Department of Defence, Whole Building Design Guide, www.wbdg.org

Von Post, Lennart (1921), "Sveriges Geologiska Undersöknings Torvinventering Ocj Några Av dess Hittills Vunna Resultat," Svenska Mosskulturfor. Tidskrift, vol. 36.

Wikipedia Encyclopedia

\\pit1-s-fs1-vm\data\projects\golder\soil classification tp\gai tp001 soil technical procedure rev 1\tg-1.2-6 gai soil description-rev 1.docx

APPENDIX A

Example Grain Size Distributions for USCS Coarse-Grained Soil Groups

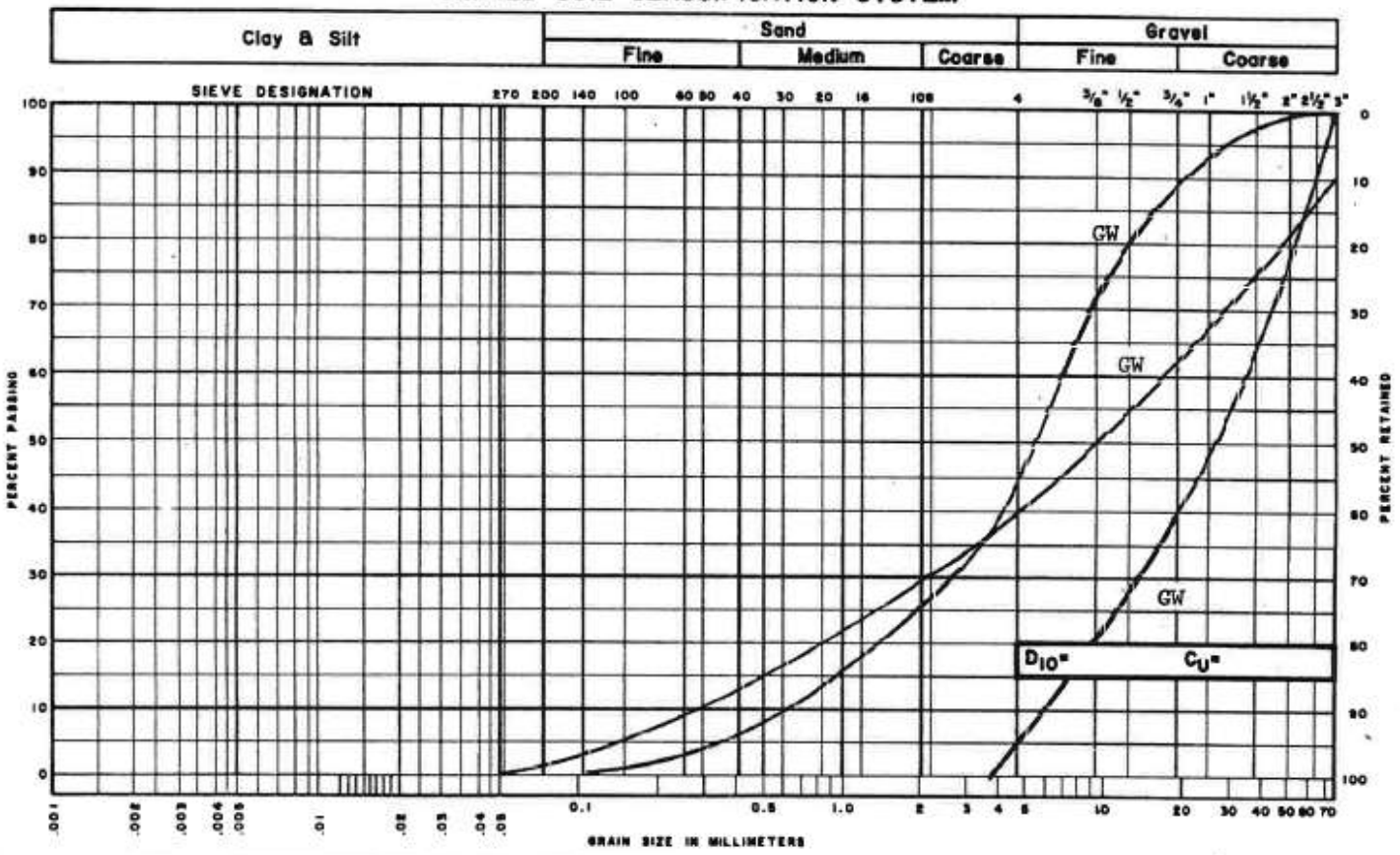
(Reference: Ministry of Environment, 1982)

MAY 1982

6.3.13

App. 6.3.2
p. 1 of 14

UNIFIED SOIL CLASSIFICATION SYSTEM



GW - Well-graded gravels, gravel-sand mixtures
- Less than 5% finer than 0.074 mm
- Uniformity coefficient > 4

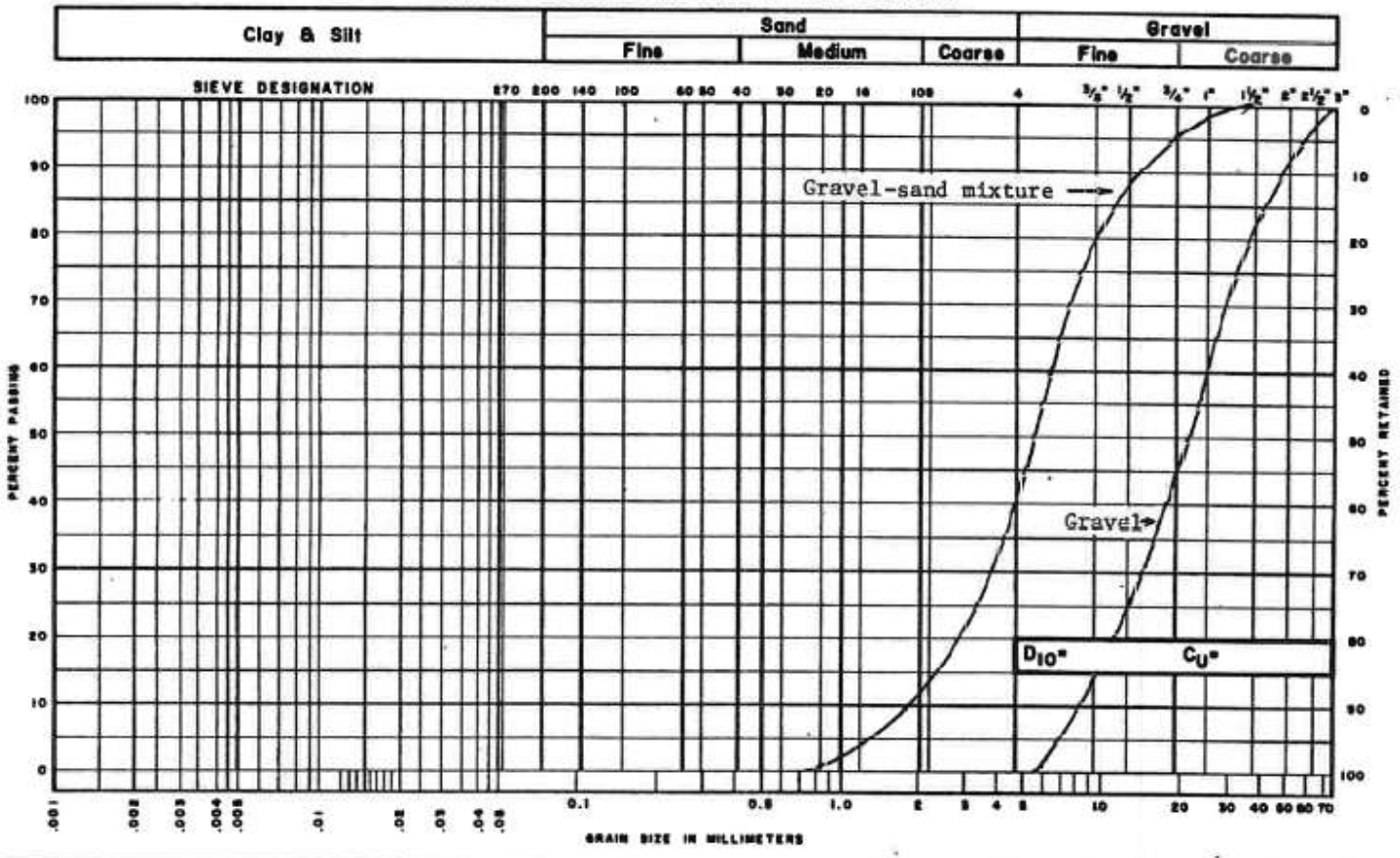
0803 3/82

MAY 1982

6.3.14

App. 6.3.2
p. 2 of 14

UNIFIED SOIL CLASSIFICATION SYSTEM



GP - Poorly-graded gravel, gravel-sand mixtures
- Less than 5% finer than 0.074 mm
- Uniformity coefficient less than 4

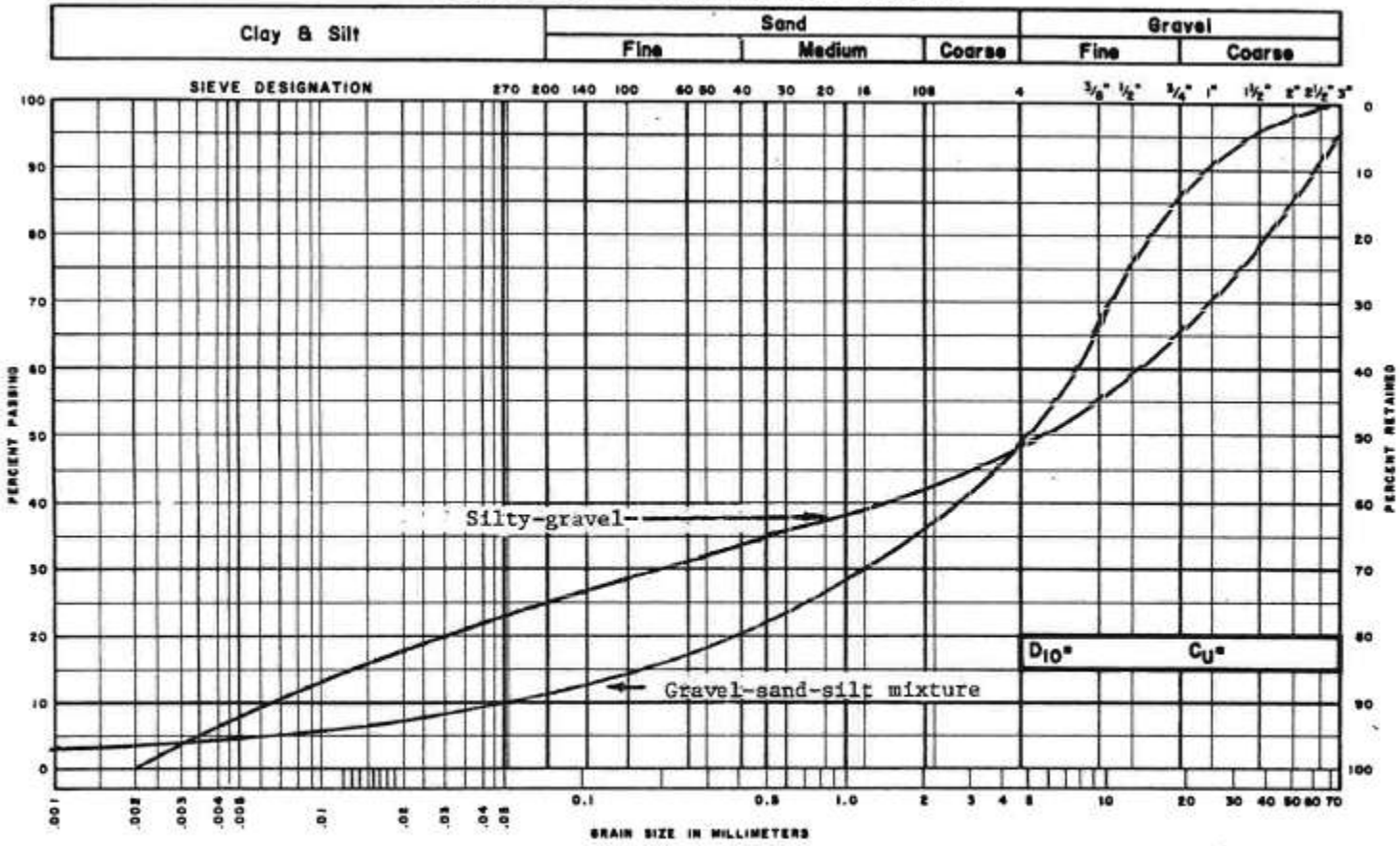
0803 3/82

MAY 1982

6.3.15

App. 6.3.2
p. 3 of 14

UNIFIED SOIL CLASSIFICATION SYSTEM



GM - Silty-gravels, gravel-sand-silt mixtures
- More than 12% finer than 0.074 mm
- Non-plastic - plasticity index less than 4
- Plots below "A" line on plasticity chart

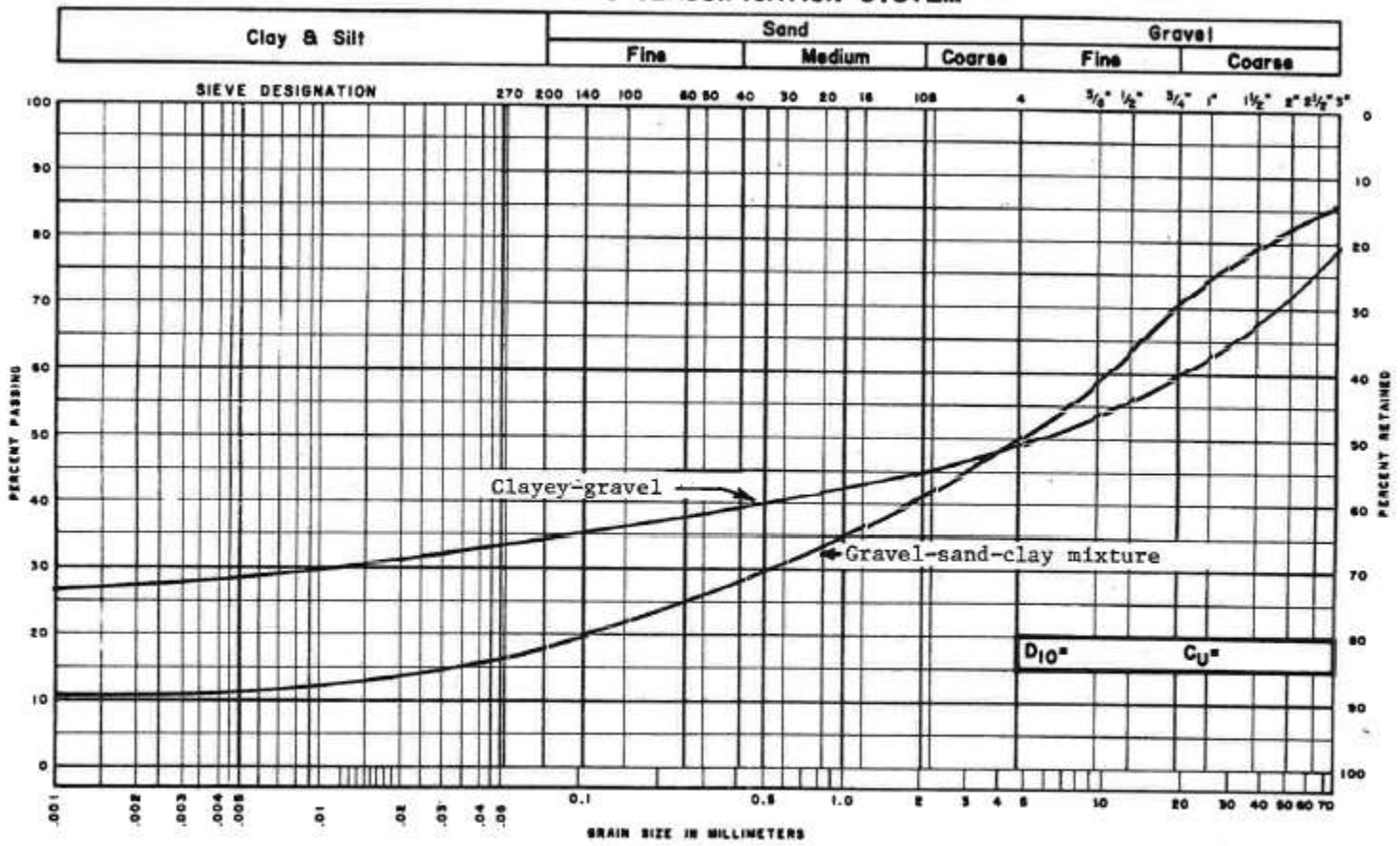
0603 3/82

MAY 1982

6.3.16

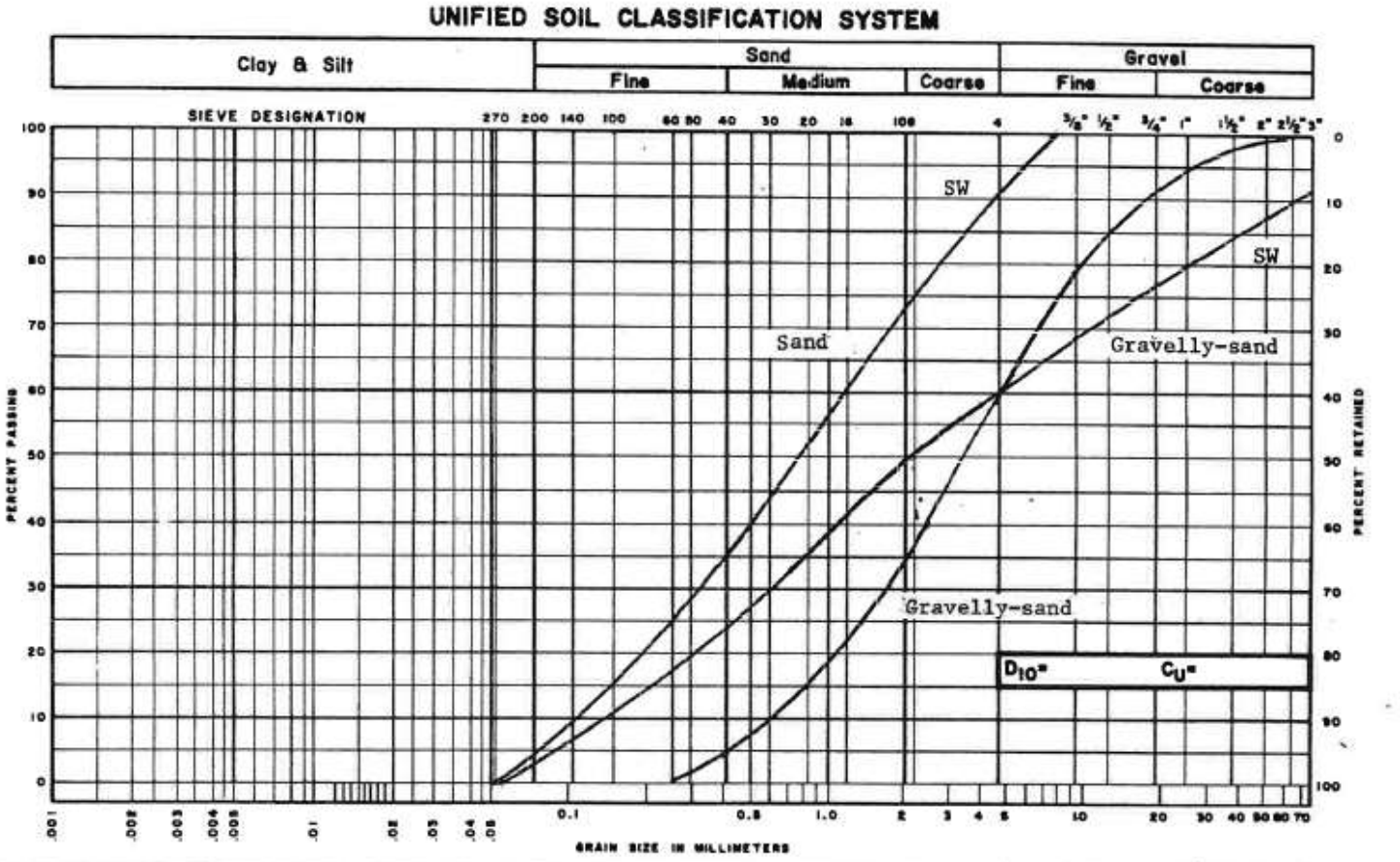
App. 6.3.2
p. 4 of 14

UNIFIED SOIL CLASSIFICATION SYSTEM



GC - Clayey-gravels, gravel-sand-clay mixtures
- More than 12% finer than 0.074 mm
- Plasticity index greater than 7
- Plots above "A" line on plasticity charts

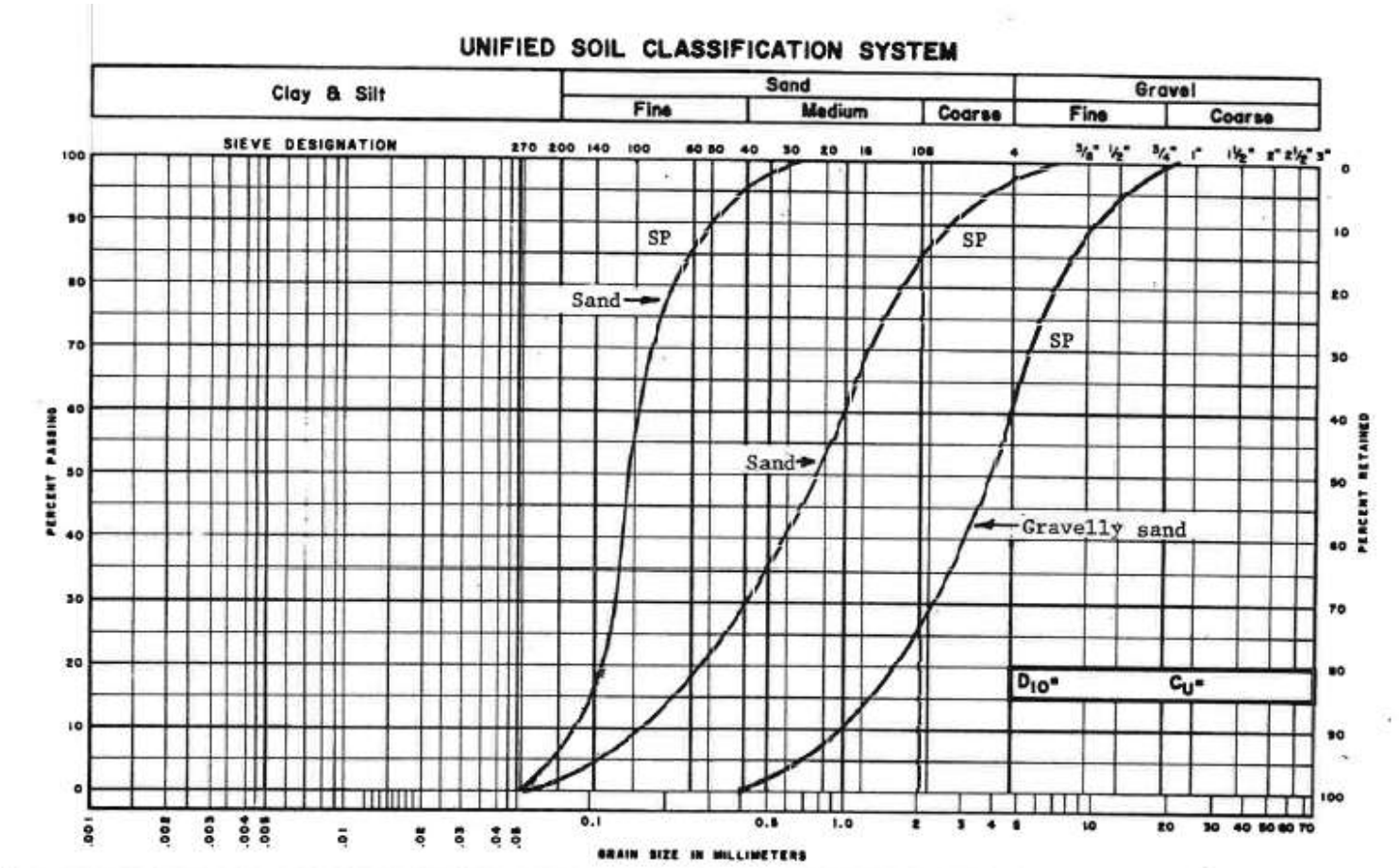
0803 3/82



MAY 1982

6.3.17

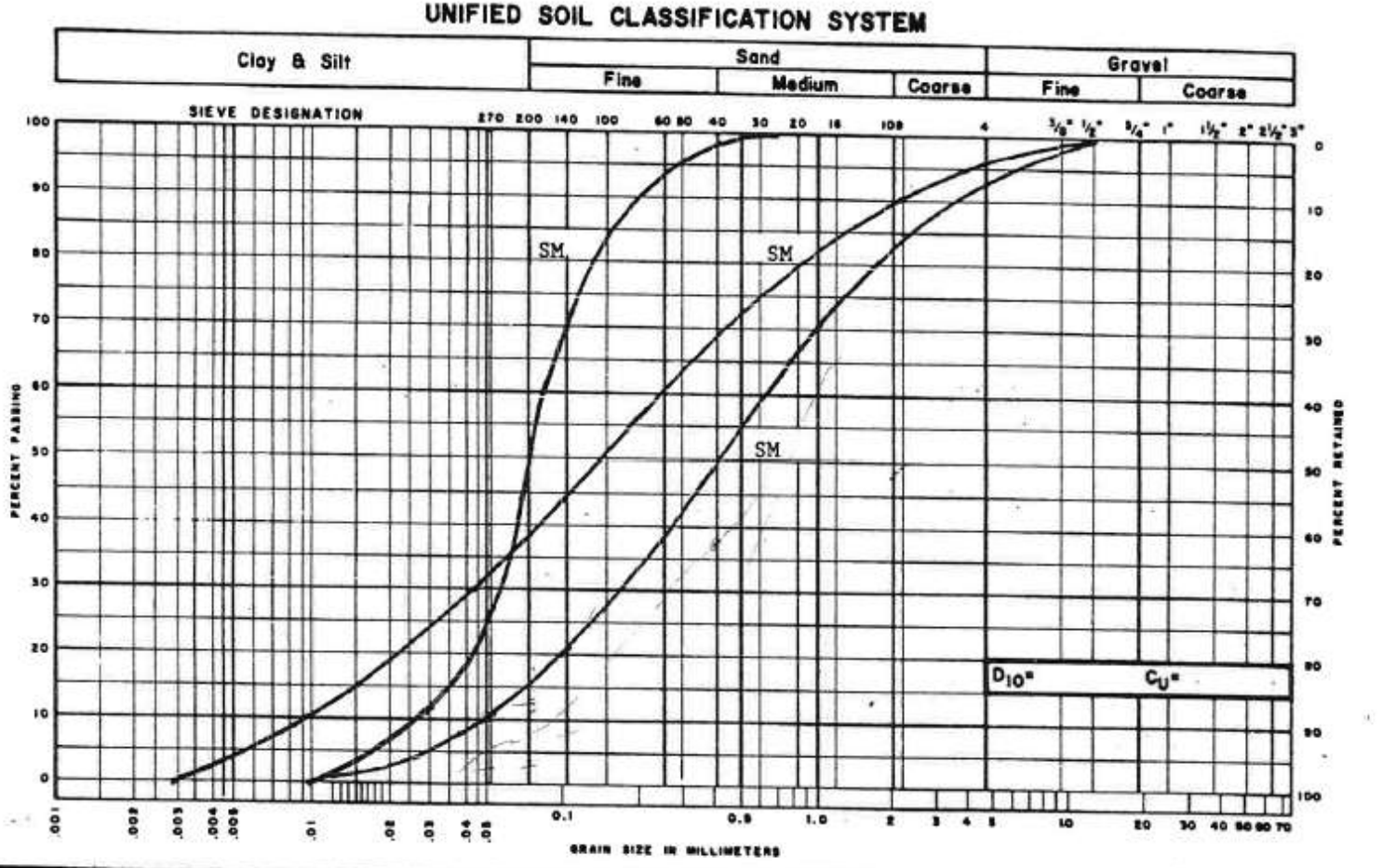
App. 6.3.2
p. 5 of 14



MAY 1982

6.3.18

App. 6.3.2
p. 6 of 14



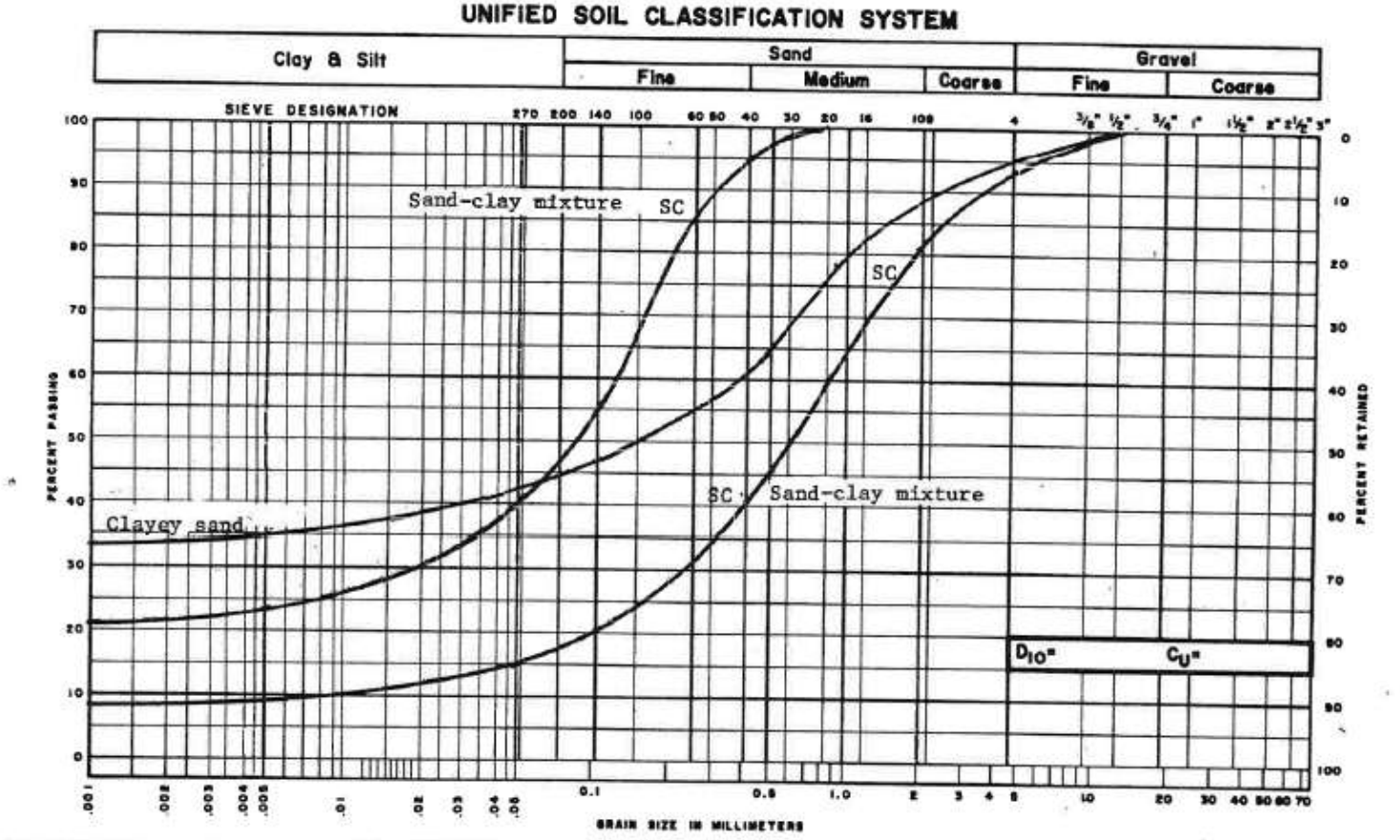
SM - Silty sands, sand-silt mixtures
- More than 12% finer than 0.074 mm
- Plasticity Index (I_p) less than 4
- Plots below "A" line on plasticity chart

0803 3/82

MAY 1982

6.3.19

App. 6.3.2
p. 7 of 14



SC - Clayey sands, sand-clay mixtures
- More than 12% finer than 0.074 mm
- Plasticity index greater than 7
- Plots above "A" line on plasticity chart

0803 3/82

MAY 1982

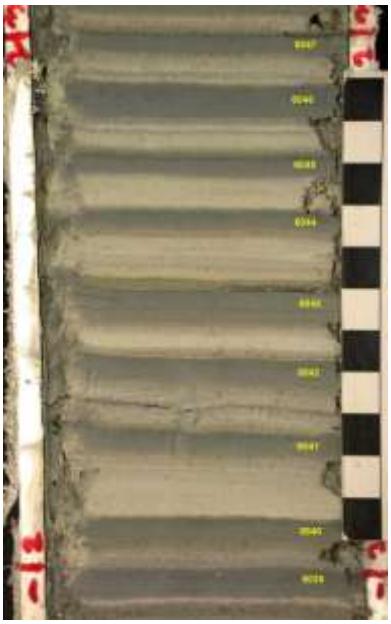
6.3.20

App. 6.3.2
p. 8 of 14

APPENDIX B

Example Descriptions for Varved/Laminated Soils

Appendix B – Example Soil Description for Varved/Laminated Soils



Example of a Varved soil:

(CH and ML) CLAY and SILT; grey and light brown, varved; w>PL, cohesive, soft

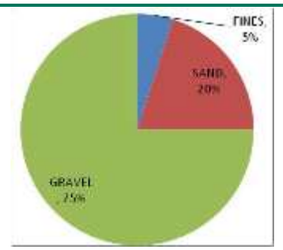
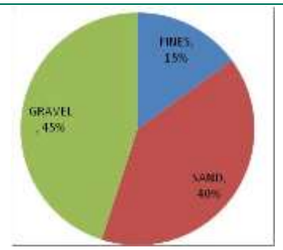
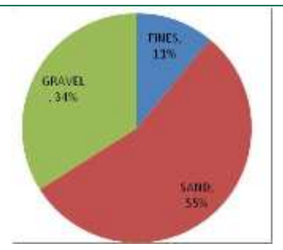
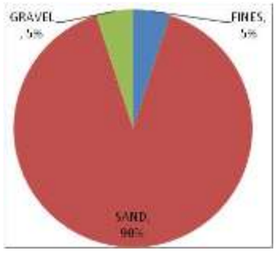
Example of a Laminated soil:

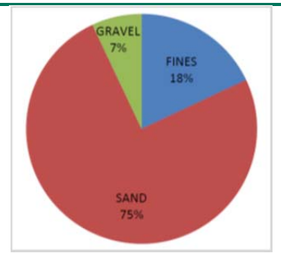
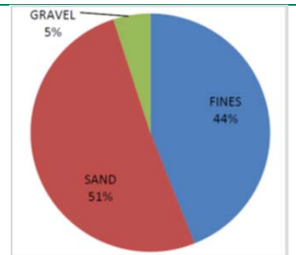
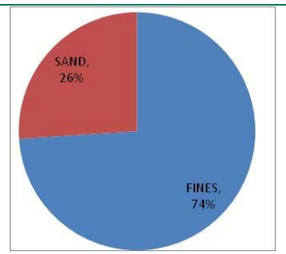
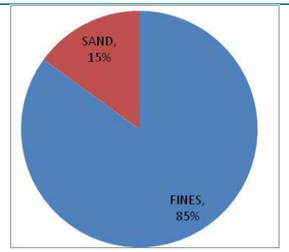
(CL, CH, ML, and SM) SILTY CLAY, CLAY, SILT, and SILTY SAND; grey and brown, thinly laminated; w>PL, cohesive, soft

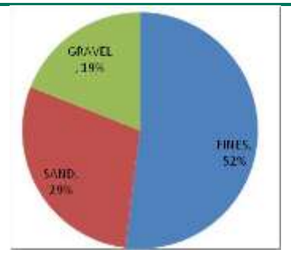
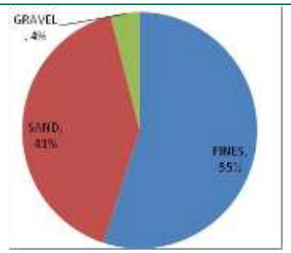
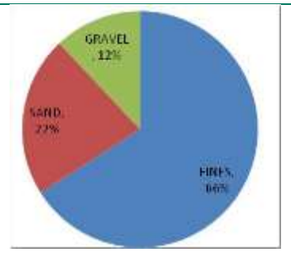
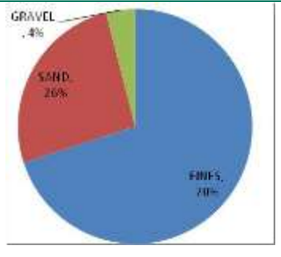
APPENDIX C

Example Soil Classifications and Descriptions

APPENDIX C – EXAMPLE SOIL CLASSIFICATIONS AND DESCRIPTIONS

% Fines (<0.075 mm)	% Sand (0.075 mm to 4.75 mm)	% Gravel (4.75 mm to 75 mm)		Well Graded or Poorly Graded	Liquid Limit (LL)	Plastic Limit (PL)	Plasticity Index (PI)	Water Content *w* (%)	Behavior	SPT (N)	Undrained Shear Strength	(Group Symbol) and Group Name, Constituents and minor components; General Characteristics; Bulk Behavior. Example soil description (comprehensive) Example soil description (abbreviated)
5%	20%	75%		Poorly Graded	Non-plastic			3	Non-cohesive	35	N/A	(GP) sandy GRAVEL, fine, poorly graded, angular, ~20% fine to coarse sand, trace non-plastic fines; light brown**;** non-cohesive, dry, dense. (GP) sandy GRAVEL, trace non-plastic fines; light brown**;** non-cohesive, dry, dense.
15%	40%	45%		Well Graded	Non-plastic			10	Non-cohesive	18	N/A	(GM/SM) SILTY GRAVEL, fine to coarse, and SAND, fine to coarse, well graded, rounded, non-plastic fines; grey; non-cohesive, moist, compact. (GM/SM) SILTY GRAVEL and SAND; grey; non-cohesive, moist, compact.
11%	55%	34%		Poorly Graded	Non-plastic			10	Non-cohesive	34	N/A	(SP-SM) gravelly SAND, fine to coarse, poorly graded, rounded; ~30 to 35% fine gravel, some non-plastic fines; light brown; non-cohesive, moist, dense. (SP-SM) gravelly SAND, some fines; light brown; non-cohesive, moist, dense.
5%	90%	5%		Well Graded	Non-plastic			18	Non-cohesive	6	N/A	(SW) SAND, fine to coarse, well graded, sub-angular, trace gravel, trace non-plastic fines; yellow brown; non-cohesive, wet, loose. (SW) SAND, trace gravel, trace non-plastic fines; yellow brown; non-cohesive, wet, loose.

% Fines (<0.075 mm)	% Sand (0.075 mm to 4.75 mm)	% Gravel (4.75 mm to 75 mm)		Well Graded or Poorly Graded	Liquid Limit (LL)	Plastic Limit (PL)	Plasticity Index (PI)	Water Content *w* (%)	Behavior	SPT (N)	Undrained Shear Strength	(Group Symbol) and Group Name, Constituents and minor components; General Characteristics; Bulk Behavior. Example soil description (comprehensive) Example soil description (abbreviated)
18%	75%	7%		Well graded	15	10	5	10	Non-cohesive	14	N/A	(SC) CLAYEY SAND, fine to coarse, subrounded sand, ~15% to 20% low plasticity fines, some fine sub-angular gravel; grey brown; non-cohesive, moist, compact. (SC) CLAYEY SAND, some gravel; grey brown; non-cohesive, moist, compact.
44%	51%	5%		Well graded	30	20	10	20	Cohesive	-	175 kPa 1.75 tsf	(SC) CLAYEY SAND, fine to coarse, ~40% to 45% medium plasticity fines, trace fine angular gravel; light brown; cohesive, w~PL, very stiff. (SC) CLAYEY SAND, trace gravel; light brown; cohesive, w~PL, very stiff.
74%	26%	0%		N/A	25	-	-	15	Non-cohesive	12	N/A	(ML) sandy SILT, non-plastic, ~30% fine to medium sand; grey brown; non-cohesive, moist, compact. (ML) sandy SILT; grey brown; non-cohesive, moist, compact.
85%	15%	0%		N/A	40	32	8	30	Cohesive	-	70 kPA 0.7 tsf	(ML) sandy CLAYEY SILT, medium plasticity, fine to medium; light brown, fissured; cohesive, w~PL, stiff. (ML) sandy CLAYEY SILT; light brown; cohesive, w~PL, stiff.

% Fines (<0.075 mm)	% Sand (0.075 mm to 4.75 mm)	% Gravel (4.75 mm to 75 mm)		Well Graded or Poorly Graded	Liquid Limit (LL)	Plastic Limit (PL)	Plasticity Index (PI)	Water Content *w* (%)	Behavior	SPT (N)	Undrained Shear Strength	(Group Symbol) and Group Name, Constituents and minor components; General Characteristics; Bulk Behavior. Example soil description (comprehensive) Example soil description (abbreviated)
52%	29%	19%		N/A	40	18	22	20	Cohesive	-	150 kPa 1.5 tsf	(CL/SC) gravelly sandy SILTY CLAY, medium plasticity, ~30% fine to coarse sand, ~ 20% fine gravel; orange brown; cohesive, w~PL, very stiff. (CL/SC) gravelly sandy SILTY CLAY; orange brown; cohesive, w~PL, very stiff.
55%	41%	4%		N/A	37	22	15	18	Cohesive	-	80 kPa 0.8 tsf	(CL) SILTY CLAY and SAND, medium plasticity, ~30 to 40% fine to coarse sand, trace fine sub-angular gravel; grey brown; cohesive, w<PL, stiff. (CL) SILTY CLAY and SAND, trace gravel; grey brown; cohesive, w<PL, stiff.
66%	22%	12%		N/A	48	23	25	20	Cohesive	-	250 kPa 2.5 tsf	(CL/CH) sandy SILTY CLAY, medium to high plasticity, ~ 20 to 30% fine to coarse sand, some fine angular gravel; orange brown; cohesive, w<PL, hard. (CL/CH) sandy SILTY CLAY, some gravel; orange brown; cohesive, w<PL, hard.
70%	26%	4%		N/A	60	25	35	35	Cohesive	-	75 kPa 0.75 tsf	(CH) sandy CLAY, high plasticity, ~20 to 30% fine to coarse sand, trace fine gravel; light brown; cohesive, w>PL, stiff. (CH) sandy CLAY, trace gravel; light brown; cohesive, w>PL, stiff.

APPENDIX D

Example Field Borehole Log (to be done), Field Borehole Log Templates, and Field "Cheat Sheets"

Golder Associates

Field Boring Log

DEPTH HOLE _____ **PROJ. NO.** _____ **PROJECT** _____ **BORING NO.** _____

DEPTH SOIL DRILL _____ **GA INSP.** _____ **DRILLING METHOD** _____ **SHEET** _____ **OF** _____

DEPTH ROCK CORE _____ **WEATHER** _____ **DRILLING COMPANY** _____ **SURFACE ELEV.** _____

ABANDONMENT _____ **DRILL RIG** _____ **DRILLER** _____ **DATUM** _____

DEPTHS _____/_____/_____/_____ **SAMPLER HAMMER TYPE** _____ **WT.** _____ **DROP** _____ **STARTED** _____/_____
WATER LEVEL CAVE-IN DATE-TIME NOTE TIME DATE

DEPTHS _____/_____/_____/_____ **HOLE LOCATION** _____ **COMPLETED** _____/_____
(DELAYED) WATER LEVEL CAVE-IN DATE-TIME NOTE TIME DATE

SAMPLE TYPES

	A.S.	AUGER SAMPLE
	C.S.	CHUNK SAMPLE
*	D.O.	DRIVE OPEN (SPT)
	D.S.	DENISON SAMPLE
	F.S.	FOIL SAMPLE
	P.S.	PITCHER SAMPLE
	S.C.	SOIL CORE
*	T.O.	THIN-WALLED, OPEN
*	T.P.	THIN-WALLED, PISTON
	W.S.	WASH SAMPLE
*	___	_____
*	___	_____
*	___	_____

* NOTE SIZE

ABBREVIATIONS

ANG	ANGULAR	GR	GRAY	R	RED
BL	BLACK	HE	HETEROGENEOUS	RES	RESIDUAL
BR	BROWN	HO	HOMOGENEOUS	RX	ROCK
C	COARSE	LYD	LAYERED	RND	ROUNDED
CIN	CAVE-IN	M	MEDIUM	SAT	SATURATED
CO	COHESIVE	MIC	MICACEOUS	SD	SAND
CL	CLAY	MOT	MOTTLED	SI	SILT
CLY	CLAYEY	MST	MOIST	SIY	SILTY
D	DRY	NC	NON-COHESIVE	SM	SOME
EL	ELONGATED	NP	NON-PLASTIC	TR	TRACE
F	FINE	OG	ORANGE	WL	WATER LEVEL
FL	FLAT	ORG	ORGANIC	WH	WEIGHT OF HAMMER
FRAG	FRAGMENTS	PP	POCKET PEN.	WR	WEIGHT OF RODS
GL	GRAVEL	PL	PLASTIC LIMIT	Y	YELLOW

ORDER OF DESCRIPTION

1) GROUP SYMBOL
2) SOIL GROUP NAME
3) PRIMARY COMPONENTS
4) SECONDARY COMPONENTS
5) MINOR COMPONENTS ;
6) COLOR
7) WEATHERING
8) STRUCTURE
9) SENSITIVITY
10) CONTAMINATION
11) MINEROLOGY
12) ORIGIN ;
13) BEHAVIOR (CO/NC)
14) MOISTURE/WATER CONTENT
15) DENSITY/CONSISTENCY

PLUS DESCRIPTION:
CL/SI: PLASTICITY
SD: SIZE, GRADING
GL: SIZE, GRADING, SHAPE, ROCK TYPE

PROPORTIONS

"TRACE"	0 – 5%
"SOME"	5 – 12%
PREFIX "-Y"	12 – 35%
"AND"	35 – 50%

NON-COHESIVE SOILS

RELATIVE DENSITY		BLOWS
VERY LOOSE	VLS	0 – 4
LOOSE	LS	4 – 10
COMPACT	CP	10 – 30
DENSE	DN	30 – 50
VERY DENSE	VDN	>50

MOISTURE CONDITION

DRY	SOIL FLOWS
MOIST	FEELS COOL
WET	WITH FREE WATER

COHESIVE SOILS

CONSISTENCY		PP(TSF)	FINGER PRESSURE
VERY SOFT	VS	<0.25	EXTRUDES
SOFT	S	0.25 - 0.5	MOLDS EASILY
FIRM	FM	0.5 – 1	MOLDS
STIFF	ST	1 – 2	THUMB INDENTS
VERY STIFF	VST	2 – 4	THUMBNAIL INDENTS
HARD	H	>4	RESISTS THUMBNAIL

WATER CONTENT - W

W < PL	CANNOT ROLL 4 mm THREAD
W ~ PL	CAN ROLL THREAD 2 – 4 mm
W > PL	CAN ROLL THREAD <2 mm

ELEV. / DEPTH	LITHOLOGY	SPT N / PP (TSF)	SAMPLES NO.	SAMPLES TYPE	SAMPLES HAMMER BLOWS PER 6 IN	SAMPLES REC / ATT	DEPTH	SAMPLE DESCRIPTION AND DRILLING NOTES

Golder Associates

Golder Associates

Field Boring Log

DEPTH HOLE _____ **PROJ. NO.** _____ **PROJECT** _____ **BORING NO.** _____

DEPTH SOIL DRILL _____ **GA INSP.** _____ **DRILLING METHOD** _____ **SHEET** _____ **OF** _____

DEPTH ROCK CORE_____ **WEATHER** _____ **DRILLING COMPANY** _____ **SURFACE ELEV.** _____

ABANDONMENT_____ **DRILL RIG** _____ **DRILLER** _____ **DATUM** _____

DEPTHS_____/_____/_____/_____ **SAMPLER HAMMER TYPE** _____ **WT.** _____ **DROP**_____ **STARTED** _____/_____
WATER LEVEL CAVE-IN DATE-TIME NOTE TIME DATE

DEPTHS_____/_____/_____/_____ **HOLE LOCATION** _____ **COMPLETED** _____/_____
(DELAYED) WATER LEVEL CAVE-IN DATE-TIME NOTE TIME DATE

SAMPLE TYPES

	A.S.	AUGER SAMPLE
	C.S.	CHUNK SAMPLE
*	D.O.	DRIVE OPEN (SPT)
	D.S.	DENISON SAMPLE
	F.S.	FOIL SAMPLE
	P.S.	PITCHER SAMPLE
	S.C.	SOIL CORE
*	T.O.	THIN-WALLED, OPEN
*	T.P.	THIN-WALLED, PISTON
	W.S.	WASH SAMPLE
*	____	_____
*	____	_____
*	____	_____

* NOTE SIZE

ABBREVIATIONS

ANG	ANGULAR	GR	GRAY	R	RED
BL	BLACK	HE	HETEROGENEOUS	RES	RESIDUAL
BR	BROWN	HO	HOMOGENEOUS	RX	ROCK
C	COARSE	LYD	LAYERED	RND	ROUNDED
CIN	CAVE-IN	M	MEDIUM	SAT	SATURATED
CO	COHESIVE	MIC	MICACEOUS	SD	SAND
CL	CLAY	MOT	MOTTLED	SI	SILT
CLY	CLAYEY	MST	MOIST	SIY	SILTY
D	DRY	NC	NON-COHESIVE	SM	SOME
EL	ELONGATED	NP	NON-PLASTIC	TR	TRACE
F	FINE	OG	ORANGE	WL	WATER LEVEL
FL	FLAT	ORG	ORGANIC	WH	WEIGHT OF HAMMER
FRAG	FRAGMENTS	PP	POCKET PEN.	WR	WEIGHT OF RODS
GL	GRAVEL	PL	PLASTIC LIMIT	Y	YELLOW

ORDER OF DESCRIPTION

CONSTITUENTS
1) GROUP SYMBOL
2) SOIL GROUP NAME
3) PRIMARY COMPONENTS
4) SECONDARY COMPONENTS
5) MINOR COMPONENTS;

PLUS DESCRIPTION:
CL/SI: PLASTICITY
SD: SIZE, GRADING
GL: SIZE, GRADING, SHAPE, ROCK TYPE

GENERAL
6) COLOR
7) WEATHERING
8) STRUCTURE
9) SENSITIVITY
10) CONTAMINATION
11) MINEROLOGY
12) ORIGIN;

BEHAVIOR
13) BEHAVIOR (CO/NC)
14) MOISTURE/WATER CONTENT
15) DENSITY/CONSISTENCY

PROPORTIONS

"TRACE"	0 – 5%
"SOME"	5 – 12%
PREFIX "-Y"	12 – 35%
"AND"	35 – 50%

NON-COHESIVE SOILS

RELATIVE DENSITY		BLOWS
VERY LOOSE	VLS	0 – 4
LOOSE	LS	4 – 10
COMPACT	CP	10 – 30
DENSE	DN	30 – 50
VERY DENSE	VDN	>50

MOISTURE CONDITION

DRY	SOIL FLOWS
MOIST	FEELS COOL
WET	WITH FREE WATER

COHESIVE SOILS

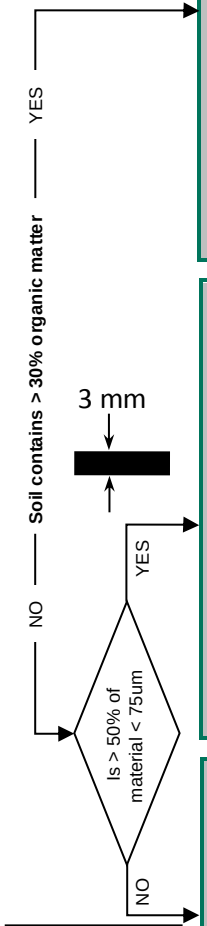
CONSISTENCY		PP(TSF)	FINGER PRESSURE
VERY SOFT	VS	<0.25	EXTRUDES
SOFT	S	0.25 - 0.5	MOLDS EASILY
FIRM	FM	0.5 – 1	MOLDS
STIFF	ST	1 – 2	THUMB INDENTS
VERY STIFF	VST	2 – 4	THUMBNAIL INDENTS
HARD	H	>4	RESISTS THUMBNAIL

WATER CONTENT - W

W < PL	CANNOT ROLL 4 mm THREAD
W ~ PL	CAN ROLL THREAD 2 – 4 mm
W > PL	CAN ROLL THREAD <2 mm

ELEV. / DEPTH	LITHOLOGY	SAMPLES						CONSTITUENTS			BEHAVIOR			USCS	SAMPLE DESCRIPTION AND DRILLING NOTES
		NO.	TYPE	DEPTH	SPT N / PP(TSF)	BLOWS PER 6 IN	REC / ATT	GL	SD	CL/SI	CO or NC	MOIST. or W	DENS./ CONS.		
								PROPORTION; SIZE, SHAPE, GRADING; PLASTICITY							

Golder Associates



*If soil has between 5% and 12% fines, dual symbols are required (e.g., SP-SM, SP-SC, GW-GM, GW-GP) .

	Category	Group Symbol and Name	Organic Content (% by mass)	Distinguishing Characteristics for Visual Identification
ORGANIC SOILS	Highly Organic Soils	(PT) Fibrous PEAT	75% to 100%	Light weight, spongy, and often elastic at natural water content. Plant structure easily identifiable. Shrinks considerably upon air drying. Much water squeezes from sample.
		(PT) Amorphous PEAT	75% to 100%	Light weight, spongy, but not usually elastic at natural water content. Plant structure visibly altered to unidentifiable. Shrinks considerably on air drying. Much water squeezes from sample.
		(PT) SILTY PEAT	30% to 75%	Relatively light weight, spongy. Thread usually weak and spongy near plastic limit. Shrinks on air drying; medium dry strength. Usually can squeeze water from sample readily. Low dilatancy.
		(PT) SANDY PEAT	30% to 75%	Sand fraction visible. Thread weak and friable near plastic limit, shrinks on air drying, low dry strength. Usually can squeeze water from sample readily, high dilatancy, "gritty."
	Organic Soils	(OH) ORGANIC SILT	5% to 30%	Often has strong H_2S odor. Thread may be tough depending on clay fraction. Medium dry strength, low dilatancy.
		(OL) ORGANIC SILT	5% to 30%	Threads weak and friable near plastic limit, or threads may not be rolled. Low dry strength; medium to high dilatancy.

FINE GRAINED SOILS	**Dilatancy**	None	None	None	None	Slow to V. Slow	Slow to V. Slow	Slow	Rapid
	Dry strength	High	Medium to High	Low to Medium	Medium to High	Low to Medium	Low to Medium	None to Low	None
	Shine Test	Shiny	Slight to Shiny	Slight	Dull to Slight	Slight	Dull to Slight	Dull	None
	Thread diameter (mm)	< 1	1 to 3	~ 3	1 to 3	3 to 6	3 to 6	3 to 6	> 6
	Toughness (of 3mm thread)	High	Medium	Low to Medium	Medium to High	Low to Medium	Low	None to Low	can't roll 3mm
	Organic content (%)	0 to 30	0 to 30	0 to 30	5 to 30	< 5	5 to 30	<5	<5
		↓	↓	↓	↓	↓	↓	↓	↓
	USCS Group Symbol	**CH**	**CL**	**CL**	**OH**	**MH**	**OL**	**ML**	**ML**
	Soil group name	CLAY	SILTY CLAY	SILTY CLAY	ORGANIC SILT	CLAYEY SILT	ORGANIC SILT	CLAYEY SILT	SILT

COARSE GRAINED SOILS	**Size of coarse fraction**	(>50% of coarse fraction is smaller than 4.75mm) SANDS				(>50% of coarse fraction is larger than 4.75mm) GRAVELS			
	Fines content	<12% fines*		>12% fines		<12% fines*		>12% fines	
	Gradation or plasticity	Well graded	Poorly graded	Plastic fines	Non-plastic fines	Well graded	Poorly graded	Plastic fines	Non-plastic fines
		↓	↓	↓	↓	↓	↓	↓	↓
	USCS Group Symbol	**SW**	**SP**	**SC**	**SM**	**GW**	**GP**	**GC**	**GM**
	Soil group name	SAND	SAND	CLAYEY SAND	SILTY SAND	GRAVEL	GRAVEL	CLAYEY GRAVEL	SILTY GRAVEL

Secondary and minor constituents

Constituents	% (by mass)	Modifier
Minor	≤ 5	Use "trace" or omit
	>5 to 12	Use "some"
Secondary	>12 to 35	Prefix primary soil name with "gravelly, clayey" as applicable.
	>35	Use 'and' to combine major constituents (i.e., SAND and GRAVEL, SILTY CLAY and SAND)

Order/Item/Parameter
1) FILL, REFUSE/WASTE, ROCK FILL, TAILINGS, TOPSOIL (if applicable).
2A) USCS Group Symbol – in (BLOCK LETTERS) e.g., (ML)
2B) Soil Group Name - in BLOCK LETTERS. e.g., CLAYEY SILT
3) Description of Primary Constituents; Particle size, grading (for sands and gravels containing <12% fines) and shape (optional) for coarse-grained soils. Plasticity for fine-grained soils.
4) Description of Secondary Constituents; including modifiers, with an estimate of their percentage (optional), and giving their particle size for coarse-grained or plasticity for fine-grained.
5) Other minor constituents including modifiers.
6) Color.
7) Weathering (if applicable).
8) Structure: e.g., zoning, fissuring, cementation, lamination, stratification, discrete layers or features (if applicable).
9) Sensitivity (if applicable).
10) Contamination, staining, and odor (if applicable).
11) Additional observations: origin or geological notes (e.g., TILL, ALLUVIUM, LOESS) or presence of cobbles/boulders, deleterious materials (e.g., with pieces of brick, asphalt and concrete) or mineralogy (e.g., calcareous, micaceous) (if applicable).
12) Behavior (non-cohesive or cohesive).
13) Field Moisture Condition (non-cohesive soils). Water Content (cohesive soils).
14) Compactness (non-cohesive soils). Consistency (cohesive soils).

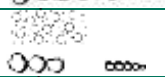
Particle sizes

Soil constituent	Particle size description	Particle Size	
		Millimetres	Inches (US std. sieve size)
BOULDERS	Not applicable	>300	>12
COBBLES	Not applicable	75 to 300	3 to 12
GRAVEL	Coarse	19 to 75	0.75 to 3
	Fine	4.75 to 19	(4) to 0.75
SAND	Coarse	2.00 to 4.75	(10) to (4)
	Medium	0.425 to 2.00	(40) to (10)
	Fine	0.075 to 0.425	(200) to (40)
SILT/CLAY	Not applicable	<0.075 mm	< (200)

Particle shape and angularity

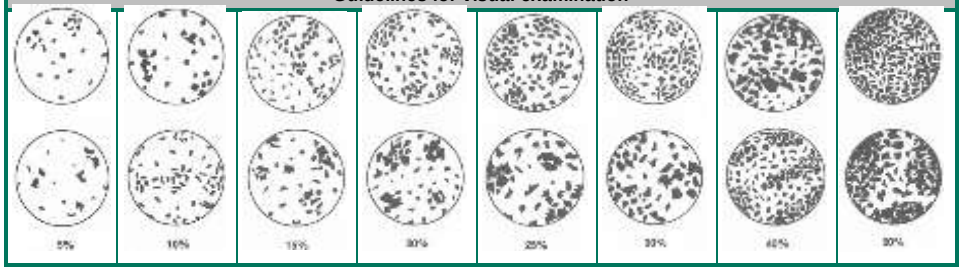
Shape and Angularity	Term	Description	Visual Example
Angularity	Rounded	Smoothly curved sides No edges Smooth or polished surfaces	
	Sub-rounded	Plane sides Well-rounded edges Partially polished surfaces	
	Sub-angular	Plane Sides Partially rounded edges Unpolished surfaces	
	Angular	Plane sides Sharp edges Unpolished surfaces	
Shape (Typically gravel)	Flat	Width to thickness ratio >3	
	Elongated	Length to width ratio >3	

Particle size distribution

Particle Size Distribution	Sub category	Description	Visual Example
Well Graded	-	Even distribution of particle sizes	
Poorly Graded	-	Uneven distribution of particle sizes	
	Gap Graded	Intermediate particle sizes absent	
	Uniformly Graded	Primarily one particle size	

Secondary and minor constituents

Guidelines for visual examination



Color

Color
Describe the color of the soil in its moist condition – Note if soil represents dry condition. Use primary color modified, if appropriate, with single adjective (e.g., grey, dark/pale grey). Borderline cases can be hyphenated, (e.g., grey-brown). Describe streaks or splotches of other colors as "mottled" (e.g., grey mottled red-brown). The suffix "ish" may be used if appropriate (reddish-brown).

Zoning and Fissures

Term	Description
Heterogeneous	Soil mass is of non-uniform, variable composition or structure.
Homogeneous	Soil mass is of uniform composition or structure.
Repetitive Structures	
Laminated	Closely spaced, alternating layers of differing soils and/or differing colors or shades of soils of similar gradation, usually arranged in a regular pattern. The following terms may be used: Thinly laminated – mean spacing under 6 mm (1/4 in) Thickly laminated – 6 to 20 mm (1/4 to 3/4 in)
Stratified, Layered, or Bedded	Differing soils or visible variations in soil constituents or color arranged in layers, generally but not necessarily parallel to one another. The following terms may be used: Very thinly bedded – 20 to 60 mm (3/4 to 2 1/2 in) Thinly bedded – 60 to 200 mm (2 1/4 to 8 in) Medium bedded – 200 to 600 mm (8 in to 2 ft) Thickly bedded – 600 to 2 m (2 to 6 ft) Very thickly bedded – over 2 m (over 6 ft)
Discrete layers or features	
Varved	A laminated soil consisting of two distinct soils (usually clay and silt) occurring in a regularly repeating pattern resulting from seasonal variations in sediment load in a lacustrine environment.
Lens	An inclusion of a different soil type within surrounding soils, which thins out laterally (horizontally) and may not be continuous over any significant distance. Typically identified by test pits or correlation between boreholes.
Parting	Paper thin separation of one soil type by another. Usually applied to fine grained soils.
Pocket	A different soil type of very limited thickness or lateral extent (a small lens).
Seam	A soil layer of considerable extent but with a thickness of less than about 10 mm (1/2 inch).
Term	**Field identification**
Fissured	Generally applied to dried or overconsolidated fine grained soils (silts and clays) containing cracks or physical discontinuities which can be vertical, horizontal, or inclined. Described as highly, moderately, and slightly fissured.
Friable, blocky, platy	Otherwise cohesive soil breaks into small (friable), larger (blocky) or thin, plate like (platy) fragments with little effort.
Slickensided	Polished or striated surfaces. Often an indication of an existing failure or slip surface. If continuous slickensided shear zones are found an estimate should be made of their angle in relation to the horizontal plane

Consistency (Cohesive Soils)

Term	Field identification	Unconfined compressive strength (tsf)	Unconfined compressive strength (kPa)	SPT 'N' (blows/foot) *
Very soft	Extrudes between fingers when squeezed	<0.25	<25	0 to 2
Soft	Molded by light finger pressure	0.25 to 0.5	25 to 50	2 to 4
Firm	Molded by strong finger pressure	0.5 to 1	50 to 100	4 to 8
Stiff	Indented by thumb	1 to 2	100 to 200	8 to 15
Very stiff	Indented by thumbnail	2 to 4	200 to 400	15 to 30
Hard	Difficult to indent with thumbnail	>4	>400	30

* Uncorrected for overburden pressure. 'N'-values should be considered ONLY an approximate guide to consistency; for sensitive clays the 'N'-value approximation for consistency terms does not apply.

Water content (Cohesive Soils)

Term	Field Identification
w < PL	Material is estimated to be drier than the Plastic Limit (cannot be rolled to a thread diameter of 4 mm).
w ~ PL	Material is estimated to be close to the Plastic Limit (can be rolled a thread diameter of between 2 mm and 4 mm).
w > PL	Material is estimated to be wetter than the Plastic Limit (can be rolled to a thread diameter of less than 2 mm).

Compactness (Non-cohesive soils)

Term	SPT 'N' (blows/foot) *	Field Identification of soil exposures
Very loose	0 to 4	Easily penetrated with shovel handle.
Loose	4 to 10	Easily penetrated with 12 mm (1/2 in) rebar pushed by hand. Easily excavated with hand shovel.
Compact	10 to 30	Easily penetrated with 12 mm (1/2 in) rebar driven with 2.3 kg (5 lb) hammer. Difficult to excavate with hand shovel.
Dense	30 to 50	Penetrated to a max depth of 300 mm (1 ft) with 12 mm (1/2 in) driven rebar. Must be loosened with pick to excavate.
Very dense	>50	Penetrated to a max depth of 50 mm (2 in) with 12 mm (1/2 inch) driven rebar. Very difficult to excavate even with pick.

* Uncorrected for overburden pressure

Field moisture condition (Non-cohesive soils)

Term	Field Identification
Dry	Soil flows freely through fingers.
Moist	Soils are darker than in the dry condition and may feel cool.
Wet	As moist, but with free water forming on hands when handled.

Notes:

- **<u>Odor of samples should only be recorded if incidentally noticed during regular inspection – Do not deliberately smell samples.</u>**
- **SPT**: The number of blows by a 63.5 kg. (140 lb.) hammer dropped 760 mm (30 in.) required to drive a 50 mm (2 in.) drive open sampler for a distance of 300 mm (12 in). The first interval is to "seat" the sampler, the second and third intervals are added together for the SPT N, (e.g., 4, 7, 11; N=18 4, 7, 11=Blows per 150mm (6 in.)). Where practical refusal occurs, the blows and penetration for that interval are reported.
  WR-Penetration occurred under the rod weight only. WH-Penetration occurred under the hammer and rod weight only.
- **<u>It is essential to note the type and weight of hammer used.</u>**
- **Soil description examples** ((GROUP symbol) Group Name, Constituents; Gen. Characteristics; Bulk Behavior).
  FILL - (SC) gravelly CLAYEY SAND; grey brown; non-cohesive, moist, compact.
  (GP) sandy GRAVEL, fine to coarse, subangular; light brown; non-cohesive, dry, dense.
  (CL) SILTY CLAY and SAND, trace fine gravel; grey brown; cohesive, w<PL, stiff.
  (ML) sandy SILT, trace gravel; brown, hydrocarbon odor; non-cohesive, moist, dense.
- **Groundwater not observed:** The observation of groundwater, whether present or not, was not possible due to drilling water, surface seepage, or cave in of the borehole/testpit.
- **Groundwater not encountered:** The borehole/test pit was dry after excavation.
- **Sample labeling**: Project #, Borehole/Testpit ID, Sample #, Depth, Date of sampling, Engineer or Technician name initials

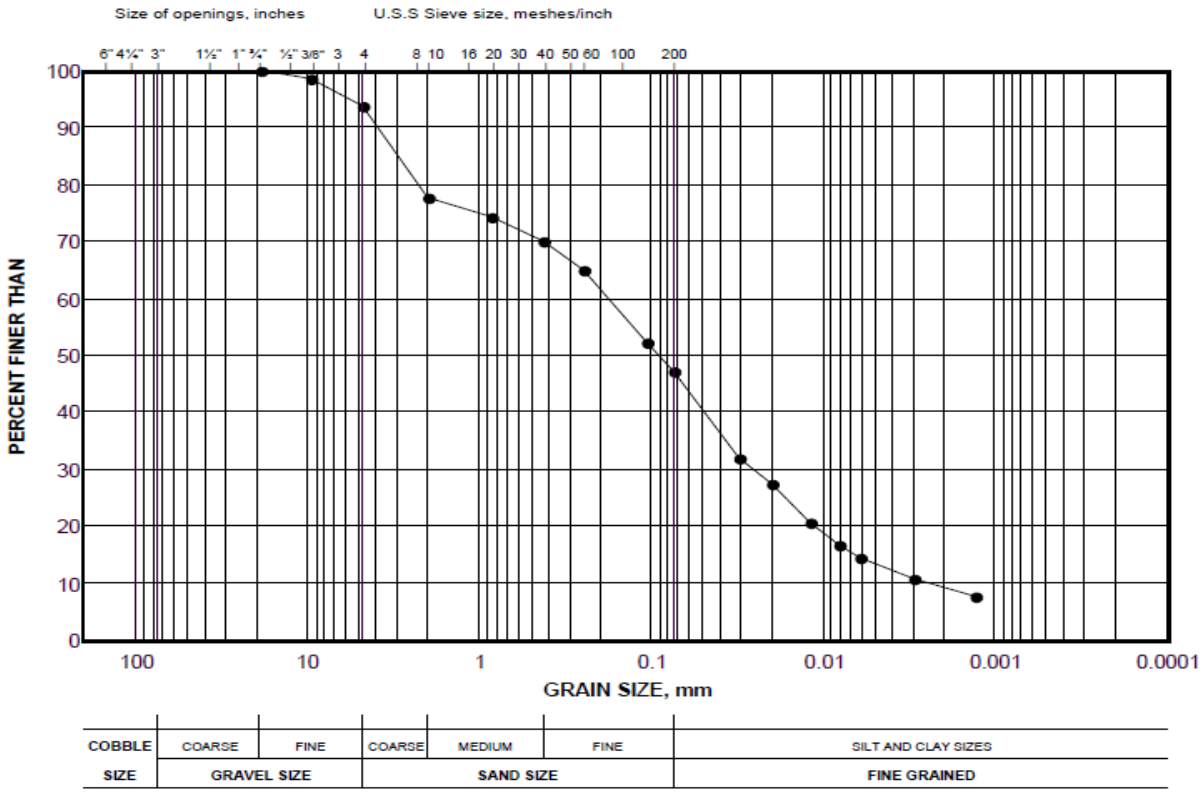
Sample labeling
Project Number Borehole/Testpit ID Sample Number Depth Date Initials

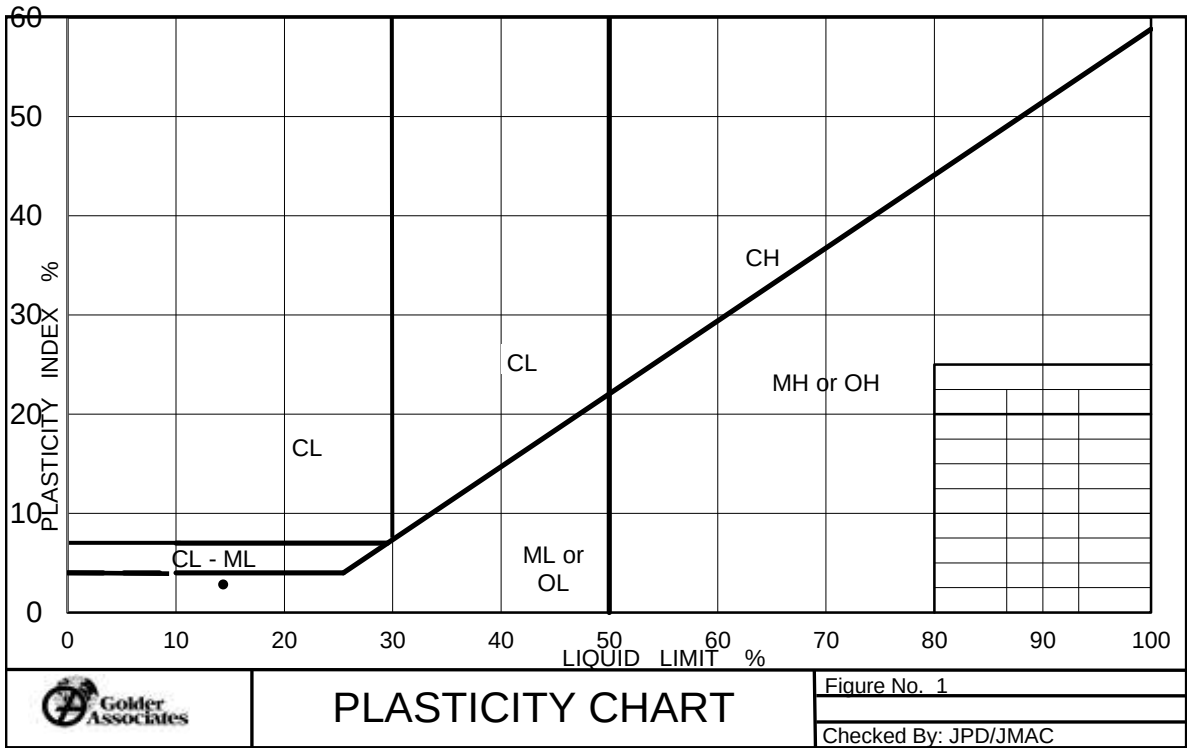
Samples			
AS	Auger sample	RC	Rock core
CS	Chunk sample	SC	Soil core
DO	Drive Open (SPT)*	ST	Slotted tube
DP	Direct Push	TO	Thin-walled, open*
DS	Denison type sample	TP	Thin-walled, piston*
FS	Foil sample	WS	Wash sample
PS	Pitcher type sample		

*Note size

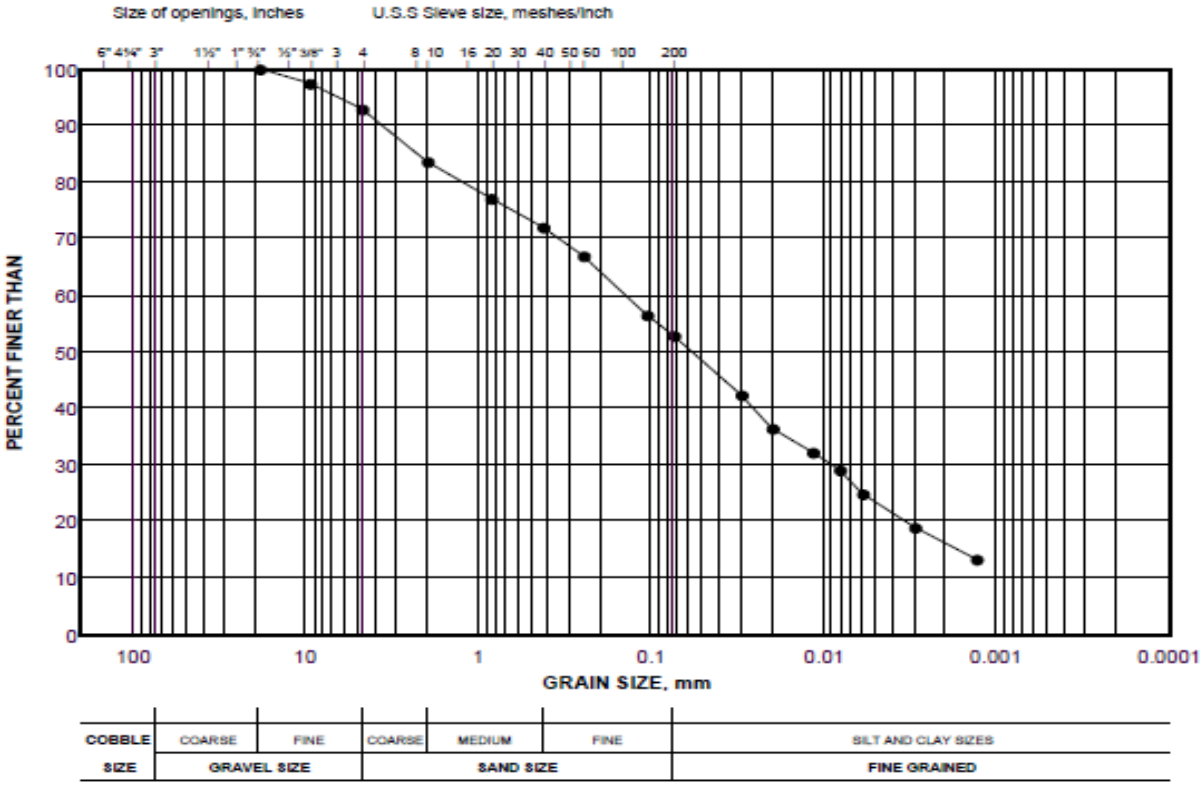
APPENDIX E

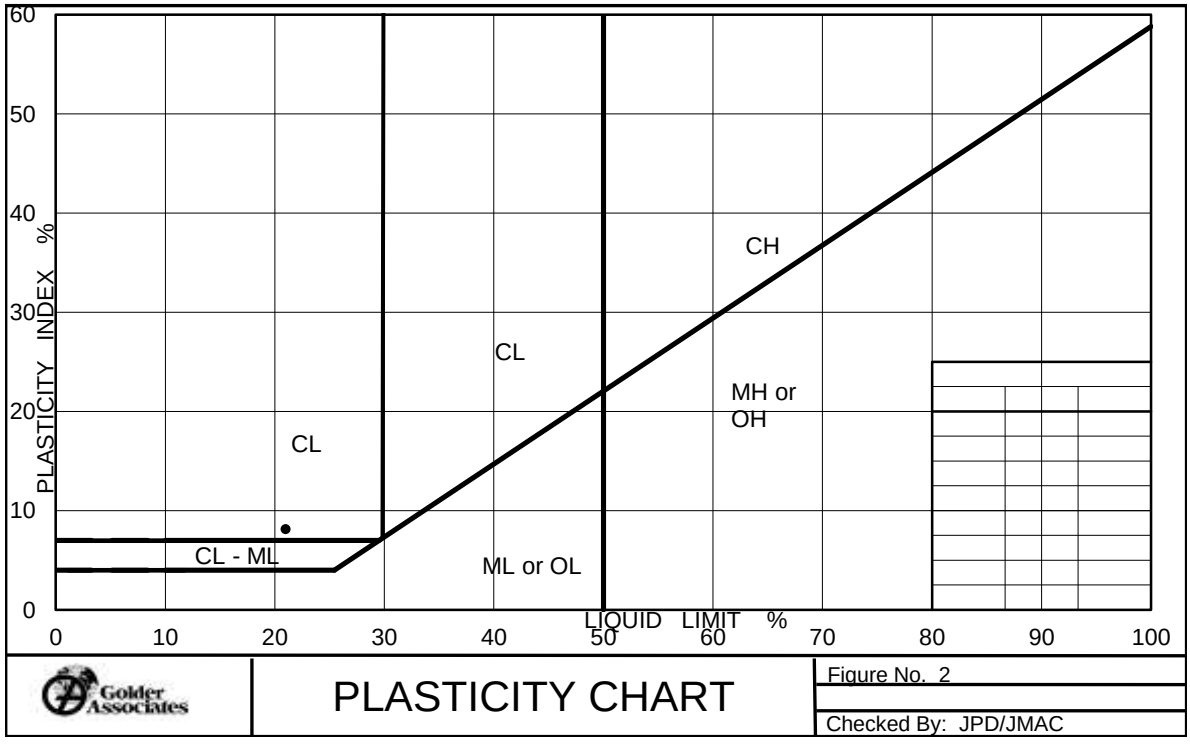
Example Soil Classifications based on Laboratory Test Results



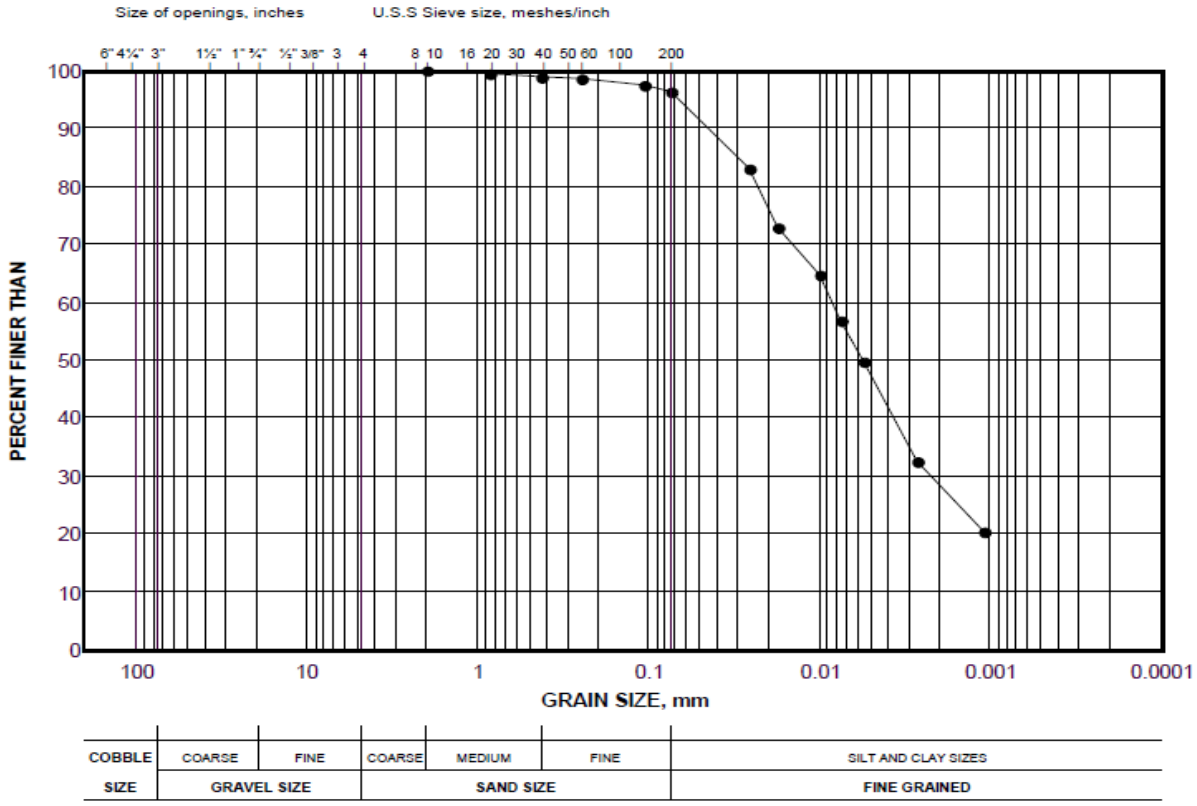


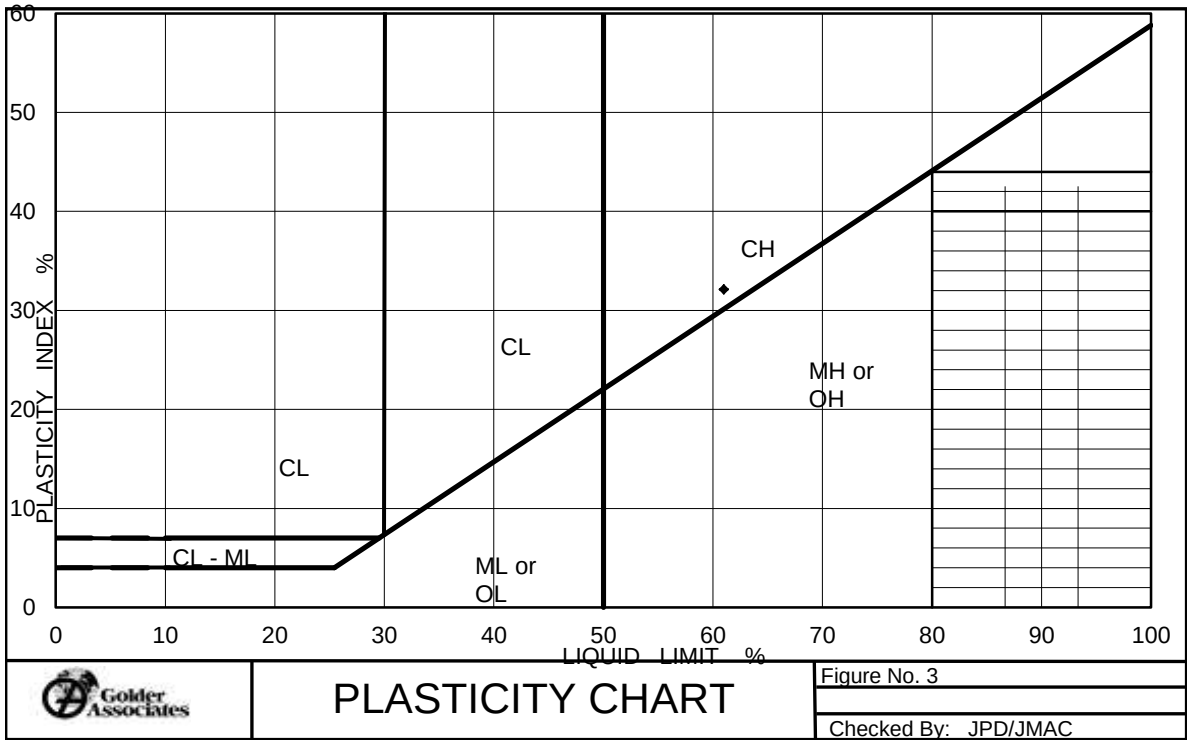
(SM) SAND and SILT, fine to coarse sand, slight plasticity, some fine gravel; (TILL)



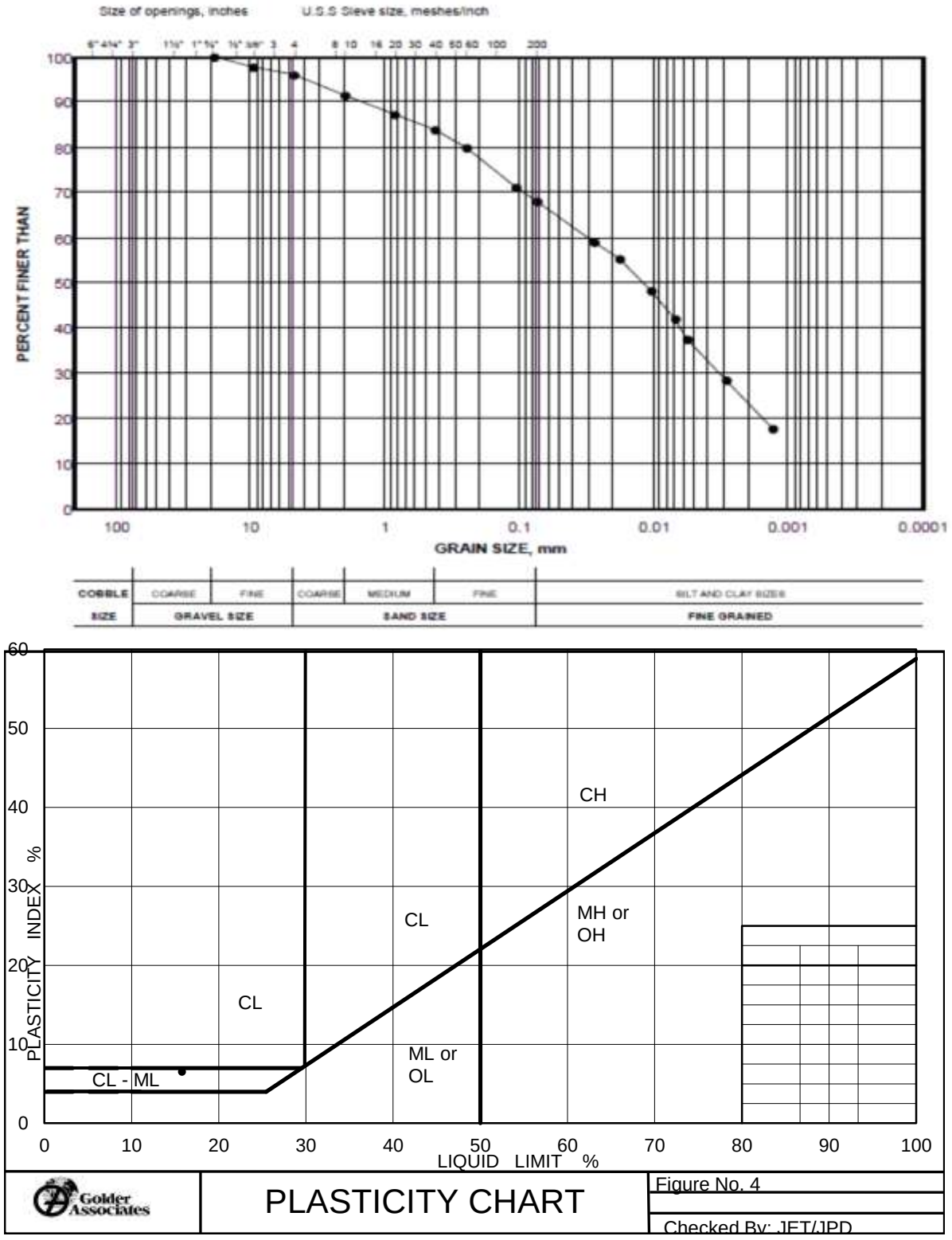


(CL) SILTY CLAY and SAND, low plasticity, fine to coarse sand, some fine gravel; (TILL)

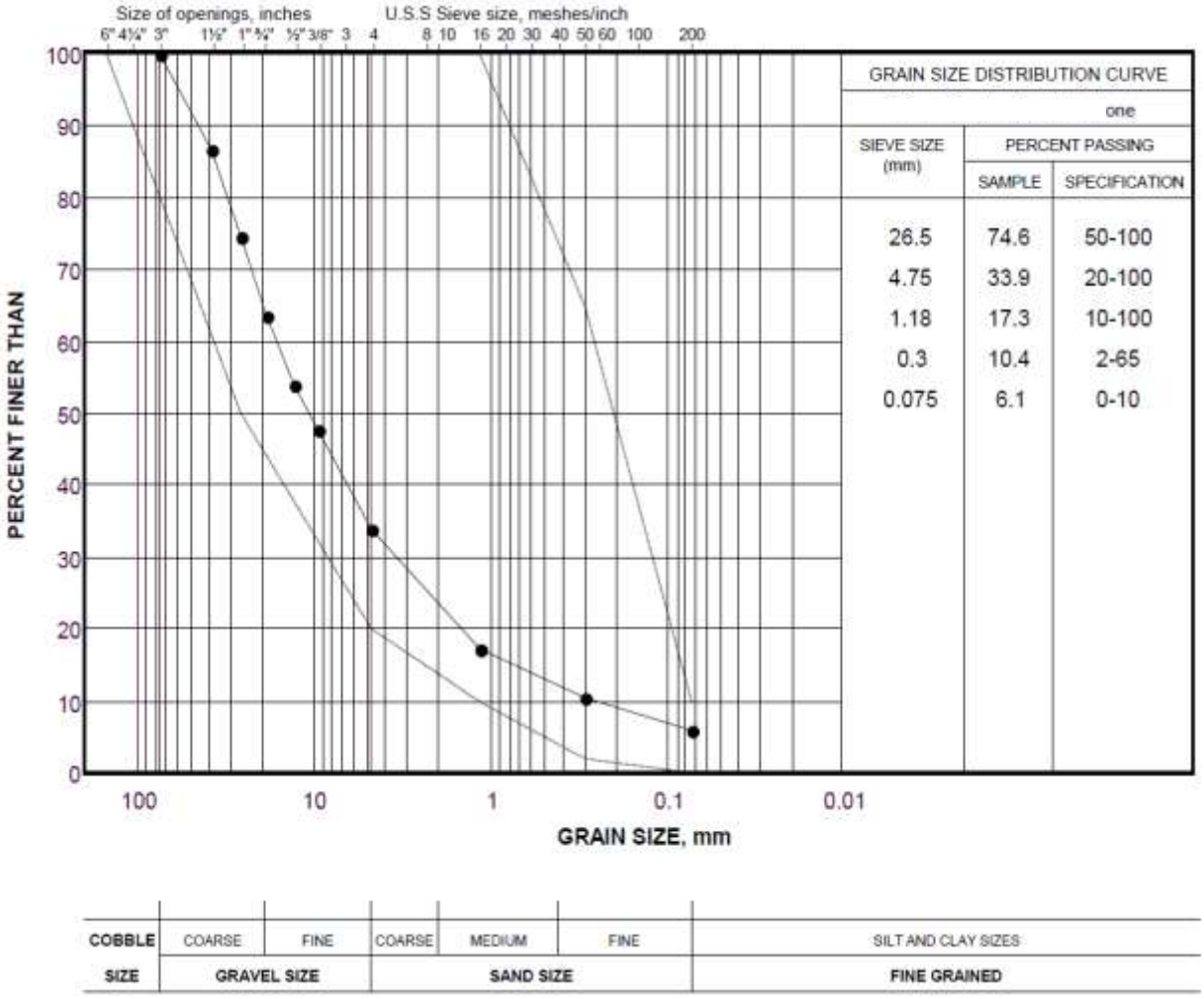




(CH) CLAY, trace sand



(CL-ML) sandy SILTY CLAY to CLAYEY SILT, low plasticity, fine to coarse sand, trace fine gravel

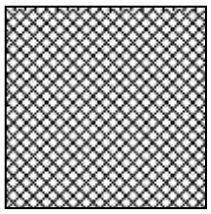


Limits – non-plastic

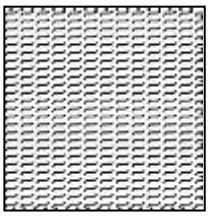
(GW-GM) sandy GRAVEL, fine to coarse gravel, fine to coarse sand, some non-plastic fines

APPENDIX F

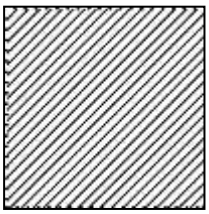
Example Record of Borehole Sheet (to be done) and Strata Symbols



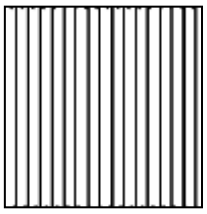
Fill/Overburden (1)



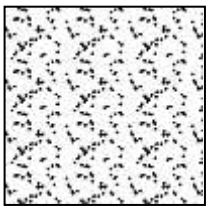
Organics/Topsoil (2)



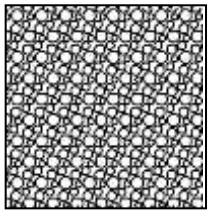
Clay (3)



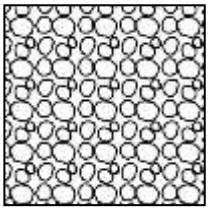
Silt (4)



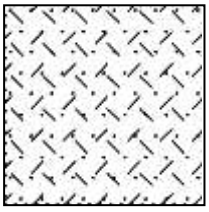
Sand (5)



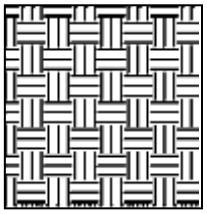
Gravel (6)



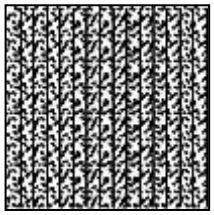
Cobbles (7)



Waste (27)



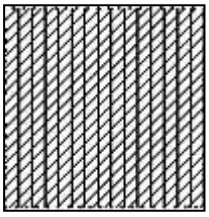
Bedrock (39)



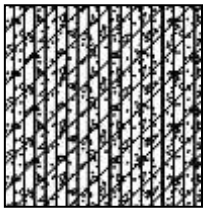
Sandy Silt (43)

Asphalt (45)

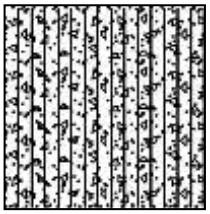
Bentonite Seal/Gypsum (61)



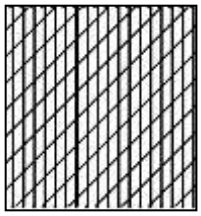
Silty Clay (65)



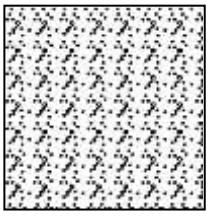
Clayey Silt Till (66)



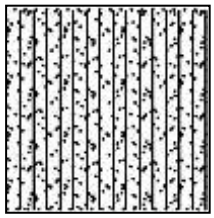
Sandy Silt Till (67)



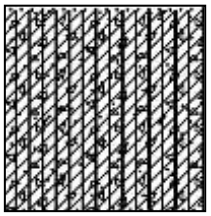
Clayey Silt (68)



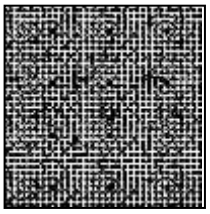
Sand and Gravel (69)



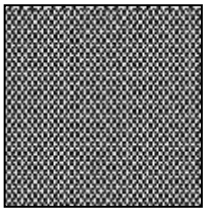
Silty Sand (70)



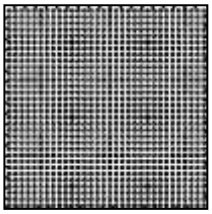
Silty Clay Till (71)



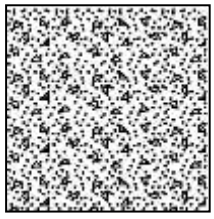
Cement Grout (72)



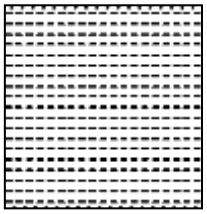
Bentonite Grout (73)



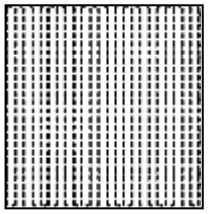
Grout (75)



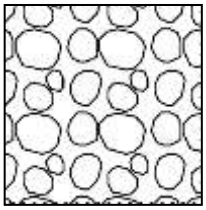
Concrete (82)



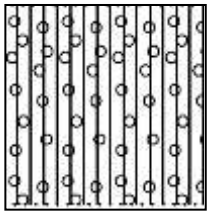
Water (83)



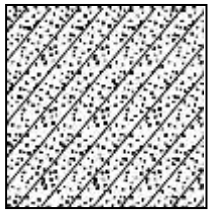
Ice (84)



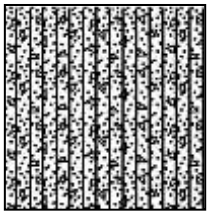
Boulders (90)



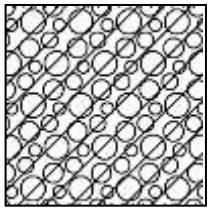
Silty Gravel (94)



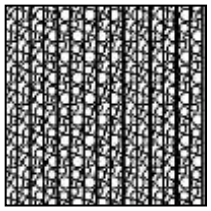
Clayey Sand (95)



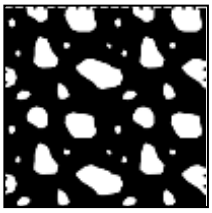
Silt Till (96)



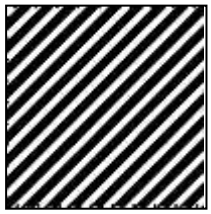
Clayey Gravel (126)



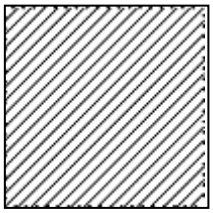
Silty Gravel (127)



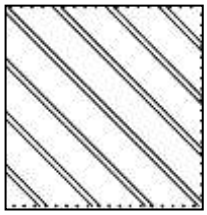
Boulders and Cobbles (BLDRCBBL)



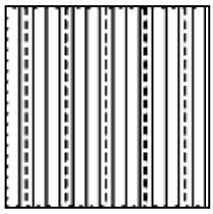
USCS High Plasticity Clay (CH)



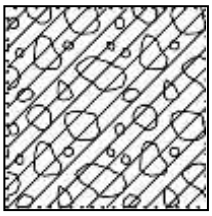
USCS Low Plasticity Clay (CL)



USCS Low to High Plasticity Clay (CL-CH) or Intermediate Plasticity Clay (CI)



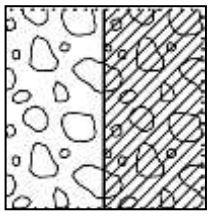
USCS Low Plasticity Silty Clay (CL-ML)



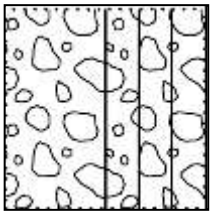
USCS Clayey Gravel (GC)

USCS Silty Gravel (GM)

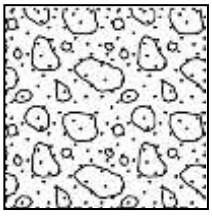
USCS Poorly-graded Gravel (GP)



USCS Poorly-graded Gravel with Clay (GP-GC)



USCS Poorly-graded Gravel with Silt (GP-GM)



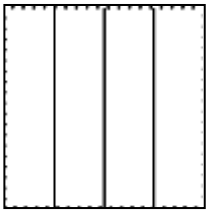
USCS Poorly-graded Sandy Gravel (GPS)

USCS Well-graded Gravel (GW)

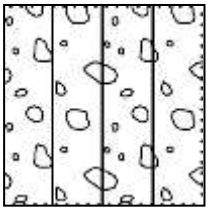
USCS Well-graded Gravel with Clay (GW-GC)

USCS Well-graded Gravel with Silt (GW-GM)

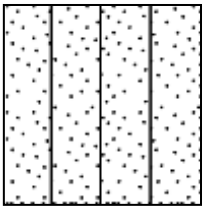
USCS Well-graded Sandy Gravel (GWS)



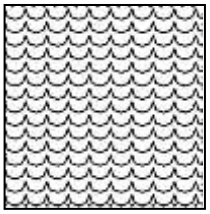
USCS Silt (ML)



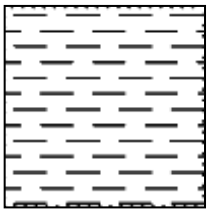
USCS Gravelly Silt (MLG)



USCS Sandy Silt (MLS)

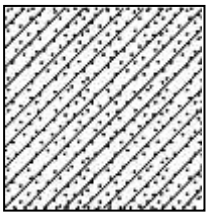


USCS High Plasticity Organic Silt or Clay (OH)

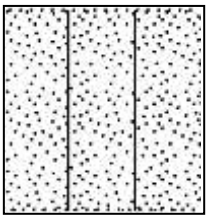


USCS Low Plasticity Organic Silt or Clay (OL)

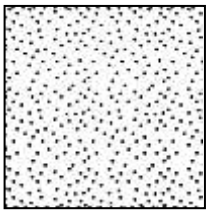
USCS Peat (PT)



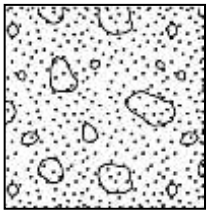
USCS Clayey Sand (SC)



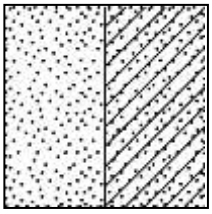
USCS Silty Sand (SM)



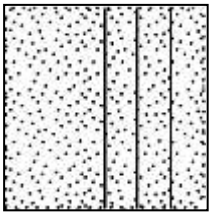
USCS Poorly-graded Sand (SP)



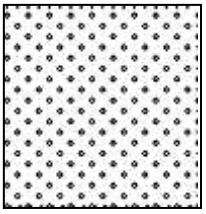
USCS Poorly-graded Gravelly Sand (SPG)



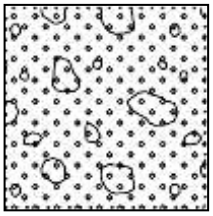
USCS Poorly-graded Sand with Clay (SP-SC)



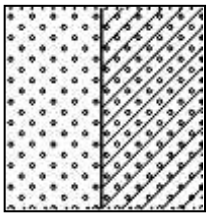
USCS Poorly-graded Sand with Silt (SP-SM)



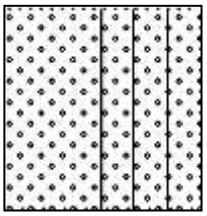
USCS Well-graded Sand (SW)



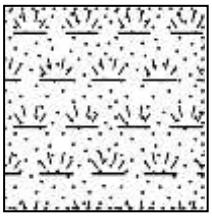
USCS Well-graded Gravelly Sand (SWG)



USCS Well-graded Sand with Clay (SW-SC)



USCS Well-graded Sand with Silt (SW-SM)



Topsoil (TOPSOIL)

APPENDIX G

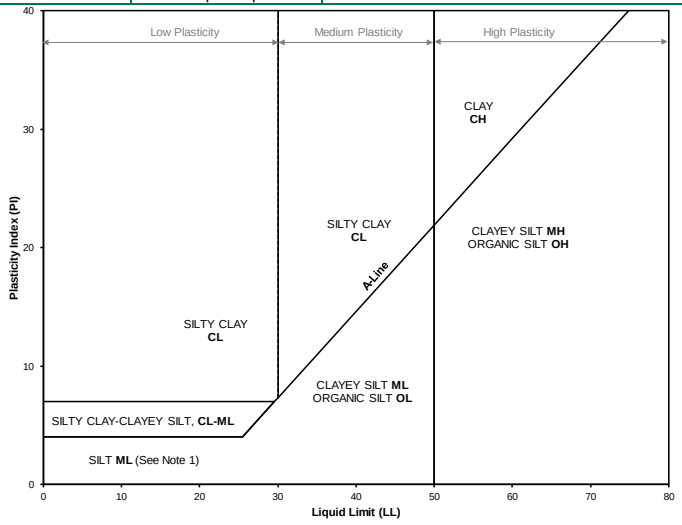
"Method of Soil Classification,"
"Abbreviations and Terms used on Record of Boreholes and Test Pits," and
"List of Symbols" Sheets

METHOD OF SOIL CLASSIFICATION

The Golder Associates Inc. Soil Classification System is based on the Unified Soil Classification System (USCS)

Organic or Inorganic	Soil Group	Type of Soil		Gradation or Plasticity	$Cu = \frac{D_{60}}{D_{10}}$	$Cc = \frac{(D_{30})^2}{D_{10} x D_{60}}$	Organic Content	USCS Group Symbol	Group Name
INORGANIC (Organic Content <30% by mass)	COARSE-GRAINED SOILS (>50% by mass is larger than 0.075 mm)	GRAVELS (>50% by mass of coarse fraction is larger than 4.75 mm)	Gravels with <12% * fines (by mass)	Poorly Graded	<4	≤1 or ≥3	<30%	GP	GRAVEL
				Well Graded	≥4	1 to 3		GW	GRAVEL
			Gravels with >12% fines (by mass)	Below A Line	n/a			GM	SILTY GRAVEL
				Above A Line	n/a			GC	CLAYEY GRAVEL
		SANDS (>50% by mass of coarse fraction is smaller than 4.75 mm)	Sands with <12% * fines (by mass)	Poorly Graded	<6	≤1 or ≥3		SP	SAND
				Well Graded	≥6	1 to 3		SW	SAND
			Sands with >12% fines (by mass)	Below A Line	n/a			SM	SILTY SAND
				Above A Line	n/a			SC	CLAYEY SAND

Organic or Inorganic	Soil Group	Type of Soil	Laboratory Tests	Field Indicators					Organic Content	USCS Group Symbol	Primary Name
				Dilatancy	Dry Strength	Shine Test	Thread Diameter	Toughness (of 3 mm thread)			
INORGANIC (Organic Content <30% by mass)	FINE-GRAINED SOILS (≥50% by mass is smaller than 0.075 mm)	SILTS (Non-Plastic or PI and LL plot below A-Line on Plasticity Chart below)	Liquid Limit <50	Rapid	None	None	>6 mm	N/A (can't roll 3 mm thread)	<5%	ML	SILT
				Slow	None to Low	Dull	3 mm to 6 mm	None to low	<5%	ML	CLAYEY SILT
				Slow to very slow	Low to medium	Dull to slight	3 mm to 6 mm	Low	5% to 30%	OL	ORGANIC SILT
			Liquid Limit ≥50	Slow to very slow	Low to medium	Slight	3 mm to 6 mm	Low to medium	<5%	MH	CLAYEY SILT
				None	Medium to high	Dull to slight	1 mm to 3 mm	Medium to high	5% to 30%	OH	ORGANIC SILT
		CLAYS (PI and LL plot above A-Line on Plasticity Chart below)	Liquid Limit <30	None	Low to medium	Slight to shiny	~ 3 mm	Low to medium	0% to 30% (see Note 2, below)	CL	SILTY CLAY
			Liquid Limit 30 to <50	None	Medium to high	Slight to shiny	1 mm to 3 mm	Medium		CL	SILTY CLAY
			Liquid Limit ≥50	None	High	Shiny	<1 mm	High		CH	CLAY
HIGHLY ORGANIC SOILS (Organic Content >30% by mass)		Peat and mineral soil mixtures							30% to 75%	PT	SILTY PEAT, SANDY PEAT
		Predominantly peat, may contain some mineral soil, fibrous, or amorphous peat							75% to 100%		PEAT



Note 1 – Fine-grained materials with PI and LL that plot in this area are named (ML) SILT with slight plasticity. Fine-grained materials which are Non-plastic (i.e. a PL cannot be measured) are named SILT.
Note 2 – For soils with <5% organic content, include the descriptor "trace organics." For soils with between 5% and 30% organic content include the prefix "organic" before the Primary name.

* Dual Symbol — A dual symbol is two symbols separated by a hyphen, for example, GP-GM, SW-SC, and, CL-ML. For non-cohesive soils, the dual symbols must be used when the soil has between 5% and 12% fines (i.e. to identify transitional material between "clean" and "dirty" sand or gravel). For cohesive soils, the dual symbol must be used when the liquid limit and plasticity index values plot in the CL-ML area of the plasticity chart (see plasticity chart at left).

Borderline Symbol — A borderline symbol is two symbols separated by a slash, for example, GM/SM, CL/ML. A borderline symbol should be used to indicate that the soil has been identified as having properties that are on the transition between similar materials. In addition, a borderline symbol may be used to indicate a range of similar soil types within a stratum.

METHOD OF SOIL CLASSIFICATION

PARTICLE SIZES OF CONSTITUENTS

Soil Constituent	Particle Size Description	Millimeters	Inches (US Std. Sieve Size)
BOULDERS	Not Applicable	> 300	> 12
COBBLES	Not Applicable	75 to 300	3 to 12
GRAVEL	Coarse Fine	19 to 75 4.75 to 19	0.75 to 3 (4) to 0.75
SAND	Coarse Medium Fine	2.00 to 4.75 0.425 to 2.00 0.075 to 0.425	(10) to (4) (40) to (10) (200) to (40)
SILT/CLAY	Classified by plasticity	< 0.075	< (200)

MODIFIERS FOR SECONDARY AND MINOR CONSTITUENTS

Percentage by Mass	Modifier
≤ 5	trace
> 5 to 12	some
> 12 to 35	Primary soil name prefixed with "gravelly, sandy, SILTY, CLAYEY" as applicable
> 35	Use 'and' to combine major constituents (*i.e.,* SAND and GRAVEL, SAND and CLAY)

PENETRATION RESISTANCE

Standard Penetration Resistance (SPT), N:

N = the number of blows required to drive a 2 inch (50 mm) split-spoon sampler one foot (300 mm) using a 140 lb (63.5 kg) hammer falling 30 inches (760 mm) after an initial 6 inch (150 mm) seating (ASTM D1586).

Cone Penetration Test (CPT):

An electronic cone penetrometer with a 60° conical tip and a typical projected end area of 10 or 15 cm^2 pushed through ground at a penetration rate of 2 cm/s. Measurements of tip resistance (q_t), porewater pressure (u) and sleeve friction (f_s) are recorded electronically in real time during penetration. The seismic CPT (SCPT) adds measurement of shear wave velocity (V_S) to the standard CPT.

Dynamic Cone Penetration Test (DCP), N_d:

The penetration rate by an 8 kg (17.6 lb) hammer dropped 575 mm (22.6 in.) to drive uncased a 20 mm (0.79 in.) diameter, 60° cone attached to 16 mm (5/8 in.) drive rods (ASTM D6951). Other test methods exist for DCPs with different configurations and different correlations.

PH: Sampler advanced by hydraulic pressure
PM: Sampler advanced by manual pressure
WH: Sampler advanced by static weight of hammer
WR: Sampler advanced by weight of sampler and rod

SAMPLE TYPES

AS	Auger sample
CS	Chunk sample
DO or DP	Drive open (SPT) or direct pushed tube sampler
DS	Denison type sample
FS	Foil sample
PS	Pitcher type sample
RC	Rock core
SC	Soil core
ST	Slotted tube
TO	Thin-walled, open
TP	Thin-walled, piston
WS	Wash sample

SOIL TESTS

M	water content
A	Atterberg limits (plastic and liquid limits)
G, H	grain size, hydrometer
UW	unit weight
Com	compaction
C	consolidation (oedometer) test
U	unconfined compression test
UU	unconsolidated undrained triaxial test
CD	consolidated isotropically drained triaxial test[1]
CU	consolidated isotropically undrained triaxial test with porewater pressure measurement[1]
D	direct shear test
V (FV)	field vane (LV-laboratory vane test)
SG	specific gravity
P	permeability
PD	pinhole dispersion
O	organic content test
PH	pH
CHEM	chemical analysis (refer to text)

1. Tests which are anisotropically consolidated prior to shear are shown as CAD, CAU.

NON-COHESIVE (COHESIONLESS) SOILS

Compactness[1]

Term	SPT 'N' (blows/foot)[2]
Very Loose	0 - 4
Loose	4 to 10
Compact	10 to 30
Dense	30 to 50
Very Dense	>50

1. Definition of compactness descriptions based on SPT 'N' ranges from Terzaghi and Peck (1967) and correspond to typical average N_{60} values.
2. SPT 'N' in accordance with ASTM D1586, uncorrected for overburden pressure effects. 'N'-values should be considered ONLY an approximate guide to consistency; for sensitive clays the 'N'-value approximation for consistency terms does not apply.

Field Moisture Condition

Term	Description
Dry	Soil flows freely through fingers.
Moist	Soils are darker than in the dry condition and may feel cool.
Wet	As moist, but with free water forming on hands when handled.

COHESIVE SOILS

Consistency

Term	Undrained Shear Strength (kPa)	Undrained Shear Strength (tsf)	SPT 'N'[1] (blows/foot)
Very Soft	<12	<0.12	0 to 2
Soft	12 to 25	0.12 to 0.25	2 to 4
Firm	25 to 50	0.25 to 0.5	4 to 8
Stiff	50 to 100	0.5 to 1	8 to 15
Very Stiff	100 to 200	1 to 2	15 to 30
Hard	>200	>2	>30

1. SPT 'N' in accordance with ASTM D1586, uncorrected for overburden pressure effects; approximate only.

Water Content

Term	Description
w < PL	Material is estimated to be drier than the Plastic Limit.
w ~ PL	Material is estimated to be close to the Plastic Limit.
w > PL	Material is estimated to be wetter than the Plastic Limit.

LIST OF SYMBOLS

Unless otherwise stated, the symbols employed in the report are as follows:

I. GENERAL

π	3.1416
ln x	natural logarithm of x
$\log_{10}$	x or log x, logarithm of x to base 10
g	acceleration due to gravity
t	time
ρ	density
γ	unit weight
	$\gamma = \rho g$

II. STRESS AND STRAIN

γ	shear strain
Δ	change in, e.g. in stress: $\Delta \sigma$
ε	linear strain
ε_v	volumetric strain
η	coefficient of viscosity
υ	Poisson's ratio
σ	total stress
σ'	effective stress
	$\sigma' = \sigma - u$
σ'_{vo}	initial effective overburden stress
$\sigma_1, \sigma_2, \sigma_3$	principal stress (major, intermediate, minor)
σ_{oct}	mean stress or octahedral stress
	$\sigma_{oct} = (\sigma_1 + \sigma_2 + \sigma_3) / 3$
τ	shear stress
u	porewater pressure
E	modulus of deformation
G	shear modulus of deformation
K	bulk modulus of compressibility

III. SOIL PROPERTIES

(a) Index Properties

ρ_t (γ_t)	bulk density (bulk unit weight)*
ρ_d (γ_d)	dry density (dry unit weight)
ρ_w (γ_w)	density (unit weight) of water
ρ_s (γ_s)	density (unit weight) of solid particles
γ'	unit weight of submerged soil
	$\gamma' = \gamma - \gamma_w$
e	void ratio
G_S	specific gravity of soil solids
	$G_S = \rho_s / \rho_w$
n	porosity
S	degree of saturation
w	water content
w_l or LL	liquid limit
w_p or PL	plastic limit
I_p or PI	plasticity index
	$PI = (w_l - w_p)$
w_s	shrinkage limit
I_L	liquidity index
	$I_L = (w - w_p) / PI$
I_C	relative consistency (consistency index)
	$I_C = (w_l - w) / I_p$
ρ_{dmax} (γ_{dmax})	index density (unit weight) in densest state
ρ_{dmin} (γ_{dmin})	index density (unit weight) in loosest state

(a) Index Properties (continued)

e_{max}	void ratio in loosest state
e_{min}	void ratio in densest state
D_R	relative density
	$D_R = (e_{max} - e) / (e_{max} - e_{min})$ or
	$D_R = \rho_{dmax} (\rho_d - \rho_{dmin}) / \rho_d (\rho_{dmax} - \rho_{dmin})$
I_D	density index
	$I_D = (\rho_d - \rho_{dmin}) / (\rho_{dmax} - \rho_{dmin})$

(b) Hydraulic Properties

h	hydraulic head or potential
q	rate of flow
v	velocity of flow
i	hydraulic gradient
k	hydraulic conductivity
	(coefficient of permeability)
j	seepage force per unit volume

(c) Consolidation (one-dimensional)

C_c	compression index
	(normally consolidated range)
C_r	recompression index
	(over-consolidated range)
C_s	swelling index
C_α	secondary compression index
m_v	coefficient of volume change
c_v	coefficient of consolidation
	(vertical direction)
c_h	coefficient of consolidation
	(horizontal direction)
T_v	time factor (vertical direction)
U	degree of consolidation
σ'_p	pre-consolidation stress
OCR	over-consolidation ratio
	$OCR = \sigma'_p / \sigma'_{vo}$

(d) Shear Strength

τ	shear strength
	$\tau = c' + \sigma' \tan \phi'$
τ_p, τ_r	peak and residual shear strength
c'	effective cohesion
ϕ'	effective angle of internal friction
μ	coefficient of friction
	$\mu = \tan \delta$
δ	angle of interface friction
c_u, s_u	undrained shear strength ($\phi = 0$ analysis)
	$s_u = q_u / 2$
q_u	unconfined compressive strength
	$q_u = (\sigma_1 - \sigma_3)$
S_t	sensitivity
p-q	diagram stress point
p	$(\sigma_1 + \sigma_3) / 2$
p'	$(\sigma'_1 + \sigma'_3) / 2$
q	$(\sigma_1 - \sigma_3) / 2$ or $(\sigma'_1 - \sigma'_3) / 2$

At Golder Associates we strive to be the most respected global company providing consulting, design, and construction services in earth, environment, and related areas of energy. Employee owned since our formation in 1960, our focus, unique culture and operating environment offer opportunities and the freedom to excel, which attracts the leading specialists in our fields. Golder professionals take the time to build an understanding of client needs and of the specific environments in which they operate. We continue to expand our technical capabilities and have experienced steady growth with employees who operate from offices located throughout Africa, Asia, Australasia, Europe, North America, and South America.

Africa	+ 27 11 254 4800
Asia	+ 86 21 6258 5522
Australasia	+ 61 3 8862 3500
Europe	+ 356 21 42 30 20
North America	+ 1 800 275 3281
South America	+ 55 21 3095 9500

solutions@golder.com
www.golder.com

Golder Associates

Date	December 15, 2017		
Revision by	None	Revision Date	None
RE: LOW-FLOW GROUNDWATER SAMPLING - STANDARD OPERATING PROCEDURES			

This groundwater sampling standard operating procedure (SOP) provides guidance for low-flow groundwater sampling.

1.0 REQUIRED EQUIPMENT AND MATERIALS

The following summarizes the required equipment for groundwater sampling at the Site:

- Groundwater Sampling Specific Items
  - Grundfos Redi-Flow II submersible pump (or equivalent)
  - Grundfos Redi-Flow II control box (or equivalent)
  - Water level meter
  - Interface probe
  - High-density polyethylene tubing
  - Water quality meter capable of in-line measurements for pH, specific conductance, dissolved oxygen (DO), temperature, turbidity, and oxidation-reduction potential (ORP)
  - Generator
  - Ferrous iron measurement field kit
  - Hose clamps

- General Items
  - Latex and/or nitrile gloves
  - Protective eye wear
  - Steel-toed boots
  - Reflective or high visibility clothing
  - Groundwater Sample Field Information Form
  - Chain of Custody (COC) form
  - Sample bottles and preservatives (provided by analytical laboratory)
  - Sample cooler (provided by analytical laboratory)
  - Deionized or distilled water
  - Alconox (or similar)
  - Isopropanol
  - 5-gallon buckets
  - Ice
  - Work Plan with sample location map
  - Well construction information

- Sample collection forms from prior event
- Site Specific Health and Safety Plan (HASP)

2.0 DOCUMENTATION

2.1 Field Books

Information pertinent to the field work will be recorded in bound and numbered project specific field notebooks. All field notes must be legible. Errors should be crossed out with a single line and initialed. Field records should at a minimum contain the following information for each day in the field:

- Date
- Project or site name
- Time of each data entry
- Description of work being performed that day
- Names and affiliations of personnel at location
- Weather conditions on site
- Location and type of activity
- Pertinent visual observations
- Pertinent field data measurements
- Serial numbers, if any, on custody seals, and transportation cases of equipment
- Name of field custodian
- Photographs taken, if any, including date, time, direction faced, description of subject or activity, sequential number of the photo and film roll number will be recorded in the field notebook
- Any deviations from the project scope of work, health and safety issues, or other relevant information should be recorded in the fieldbook. The project manager and health and safety officer should be contacted immediately in the event of any health and safety concerns

2.2 Field Forms

The following information should be recorded on the field forms for each monitoring location:

- Site name, location, and project number
- Date, time
- Well ID
- Depth to water
- Overall well depth
- Well construction (depth and screened interval)
- Water quality meter model and calibration time

- Start and end time for purging
- Pump intake depth
- Purge device
- Purge rate
- Depth to water, pH, ORP, temperature, specific conductance, turbidity and dissolved oxygen (every 5 minutes)
- Parameters to be analyzed
- Start and end time for sampling
- Pertinent observations regarding sample characteristics (turbidity, color, etc.)
- Results of field test kit measurements

2.3 Chains of Custody

A chain of custody form (COC) is a laboratory supplied form that documents sample collection information. The following information should be documented on the COC:

- Company and contact information
- Sample identification
- Sample date
- Sample time
- Sample matrix
- Analyses requested
- Sample turn-around-time
- Signature (with date and time) of person who is relinquishing the samples to the laboratory

Generally, the COCs include carbon copies. After signing the COC to relinquish the samples to the laboratory, the pink carbon copy should be removed from the stack and saved for record documentation. If no carbon copy is available, a photo of the completed form should be saved.

3.0 SYNOPTIC GROUNDWATER LEVEL MEASUREMENTS

Prior to groundwater sampling activities, a synoptic groundwater level round should be performed. Depth to water should be measured in each monitoring well to the nearest 0.01-foot using an electronic depth-indicating sounder (water level meter). Additionally, well depths should be gauged at least annually to evaluate the integrity of the well.

When measuring the groundwater levels, the water level meter should be lowered slowly until the sounder initiates. When the sounder initiates, the field measurement should be duplicated three times for accuracy. A second person should visibly confirm the field measurement and then record it in the field book. Previous

records of groundwater level measurements should also be utilized to confirm the reasonableness of the field measurements.

4.0 GENERAL GROUNDWATER SAMPLING PROCEDURES

4.1 Overview

Groundwater samples should be collected using the following modified "low-flow sampling technique" based on USEPA-recommended procedures (i.e., *Low Flow (Minimal Drawdown) Groundwater Sampling Procedures*, EPA/540/S-95/504, April 1996.

Wells should be purged and sampled using a stainless-steel submersible positive displacement pump that is thoroughly decontaminated between sample locations. Dedicated HDPE tubing should be used in the wells: Tubing that is reused during each monitoring event, should not be allowed to come in contact with the ground surface or any other location that could introduce contamination to the surface of the tubing.

The tubing should be fastened to the pump with a barbed fitting and hose clamp. The pump should be lowered to a depth such that the intake is within the open or screened interval of the well. The pump and tubing should be lowered slowly into position to minimize mixing of the groundwater in the well casing and agitation of any solids. Pumps should not be placed at a depth greater than 300 feet, to avoid exceeding the functional capacity of the pump. A minimum vertical separation of two feet should be maintained between the pump intake and the well bottom. Wells should be purged according to procedures detailed below in Section 5.4.

The tubing that coming out of the well that is connected to the pump is connected to the flow-through cell of a water quality meter. After purging, the tubing should be disconnected from the intake of the flow-through cell, the flow rate should be reduced to less than 0.5 gpm (e.g., 400 mL/min), and groundwater samples should be collected directly from the tubing into laboratory-supplied containers.

After all the sampled bottles are filled and prior to turning the pump off, field samples for ferrous iron analysis should collected directly from the tubing via a Hach® color disc field kit (or similar) and the results should be documented on the field forms.

Following sampling, tubing should be either left dedicated in individual wells or stored separately in dedicated bags and labeled for use at the same well during future events. Expended polyethylene tubing should be disposed of in an on-Site general refuse dumpster.

4.2 Equipment Decontamination

Prior to each use, the following procedures will be used for all non-dedicated sampling equipment used to collect groundwater samples:

1. Rinse with tap water and soap using a brush to remove obvious particulate matter and surface films
2. Cycle a tap water and Alconox (or similar) solution through the pump for approximately two to five minutes
3. Purge deionized or distilled water through the pump to remove Alconox solution until discharged water is visibly free of solution
4. Cycle thoroughly with pesticide-grade isopropanol and distilled water solution
5. Purge deionized or distilled water through the pump to remove isopropanol solution until discharged water is visibly free of solution
6. Equipment should be allowed to air dry then wrapped in aluminum foil until needed for sampling.

Water level meters will be thoroughly rinsed with distilled or deionized water prior to each use.

4.3 Equipment Set-up

All monitoring wells will be inspected for signs of damage, tampering, or unauthorized access. Prior to purging the monitoring well, a field decontaminated electronic water level meter will be used to measure the depth from the top of the inner well casing (or equivalent measuring point) to the top of water surface to the nearest 0.01 feet.

Periodically (e.g. annually), the depth to the bottom of the well should be measured to confirm well construction details, however such measurements should not be made immediately prior to well purging or sample collection in order to minimize potential cross-contamination and disturbance to sediments which may have accumulated in the bottom of the well.

The pump should be lowered into the well. The pump should then be connected to the control box. The control box is then connected to the generator for power. Once the pump is started, the power to the pump should be increased slowly by using the controls until an adequate flow rate is achieved.

4.4 Groundwater Purging

A modified “low-flow sampling technique” should be practiced at the Site. Because of the large vertical length of the open boreholes that are being monitored, a flow rate of approximately 0.5 gallons per minute (gpm) should be used to ensure that formation water is being appropriately monitored. This flow rate can be reduced if slower groundwater recharge rates are observed.

During purging, monitor the water level and adjust pumping rate such that the water level drawdown is minimized (ideally no more than 0.3 feet drawdown during purge). If drawdown is excessive, the pump rate should be reduced (noting the purge rates on the field form). There must be at least 2 feet of water maintained over the pump intake to prevent pump suction being broken, or entrainment of air in the sample. Record all pumping rate adjustments and depth(s) to water on the sample collection form.

Attach a Horiba U-52 water quality meter (or equivalent), has been calibrated to the manufacturer's specifications and in-line flow-through cell to the discharge tubing. Measurements are taken with a flow-through cell so that they are recorded prior to the sample being exposed to the atmosphere. During purging, monitor the field parameters (temperature, pH, turbidity, specific conductance, Oxygen Reduction Potential [ORP], and dissolved oxygen [DO]) with the Horiba approximately every 5 minutes (or at a minimum of one flow cell volume exchange) until the parameters have stabilized over three (3) consecutive readings. In general, stabilization is considered achieved if:

- pH is within +/- 0.1;
- conductivity is within +/- 3%;
- temperature is within +/- 10%;
- ORP is within +/- 10 mV;
- turbidity is within +/- 10% (or is less than 50 NTU); and
- DO is within +/- 10% (or within 0.1 mg/l when the DO is less than 1 mg/l).

All measurements will be recorded on the sample collection form or in field notebooks.

4.5 Groundwater Sampling

Once all measured groundwater parameters have stabilized for three consecutive readings, lower the purge rate to approximately 400 milliliters per minute (ml/min). Detach the tubing from the water quality meter and begin filling the laboratory supplied bottles directly from the discharge of the tubing. Since the groundwater is not treated once detached from the carbon filtration units, capture all purge water in buckets or similar containers. Groundwater sample bottles should be filled in the following sequence of parameters:

- Volatile organic compounds (VOCs)
- Semi-VOCs (SVOCs)
- Metals (dissolved and total)
- Natural Attenuation Parameters (NAPs)

Sample bottleware should correspond with the requested analysis of the groundwater sample. A list of sample parameters and their associated bottleware should be provided by laboratory. This list should be checked against the bottleware that is being used to collect the sample.

Laboratory supplied sample labels should be filled out completely and placed on the appropriate bottles. Typical items on the sample label include the following information: sample identification, project, analysis, date, time, and sample preservation.

4.6 Groundwater Sample Storage and Shipment

After collection, all groundwater samples should be appropriately labeled and stored in laboratory supplied coolers with ice at a temperature of approximately 4 degrees Celsius. The samples should remain in the cooler until the COC has been appropriately filled out and signed and the samples are relinquished to the laboratory by courier for analysis.

4.7 Quality Assurance/Quality Control

Rinsate blanks (RBs), field duplicates (FDs), and matrix spike/matrix spike duplicates (MS/MSDs) should be collected at a frequency of one (1) per twenty (20) primary groundwater samples. Daily trip blanks (TBs) should also be included to detect any potential sample contamination during transport of samples.

5.0 ADDITIONAL PROCEDURES

5.1 Investigation Derived Waste

Any liquid investigation derived waste (IDW), such as purge or decontamination liquids, should be containerized (i.e., 5-gallon buckets) at the source of generation. The containerized liquid IDW should then be emptied into the sump connected to the 10,000-gallon liquid storage tank in the on-site water treatment building.

Solid IDW (i.e., gloves, tubing, sample containers, etc.) will be disposed of in an on-Site general refuse dumpster.

5.2 Water Quality Meter Calibration

The Horiba U-52 (Horiba) provides real time field measurements for the following parameters: temperature, pH, DO, turbidity, specific conductance, and ORP. This meter requires daily calibration and periodic checks throughout the work day.

To calibrate the Horiba, a vendor supplied calibration liquid (AutoCal) is utilized. The Horiba should be powered on. On the control face of the Horiba a “Cal” function is present and should be pressed. On the screen two options will appear, Auto Calibration or Manual Calibration. Auto Calibration should be selected

using the arrows on the control face. The screen instructions will require immersion of the meter in the AutoCal solution using the supplied calibration cup. The Horiba sensors should be placed into the cup and the AutoCal solution should be poured into the cup so that every sensor is submerged in the solution. Once the sensors are submerged, press Enter and the Horiba will begin to calibrate based on the AutoCal solution. The readings will blink on the screen as the calibration is taking place. Once the readings stop blinking, calibration of the AutoCal solution is complete.

Additional calibration of DO is required using a zero-percent DO solution. The solution should be applied to the sensor in the same manner as the AutoCal. But the procedure requires manual calibration to be selected. Then select DO as the parameter and a single point calibration. Press enter and wait for the reading to reach zero and then hit enter again. The Horiba is now calibrated.

5.3 Water Level Meter Calibration

The graduated tape of an individual electronic water level meter should be checked against a similarly graduated engineer's measuring tape.

If multiple meters/probes will be used during the event, they should be compared by measuring the water level at a specific well to verify measurement consistency. If possible, this calibration procedure should be conducted in two wells: one of the shallowest at the site and one of the deepest. These calibrations must be performed prior to each field event. Any deviations from the engineer's tape, or differences between water level meters, should be documented and taken into account when the measured water levels or product levels are converted to elevations.

golder.com

golder.com