QUALITY ASSURANCE PROJECT PLAN

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Revised/Updated
December 2016

TABLE OF CONTENTS

SECTION **PAGE NO.**

TABLES

FIGURES

APPENDICES

1. INTRODUCTION

This Quality Assurance Project Plan (QAPP) has been prepared in conjunction with approved work plans for the Safety-Kleen Service Center N. Amityville, NY site (**Figure 1**), including the Interim Corrective Measures Work Plan, dated November 1, 2004 and the Supplemental Corrective Measures Work Plan dated February 9, 2014, hereafter collectively referred to as the Work Plans. This QAPP has been prepared in accordance with the general guidelines and requirements of the document "EPA Requirements for Quality Assurance Project Plans for Environmental Data Operations" (EPA QA/R-5) dated March 2001. The QAPP focuses on specific Quality Assurance and Quality Control (QA/QC) activities, policies, applicable organization, objectives, and functional activities that are designed to achieve data quality goals for this project. The QAPP contains detailed procedures and references to approved documents to be utilized during field investigation, monitoring, and analytical laboratory activities relating to the site. The following sections briefly describe the site and the purpose and objectives of this QAPP.

1.1 Project Description

This site-specific QAPP describes the QA/QC procedures employed to ensure the integrity, validity and usability of the analytical results. The analytical procedures utilized to support this project will include sample analysis using United States Environmental Protection Agency (EPA) "Test Methods for Evaluating Solid Waste, Physical/Chemical Methods (SW-846), Third Edition, Update III, December 1996, as well as the American Public Health Association (APHA) publication "Standard Methods for the Examination of Water and Wastewater, 18th Edition, 1992" and its subsequent editions. The primary scope includes analysis of select soil and water samples for the chemical parameters listed in **Section 1.6** for performance and progress monitoring.

1.2 Site Description

The site formerly operated as hazardous waste storage facility in accordance with the requirements of a New York State Department of Environmental Conservation (NYSDEC) Hazardous Waste Management Program RCRA Part B permit (EPA ID#: NYD 000708198). In 2008, the facility was RCRA-closed as a hazardous waste storage facility in accordance with the closure requirements of 6NYCRR Section 373-2.7, and transitioned to a less than 10-day transfer station and used oil storage. A facility site plan is presented as **Figure 2**.

1.3 Project Objectives

The Work Plans define an approach to implement additional remedial measures to degrade subsurface residual mineral spirit range organics (MSRO) and volatile organic compounds (VOCs). Data to be collected for the Work Plans will be retained in accordance with the field techniques for sample collection and handling as outlined in this QAPP. These procedures will be followed for all scopes implemented at the site, until such time as site work scope modifications require that the QAPP be amended.

A National Environmental Laboratory Accreditation Program (NELAP) and New York State Department of Health (NYSDOH) ELAP certified laboratory (for the parameters of interest) will perform the analytical work. The laboratory will be able to provide the equivalent of an NYSDEC ASP Category B data package. At this time, samples are sent for laboratory analysis to TestAmerica, Inc. (TestAmerica). TestAmerica's Edison, NJ laboratory performs MSRO as well as VOC analyses. Monitored Natural Attenuation (MNA) parameter analyses are conducted by TestAmerica's laboratories in Edison, NJ, Buffalo, NY, and Nashville, TN. TestAmerica holds both NY NELAP and NYSDOH ELAP certifications.

The technical standards to be used to evaluate groundwater results are based on NYSDEC Technical & Operational Guidance Series (TOGS) Section 1.1.1 class GA standards. Soil sample results will be compared to the values in Table 375-6.8(a) in 6 NYCRR Part 375. Previously reported laboratory reporting limits will be used where no standards exist.

1.4 Data Gathering Objectives

Definitions of specific work activities and their objectives are derived from the data quality objectives (DQOs), which are reflected in the overall project approach.

1.4.1 Investigation Synopsis

The investigation and monitoring programs designed for the site will document remedial efforts and natural attenuation of constituents, as necessary, in groundwater and soils.

1.4.2 Physical Characterization Activities

The base tasks required to collect the respective work scope samples are detailed in this QAPP.

1.5 Project Schedule

Monitoring is conducted quarterly, normally in the months of March, June, September and December. Investigation activities are conducted as needed.

1.6 Chemical Parameters to be Measured

Chemical and MNA parameters to be measured are detailed in the Work Plans and include:

1. VOCs
2. MSRO - Total
3. Alkalinity/Bicarbonate
4. Sulfate/Nitrate/Nitrite
5. Ammonia
6. Methane
7. Total Organic Carbon (TOC)
8. Hydrogen Sulfide
9. Carbon Dioxide
10. Phosphate
11. Dissolved Iron/Manganese

The NYSDEC 2005 Analytical Services Protocol Target Compound List and the project specific reporting limits for groundwater and soil samples are provided in **Appendix A**.

2. PROJECT ORGANIZATION

This section describes the project team that has been assembled in order to complete investigative and monitoring programs. In addition, the responsibilities of key project personnel have also been outlined. Please note that multiple project team positions may be assigned to a single project team member. The following is a listing of selected/key project positions:

- NYSDEC Project Manager;
- Client Project Manager;
- Consultant Project Manager;
- Site Manager;
- Project QA Officer;
- Health and Safety Officer;
- Site Supervisor;
- Field Personnel Team; and
- Analytical Laboratory Subcontractor.

The specific project team positions and responsibilities are detailed below. The following is a brief description of selected/key project personnel and their roles within the investigation and monitoring programs.

2.1 NYSDEC Project Manager

The NYSDEC Project Manager (NYSDEC PM) will interface directly with the Client Project Manager and will be the central point of contact for intra-agency, and other regulatory bodies who have an interest in this project. The NYSDEC PM will also monitor the progress of program(s), and ensure that it is being implemented to the Department's satisfaction. All report comments, NYSDEC correspondence, and requests from any regulatory agency will be provided through the NYSDEC point-of-contact. The NYSDEC PM for the site is Kent Johnson.

2.2 Client Project Manager

The Client Project Manager (Client PM) will interface directly with the consultant to monitor that the overall program is being implemented in accordance with all regulatory requirements. The Client PM is also responsible for the timely submittal of reports and other project documentation to the NYSDEC. The Client PM for the site is Mr. Stephen D. Fleming, PE, Safety-Kleen Systems, Inc.

2.3 Consultant Project Manager

The Consultant Project Manager (Consultant PM) is responsible for the overall technical and logistical aspects of the program implementation. The Consultant PM will provide overall guidance to the project staff to ensure that all tasks are completed in accordance with the objectives, procedures, and project schedule established in the Work Plans. In addition, the Consultant PM will review and assess the performance of the project staff and subcontractors, serve as the primary point of contact for the Client PM, maintain the project files, and disseminate project data and results for analysis. All consultant communications with the Client PM will be cleared through the Consultant PM, or at the Consultant PM's direction. The Consultant PM is Anne E. Proctor, PE, Woodard & Curran, Inc.

2.4 Site Manager

The Site Manager (SM) is responsible for the coordination and day-to-day operations of the personnel assigned to the field team. The SM will provide technical work scope implementation consultation to both the field team and the Consultant PM. The SM will be designated by the sampling contractor, Clean Harbors Environmental Systems.

2.5 Project Quality Assurance Officer

The QA Officer provides in-house consultant review of field methods/procedures and laboratory data, but is not involved with the day-to-day execution of the work scopes. The QA Officer's responsibility will be to review field and lab data for compliance with QAPP requirements. The QA Officer will also interact with the laboratory chemist regarding issues specifically germane to laboratory data generation. The Consultant PM or a designee will act as the QA Officer.

2.6 Health and Safety Officer

The Health and Safety Officer (HSO) is responsible for providing hazard communication information, oversight of training employees (as directed by the SM) in safe operating procedures, and advising the SM on any matters which involve the health and safety of personnel completing the field programs. The HSO will be consulted prior to any changes made in the project-specific Health and Safety Plan (HSP) and will ensure that the HSP complies with local, state and federal safety requirements. The SM or a designee will act as the HSO.

2.7 Site Supervisor

The Site Supervisor (SS) will coordinate the on-site field investigative and monitoring activities and serve as the on-site HSO. The SS will supervise on-site field/subcontractor personnel and will monitor that the QAPP is being implemented. The SS will also be part of the Field Personnel Team. The SM or a designee will act as the SS.

2.8 Field Personnel Team

The Field Personnel Team (FPT) will be responsible for the implementation for the field programs, as directed by the SM. The FPT may include, but is not restricted to, one or more field and staff level professionals, environmental technicians and contractors. Personnel whose experience is germane to the field activity assigned will complete sample collection activities.

2.9 Analytical Laboratory Subcontractor

The analytical laboratory will be responsible for the chemical characterization of all samples collected for fixed base analysis (versus field analyzed samples). The designated laboratory will be a contractor able to complete the prescribed analyses in accordance with EPA SW-846, third edition requirements as well as NYSDOH ELAP certifications. Further, the laboratory will maintain an internal QA/QC and data validation program in accordance with all applicable certifications. The laboratory selected for the program is TestAmerica Laboratories, Inc. (TestAmerica). The standard operating procedures (SOPs) for each chemical parameter listed in **Section 1.6** are included in **Appendix B**.

3. QUALITY ASSURANCE OBJECTIVES AND CRITERIA

It is expected that by the design of separate data quality requirements for field sampling and laboratory analysis, clear distinctions between any problems found in the system can be isolated with respect to the cause. The data quality requirements are also designed to provide an indication of the variability inherent to the overall system. The QC samples, field sampling protocols, and project-specific work plan requirements are utilized to identify and to address the variability in the system, as necessary.

The investigative and monitoring programs designed for the site will address the presence or absence of chemicals, as necessary, in various media with respect to the site. Work activities planned as part of the remedial program are or will be included in the Work Plans.

The assessment and analytical strategies have been selected to achieve the various project goals of site characterization and remediation. The seven steps included in EPA's "Guidance on Systematic Planning Using the Data Quality Objective Process" (EPA QA/G-4) are noted below:

1. Statement of the Problem.
2. Identification of the Goals of the Study.
3. Identification of the Information Inputs.
4. Definition of the Boundaries of the Study.
5. Development of an Analytical Approach.
6. Specification of Performance or Acceptance Criteria.
7. Development of a Detailed Plan for Obtaining Data.

To achieve the overall objectives, specific data quality requirements will need to be met. The DQOs are outlined below and represent quantitative and qualitative statements specifying the quality of the environmental data required to support the decision making process for this project. The DQOs define the acceptable limits for individual activities or for overall sequences of processes when the individual activities cannot be isolated. The indicators of data quality can be determined through evaluation of a number of key parameters. Precision, accuracy, representativeness, completeness and comparability, more commonly known as PARCC parameters, are utilized to determine data quality. The general definitions of the data quality indicators used in the subsequent sections are defined below.

Precision - Precision is the degree of agreement between a set of measurements, irrespective of the true value. Precision or reproducibility is determined through the evaluation of duplicate or replicate samples. The relative percent difference (RPD) of the two sample results is calculated and evaluated in relation to acceptable limits for the analytical method and sample matrix. Precision will be calculated as defined in **Section 12.0** of the QAPP.

Accuracy - Accuracy is a measure of bias, or the difference between a measurement and an accepted or true value. This difference can be expressed as a percent of the true value and/or % Recovery, as defined in **Section 12.0**. A central goal of the accuracy objective is to identify, evaluate, and limit systematic and random errors in the measurement system.

Representativeness - Representativeness expresses the degree to which data accurately and precisely represents the characteristic of the environmental area from which it was obtained. Multiple random measurements or samplings improve the accuracy of a determination when random errors are involved. In this way, the effects of random error are minimized. Systematic errors affect the representativeness as well and are covered by the accuracy DQOs. The

precision of random samplings is the primary measure to be used to monitor the effects of the various factors, such as medium homogeneity, which affect representativeness.

Completeness - Completeness is the ratio of the amount of valid data obtained from a measurement system compared to the amount expected under ideal conditions. For the current project, this ratio is expressed as a percent, as indicated in **Section 12.0**. The amount of data expected from a measurement system is by design a sufficient amount to achieve the overall project objectives. A sufficient amount of data of known quality must be generated in order to properly assess the study area and to substantiate further decision making.

Comparability - Comparability expresses the confidence with which one data set can be compared with another data set from a different phase or from a different program. Comparability involves a review of a composite of the parameters generated above as well as review of design factors such as sampling and analytical protocols. Inter-laboratory evaluations can play a role in the evaluation of analytical comparability.

4. SAMPLING PROCEDURES

4.1 Media Sampling

This section specifies sampling equipment, sampling methods, and analytical procedures that will be used for the field activities at the site. Inspection and calibration procedures have been established to ensure that all equipment is in proper working order prior to going into the field. Decontamination procedures are also detailed. Prior to sampling, the following steps must be taken by the persons responsible for the collection of the various media samples.

1. Thoroughly review the Work Plans and the QAPP, sampling procedures and required analyses.

2. Assemble and inspect all field equipment necessary for sample collection, verify that the equipment is clean and in proper working order. Note and replace any items that are in short supply or that are showing indications of wear. Calibrate all equipment to manufacturer's specifications.

3. Procure all pre-cleaned sampling containers. Examine containers to ensure that their integrity is not compromised. Contact the laboratory if any problems are found.

4. Confirm sample delivery date and method of shipment with the laboratory.

5. Review the site-specific HSP.

4.1.1 Review of Sampling Procedures

The FPT involved in the sample collection process will be thoroughly familiar with the procedures outlined in the Work Plans and QAPP, and any questions will be addressed before going to the site. The checklists provided in **Appendix C** will help to ensure that all resources necessary for the collection of representative samples will be available.

4.1.2 Field Equipment Calibration and Corrective Action Procedures

Field equipment anticipated to be used during this project includes multi-meter water quality instruments, turbidity meters, and water level meters/interface probes. Field instruments and equipment will be calibrated, operated, serviced, and maintained in accordance with the manufacturer's specified recommendations as outlined in equipment manuals. It will be the responsibility of the equipment supplier/rental company to adhere to the maintenance schedule and to arrange for service as required. The FPT will only perform calibration and basic maintenance (e.g., cleaning) on field equipment. In the event that an instrument is not operating correctly and basic maintenance does not correct the problem, replacement equipment will be obtained either from the contractor's equipment supply or from an equipment rental vendor, depending on availability. Service to the equipment, instruments, tools, gauges, etc. shall be performed only by qualified personnel. **Table 4-1** below presents a summary of the field instrument calibration procedures.

Table 4-1 Field Equipment Calibration and Corrective Action Procedures					
Instrument	**Activity**	**Frequency**	**Calibration Standard and Concentration**	**Acceptance Criteria**	**Corrective Action**
Turbidity Meter	Groundwater Sampling	Daily	10 NTU and 0 NTU Solutions	Refer to Manufacturer's Specifications	Clean sample vials, check charge/battery, then recalibrate instrument. If still out of range call vendor for troubleshooting guidance or a replacement instrument.
Water Level Meter/ Interface Probe	Groundwater Sampling	NA	NA	NA	Perform battery check prior to each use.
Multi-Parameter Water Quality Meter	Groundwater Sampling	Calibrate beginning and end of day Calibration Checks as needed	pH - 4.00, 7.00, and 10.00 solutions Conductivity - 1,000 uS/cm (at STP) solution ORP - 100 mV (at STP) solution DO - 100% saturated environment	Refer to Manufacturer's Specifications	Perform charge/battery check, inspect probes for damage/ sediment buildup, recalibrate instrument. Inspect DO probe for torn membrane and/or bubbles in fluid. Correct and recalibrate instrument. If still out of range, contact vendor for trouble shooting guidance and/or replacement instrument.

Note: DO = Dissolved Oxygen, ORP = Oxygen/Reduction Potential, mV = Millivolts, NTU = Nephelometric Turbidity Units, STP = Standard Temperature and Pressure, uS/cm = Microsiemens/Centimeter

4.1.3 Sample Containers

Sample containers used will not distort, rupture, leak, adsorb, or react with constituents of the samples. The containers will have adequate wall thickness to withstand handling, sample collection, and transportation to the laboratory. Containers will be large enough to contain the optimum sample volume. All sample containers will be pre-cleaned. The cleanliness of a batch of containers will be verified and documentation will be provided by the supplier prior to use on request. The sample container size, type, and preservative necessary for sampling visits are found in the laboratory SOPs in **Appendix B**.

4.1.4 Laboratory Scheduling

The laboratory selected to analyze the soil, water and blank samples will be notified as to the types of analysis to be run, number of samples per analyses, sampling date, and expected arrival date at the laboratory. This information will be utilized by the laboratory for scheduling purposes to insure that all samples are analyzed within allowable holding times.

4.1.5 Health and Safety

A site-specific HSP will be available for this facility in accordance with the Occupational Safety and Health Administration (OSHA) Hazardous Waste Operations and Emergency Response (HAZWOPER) regulation 29 CFR 1910.120. The FPT involved with the field programs will be thoroughly familiar with the HSP. Field personnel will sign off on the document acknowledging that they have read and fully understand its contents. All members of the FPT working at this site will have a minimum of 40 hours of initial hazardous waste activity instruction and an annual 8 hours

of refresher training, as well as a minimum of three days of field experience under the direct supervision of an experienced person. On-site managers and supervisors directly responsible for the employees engaged in the sampling activities will also receive an additional 8 hours of supervisory training. These training requirements comply with the OSHA HAZWOPER regulation.

4.2 Field Sampling Protocols

This section details procedures which will be followed to collect representative media samples from the proposed locations. A summary of the proposed sampling for each media is also provided in the Work Plans.

4.2.1 Groundwater Gauging and Sampling

4.2.1.1 Groundwater Gauging

Prior to the start of groundwater monitoring activities, monitoring wells will be gauged using a water level indicator instrument to determine depth to water. The following work steps will be followed during the well gauging:

1. Remove the monitoring well cover from the well utilizing the proper lifting equipment and techniques, record pertinent observations.

2. Using a water level indicator instrument capable of measuring the depth to water to 0.01 foot. Use the top of the monitoring well casing or sampling point as the reference point and record in the field log.

3. Record all monitoring and observation data in the field log. Check results for consistency with previously measured values before proceeding.

4.2.1.2 Groundwater Sampling

1. Remove the monitoring well cover from the well utilizing the proper lifting equipment and techniques, record pertinent observations.

2. Using a dedicated, disposable polyethylene bailer, purge 3 to 5 well volumes of groundwater prior to sampling.

3. Collect groundwater sample using the dedicated, disposable polyethylene bailer and place sample into containers provided by the Analytical Laboratory Subcontractor as specified for each analysis.

4. Label the sample containers with a self-adhesive label and waterproof ink and seal the containers. Labels will include the following information:

    a. Sample identification number,

    b. Job name and identification number,

    c. Date and time of sample collection,

    d. Type of analysis requested, and

    e. Name of sampler.

5. Repeat steps 1-4 for all of the monitoring well locations and collect the appropriate blanks and duplicate samples as discussed in the QAPP and the Work Plans.

6. Fill out the chain-of-custody form and reference the preservation techniques in the remarks section.

7. Store the collected samples obtained during the sampling activities together under refrigeration and in an area known to be free of contamination.

8. Enter into the field log, at a minimum, the information requested in the QAPP.

9. Ship the iced sample set via overnight courier within 24 hours of collection, maintaining chain-of-custody as described in **Section 5.0** of the QAPP. Each cooler shall contain a temperature blank, which the laboratory will use to verify that the samples are chilled to 4 C.

4.2.2 Soil Sampling Procedures

Monitoring point DW-1 is known to be occasionally dry. Based on a previously approved catch basin cleanout and media sampling program, a soil sample is collected from the bottom of DW-1 if dry.

The work steps listed below will be followed during the soil sample collection process:

1. Using a clean, stainless steel auger, collect the soil sample.

2. Upon retrieval of the stainless steel auger, soil sample aliquots will be collected immediately using 5-gram En Core Samplers and containers provided by the Analytical Laboratory Subcontractor as specified for each analysis.

3. Samples selected for laboratory analysis will be shipped to the laboratory within 24 hours of collection via an overnight courier. The detailed procedure for sample handling and chain-of-custody procedures is described in **Section 5.0** of this QAPP.

4. Label the sample containers with a self-adhesive label and waterproof ink and seal the containers. Labels will include the following information:

    a. Sample identification number,

    b. Job name and identification number,

    c. Date and time of sample collection,

    d. Type of analysis requested, and

    e. Name of sampler.

5. Decontaminate all non-disposable sampling equipment as detailed in the QAPP.

6. Fill out the chain-of-custody form and reference the preservation techniques in the remarks section.

7. Store the collected samples obtained during the sampling activities together under refrigeration and in an area known to be free of contamination.

8. Enter into the field log, at a minimum, the information requested in the QAPP.

9. Ship the iced sample set via overnight courier within 24 hours of collection, maintaining chain-of-custody as described in **Section 5.0** of the QAPP. Each cooler shall contain a temperature blank, which the laboratory will use to verify that the samples are chilled to 4 C.

4.2.3 Waste Disposal Sampling

Wastes will be contained in drums for disposal or managed at the direction of the facility manager. Wastes, including soil and liquid, resulting from field investigation and monitoring activities may require sampling for characterization to facilitate proper disposal. Accumulated soils and liquid wastes will be sampled by collecting grab samples using hand sampling equipment. Any required waste disposal with be done in accordance with regulatory requirements.

4.2.4 Equipment Decontamination

Soil Sampling Equipment Decontamination: Before sampling activities begin, a decontamination area will be established. All decontaminated equipment will be kept in this area on clean plastic. The following decontamination procedures will be followed for cleaning the soil sampler (stainless steel auger):

1. Wash and scrub with low phosphate detergent (Liqui-Nox or equivalent);
2. Tap water rinse;
3. Thoroughly rinse with deionized water;
4. Air dry; and
5. Wrap in aluminum foil for transport.

Water Sampling Equipment Decontamination:

The following procedures shall be used for decontamination of sampling pumps, purge pumps, water level/interface probes, flow-through cells and bailers (if used):

1. Wash and scrub with low phosphate detergent;
2. Tap water rinse;
3. Thoroughly rinse with deionized water;
4. Air dry; and
5. Wrap in aluminum foil for transport.

NOTE: Field instrumentation will follow the decontamination procedure described above unless manufacturer's instructions explain otherwise.

Disposable sampling equipment (e.g. latex gloves, disposable bailers) will be collected in plastic bags. Bags will be placed in a designated storage area as directed by the facility manager.

4.2.5 Blank Samples

Blank samples will be collected as a means to assess field QC. Blanks will be clearly labeled and distinguishable to the laboratory to the extent possible. The following control samples, as discussed in **Section 9.0** of this QAPP, shall be collected: equipment (rinsate) blanks, travel (trip) blanks and temperature blanks. Deionized water will be used for blank preparation.

Equipment blanks will be collected by passing deionized water through and over cleaned sampling equipment. Equipment blanks will be collected at a frequency specified in **Section 9.0**. Equipment blanks will be collected in sample containers and preserved in accordance with routine procedures as specified in **Section 9.0**.

Trip blanks are comprised of 40 ml VOA vials filled with deionized water and will be supplied by the laboratory within one week of the scheduled sampling date. Trip blanks will be placed in sample coolers prior to sample collection and will remain unopened until they and the samples are returned to the laboratory. Trip blanks will be included in all coolers containing samples to be analyzed for VOC parameters at the frequency described in the QAPP.

Temperature blanks will be comprised of at least 100 ml plastic bottles filled with deionized water. Temperature blanks will be placed in sample coolers along with the samples and will remain unopened until they and the samples are returned to the laboratory. Each cooler shall contain a temperature blank which the laboratory will use to verify that the samples are chilled to 4 C.

All blank samples shall be given an arbitrary identification (ID) number which must be logged in the field log. Blank sampling frequency is indicated in **Section 9.0**.

4.2.6 Documentation

FPT members will log all measurements, observations, and field instrument calibrations in bound, waterproof field notebooks or on prescribed forms (**Appendix D**). Each individual making an entry into the field log will date and sign their entry. It is anticipated that the data reduction for investigation and monitoring will be minimal and will consist primarily of tabulating analytical results. Entries in the log will include the following:

- Date and time of entry;
- Weather;
- Purpose of sampling;
- Name and address of field contact;
- All members of the FPT on-site on that date;
- Description of sample (note if sample is replicate, blank, composite or grab);
- Sample date, time and source of sample containers;
- Sampling location (and map referenced);
- Screening concentrations and depth of sample;
- Number and size of samples taken;
- Description of sample point;
- Collector's sample ID number(s);
- Field observations;
- Any field measurements;

- Sampling point purging information (volume purged, temperature, pH, DO, ORP, turbidity, conductivity, depth to water);
- The method of sample shipment, name of courier with tracking number (if applicable), and laboratory receiving the samples;
- All samples shipped on the chain-of-custody;
- Calibration of all instruments (pH probes, conductivity meters, turbidity meters, etc.);
- A description of any circumstances or observations that might affect analytical results; and
- A description of any deviations from the normal or written sampling procedures.

Because sampling situations vary, notes will be as descriptive and inclusive as possible. Language will be objective, factual, and free of personal feelings or inappropriate terminology. If anyone other than the persons to whom the log was assigned makes an entry, they will date and sign that entry.

5. SAMPLE CUSTODY

A sample is the physical evidence collected from a site or the environment. An important part of the investigation and monitoring programs is the control and tracking of the evidence collected. The primary objectives of sample custody procedures are to create accurately written records that can be used to trace the possession and handling of all samples from the moment of their collection, through analysis, until their final disposition. Sample custody for samples collected will be maintained by the SM or personnel collecting the samples. The FPT are responsible for documenting each sample transfer and maintaining custody of all samples until they are shipped to the laboratory.

5.1 Field Custody Procedures

All necessary sample containers will be shipped by the laboratory to the SM or designee. Sample containers needed for a specific sampling task will then be relinquished by the SM to the sampling team after the SM (or designee) has checked the integrity of the containers and assured that the proper containers are available for the planned task.

Immediately after the sample collection, each sample container will be sealed. The samples will then be placed into an insulated cooler for shipment to the laboratory. Field chain-of-custody (**Figure 3**) will be completed at the time of the sample collection and will accompany the sample cooler, placed inside the cooler in a zip-lock bag. The samples will be properly relinquished on the field COC record by each sampling team. Each cooler will contain sufficient ice to insure proper temperature is maintained, and will be packed in a manner to prevent damage to sample containers. A temperature blank will be included in each cooler shipped to the laboratory. The SM or designee will seal each sample cooler relinquished to an overnight courier. The name of the overnight courier will be written on the COC.

5.1.1 Sample Identification

Each separate sample will be identified using a waterproof pen, as follows:

1. The sample ID number will be the number assigned to the particular sampling location.

   Example: Well/Boring I.D.: GT-1 Matrix: Groundwater

2. The particular site.

   Example: SK N Amityville

3. The analysis required will be indicated for each sample.

   Example: EPA Method 8260

4. Date taken will be the date the sample was collected, using the format: MM-DD-YYYY.

   Example: 03-01-2017

5. Time will be the time the sample was collected, using military time.

   Example: 1201

6. The sampler's name will be printed in the "Sampled by" section.

7. Other information relevant to the sample.

Prior to going to the field, this identification procedure will be further refined if necessary, to facilitate accurate and simple sample identification.

The sample label contains the authoritative information for the sample. Inconsistencies with other documents will be settled in favor of the vial or container label unless otherwise corrected in writing by the FPT member collecting samples.

All samples analyzed by the laboratory are considered to be of an evidentiary nature. The possession of samples must be traceable from the time samples are collected in the field until the analysis is completed and the data are entered as evidence. The tracing of the samples is accomplished by "chain-of custody" procedures as follows:

1. A chain-of-custody record (**Figure 3**) will be completed for each set of samples.
2. Samples will not leave custody of the FPT until relinquished to another responsible party.

Custody is defined as:

1. In the actual physical possession of the samples by the FPT.
2. In a FPT member's view after being in physical possession.
3. In a locked area after being in physical possession.

5.2 Laboratory Chain-of-Custody

Upon arrival at the laboratory, the sample custodian at the Analytical Laboratory Subcontractor must maintain possession of the chain-of-custody samples and all records documenting that possession. Upon receipt of samples, the sample custodian removes the COC from the sealed cooler and must sign the shipping report accompanying each sample and record the date and time. Samples as received are verified to match those listed on the COC. A copy of this record becomes part of the report file. The sample custodian must sign the COC "Received by Laboratory" space. The samples are then secured in refrigerated storage.

After each extraction or analysis of a sample fraction, the custody record must be signed by the analyst, indicating the date and time of completion, which samples were used, and to which location they were returned.

By signing the custody record, the individual affirmed that s/he was completely responsible for the sample fraction during the period of time it was not in secure storage.

5.3 Laboratory Sample Tracking and Management

The laboratory will maintain sample information records in a LIMS (Laboratory Information Management System) computer system. The sample receipt and data entry activity (called "login") is reflected in a daily report, which is immediately entered into a master log. The chronological file contains all samples.

Each laboratory manager gets a report of pertinent analyses not yet completed including the daily update from the login activity. The tracking continues until the LIMS registers the completion of the report.

6. CALIBRATION PROCEDURES AND FREQUENCY

6.1 Laboratory Calibration

The confirmation calibration frequency required by the laboratory methods is dependent on the outcome of daily calibration checks made with QC standards. Reference materials are a minimum of 97% purity, certified by a reliable source. Spiked reference samples (spiked into reagent water) are introduced into the analytical system to determine recovery and to further validate calibration at a frequency dependent on the matrix spike performance. All calibrations and frequency are detailed in the individual methods and will be followed for all analytical work completed.

6.2 Field Calibration

In addition to the laboratory analysis conducted during the course of investigation and monitoring activities, field measurements may be taken utilizing field instruments during sampling. Specific conductance, pH, DO, ORP, turbidity, and temperature will be measured in water samples.

Daily field calibration procedures will, at a minimum, include the following:

1. Each instrument/meter shall have a dedicated log to record all calibrations, maintenance/repair work and usage in a standard format including the information below.

2. Entries to the instrument log shall be made at least daily whenever the instrument is in use.

3. Daily calibration records shall include:

    - calibrator's name
    - standards used and source
    - date/time of calibration
    - corrective actions taken (if any)
    - instrument name/model
    - temperature/barometric pressure/humidity conditions (if known)

4. All standards used shall be checked periodically to determine stability and operating condition, and a record kept of these inspections.

5. All personnel performing instrument calibrations shall be trained in its operation and calibrating procedures.

Refer to **Table 4-1** for Instruments, activity, frequency of calibration, calibration standards, acceptance criteria, and corrective action.

7. ANALYTICAL PROCEDURES

7.1 Laboratory Analyses

The methods and technical information indicated in this QAPP are derived from the following referenced manuals:

1. United States Environmental Protection Agency. Test Methods for Evaluating Solid Waste: Physical/Chemical Methods. SW-846, Third Edition, Update III, December 1996 and all subsequent revisions.

2. TestAmerica Laboratories, Inc. (TestAmerica) Standard Operating Procedures (**Appendix B** of this QAPP) and Test America's QA Manuals.

3. American Public Health Association. Standard Methods for the Examination of Water and Wastewater. 18th edition and all subsequent revisions.

The laboratory is under obligation to perform analytical cleanups when matrix interferences are present in order to achieve the required detection limits. Re-extraction, re-sonification or re-steam distillation is warranted along with cleanup to provide the best prepared sample possible.

Analyte detection limits are provided in **Appendix A**. The QC limits are provided in **Appendix B**. Low level detection limits will be utilized whenever possible for all analyses. However, if samples are heavily impacted with matrix interferences, then the low level detection limits may not be achievable. The affected samples will be diluted and re-analyzed. Medium level detection limits will be utilized, if warranted.

The laboratory is expected to perform all analyses to provide the best possible representation of the sampling point. The laboratory will document any analytical problems encountered during the course of analysis and report the problems and corrective actions in the Case Narrative of the individual data packages.

The methods described above will be used by the designated laboratory to analyze all samples, where possible. All laboratory reporting will utilize standardized forms for reporting results.

7.2 Method Selection

The methods specified include:

1. VOCs
2. MSRO - Total
3. Alkalinity/Bicarbonate
4. Sulfate/Nitrate/Nitrite
5. Ammonia
6. Methane
7. TOC
8. Hydrogen Sulfide
9. Carbon Dioxide
10. Phosphate
11. Dissolved Iron/Manganese

All methods are selected to obtain the most accurate representation of the sampling point possible. Method detection limit selection is based on the real assessment and analytical characterization DQOs. A comprehensive deliverables package will be generated to ensure that sufficient QC data and documentation is available if needed for external data validation. The methods selected specify the frequency and acceptance criteria for all associated QC samples.

QC samples include one method blank, matrix spike, and matrix spike duplicate every twenty samples or one per analytical batch, whichever occurs most frequently. Surrogate spikes are compounds that are added to every sample, method, field or trip blank, matrix spike and matrix spike duplicate sample. Surrogate recoveries are monitored to indicate the necessity for re-analysis based upon very low or very high recovery.

8. DATA REDUCTION, VALIDATION, AND REPORTING

Internal data validation practices will be followed to insure that new data is recorded and reported accurately and that an audit trail is developed for data that will be reduced. Data validation practices occur in the field and laboratory, and a QA check will be done by the Consultant PM or designee.

8.1 Data Reduction

8.1.1 Field Data Collection and Reduction

The FPT will log all field measurements, observations, and field instrument calibrations in bound, field notebooks or on prescribed forms (**Appendix D**). Entries will be dated, legible, and contain accurate and inclusive documentation of an individual's project activities. Because the log will be used to write reports, it will contain only facts and observations. Language will be objective, factual, and free of personal feelings or other terminology that may prove inappropriate. Each individual making an entry into the field log will date and sign their entry.

8.1.2 Laboratory Data Collection and Reduction

The data reduction scheme used in the laboratory form each of the measurement parameters, including the formulas used for calculating concentration for water will be that stated in the SOP for the analytical method used. All analyses will utilize a log into which will be recorded the following items, at a minimum:

1. analyst;
2. date;
3. sample number (lab #); and
4. analysis set-up conditions, e.g. dilutions, auto-sampler position number, or other instrument specifics not covered by an SOP.

For instrumental analyses, this analysis log will be instrument-specific and referred to as an instrument log. For other types of analyses, this analysis log will also contain all raw data collected by the analyst. All analyses will involve electronic data handling, resulting in values bearing the conventional units used by the laboratory, and already corrected for dilutions. The analyst will need to round the answers appropriately and sometimes sum the columns of data.

For spectrophotometric analyses, the raw data output will be in proportional units, such as peak height. This output will be recorded with sample identification, date and dilutions. Subsequent data reduction will involve the use of a fully-documented calibration curve, as indicated in the specific method SOP. For extractable organics, sample volume, and amount of injection enter into the calculation. In this case the electronic data handling system will report in-solution concentration, and the analyst will apply the corresponding correction using the sample volume recorded on the extractions lab bench sheet and the injection volume recorded in the analysis log. The resulting run factor and how it is derived will be transcribed onto the in-process data form.

For all analyses, the data will not be blank-corrected and will be flagged with a 'B' qualifier if blanks do not meet acceptability criteria. Additionally, any result that is less than ten times the value of the blank for common laboratory contaminants (2-butanone, methylene chloride, and acetone only) will be considered questionable. All other compounds are considered questionable if the result is less than five (5) times the value of the blank.

Chemists and technicians will be responsible for the measurement/analysis of any specific parameter, and for any calculations associated with the determination of parameter concentrations. All calculations are listed in the reference method. The chemists and their supervisors will be responsible for reviewing all results, applying calculation checks on a minimum of 10 percent of the results on each report. These individuals will be responsible for determining whether or not the results are acceptable, though the ultimate authority to determine acceptability will be the Director of QA. The laboratory section manager will be responsible for the final review of all data and for proofing of reports prior to submittal of the final reports.

Final reports will be typed from the in-process report forms approved by the supervisor after the review of all supporting data. The data output will be stored in a secure manner, indexed by project number, for at least five years.

8.2 Field and Laboratory Validation

8.2.1 Field Validation

The QA Officer will proof ten percent of the entries in the field log for completeness and correctness. Additionally, the QA Officer will check field calculations to ensure correctness.

8.2.2 Laboratory Validation

A complete record of each sample's history will be available for documenting its progress from the time of sample collection to arrival at the laboratory, and through the laboratory from sample receipt to reporting. Data validation will include the use of dated entries, signed by analysts and supervisors, on worksheets and logs used for all samples; the use of sample tracking and numbering systems to logically follow the progress of samples through the laboratory; and the use of QC criteria to reject or accept specific data (see **Section 9.0** of this QAPP).

8.3 Identification and Treatment of Outliers

Outliers are unusually large or unusually small values in a population of observations. Outliers may be the result of a variety of circumstances, including any of the following:

1. errors in recording of data,
2. calculation errors,
3. analytical errors,
4. inaccurate reading of meters,
5. faulty or defective instruments,
6. actual values due to comparatively unique conditions,
7. sample identification incorrectly transcribed in the field or lab,
8. sampling artifacts, or
9. sample integrity problems.

8.3.1 Identification of Outliers

Procedures for the identification of outliers will be followed at both the analytical stage and at the ensuing data reduction stage.

Outliers in laboratory data can arise from errors in analysis or from site-specific conditions that are out of control of the laboratory. Errors in the laboratory are most often detected in the data review and validation process. It is necessary to eliminate outliers from QC data because of the skewing effect which can destroy the effectiveness of the QC data. Extreme values will not be automatically eliminated as outliers. Those data will be validated and evaluated as part of the data set.

Outliers will be identified at the data reduction stage by the Consultant PM and reviewed by the QA Officer. When any particular value is suspected to be an outlier, the following steps will be taken:

1. Other data from the same sample will be checked to see if they are also anomalous.
2. The Consultant PM will interview individuals involved in generating the anomalous value. This will include questioning the FPT and the analyst.
3. If samplers demonstrate standard competency in the sampling procedure used at the time the sample with the anomalous value was obtained, the sampling error will be dismissed as a possible cause of the outlier.
4. The analyst will be asked to examine his/her notes and calculations and, if possible, to rerun the sample, for the specific parameter in question.
5. The following procedure may be used to identify and handle extreme values and outliers identified in the data collected. After the samples have been analyzed and verified according to the QA/QC requirements, a statistical program will be employed to evaluate the significance of the data. This data will be compiled into two subsets: one representing total VOCs and one inorganic parameters. After the data have been segregated, a statistical test will be conducted to evaluate the appropriate statistical distribution (e.g., the Shapiro-Wilk's or the Lilliefors's goodness-of-fit test). The distribution will be used to decide on the appropriate method to assess extreme values or outliers.
6. Based on the results from the outlier study, a map may be generated locating the potential "hot spots" to evaluate the need for supplemental data.

8.3.2 Treatment of Outliers

Rejection of any suspect data or outliers will only be done by the Consultant PM or an outside validator. The data will be rejected as an unacceptable outlier if:

1. A problem with equipment or an incorrect procedure during the sampling stage is identified.
2. The rerun of the analyst generates a value that significantly differs from the value being examined.
3. Additional data does not support the result(s).

8.4 Data Reduction, Reporting, and Report Storage

8.4.1 Data Reduction

Analytical data will be generated from direct-reading instruments, reporting integrators or data management computer software. The automated outputs will include identifications of compounds, concentrations and retention times. Outputs will be in graphic form (chromatograms), spectra, recorder charts, and in printer tabular form. The outputs will be in a standard format specified for each analysis and monitored for consistency. For direct reading instruments, the analyst will be required to record all results into a log.

Auxiliary data produced for internal records, which will not normally be reported to customers as part of the analytical data, will include the following: laboratory worksheets, laboratory notebooks, sample tracking system forms, instrument logs, standard records, maintenance records, calibration records, and associated QC records. These sources will be available; however, for inspection during audits to determine the validity of data.

8.4.2 Data Reporting

In addition to analytical data, the laboratory will report matrix spike, matrix duplicate, surrogate recoveries and method blanks, as applicable. This data package will be presented as an appendix to any project report. The laboratory will provide, electronically and in PDF ® file format, data deliverables packages for all analyses submitted under this QAPP as required.

8.4.3 Report Staging

All final customer report folders will be filed in a secure area in the laboratory documentation office. QC sample reports are maintained in separate files. All data, chromatograms, calculations, and reports will be stored for a minimum of five years in the Analytical Laboratory Subcontractor's office.

8.5 Reference

Dixon, W.J., Processing Data for Outliers, Biometrics, Vol. 9, No. 1, March 1953, pp. 74-89.

9. INTERNAL QUALITY CONTROL CHECKS

The intent of the internal QC program is to detect potential problems at the source and, if necessary, trace the sample analytical pathways for introduction of contamination. The QC data generated in the field will be used to monitor sampling technique reproducibility and cleanliness. QC data generated by the laboratory will not only monitor reproducibility (precision) in laboratory methods and cleanliness, but accuracy in samples submitted for analysis. If required, a formal data validation process will be performed by an external data validation contractor to separately assess variability in sampling technique and laboratory performance.

9.1 Field Quality Control Checks

The field QC checks monitor the data quality as it is affected by field procedures and conditions to the degree feasible as discussed in **Section 4.0** of this QAPP. The degree of effort (number of check samples per total samples taken) is stated in this section.

9.1.1 Blanks

Trip Blank – Trip blanks consist of reagent water prepared by the laboratory and sealed in the proper sampling container. It is handled as other samples except that it is not opened. This sample focuses on external sources of contamination and sampling container quality and cleanliness. For each shipment to the laboratory, one trip blank will be submitted. A minimum of one trip black will be submitted for each batch of glassware received by the field crew from the laboratory. The trip blank is never opened in the field and is only analyzed for VOCs.

Equipment Rinse Blank – One rinsate blank will be collected for each type of equipment used. The rinsate blank is collected by pouring de-ionized water over decontaminated/pre-cleaned sampling equipment to determine the possibility of cross contamination.

Temperature Blank - Each cooler shall contain a temperature blank which the laboratory will use to verify that the samples are chilled to 4 C.

The acceptability limits for all blanks are to be below the quantification limits of less than one-tenth of the level in the lowest sample in the batch.

9.1.2 Duplicates

Blind field duplicates (as opposed to duplicate containers full of sample intended as backup) are sequential or collocated grab samples collected to monitor overall precision as detailed in **Section 3.0** of this QAPP. One duplicate will be collected and submitted per twenty (20) samples, or one (1) per matrix type, as described in **Section 3.0**.

9.2 Laboratory Quality Control

All analytical procedures and QA/QC protocols will be followed as per EPA methods and/or the Analytical Laboratory Subcontractor's QA Manuals (**Appendix E**). The QC criteria are summarized in each approved analytical method, and are also presented in **Appendix B**.

10. PERFORMANCE AND SYSTEM AUDITS

10.1 Performance Audits

Laboratory QC audits are to be carried out by the Laboratory QA staff annually for each Laboratory Department. Results of QC audits will be reviewed by the QA Officer and will be reported as part of the QA reports.

Laboratory QC audits are to be carried out by the Laboratory QA staff semi-annually for each method. Blind audit samples will be utilized. Results of blind audit sample analyses will be reviewed by the QA Officer and will be reported as part of the QA reports.

10.2 System Audits

The Laboratory QA Officer will conduct a systems audit of laboratory QC procedures shortly after these systems are operational.

10.3 On-Site Inspections

The laboratory should be regularly inspected by state agencies in order to document compliance with the various certification programs in which the laboratory participates. The laboratory will maintain the proper certifications for all sub-categories of solid and hazardous waste.

11. PREVENTATIVE MAINTENANCE

11.1 Laboratory Maintenance

To assure minimum storage times for samples, the laboratory will maintain equipment to the manufacturer's specifications and keep enough overcapacity to have instruments available should one fail.

The laboratory will maintain written logs defining specific routine and preventive procedures for all instruments. Also, all instruments will be maintained through service contracts with the manufacturers as required.

11.2 Field Maintenance

The consultant's field equipment is maintained through the use of a tracking system incorporating the tagging of each equipment item. When damaged equipment in need of repair is returned to the equipment warehouse, it is appropriately flagged for the required maintenance to be performed. This process assures only operable and properly maintained equipment enters the fled. Routine daily maintenance procedures conducted in the field will include:

1. Removal of surface dirt and debris from exposed surfaces of sampling equipment and measurement systems;
2. Storage of equipment away from the elements;
3. Daily inspections of sampling equipment and measurement systems for possible problems (e.g., cracked or clogged lines or tubing or weak batteries);
4. Check instruments calibration; and
5. Charging any battery packs for equipment when not in use.

Spare replacement parts to be available in the field to minimize downtime include:

1. appropriately sized batteries;
2. locks;
3. extra sample container and preservatives;
4. extra sample coolers, packing materials, and sample locations stakes;
5. additional supply of health and safety equipment (i.e., boots, gloves, etc.); and
6. additional equipment as necessary for the field tasks.

12. SPECIFIC ROUTINE PROCEDURES USED TO ASSESS DATA PRECISION, ACCURACY, AND COMPLETENESS

Performance of the following calculations will be documented and included in the QC section of the analytical report, where appropriate.

12.1 Precision

Relative Percent Difference (RPD) is a measure of the difference between two samples assumed to be identical through dividing (splitting) the original sample. Each portion is analyzed as a unique sample, identifying the values of the first replicate (X1) and that of the second replicate (X2), and dividing the difference by the mean (X) of (X1) and (X2).

$$RPD = \frac{|x_1 - x_2|}{(x_1 + x_2)/2} \times 100$$

The RPDs will be compared to QC criteria included in USEPA CLP Functional Guidelines for Data Validation. If RPDs are generated outside of the acceptable limits, then the data will be qualified in accordance with the appropriate Region III Data Validation Criteria. Standard Deviation (SD) is the most widely used measure to describe the dispersion of a set of data. The estimate of precision of a series of replicate measurements will usually be expressed as the Relative Standard Deviation (RSD). SD and RSD are calculated as follows:

$$\%RSD = \frac{SD}{\overline{x}} \times 100 \qquad SD = \sqrt{\sum_{i=1}^{N} \frac{(x_i - \overline{x})^2}{(N-1)}}$$

Where:
N = total number of values
X_i $\overline{X}$ = each observed value
$\overline{X}$ = arithmetic mean of all observed value

The %RSD is primarily used to evaluate data sets such as those for the initial calibration of the instruments to be used for the analyses. The QC criteria for the %RSD are described in the analytical methods and Region III Data Validation Criteria. If the %RSD is generated outside the acceptance limits, then the associated data will be qualified in accordance with Region III Data Validation Criteria.

QC charts are maintained by the laboratory as a measure of precision and accuracy. The charts are prepared by determining the mean values generated and setting warning limits of 2 standard deviations from the mean and control limits of 3 standard deviations from the mean. The limits are updated on a regular basis for the analytical procedures performed. Corrective action such as re-analysis is initiated if data is generated outside of the acceptable limits. Acceptance criteria and appropriate corrective action are described in the analytical methods.

12.2 Accuracy

Percent recovery (%R) of a "known" amount of analyte (spike) added to a sample of known value is calculated as follows:

$$\%R = \frac{SSR - SR}{SA} \times 100$$
(Spike)

Where:
SSR = spiked sample results
SR = sample results
SA = amount of spike added

Percent recovery of a standard of "known" value is calculated as follows:

$$\%R = \frac{\text{Observed value}}{\text{True value (Standard)}} \times 100$$

Reference materials and stock solutions used for accuracy spikes will be traceable, independent and acceptable EPA solutions. Spike recovery will be evaluated in relation to the criteria presented in the methods and Region III criteria. If the recoveries are generated outside of the acceptable limits, then the data will be qualified in accordance with Region III Data Validation Criteria.

12.3 Completeness

Completeness is a measure of the amount of valid data obtained from a measurement system compared to the total amount expected to be obtained. It is calculated as follows:

$$\text{Completeness (\%)} = \frac{\text{Number of valid analysis}}{\text{number of analysis required}} \times 100$$

13. CORRECTIVE ACTION

13.1 Laboratory Corrective Action

The QC samples are designed to imitate samples and indicate possible sources of error or laboratory contamination. Any corrective actions taken by the laboratory will all be documented and initialed. The respective laboratory manager will provide documentation as to what, if any, corrective actions (i.e., re-analysis) were initiated during this study and report them to the QA Officer and/or Consultant PM.

The laboratory must take corrective action if any of the blank QC data generated during the laboratory analysis is outside criteria. By comparison of blank results, contamination may be attributed to either laboratory or field sampling techniques.

Corrective action for out-of-control calibrations is to re-calibrate the instrument and reanalyze the samples. A sequence is specified in the procedure to be used to analyze the sample when problems in analyses are encountered. The laboratory will be expected to follow this procedure exactly and document the problems encountered and corrective action taken in a case narrative enclosed with each deliverables package. Corrective action is described in the appropriate methods.

If any laboratory data fail to meet the precision and accuracy criteria objectives described in the analytical methods, the laboratory group manager will be notified and will be required to make any necessary systematic changes and then re-analyze the samples in question. If the samples in questions cannot be re-analyzed for some reason, the Client and Consultant PMs will decide whether or not the suspect data will be used, or if re-sampling is necessary.

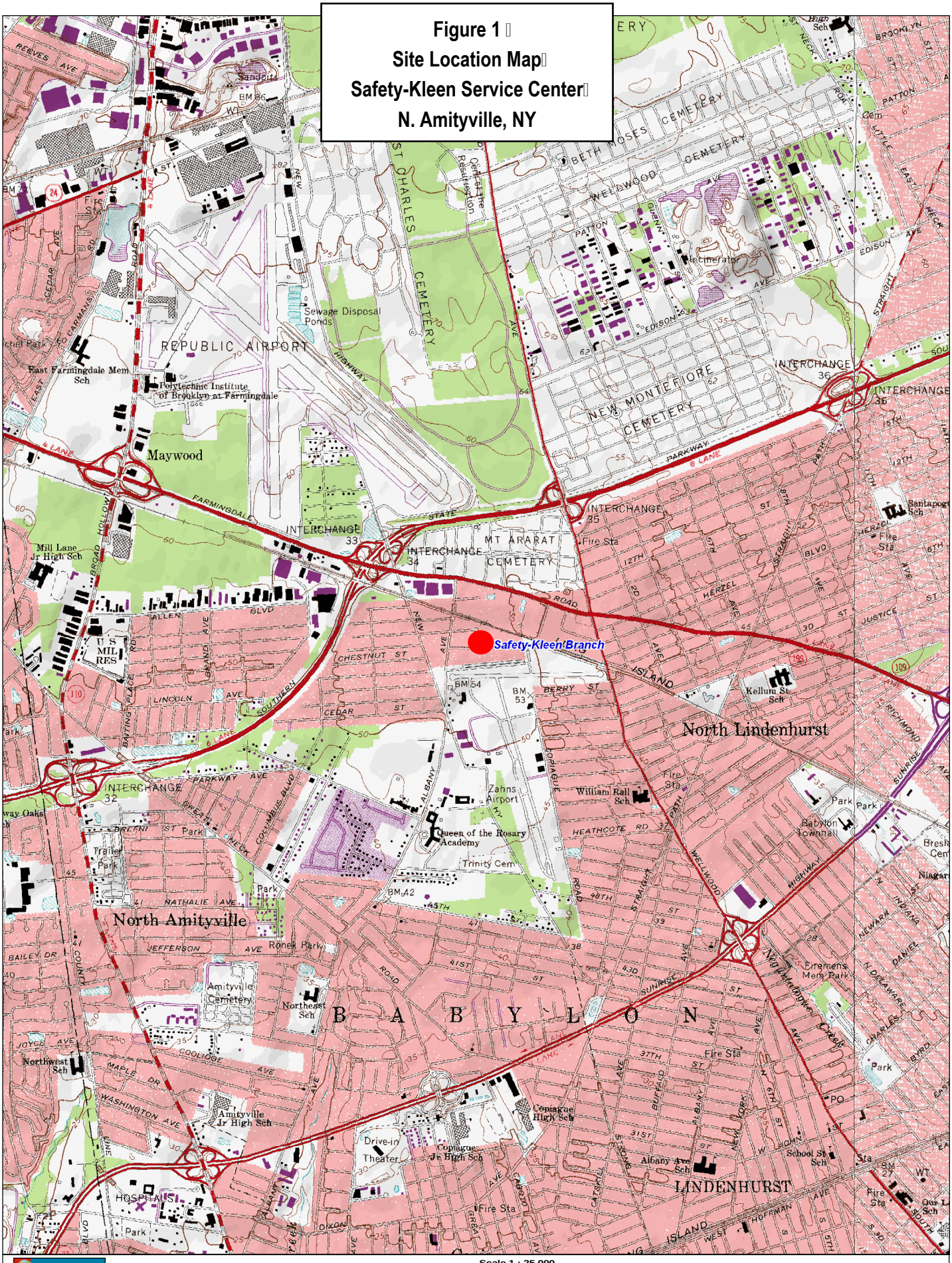
13.2 Client/Consultant Corrective Action

Field QA activities will be reported to the Consultant PM. Problems encountered during the study affecting QA will be reported to the QA Officer, who will notify the Client and Consultant PMs to decide whether or not the suspect data will be used, or if re-sampling is necessary.

The Consultant PM will be responsible for initiating the corrective actions and for insuring that the actions are taken in a timely manner, and also that the desired results are produced. The Consultant PM will report to the Client PM and QA Officer all the necessary corrective actions taken, the outcome of these actions, and their effect on data produced. All corrective actions taken will be reported to the appropriate regulatory agencies in the final report.

Figures

Figure 1
Site Location Map
Safety-Kleen Service Center
N. Amityville, NY



Scale 1 : 25,000
1" = 2080 ft

TN
MN
13.8°W

LONG ISLAND RAILROAD (CENTRAL BRANCH)

72 SEABRO AVENUE

CONTAINER STORAGE AREA # 4

70 SEABRO AVENUE

OFFICES

FLAM SHED

C.S. AREA # 05

DOCK #1

RAMP DOWN

38 ROOF DRAINS

FUTURE C.S. AREA # 06

60 SEABRO AVENUE

OFFICES

SMH

STC

GT-3

GT-2

GT-4

GT-5

GT-6

GT-7

GT-1

CB-5

EXISTING DRYWELLS

CB-4

BERMED STORM DRAIN

PARKING

EXISTING STORM DRAIN

EXISTING 6'-0" CHAIN LINK FENCE ON CURB

EXISTING DRYWELL

GATE

24

22

19

20

RAMP DN

DOCK #2

DOCK #3

DEMIZING WALL

FUTURE C.S. AREA #06

RECYCLABLES STORAGE

OFFICES

OTHER TENANT

84 SEABRO AVENUE

86 SEABRO AVENUE

88 SEABRO AVENUE

82 SEABRO AVENUE

OTHER TENANT

EXISTING CONC. SIDEWALK

EDGE OF PAVEMENT

EXIST. 6" CONCRETE CURB

CB-3

PIER

3.5' DIKE WALL

CMS 6K

C.S. AREA #01

C.S. AREA #02

C.S. AREA #03

R/F

43'-3" C TO C OF COLUMNS

UMS 6K

OFFICES

RAMP

CB-2C

CB-2A

CB-2

CB-2B

CB-1A

CB-2D

DW-1

VE-5

VE-1

BERMED STORM DRAIN DW-1

GATE

EXISTING CONC. SIDEWALK

EDGE OF PAVEMENT

SEABRO AVENUE

Rev-2 (VPA/B Loc)-061810
Rev-1 (VE1/5 Loc)-012109

CSA-03A

VP-B

CSA-03

CSA-03B

VP-A

R/F

OVERHEAD DOORS

C.S. AREA #03

CSA-03C

RAISED STEEL DECK FLOOR COVERS AREA

CSA-03D

OFFICES

C.S. AREA #03 DETAIL AREA

0 10 20 30 40

Scale in Feet

SOURCE:

MAP BASED ON SAFETY KLEEN BASE MAP ENTITLED "SITE PLAN EXISTING" DATED 8/26/00; DRAWING NO. 7039-SPOO-001, REV. 1 BY RM - SCALE: 1"=20'

0 20 40 60 80

Scale in Feet

LEGEND

- GT-3 — MONITORING WELL LOCATION
- DRYWELL (ADDRESSED IN CLOSURE)
- 23 — DRYWELL (EXISTING)
- CB-2B — OVER-FLOW POOL
- NEW PROPOSED BORING
- ALREADY COMPLETED BORING
- 6" LINE CONNECTING OFP TO DRYWELL
- PROPERTY LINE
- VE/VP-x — Vapor Extraction Well

Basile Environmental Solutions, LLC
1188 Hillside Dr.
Cortland, NY 13045

5/23/12

SCALE: AS SHOWN

CAD FILE: 7039-1A

DRAWN BY: JB

CHECKED BY: J.B.

TITLE: SITE PLAN

FIGURE No: 2

CLIENT: SAFETY-KLEEN SYSTEMS INC.
60 SEABRO AVENUE
NORTH AMITYVILLE, NY

TestAmerica Edison
777 New Durham Road
Edison, NJ 08817
Phone (732)549-3900 Fax (732)549-8913

Figure 3 - Chain of Custody Record (1 of 2)

TestAmerica
THE LEADER IN ENVIRONMENTAL TESTING

Client Information	Sampler:	Lab PM: Haas, Melissa (203-944-1310)	Carrier Tracking No(s):	COC No: 460-35545-23035.1
Client Contact: Stephen Fleming	Phone:	E-Mail: melissa.haas@testamericainc.com		Page: Page 1 of 2
Company: Safety Kleen				Job #:

Address: 4120 Thunderbird Lane	Due Date Requested:	
City: Fairfield	TAT Requested (days): **10 business days**	
State, Zip: OH, 45014		
Phone: 513-956-2172	PO #:	
Email: stephen.fleming@safety-kleen.com	WO #:	
Project Name: 2016 Safety-Kleen Amityville	Project #: 46008953 (VOC/Mineral Spirits/Natural Atten)	
Site:	SSOW#:	

Analysis Requested

Preservation Codes:

A - HCL	M - Hexane
B - NaOH	N - None
C - Zn Acetate	O - AsNaO2
D - Nitric Acid	P - Na2O4S
E - NaHSO4	Q - Na2SO3
F - MeOH	R - Na2S2SO3
G - Amchlor	S - H2SO4
H - Ascorbic Acid	T - TSP Dodecahydrate
I - Ice	U - Acetone
J - DI Water	V - MCAA
K - EDTA	W - ph 4-5
L - EDA	Z - other (specify)

Other:

Sample Identification	Sample Date	Sample Time	Sample Type (C=comp, G=grab)	Matrix (W=water, S=solid, O=waste/oil, BT=Tissue, A=Air)	Field Filtered Sample (Yes or No)	Perform MS/MSD (Yes or No)	8260C VOC	8015D Mineral Spirits	2320B-Alkalinity/Bicarbonate	300.0-Sulfate	SM4500_NH3-Ammonia	RSK_175-Methane	300.0 - Nitrate/Nitrite	SM5310B_TOC	SM4500_S2_D - Hydrogen Sulfide	SM4500_CO2_D-Carbon Dioxide	4500_P_E - Phosphate	200.7 - Dissolved Fe/Mn	8015D Mineral Spirits - Dissolved	Total Number of containers	Special Instructions/Note:
			Preservation Code:				A	N													
GT-1				Wat																	
GT-2				Water																	
GT-3				Water																	
GT-5				Water																	
GT-6				Water																	
GT-7				Water																	
VE-1R				Water																	
VE-5				Water																	
VP-A				Water																	
VP-B				Water																	
GW-DUP				Water																	

Possible Hazard Identification
☐ *Non-Hazard* ☐ *Flammable* ☐ *Skin Irritant* ☐ *Poison B* ☐ *Unknown* ☐ *Radiological*

Sample Disposal (A fee may be assessed if samples are retained longer than 1 month)
☐ *Return To Client* ☐ *Disposal By Lab* ☐ *Archive For _____ Months*

Deliverable Requested: I, II, III, IV, Other (specify)

Special Instructions/QC Requirements:

Empty Kit Relinquished by:	Date:	Time:	Method of Shipment:

Relinquished by:	Date/Time:	Company	Received by:	Date/Time:	Company
Relinquished by:	Date/Time:	Company	Received by:	Date/Time:	Company
Relinquished by:	Date/Time:	Company	Received by:	Date/Time:	Company

Custody Seals Intact: Δ Yes Δ No	Custody Seal No.:	Cooler Temperature(s) °C and Other Remarks:

TestAmerica Edison
777 New Durham Road
Edison, NJ 08817
Phone (732)549-3900 Fax (732)549-8913

Figure 3 - Chain of Custody Record (2 of 2)

TestAmerica
THE LEADER IN ENVIRONMENTAL TESTING

Client Information	Sampler:	Lab PM: Haas, Melissa (203-944-1310)	Carrier Tracking No(s):	COC No: 460-35545-23035.2
Client Contact: Stephen Fleming	Phone:	E-Mail: melissa.haas@testamericainc.com		Page: Page 2 of 2
Company: Safety Kleen				Job #:

Address: 4120 Thunderbird Lane	Due Date Requested:
City: Fairfield	TAT Requested (days): 10 business days
State, Zip: OH, 45014	
Phone: 513-956-2172	PO #:
Email: stephen.fleming@safety-kleen.com	WO #:
Project Name: 2016 Safety-Kleen Amityville	Project #: 46008953
Site:	SSOW#:

Analysis Requested

Preservation Codes:

A - HCL	M - Hexane
B - NaOH	N - None
C - Zn Acetate	O - AsNaO2
D - Nitric Acid	P - Na2O4S
E - NaHSO4	Q - Na2SO3
F - MeOH	R - Na2S2SO3
G - Amchlor	S - H2SO4
H - Ascorbic Acid	T - TSP Dodecahydrate
I - Ice	U - Acetone
J - DI Water	V - MCAA
K - EDTA	W - ph 4-5
L - EDA	Z - other (specify)

Other:

Sample Identification	Sample Date	Sample Time	Sample Type (C=comp, G=grab)	Matrix (W=water, S=solid, O=waste/oil, BT=Tissue, A=Air)	Field Filtered Sample (Yes or No)	Perform MS/MSD (Yes or No)	8260C VOC	8015D Mineral Spirits - Total	2320B-Alkalinity/Bicarbonate	300.0-Sulfate	SM4500_NH3-Ammonia	RSK_175-Methane	300.0 - Nitrate/Nitrite	SM5310B_TOC	SM4500_S2_D - Hydrogen Sulfide	SM4500_CO2_D-Carbon Dioxide	4500_P_E - Phosphate	200.7 - Dissolved Fe/Mn	8015D Mineral Spirits - Dissolved	Total Number of containers	Special Instructions/Note:
			Preservation Code:				A	N													
Rinse-GW				Water																	
Rinse-Soil				Water																	
TRIP BLANK				Water																	
DW-1				Soil/Water																	
DW-1 DUP				Soil/Water																	

Possible Hazard Identification
☐ *Non-Hazard* ☐ *Flammable* ☐ *Skin Irritant* ☐ *Poison B* ☐ *Unknown* ☐ *Radiological*

Sample Disposal (A fee may be assessed if samples are retained longer than 1 month)
☐ *Return To Client* ☐ *Disposal By Lab* ☐ *Archive For _____ Months*

Deliverable Requested: I, II, III, IV, Other (specify)

Special Instructions/QC Requirements:

Empty Kit Relinquished by:	Date:	Time:	Method of Shipment:

Relinquished by:	Date/Time:	Company	Received by:	Date/Time:	Company
Relinquished by:	Date/Time:	Company	Received by:	Date/Time:	Company
Relinquished by:	Date/Time:	Company	Received by:	Date/Time:	Company

Custody Seals Intact: Δ Yes Δ No	Custody Seal No.:	Cooler Temperature(s) °C and Other Remarks:

APPENDIX A: TARGET COMPOUND LIST AND SITE-SPECIFIC REPORTING LIMITS

Project Specific Reporting Limits – Aqueous Samples

For aqueous samples, please note that the reporting limits listed below may vary for each sample analyzed based on sample volume, and/or sample dilution. The aqueous laboratory reporting limits are based on the New York State Department of Environmental Conservation (NYSDEC) Technical & Operational Guidance Series (TOGS) section 1.1.1 class GA standards, and ASI's previously reported laboratory reporting limits where no TOGS class GA standard exists.

Analyte	Aqueous Project Specific Reporting Limits	Units
Acetone	50	ug/L
Acetonitrile	10	ug/L
Allyl chloride	5	ug/L
Benzene	1	ug/L
Benzyl chloride	10	ug/L
Bromodichloromethane	50	ug/L
Bromoform	5	ug/L
Bromomethane	5	ug/L
2-Butanone (MEK)	50	ug/L
Carbon disulfide	60	ug/L
Carbon tetrachloride	5	ug/L
Chlorobenzene	5	ug/L
Chloroethane	5	ug/L
2-Chloroethyl vinyl ether	20	ug/L
Chloroform	7	ug/L
Chloromethane	5	ug/L
cis-1,2-Dichloroethene	5	ug/L
cis-1,3-Dichloropropene	0.2	ug/L
Dibromochloromethane	50	ug/L
1,2-Dibromo-3-Chloropropane	0.04	ug/L
1,2-Dibromoethane	5	ug/L
Dibromomethane	5	ug/L
1,3-Dichlorobenzene	3	ug/L
1,4-Dichlorobenzene	3	ug/L
1,2-Dichlorobenzene	3	ug/L
Dichlorodifluoromethane	5	ug/L
1,1-Dichloroethane	5	ug/L
1,2-Dichloroethane	0.6	ug/L
1,1-Dichloroethene	5	ug/L
1,2-Dichloroethene, Total	2	ug/L
1,2-Dichloropropane	1	ug/L
Ethylbenzene	5	ug/L
Ethyl methacrylate	5	ug/L
2-Hexanone	50	ug/L
Iodomethane	5	ug/L
Isobutyl alcohol	250	ug/L
Methacrylonitrile	5	ug/L
Methylene Chloride	5	ug/L
Methyl methacrylate	50	ug/L
4-Methyl-2-pentanone (MIBK)	5	ug/L
m&p-Xylene	10	ug/L
o-Xylene	5	ug/L
Styrene	5	ug/L
1,1,1,2-Tetrachloroethane	5	ug/L
1,1,2,2-Tetrachloroethane	5	ug/L
Tetrachloroethene	5	ug/L
Toluene	5	ug/L
trans-1,4-Dichloro-2-butene	5	ug/L
trans-1,2-Dichloroethene	5	ug/L

Analyte	Aqueous Project Specific Reporting Limit	Units
trans-1,3-Dichloropropene	0.2	ug/L
1,1,1-Trichloroethane	5	ug/L
1,1,2-Trichloroethane	1	ug/L
Trichloroethene	5	ug/L
1,2,3-Trichloropropane	0.04	ug/L
Vinyl acetate	5	ug/L
Vinyl chloride	2	ug/L
Xylenes, Total	15	ug/L
Mineral Spirit Range Organics	50	ug/L

Project Specific Reporting Limits – Solid Samples

For solid samples, please note that the reporting limits listed below will vary for each sample analyzed based on sample moisture content, sample volume, and/or sample dilution. The solid laboratory reporting limits are based on the New York State Department of Environmental Conservation (NYSDEC) Subpart 375-6.8(a) Unrestricted Use Soil Cleanup Objectives and TestAmerica Edison's laboratory reporting limits where no part 375 cleanup objectives exist.

Analyte	Solid Project Specific Reporting Limits	Units
Acetone	50	ug/Kg
Acetonitrile	50	ug/Kg
Allyl chloride	5	ug/Kg
Benzene	60	ug/Kg
Benzyl chloride	5	ug/Kg
Bromodichloromethane	5	ug/Kg
Bromoform	5	ug/Kg
Bromomethane	5	ug/Kg
2-Butanone (MEK)	120	ug/Kg
Carbon disulfide	5	ug/Kg
Carbon tetrachloride	760	ug/Kg
Chlorobenzene	1100	ug/Kg
Chloroethane	5	ug/Kg
2-Chloroethyl vinyl ether	5	ug/Kg
Chloroform	370	ug/Kg
Chloromethane	5	ug/Kg
cis-1,2-Dichloroethene	250	ug/Kg
cis-1,3-Dichloropropene	5	ug/Kg
Dibromochloromethane	5	ug/Kg
1,2-Dibromo-3-Chloropropane	10	ug/Kg
1,2-Dibromoethane	5	ug/Kg
Dibromomethane	5	ug/Kg
1,3-Dichlorobenzene	2400	ug/Kg
1,4-Dichlorobenzene	1800	ug/Kg
1,2-Dichlorobenzene	1100	ug/Kg
Dichlorodifluoromethane	5	ug/Kg
1,1-Dichloroethane	270	ug/Kg
1,2-Dichloroethane	20	ug/Kg
1,1-Dichloroethene	330	ug/Kg
1,2-Dichloroethene, Total	5	ug/Kg
1,2-Dichloropropane	5	ug/Kg
Ethylbenzene	1000	ug/Kg
Ethyl methacrylate	10	ug/Kg
2-Hexanone	10	ug/Kg
Iodomethane	10	ug/Kg
Isobutyl alcohol	150	ug/Kg
Methacrylonitrile	10	ug/Kg
Methylene Chloride	50	ug/Kg
Methyl methacrylate	10	ug/Kg
4-Methyl-2-pentanone (MIBK)	5	ug/Kg
m&p-Xylene	5	ug/Kg
o-Xylene	5	ug/Kg
Styrene	5	ug/Kg
1,1,1,2-Tetrachloroethane	5	ug/Kg
1,1,2,2-Tetrachloroethane	5	ug/Kg
Tetrachloroethene	1300	ug/Kg
Toluene	700	ug/Kg
trans-1,4-Dichloro-2-butene	10	ug/Kg
trans-1,2-Dichloroethene	190	ug/Kg

Analyte	Solid Project Specific Reporting Limits	Units
trans-1,3-Dichloropropene	5	ug/Kg
1,1,1-Trichloroethane	680	ug/Kg
1,1,2-Trichloroethane	5	ug/Kg
Trichloroethene	470	ug/Kg
1,2,3-Trichloropropane	5	ug/Kg
Vinyl acetate	20	ug/Kg
Vinyl chloride	5	ug/Kg
Xylenes, Total	260	ug/Kg
Mineral Spirit Range Organics	6700	ug/Kg

Project Specific Reporting Limits – Solid Samples

For solid samples, please note that the reporting limits listed below will vary for each sample analyzed based on sample moisture content, sample volume, and/or sample dilution. The solid laboratory reporting limits are based on the New York State Department of Environmental Conservation (NYSDEC) Subpart 375-6.8(a) Unrestricted Use Soil Cleanup Objectives and TestAmerica Edison's laboratory reporting limits where no part 375 cleanup objectives exist.

Analyte	Solid Project Specific Reporting Limits	Units
Acetone	50	ug/Kg
Acetonitrile	50	ug/Kg
Allyl chloride	5	ug/Kg
Benzene	60	ug/Kg
Benzyl chloride	5	ug/Kg
Bromodichloromethane	5	ug/Kg
Bromoform	5	ug/Kg
Bromomethane	5	ug/Kg
2-Butanone (MEK)	120	ug/Kg
Carbon disulfide	5	ug/Kg
Carbon tetrachloride	760	ug/Kg
Chlorobenzene	1100	ug/Kg
Chloroethane	5	ug/Kg
2-Chloroethyl vinyl ether	5	ug/Kg
Chloroform	370	ug/Kg
Chloromethane	5	ug/Kg
cis-1,2-Dichloroethene	250	ug/Kg
cis-1,3-Dichloropropene	5	ug/Kg
Dibromochloromethane	5	ug/Kg
1,2-Dibromo-3-Chloropropane	10	ug/Kg
1,2-Dibromoethane	5	ug/Kg
Dibromomethane	5	ug/Kg
1,3-Dichlorobenzene	2400	ug/Kg
1,4-Dichlorobenzene	1800	ug/Kg
1,2-Dichlorobenzene	1100	ug/Kg
Dichlorodifluoromethane	5	ug/Kg
1,1-Dichloroethane	270	ug/Kg
1,2-Dichloroethane	20	ug/Kg
1,1-Dichloroethene	330	ug/Kg
1,2-Dichloroethene, Total	5	ug/Kg
1,2-Dichloropropane	5	ug/Kg
Ethylbenzene	1000	ug/Kg
Ethyl methacrylate	10	ug/Kg
2-Hexanone	10	ug/Kg
Iodomethane	10	ug/Kg
Isobutyl alcohol	150	ug/Kg
Methacrylonitrile	10	ug/Kg
Methylene Chloride	50	ug/Kg
Methyl methacrylate	10	ug/Kg
4-Methyl-2-pentanone (MIBK)	5	ug/Kg
m&p-Xylene	5	ug/Kg
o-Xylene	5	ug/Kg
Styrene	5	ug/Kg
1,1,1,2-Tetrachloroethane	5	ug/Kg
1,1,2,2-Tetrachloroethane	5	ug/Kg
Tetrachloroethene	1300	ug/Kg
Toluene	700	ug/Kg
trans-1,4-Dichloro-2-butene	10	ug/Kg
trans-1,2-Dichloroethene	190	ug/Kg

Analyte	Solid Project Specific Reporting Limits	Units
trans-1,3-Dichloropropene	5	ug/Kg
1,1,1-Trichloroethane	680	ug/Kg
1,1,2-Trichloroethane	5	ug/Kg
Trichloroethene	470	ug/Kg
1,2,3-Trichloropropane	5	ug/Kg
Vinyl acetate	20	ug/Kg
Vinyl chloride	5	ug/Kg
Xylenes, Total	260	ug/Kg
Mineral Spirit Range Organics	6700	ug/Kg

EXHIBIT C

TARGET COMPOUND LISTS (TCLs)

AND

CONTRACT REQUIRED QUANTITATION LIMITS (CRQLs)

EXHIBIT C

TABLE OF CONTENTS

INTRODUCTION

NOTE: *The values in these tables are minimum quantitation limits, not absolute detection limits. The amount of material necessary to produce a detector response that can be identified and reliably quantified is greater than that needed to simply be detected above the background noise. Most of the quantitation limits in these tables are set at the concentrations in the sample equivalent to the concentration of the lowest calibration standard analyzed for each analyte.*

Specific quantitation limits are highly matrix dependent. It is expected that the laboratory make every effort possible to meet the quantitation limits listed herein but it is realized that these limits may not be achievable in all instances.

CRQL values listed on the following pages are based on the analysis of samples according to the specifications given in Exhibit D. Modifications to the sample amounts processed may deviate from those listed in Exhibit D, as long as the limits listed herein can still be achieved.

All CRQL values are rounded to two significant figures.

The term "Solids" is used to denote the following matrices: soil, sediment, sludge, tissue, ash, oil, or mixed phase samples.

CRQL values listed for solids (soil, sediments, etc., except for tissue) are all based on 100% solids content. The quantitation limits calculated by the Laboratory for soil/sediment, calculated on dry weight basis, as required by the Protocol, will be higher. Results for tissue samples should be reported on a wet weight basis, along with their Percent lipid (% Lipid) content.

Changes to the Organic Target Compound Lists (TCLs) (e.g. adding an additional analyte) or lower CRQLs may be requested under the flexibility clause in the contract.

PART I – SUPERFUND-CLP ORGANICS

Volatiles Target Compound List (TCL) and Contract Required Quantitation Limits (CRQL) for Aqueous Samples

	Volatile Analyte	CAS Number	Trace Water By SIM (µg/L)	Trace Level Water (µg/L)	Low Level Water (µg/L)
1.	Dichlorodifluoromethane	75-71-8		0.50	5.0
2.	Chloromethane	74-87-3		0.50	5.0
3.	Vinyl Chloride	75-01-4		0.50	5.0
4.	Bromomethane	74-83-9		0.50	5.0
5.	Chloroethane	75-00-3		0.50	5.0
6.	Trichlorofluoromethane	75-69-4		0.50	5.0
7.	1,1-Dichloroethene	75-35-4		0.50	5.0
8.	1,1,2-Trichloro-1,2,2-trifluoroethane	76-13-1		0.50	5.0
9.	Acetone	67-64-1		5.0	10.0
10.	Carbon Disulfide	75-15-0		0.50	5.0
11.	Methyl Acetate	79-20-9		0.50	5.0
12.	Methylene chloride	75-09-2		0.50	5.0
13.	trans-1,2-Dichloroethene	156-60-5		0.50	5.0
14.	Methyl tert-Butyl Ether	1634-04-4		0.50	5.0
15.	1,1-Dichloroethane	75-34-3		0.50	5.0
16.	cis-1,2-Dichloroethene	156-59-2		0.50	5.0
17.	2-Butanone	78-93-3		5.0	10.0
18.	Bromochloromethane	74-97-5		0.50	5.0
19.	Chloroform	67-66-3		0.50	5.0
20.	1,1,1-Trichloroethane	71-55-6		0.50	5.0
21.	Cyclohexane	110-82-7		0.50	5.0
22.	Carbon tetrachloride	56-23-5		0.50	5.0
23.	Benzene	71-43-2		0.50	5.0
24.	1,2-Dichloroethane	107-06-2		0.50	5.0
25.	1,4-Dioxane	123-91-1	1.0	25	125
26.	Trichloroethane	79-01-6		0.50	5.0

Volatiles Target Compound List (TCL) and Contract Required Quantitation Limits (CRQL) for Aqueous Samples (Continued)

	Volatile Analyte	CAS Number	Trace Water By SIM (µg/L)	Trace Level Water (µg/L)	Low Level Water (µg/L)
27.	Methylcyclohexane	108-87-2		0.50	5.0
28.	1,2-Dichloropropane	78-87-5		0.50	5.0
29.	Bromodichloromethane	75-27-4		0.50	5.0
30.	cis-1,3-Dichloropropene	10061-01-5		0.50	5.0
31.	4-methyl-2-pentanone	108-10-1		5.0	10.0
32.	Toluene	108-88-3		0.50	5.0
33.	Trans-1,3-Dichloropropene	10061-02-6		0.50	5.0
34.	1,1,2-Trichloroethane	79-00-5		0.50	5.0
35.	Tetrachloroethene	127-18-4		0.50	5.0
36.	2-Hexanone	591-78-6		5.0	10.0
37.	Dibromochloromethane	124-48-1		0.50	5.0
38.	1,2-Dibromoethane	106-93-4	0.05	0.50	5.0
39.	Chlorobenzene	108-90-7		0.50	5.0
40.	Ethylbenzene	100-41-4		0.50	5.0
41.	Xylenes (Total)	1330-20-7		0.50	5.0
42.	Styrene	100-42-5		0.50	5.0
43.	Bromoform	75-25-2		0.50	5.0
44.	Isopropylbenzene	98-82-8		0.50	5.0
45.	1,1,2,2-Tetrachloroethane	79-34-5		0.50	5.0
46.	1,3-Dichlorobenzene	541-73-1		0.50	5.0
47.	1,4-Dichlorobenzene	106-46-7		0.50	5.0
48.	1,2-Dichlorobenzene	95-50-1		0.50	5.0
49.	1,2-Dibromo-3-chloropropane	96-12-8	0.05	0.50	5.0
50.	1,2,4-Trichlorobenzene	120-82-1		0.50	5.0
51.	1,2,3-Trichlorobenzene	87-61-6		0.50	5.0

Volatiles Target Compound List (TCL) and Contract Required Quantitation Limits (CRQL) for Solid Samples

	Volatile Analyte	CAS Number	Low Level Soil (µg/Kg)	Med. Level Soil (µg/Kg)
1.	Dichlorodifluoromethane	75-71-8	5.0	500
2.	Chloromethane	74-87-3	5.0	500
3.	Vinyl Chloride	75-01-4	5.0	500
4.	Bromomethane	74-83-9	5.0	500
5.	Chloroethane	75-00-3	5.0	500
6.	Trichlorofluoromethane	75-69-4	5.0	500
7.	1,1-Dichloroethene	75-35-4	5.0	500
8.	1,1,2-Trichloro-1,2,2-trifluoroethane	76-13-1	5.0	500
9.	Acetone	67-64-1	10.0	1000
10.	Carbon Disulfide	75-15-0	5.0	500
11.	Methyl Acetate	79-20-9	5.0	500
12.	Methylene chloride	75-09-2	5.0	500
13.	trans-1,2-Dichloroethene	156-60-5	5.0	500
14.	Methyl tert-Butyl Ether	1634-04-4	5.0	500
15.	1,1-Dichloroethane	75-34-3	5.0	500
16.	cis-1,2-Dichloroethene	156-59-2	5.0	500
17.	2-Butanone	78-93-3	10.0	1000
18.	Bromochloromethane	74-97-5	5.0	500
19.	Chloroform	67-66-3	5.0	500
20.	1,1,1-Trichloroethane	71-55-6	5.0	500
21.	Cyclohexane	110-82-7	5.0	500
22.	Carbon tetrachloride	56-23-5	5.0	500
23.	Benzene	71-43-2	5.0	500
24.	1,2-Dichloroethane	107-06-2	5.0	500
25.	1,4-Dioxane	123-91-1	125	12500
26.	Trichloroethane	79-01-6	5.0	500
27.	Methylcyclohexane	108-87-2	5.0	500
28.	1,2-Dichloropropane	78-87-5	5.0	500

Volatiles Target Compound List (TCL) and Contract Required Quantitation Limits (CRQL) for Solid Samples (Continued)

	Volatile Analyte	CAS Number	Low Level Soil (µg/Kg)	Med. Level Soil (µg/Kg)
29.	Bromodichloromethane	75-27-4	5.0	500
30.	cis-1,3-Dichloropropene	10061-01-5	5.0	500
31.	4-methyl-2-pentanone	108-10-1	10.0	1000
32.	Toluene	108-88-3	5.0	500
33.	Trans-1,3-Dichloropropene	10061-02-6	5.0	500
34.	1,1,2-Trichloroethane	79-00-5	5.0	500
35.	Tetrachloroethene	127-18-4	5.0	500
36.	2-Hexanone	591-78-6	10.0	1000
37.	Dibromochloromethane	124-48-1	5.0	500
38.	1,2-Dibromoethane	106-93-4	5.0	500
39.	Chlorobenzene	108-90-7	5.0	500
40.	Ethylbenzene	100-41-4	5.0	500
41.	Xylenes (Total)	1330-20-7	5.0	500
42.	Styrene	100-42-5	5.0	500
43	Bromoform	75-25-2	5.0	500
44.	Isopropylbenzene	98-82-8	5.0	500
45.	1,1,2,2-Tetrachloroethane	79-34-5	5.0	500
46.	1,3-Dichlorobenzene	541-73-1	5.0	500
47.	1,4-Dichlorobenzene	106-46-7	5.0	500
48.	1,2-Dichlorobenzene	95-50-1	5.0	500
49.	1,2-Dibromo-3-chloropropane	96-12-8	5.0	500
50.	1,2,4-Trichlorobenzene	120-82-1	5.0	500
51.	1,2,3-Trichlorobenzene	87-61-6	5.0	500

Semivolatiles Target Compound List (TCL) and Contract Required Quantitation Limits (CRQL) for Aqueous Samples

	Semivolatile Analyte	CAS Number	Low Water By SIM[1] (µg/L)	Water (µg/L)
1.	Benzaldehyde	100-52-7		5.0
2.	Phenol	108-95-2	0.10	5.0
3.	Bis-(2-chlorothyl) ether	111-44-4		5.0
4.	2-Chlorophenol	95-57-8	0.10	5.0
5.	2-Methylphenol	95-48-7	0.10	5.0
6.	2,2'-Oxybis (1-chloropropane)[3]	108-60-1		5.0
7.	Acetophenone	98-86-2		5.0
8.	4-Methylphenol	106-44-5	0.10	5.0
9.	N-Nitroso-di-n-propylamine	621-64-7		5.0
10.	Hexachloroethane	67-72-1		5.0
11.	Nitrobenzene	98-95-3		5.0
12.	Isophorone	78-59-1		5.0
13.	2-Nitrophenol	88-75-5	0.10	5.0
14.	2,4-Dimethylphenol	105-67-9	0.10	5.0
15.	Bis (2-chloroethoxy) methane	111-91-1		5.0
16.	2,4-Dichlorophenol	120-83-2	0.10	5.0
17.	Naphthalene	91-20-3	0.10	5.0
18.	4-Chloroaniline	106-47-8		5.0
19.	Hexachlorobutadiene	87-68-3		5.0
20.	Caprolactam	105-60-2		5.0
21.	4-Chloro-3-methylphenol	59-50-7	0.10	5.0
22.	2-Methylnaphthalene	91-57-6		5.0
23.	Hexachlorocyclopentadiene	77-47-4		5.0
24.	2,4,6-Trichlorophenol	88-06-2	0.10	5.0
25.	2,4,5-Trichlorophenol[4]	95-95-4	0.20	10.0
26.	1,1'-Biphenyl	92-52-4		5.0
27.	2-Chloronaphthalene	91-58-7		5.0

Semivolatiles Target Compound List (TCL) and Contract Required Quantitation Limits (CRQL) for Aqueous Samples (Continued)

	Semivolatile Analyte	CAS Number	Low Water By SIM[1] (µg/L)	Water (µg/L)
28.	2-Nitroaniline[4]	88-74-4		10.0
29.	Dimethylphthalate	131-11-3		5.0
30.	2,6-Dinitrotoluene	606-20-2		5.0
31.	Acenaphthylene	208-96-8	0.10	5.0
32.	3-Nitroaniline[4]	99-09-2		10.0
33.	Acenaphthene	83-32-9	0.10	5.0
34.	2,4-Dinitrophenol[4]	51-28-5	0.20	10.0
35.	4-Nitrophenol[4]	100-02-7	0.20	10.0
36.	Dibenzofuran	132-64-9		5.0
37.	2,4-Dinitrotoluene	121-14-2		5.0
38.	Diethylphthalate	84-66-2		5.0
39.	Fluorene	86-73-7	0.10	5.0
40.	4-Chlorophenyl-phenyl ether	7005-72-3		5.0
41.	4-Nitroaniline[4]	100-01-6		10.0
42.	4,6-Dinitro-2-methylphenol[4]	534-52-1	0.20	10.0
43.	N-Nitrosodiphenylamine	86-30-6		5.0
44.	1,2,4,5-Tetrachlorobenzene	95-34-3		5.0
45.	4-Bromophenyl-phenylether	101-55-3		5.0
46.	Hexachlorobenzene	100-52-7		5.0
47.	Atrazine	108-95-2	0.10	5.0
48.	Pentachlorophenol	111-44-4	0.20	10.0
49.	Phenanthrene	95-57-8	0.10	5.0
50.	Anthracene	95-48-7	0.10	5.0
51.	Carbazole	108-60-1		5.0
52.	Di-n-butylphthalate	98-86-2		5.0

Semivolatiles Target Compound List (TCL) and Contract Required Quantitation Limits (CRQL) for Aqueous Samples (Continued)

	Semivolatile Analyte	CAS Number	Low Water By SIM[1] (µg/L)	Water (µg/L)
53.	Fluoroanthene	106-44-5	0.10	5.0
54.	Pyrene	621-64-7		5.0
55.	Butylbenzylphthalate	67-72-1		5.0
56.	3,3'-Dichlorobenzidine	98-95-3		5.0
57.	Benzo (a) anthracene	78-59-1		5.0
58.	Chrysene	88-75-5	0.10	5.0
59.	Bis (2-ethylhexyl) phthalate	105-67-9	0.10	5.0
60.	Di-n-octylphthalate	111-91-1		5.0
61.	Benzo (b) fluoranthene	120-83-2	0.10	5.0
62.	Benzo (k) fluoranthene	91-20-3	0.10	5.0
63.	Benzo (a) pyrene	106-47-8		5.0
64.	Indeno (1,2,3-cd) pyrene	87-68-3		5.0
65.	Benzo (a,h) anthracene	105-60-2		5.0
66.	Benzo (g,h,i) perylene	59-50-7	0.10	5.0

Semivolatiles Target Compound List (TCL) and Contract Required Quantitation Limits (CRQL) for Solid Samples

	Semivolatile Analyte	CAS Number	Low Level By SIM[1] (µg/Kg)	Low Level Solids[2] (µg/Kg)	Med. Level Solids[2] (µg/Kg)
1.	Benzaldehyde	100-52-7		170	50000
2.	Phenol	108-95-2	3.3	170	50000
3.	Bis-(2-chlorothyl) ether	111-44-4		170	50000
4.	2-Chlorophenol	95-57-8	3.3	170	50000
5.	2-Methylphenol	95-48-7	3.3	170	50000
6.	2,2'-Oxybis (1-chloropropane)[3]	108-60-1		170	50000
7.	Acetophenone	98-86-2		170	50000
8.	4-Methylphenol	106-44-5	3.3	170	50000
9.	N-Nitroso-di-n-propylamine	621-64-7		170	50000
10.	Hexachloroethane	67-72-1		170	50000
11.	Nitrobenzene	98-95-3		170	50000
12.	Isophorone	78-59-1		170	50000
13.	2-Nitrophenol	88-75-5	3.3	170	50000
14.	2,4-Dimethylphenol	105-67-9	3.3	170	50000
15.	Bis (2-chloroethoxy) methane	111-91-1		170	50000
16.	2,4-Dichlorophenol	120-83-2	3.3	170	50000
17.	Naphthalene	91-20-3	3.3	170	50000
18.	4-Chloroaniline	106-47-8		170	50000
19.	Hexachlorobutadiene	87-68-3		170	50000
20.	Caprolactam	105-60-2		170	50000
21.	4-Chloro-3-methylphenol	59-50-7	3.3	170	50000
22.	2-Methylnaphthalene	91-57-6		170	50000
23.	Hexachlorocyclopentadiene	77-47-4		170	50000
24.	2,4,6-Trichlorophenol	88-06-2	3.3	170	50000

Semivolatiles Target Compound List (TCL) and Contract Required Quantitation Limits (CRQL) for Solid Samples (Continued)

	Semivolatile Analyte	CAS Number	Low Level By SIM[1] (µg/Kg)	Low Level Solids[2] (µg/Kg)	Med. Level Solids[2] (µg/Kg)
25.	2,4,5-Trichlorophenol[4]	95-95-4	6.7	330	100000
26.	1,1'-Biphenyl	92-52-4		170	50000
27.	2-Chloronaphthalene	91-58-7		170	50000
28.	2-Nitroaniline[4]	88-74-4		330	100000
29.	Dimethylphthalate	131-11-3		170	50000
30.	2,6-Dinitrotoluene	606-20-2		170	50000
31.	Acenaphthylene	208-96-8	3.3	170	50000
32.	3-Nitroaniline[4]	99-09-2		330	100000
33.	Acenaphthene	83-32-9	3.3	170	50000
34.	2,4-Dinitrophenol[4]	51-28-5	6.7	330	100000
35.	4-Nitrophenol[4]	100-02-7	6.7	330	100000
36.	Dibenzofuran	132-64-9		170	50000
37.	2,4-Dinitrotoluene	121-14-2		170	50000
38.	Diethylphthalate	84-66-2		170	50000
39.	Fluorene	86-73-7	3.3	170	50000
40.	4-Chlorophenyl-phenyl ether	7005-72-3		170	50000
41.	4-Nitroaniline[4]	100-01-6		330	100000
42.	4,6-Dinitro-2-methylphenol[4]	534-52-1	6.7	330	100000
43.	N-Nitrosodiphenylamine	86-30-6		170	50000
44.	1,2,4,5-Tetrachlorobenzene	95-34-3		170	50000
45.	4-Bromophenyl-phenylether	101-55-3		170	50000
46.	Hexachlorobenzene	118-74-1		170	10000
47.	Atrazine	1912-24-9		170	50000
48.	Pentachlorophenol	87-86-5	6.7	330	100000

Semivolatiles Target Compound List (TCL) and Contract Required Quantitation Limits (CRQL) for Solid Samples (Continued)

	Semivolatile Analyte	CAS Number	Low Level By SIM[1] (µg/Kg)	Low Level Solids[2] (µg/Kg)	Med. Level Solids[2] (µg/Kg)
49.	Phenanthrene	85-01-8	3.3	170	50000
50.	Anthracene	120-12-7	3.3	170	50000
51.	Carbazole	86-74-8		170	50000
52.	Di-n-butylphthalate	84-74-2		170	50000
53.	Fluoroanthene	206-44-0	3.3	170	50000
54.	Pyrene	129-00-0	3.3	170	50000
55.	Butylbenzylphthalate	85-68-7		170	50000
56.	3,3'-Dichlorobenzidine	91-94-1		170	50000
57.	Benzo (a) anthracene	56-55-3	3.3	170	50000
58.	Chrysene	218-01-9	3.3	170	50000
59.	Bis (2-ethylhexyl) phthalate	117-81-7		170	50000
60.	Di-n-octylphthalate	117-84-0		170	50000
61.	Benzo (b) fluoranthene	205-99-2	3.3	170	50000
62.	Benzo (k) fluoranthene	207-08-9	3.3	170	50000
63.	Benzo (a) pyrene	50-32-8	3.3	170	50000
64.	Indeno (1,2,3-cd) pyrene	193-39-5	3.3	170	50000
65.	Benzo (a,h) anthracene	53-70-3	3.3	170	50000
66.	Benzo (g,h,i) perylene	191-24-2	3.3	170	50000

Semivolatile Notes

[1] CRQLs for optional analysis of water and soil samples using SIM (Selected Ion Monitoring) techniques for PAHs and phenols.

[2] Denotes soil, sediment, tissue, or mixed phase samples.

[3] Previously known as bis (2-Chloroisoproply) ether.

[4] Seven semivolatile compounds are calibrated using only a four point initial calibration, eliminating the lowest standard. Therefore, the CRQL values for these eight compounds are 2 times higher for all matrices and levels.

Pesticide Target Compound List (TCL) and Contract Required Quantitation Limits (CRQL) For Aqueous and Solid Samples

	Pesticide Analyte	CAS Number	Water (µg/L)	Solids[1] (µg/Kg)
1.	alpha-BHC	319-84-6	0.050	1.7
2.	beta-BHC	319-85-7	0.050	1.7
3.	delta-BHC	319-86-8	0.050	1.7
4.	gamma-BHC (Lindane)	58-89-9	0.050	1.7
5.	Heptachlor	76-44-8	0.050	1.7
6.	Aldrin	309-00-2	0.050	1.7
7.	Heptachlor epoxide[2]	1024-57-3	0.050	1.7
8.	Endosulfan I	959-98-8	0.050	1.7
9.	Dieldrin	60-57-1	0.10	3.3
10.	4,4'-DDE	72-55-9	0.10	3.3
11.	Endrin	72-20-8	0.10	3.3
12.	Endosulfan II	33213-65-9	0.10	3.3
13.	4,4'-DDD	72-54-8	0.10	3.3
14.	Endosulfan sulfate	1031-07-8	0.10	3.3
15.	4,4'-DDT	50-29-3	0.10	3.3
16.	Methoxychlor	72-43-5	0.10	3.3
17.	Endrin ketone	53494-70-5	0.10	3.3
18.	Endrin aldehyde	7421-93-4	0.10	3.3
19.	alpha-Chlordane	5103-71-9	0.050	1.7
20.	gamma-Chlordane	5103-74-2	0.050	1.7
21.	Toxaphene	8001-35-2	5.0	34

Pesticide Notes

[1] There is no differentiation between the preparation of low and medium soil samples in this method for the analysis of pesticides.

[2] Only the exo-epoxy isomer (isomer B) of heptachlor epoxide is reported on the data reporting forms (Exhibit B).

PCB Aroclor Target Compound List (TCL) and Contract Required Quantitation Limits (CRQL) For Aqueous and Solid Samples

	Aroclor Analyte	CAS Number	Water (µg/L)	Solids[1] (µg/Kg)
1.	Arochlor-1016	12674-11-2	1.0	33
2.	Arochlor-1221	11104-28-2	1.0	33
3.	Arochlor-1232	11141-16-5	1.0	33
4.	Arochlor-1242	53469-21-9	1.0	33
5.	Arochlor-1248	12672-29-6	1.0	33
6.	Arochlor-1254	11097-69-1	1.0	33
7.	Arochlor-1260	11096-82-5	1.0	33
8.	Arochlor-1262	37324-23-5	1.0	33
9.	Arochlor-1268	11100-14-4	1.0	33

Aroclor PCB Notes

[1] There is no differentiation between the preparation of low and medium soil samples in this method for the analysis of Aroclor PCBs.

PCB Congeners Target Compound List (TCL) and Contract Required Quantitation Limits (CRQL) For Aqueous and Solid Samples

	Congener Analyte	IUPAC Number	CAS Number	Water (pg/L)	Solids[1] (ng/Kg)
1.	2-MoCB	1	2051-60-7	20	2.0
2.	4-MoCB	3	2051-62-9	20	2.0
3.	2,2'-DiCB	4	13029-08-8	20	2.0
4.	4,4'-DiCB	15	2050-68-2	20	2.0
5.	2,2',6'-TrCB	19	38444-73-4	20	2.0
6.	3,4,4'-TrCB	37	38444-90-5	20	2.0
7.	2,2',6,6'-TeCB	54	15968-05-5	20	2.0
8.	3,3',4,4'-TeCB	77	32598-13-3	20	2.0
9.	3,4,4',5-TeCB	81	70362-50-4	20	2.0
10.	2,2',4,6,6'-PeCB	104	56558-16-8	20	2.0
11.	2,3,3',4,4'-PeCB	105	32598-14-4	20	2.0
12.	2,3,4,4',5-PeCB	114	74472-37-0	20	2.0
13.	2,3',4,4',5-PeCB	118	31508-00-6	20	2.0
14.	2',3,4,4',5-PeCB	123	65510-44-3	20	2.0
15.	3,3',4,4',5-PeCB	126	57465-28-8	20	2.0
16.	2,2',4,4',6,6'-HxCB	155	33979-03-2	20	2.0
17.	2,3,3',4,4',5-HxCB	156	38380-08-4	20	2.0
18.	2,3,3',4,4',5'-HxCB	157	69782-90-7	20	2.0
19.	2,3',4,4',5,5'-HxCB	167	52663-72-6	20	2.0
20.	3,3',4,4',5,5'-HxCB	169	32774-16-6	20	2.0
21.	2,2',3,4',5,6,6'-HpCB	188	74487-85-7	20	2.0
22.	2,2',3,4',5,6,6'-HpCB	189	39635-31-9	20	2.0
23.	2,2',3,3',5,5',6,6'-OcCB	202	2136-99-4	20	2.0
24.	2,3,3',4,4',5,5',6-OcCB	205	74472-53-0	20	2.0
25.	2,2',3,3',4,4',5,5',6-NoCB	206	40186-72-9	20	2.0

PCB Congeners Target Compound List (TCL) and Contract Required Quantitation Limits (CRQL) For Aqueous and Solid Samples (Continued)

	Congener Analyte	IUPAC Number	CAS Number	Water (pg/L)	Solids[1] (ng/Kg)
26.	2,2',3,3',4,5,5',6,6'-NoCB	208	52663-77-1	20	2.0
27.	DeCB	209	2051-24-3	20	2.0

Congener PCB Notes

[1]There is no differentiation between the preparation of low and medium soil samples in this method for the analysis of congener PCBs.

PCDD/F Target Compound List (TCL) and Contract Required Quantitation Limits (CRQL) For Aqueous and Solid Samples

	PCDD/F Analyte	CAS Number	Water (pg/L)	Solids[1] (ng/Kg)
1.	2,3,7,8-TCDD	1746-01-6	10	1.0
2.	1,2,3,7,8-PeCDD	40321-76-4	50	5.0
3.	1,2,3,6,7,8-HxCDD	57653-85-7	50	5.0
4.	1,2,3,4,7,8-HxCDD	39227-28-6	50	5.0
5.	1,2,3,7,8,9-HxCDD	19408-74-3	50	5.0
6.	1,2,3,4,6,7,8-HpCDD	35822-46-9	50	5.0
7.	OCDD	3268-87-9	100	10
8.	2,3,7,8-TCDF	51207-31-9	10	1.0
9.	1,2,3,7,8-PeCDF	57117-41-6	50	5.0
10.	2,3,4,7,8-PeCDF	57117-31-4	50	5.0
11.	1,2,3,6,7,8-HxCDD	57117-44-9	50	5.0
12.	1,2,3,7,8,9-HxCDD	72918-21-9	50	5.0
13.	1,2,3,4,7,8-HxCDD	70648-26-9	50	5.0
14.	2,3,4,6,7,8-HxCDD	60851-34-5	50	5.0
15.	1,2,3,4,6,7,8-HpCDD	67562-39-4	50	5.0
16.	1,2,3,4,7,8,9-HpCDD	55673-89-7	50	5.0
17.	OCDF	39001-02-0	100	10

PCDD/F Notes

[1] There is no differentiation between the preparation of low and medium soil samples in this method for the analysis of PCDDs and PCDFs.

Total PCDD/F Homologues

Data are reported for the total concentration of all detected chlorinated dibenzo-p-dioxins (CDD) or chlorinated dibenzofurans (CDF's) in the following homologues. However, because the number of non-2,3,7,8-substituted isomers that might be detected in a sample is unpredictable, it is not possible to assign Contract Required Quantitation Limits (CRQLs) values to the total homologue concentrations.

PCDD/F Homologue	CAS Number	No. of Possible Isomers	No. of 2,3,7,8-Substituted Isomers
Total TCDD	41903-57-5	22	1
Total PeCDD	36088-22-9	14	1
Total HxCDD	34465-46-8	10	3
Total HpCDD	37871-00-4	2	1
Total TCDF	55722-27-5	38	1
Total PeCDF	30402-15-4	28	2
Total HxCDD	55684-94-1	16	4
Total HpCDD	38998-75-3	4	2

There is only one isomer in both the OCDD on OCDF homologues, hence the total concentration is the same as the 2,3,7,8-substituted concentration.

Homologue	Definition
TCDD	Tetrachlorinated dibenzo-p-dioxin
PeCDD	Pentachlorinated dibenzo-p-dioxin
HxCDD	Hexachlorinated dibenzo-p-dioxin
HpCDD	Heptachlorinated dibenzo-p-dioxin
OCDD	Octachlorinated dibenzo-p-dioxin
TCDF	Tetrachlorinated dibenzofuran
PeCDF	Pentachlorinated dibenzofuran
HxCDD	Hexachlorinated dibenzofuran
HpCDD	Heptachlorinated dibenzofuran
OCDF	Octachlorinated dibenzofuran

PART II – SUPERFUND-CLP INORGANICS

Inorganic Target Compound List (TCL) and Contract Required Quantitation Limits (CRQLs) For Aqueous and Solid Samples

	Analyte	CAS Number	ICP-AES[1] CRQL for Water (µg/L)	ICP-AES[1] CRQL for Solids (mg/Kg)	ICP-MS[1] for Water (µg/L)
1.	Aluminum	7429-90-5	200	40	30
2.	Antimony	7440-36-0	60	12	2
3.	Arsenic	7440-38-2	15	3	1
4.	Barium	7440-39-3	200	40	10
5.	Beryllium	7440-41-7	5	1	1
6.	Cadmium	7440-43-9	5	1	1
7	Calcium	7440-70-2	5000	1000	--
8.	Chromium	7440-47-3	10	2	2
9.	Cobalt	7440-48-4	50	10	0.5
10.	Copper	7440-50-8	25	5	2
11.	Iron	7439-89-6	100	20	--
12.	Lead	7439-92-1	10	2	1
13.	Magnesium	7439-95-4	5000	1000	--
14.	Manganese	7439-96-5	15	3	0.5
15.	Mercury[2]	7439-97-6	0.2	0.1	--
16.	Nickel	7440-02-0	40	8	1
17.	Potassium	7440-09-7	5000	1000	--
18.	Selenium	7782-49-2	35	7	5
19.	Silver	7440-22-4	10	2	1
20.	Sodium	7440-23-5	5000	1000	--
21.	Thallium	7440-28-0	25	5	1
22.	Vanadium	7440-62-2	50	10	1
23.	Zinc	7440-66-6	60	12	1
24.	Cyanide[2]	57-12-5	10	1	--

Inorganic Notes

[1] Any analytical method specified in Exhibit D, may be utilized as long as the documented instrument or method detection limits (IDLs or MDLs) are less than one half the Contract Required Quantitation Level (CRQL) requirements. Higher quantitation levels may only be used in the following circumstance:

If the sample concentration exceeds five times the quantitation limit of the instrument or method in use, the value may be reported even though the instrument or method detection limit may not equal the Contract Required Quantitation Limit. This is illustrated in the example below:

For lead:
Method in use = ICP
Instrument Detection Limit (IDL) = 40
Sample concentration = 220
Contract Required Quantitation Level (CRQL) = 3

The value of 220 may be reported even though instrument detection limit is greater than Contract Required Quantitation Limit. The instrument or method detection limit must be documented as described in Exhibit E.

[2] Mercury is analyzed by cold vapor atomic absorption. Cyanide is analyzed by colorimetry/spectrophotometry.

PART III – REGULATORY PROMULGATED PARAMETERS

In addition to the preceding lists, the Laboratory may be asked to analyze for any or all of the conventional water quality parameters as listed in 40CFR Part 136 or for the hazardous waste parameters listed in 40CFR Part 260 through 270 (RCRA).

Quantitation limits to be achieved for 40CFR Part 136 analyses are listed on tables contained in Part A of Section III. Quantitation limits to be achieved for RCRA analyses are specified on tables contained in Part B of Section III.

40CFR Part 136 Parameters
Target Compound List (TCL) and
Contract Required Quantitation Limit (CRQL)

	Parameter	CAS Number	Contract Required Quantitation Level (µg/L)
Conventionals			
1.	Biochemical Oxygen Demand (BOD5)	N/A	2,000
2.	Chemical Oxygen Demand (COD)	N/A	3,000
3.	Total Dissolved Solids (TDS)	N/A	10,000
4.	Total Suspended Solids (TSS)	N/A	4,000
5.	Ammonia, as N	7664-41-7	10
6.	Total Kjeldahl Nitrogen, as N	7727-37-9	30
7.	Nitrate-Nitrite	14797-55-8 /14797-65-0	16
8.	Total Phosphorus	7723-14-0	10
9.	Reactive Phosphorus	7723-14-0	10
10.	Sulfate	14808-79-8	80
11.	Oil and Grease	N/A	5,000
12.	Total Organic Carbon	N/A	1,000
13.	Total Phenols	64743-03-9	2
14.	Chloride	16887-00-6	16
15.	Fluoride	16984-48-8	36
16.	Cyanide	57-12-5	5
Metals			
1.	Aluminum	7429-90-5	30
2.	Antimony	7440-36-0	2
3.	Arsenic	7440-38-2	1
4.	Barium	7440-39-3	10
5.	Beryllium	7440-41-7	1
6.	Cadmium	7440-43-9	1

40CFR Part 136 Parameters
Target Compound List (TCL) and
Contract Required Quantitation Limit (CRQL) (Continued)

	Parameter	CAS Number	Contract Required Quantitation Level (µg/L)
Metals (Continued)			
7.	Calcium	7440-70-2	5000
8.	Chromium	7440-47-3	2
9.	Cobalt	7440-48-4	0.5
10.	Copper	7440-50-8	2
11.	Gold	7440-57-5	10
12.	Iridium	7439-88-5	200
13.	Iron	7439-89-6	100
14.	Lead	7439-92-1	1
15.	Magnesium	7439-95-4	5000
16.	Manganese	7439-96-5	0.5
17.	Mercury	7439-97-6	0.02
18.	Molybdenum	7439-98-7	10
19.	Nickel	7440-02-0	1
20.	Osmium	7440-02-4	100
21.	Palladium	7440-05-3	100
22.	Platinum	7440-06-4	200
23.	Potassium	7440-09-7	5000
24.	Rhenium	7440-15-5	5000
25.	Rhodium	7440-16-6	100
26.	Ruthenium	7440-18-8	500
27.	Selenium	7782-49-2	5
28.	Silver	7440-22-4	1
29.	Sodium	7440-23-5	5000
30.	Thallium	7440-28-0	1
31.	Tin	7440-31-5	40
32.	Titanium	7440-32-6	100

40CFR Part 136 Parameters
Target Compound List (TCL) and
Contract Required Quantitation Limit (CRQL) (Continued)

	Parameter	CAS Number	Contract Required Quantitation Level (µg/L)
Metals (Continued)			
33.	Vanadium	7440-62-2	1
34.	Zinc	7440-66-6	1
Trace Level Volatile Organics (Method 524)			
1.	Acetone	67-64-1	5.0
2.	Acrylonitrile	107-13-1	5.0
3.	Allyl Chloride	107-05-1	5.0
4.	Benzene	71-43-2	0.50
5.	Bromobenzene	108-86-1	0.50
6.	Bromochloromethane	74-97-5	0.50
7.	Bromodichloromethane	75-27-4	0.50
8.	Bromoform	75-25-2	0.50
9.	Bromomethane	74-38-9	0.50
10.	2-Butanone	78-93-3	5.0
11.	tert-Butyl Alcohol (TBA)	75-65-0	5.0
12.	n-Butylbenzene	104-51-8	0.50
13.	sec-Butylbenzene	135-98-8	0.50
14.	tert-Butylbenzene	98-06-6	0.50
15.	Carbon Disulfide	75-15-0	0.50
16.	Carbon Tetrachloride	56-23-5	0.50
17.	Chloroacetonitrile	107-14-2	5.0
18.	Chlorobenzene	108-90-7	0.50
19.	1-Chlorobutane	109-69-3	5.0
20.	Chloroethane	75-00-3	0.50
21.	Chloroform	67-66-3	0.50

40CFR Part 136 Parameters
Target Compound List (TCL) and
Contract Required Quantitation Limit (CRQL) (Continued)

	Parameter	CAS Number	Contract Required Quantitation Level (µg/L)
Trace Level Volatile Organics (Method 524) (Continued)			
22.	Chloromethane	74-87-3	0.50
23.	2-Chlorotoluene (o-Chlorotoluene)	95-49-8	0.50
24.	4-Chlorotoluene (p-Chlorotoluene)	106-43-4	0.50
25.	Dibromochloromethane	124-48-1	0.50
26.	1,2-Dibromo-3-Chloropropane	96-12-8	0.50
27.	1,2-Dibromoethane	106-93-4	0.50
28.	Dibromomethane	74-95-3	0.50
29.	1,2-Dichlorobenzene	95-50-1	0.50
30.	1,3-Dichlorobenzene	541-73-1	0.50
31.	1,4-Dichlorobenzene	106-46-7	0.50
32.	trans-1,4-Dichloro-2-Butene	110-57-6	5.0
33.	Dichlorodifluoromethane	75-71-8	0.50
34.	1,1-Dichloroethane	75-34-3	0.50
35.	1,2-Dichloroethane	107-06-2	0.50
36.	1,1-Dichloroethene	75-35-4	0.50
37.	cis-1,2-Dichloroethene	156-59-2	0.50
38.	trans-1,2-Dichloroethene	156-60-5	0.50
39.	1,2-Dichloropropane	78-87-5	0.50
40.	1,3-Dichloropropane	142-28-9	0.50
41.	2,2-Dichloropropane (sec-Dichloropropane)	594-20-7	0.50
42.	1,1-Dichloropropene	563-58-6	0.50
43.	1,1-Dichloropropanone (1,1-Dichloroacetone)	513-88-2	5.0

40CFR Part 136 Parameters
Target Compound List (TCL) and
Contract Required Quantitation Limit (CRQL) (Continued)

	Parameter	CAS Number	Contract Required Quantitation Level (µg/L)
Trace Level Volatile Organics (Method 524) (Continued)			
44.	cis-1,3-Dichloropropene	10061-01-5	0.50
45.	trans-1,3-Dichloropropene	10061-02-6	0.50
46.	Diethyl Ether	60-29-7	5.0
47.	Ethylbenzene	100-41-4	0.50
48.	Ethyl Methacrylate	97-63-2	0.50
49.	Hexachlorobutadiene	87-68-3	0.50
50.	Hexachloroethane	67-72-1	0.50
51.	2-Hexanone	591-78-6	5.0
52.	Isopropylbenzene	98-82-8	0.50
53.	4-Isopropyltoluene (p-Cymene)	99-87-6	0.50
54.	Methacrylonitrile	126-98-7	5.0
55.	Methylacrylate	96-33-3	5.0
56.	Methylene Chloride	75-09-2	0.50
57.	Methyl Iodide (Iodomethane)	74-88-4	0.50
58.	Methylmethacrylate	80-62-6	5.0
59.	4-Methyl-2-Pentanone	108-10-1	5.0
60.	Methyl-t-butyl Ether (MTBE)	1634-04-4	0.50
61.	Naphthalene	91-20-3	0.50
62.	Nitrobenzene	98-95-3	5.0
63.	2-Nitropropane	79-46-9	5.0
64.	Pentachloroethane	76-01-7	0.50
65.	Propionitrile (Ethyl cyanide)	107-12-0	5.0
66.	n-Propylbenzene	103-65-1	0.50

40CFR Part 136 Parameters
Target Compound List (TCL) and
Contract Required Quantitation Limit (CRQL) (Continued)

	Parameter	CAS Number	Contract Required Quantitation Level (µg/L)
Trace Level Volatile Organics (Method 524) (Continued)			
67.	Styrene	100-42-5	0.50
68.	1,1,1,2-Tetrachloroethane	630-20-6	0.50
69.	1,1,2,2-Tetrachloroethane	79-34-5	0.50
70.	Tetrachloroethene	127-18-4	0.50
71.	Tetrahydrofuran	109-99-9	5.0
72.	Toluene	108-88-3	0.50
73.	1,2,3-Trichlorobenzene	87-61-6	0.50
74.	1,2,4-Trichlorobenzene	120-82-1	0.50
75.	1,1,1-Trichloroethane	71-55-6	0.50
76.	1,1,2-Trichloroethane	79-00-5	0.50
77.	Trichloroethene	79-01-6	0.50
78.	Trichlorofluoromethane	75-69-4	0.50
79.	1,2,3-Trichloropropane	96-18-4	0.50
80.	1,2,4-Trimethylbenzene	95-63-6	0.50
81.	1,3,5-Trimethylbenzene (Mesitylene)	108-67-8	0.50
82.	Vinyl Chloride	75-01-4	0.50
83.	o-Xylene	95-47-6	0.50
84.	m-Xylene	108-38-3	0.50
85.	p-Xylene	106-42-3	0.50
Volatile Organics (Method 624)			
1.	Chloromethane	74-87-3	10
2.	Bromomethane	74-83-9	10
3.	Vinyl chloride	75-01-4	10

40CFR Part 136 Parameters
Target Compound List (TCL) and
Contract Required Quantitation Limit (CRQL) (Continued)

	Parameter	CAS Number	Contract Required Quantitation Level (µg/L)
Volatile Organics (Method 624) (Continued)			
4.	Chloroethane	75-00-3	10
5.	Methylene chloride	75-09-2	5
6.	1,1-Dichloroethene	75-35-4	5
7.	1,1-Dichloroethane	75-35-3	5
8.	trans-1,2-Dichloroethene	156-60-5	5
9.	Chloroform	67-66-3	5
10.	1,2-Dichloroethane	107-06-2	5
11.	1,1,1-Trichloroethane	71-55-6	5
12.	Carbon tetrachloride	56-23-5	5
13.	Bromodichloromethane	75-27-4	5
14.	1,1,2,2-Tetrachloroethane	79-34-5	5
15.	1,2-Dichloropropane	78-87-5	5
16.	trans-1,3-Dichloropropene	10061-02-6	5
17.	Trichloroethene	79-01-6	5
18.	Dibromochloromethane	124-48-1	5
19.	1,1,2-Trichloroethane	79-00-5	5
20.	Benzene	71-43-2	5
21.	cis-1,3-Dichloropropene	10061-01-5	5
22.	2-Chloroethyl vinyl ether	110-75-8	10
23.	Bromoform	75-25-2	5
24.	Tetrachloroethene	127-18-4	5
25.	Toluene	108-88-3	5
26.	Chlorobenzene	108-90-7	5

40CFR Part 136 Parameters
Target Compound List (TCL) and
Contract Required Quantitation Limit (CRQL) (Continued)

	Parameter	CAS Number	Contract Required Quantitation Level (µg/L)
Volatile Organics (Method 624) (Continued)			
27.	Ethylbenzene	100-41-4	5
28.	1,3-Dichlorobenzene	541-73-1	10
29.	1,4-Dichlorobenzene	106-46-7	10
30.	1,2-Dichlorobenzene	95-50-1	10
31.	Trichlorofluoromethane	75-69-4	10
Volatile Organics (Method 601)			
1.	Chloromethane	74-87-3	0.5
2.	Bromomethane	74-83-9	5
3.	Vinyl chloride	75-01-4	1.0
4.	Chloroethane	75-00-3	5
5.	Methylene chloride	75-09-2	1.0
6.	1,1-Dichloroethene	75-35-4	0.1
7.	1,1-Dichloroethane	75-35-3	0.5
8.	trans-1,2-Dichloroethene	156-60-5	0.5
9.	Chloroform	67-66-3	0.5
10.	1,2-Dichloroethane	107-06-2	0.1
11.	1,1,1-Trichloroethane	71-55-6	0.1
12.	Carbon tetrachloride	56-23-5	0.5
13.	Bromodichloromethane	75-27-4	0.5
14.	1,1,2,2-Tetrachloroethane	79-34-5	0.1
15.	1,2-Dichloropropane	78-87-5	0.5
16.	trans-1,3-Dichloropropene	10061-02-6	1.0

40CFR Part 136 Parameters
Target Compound List (TCL) and
Contract Required Quantitation Limit (CRQL) (Continued)

	Parameter	CAS Number	Contract Required Quantitation Level (µg/L)
Volatile Organics (Method 601) (Continued)			
17.	Trichloroethene	79-01-6	0.5
18.	Dibromochloromethane	124-48-1	0.5
19.	1,1,2-Trichloroethane	79-00-5	0.1
20.	cis-1,3-Dichloropropene	10061-01-5	0.5
21.	2-Chloroethyl vinyl ether	110-75-8	0.5
22.	Bromoform	75-25-2	1.0
23.	Tetrachloroethene	127-18-4	0.1
24.	Chlorobenzene	108-90-7	1.0
25.	1,2-Dichlorobenzene	95-50-1	1.0
26.	1,3-Dichlorobenzene	541-73-1	1.0
27.	1,4-Dichlorobenzene	106-46-7	1.0
28.	Trichlorofluoromethane	75-69-4	2.0
Volatile Organics (Method 602)			
1.	Benzene	71-43-2	1.0
2.	Toluene	108-88-3	1.0
3.	Chlorobenzene	108-90-7	1.0
4.	Ethyl Benzene	100-41-4	1.0
5.	1,3-Dichlorobenzene	541-73-1	1.0
6.	1,4-Dichlorobenzene	106-46-7	1.0
7.	1,2-Dichlorobenzene	95-50-1	1.0

40CFR Part 136 Parameters
Target Compound List (TCL) and
Contract Required Quantitation Limit (CRQL) (Continued)

	Parameter	CAS Number	Contract Required Quantitation Level (µg/L)
Semivolatile Organics (Method 625)			
1.	N-Nitrosodimethylamine	62-75-9	5.0
2.	Phenol	108-95-2	5.0
3.	bis(2-Chloroethyl) ether	111-44-4	5.0
4.	2-Chlorophenol	95-57-8	5.0
5.	1,3-Dichlorobenzene	541-73-1	5.0
6.	1,4-Dichlorobenzene	106-46-7	5.0
7.	1,2-Dichlorobenzene	95-50-1	5.0
8.	2,2'-oxybis (1-Chloropropane)	108-60-1	5.0
9.	N-Nitrosodi-n-propylamine	621-64-7	5.0
10.	Hexachloroethane	67-72-1	5.0
11.	Nitrobenzene	98-95-3	5.0
12.	Isophorone	78-59-1	5.0
13.	2-Nitrophenol	88-75-5	5.0
14.	2,4-Dimethylphenol	105-67-9	5.0
15.	bis(2-Chloroethoxy) methane	111-91-1	5.0
16.	2,4-Dichlorophenol	120-83-2	5.0
17.	1,2,4-Trichlorobenzene	120-82-1	5.0
18.	Naphthalene	91-20-3	5.0
19.	Hexachlorobutadiene	87-68-3	5.0
20.	4-Chloro-3-methylphenol (p-chloro-m-cresol)	59-50-7	5.0
21.	Hexachlorocyclopentadiene	77-47-4	5.0
22.	2,4,6-Trichlorophenol	88-06-2	5.0

40CFR Part 136 Parameters
Target Compound List (TCL) and
Contract Required Quantitation Limit (CRQL) (Continued)

	Parameter	CAS Number	Contract Required Quantitation Level (µg/L)
Semivolatile Organics (Method 625) (Continued)			
23.	2-Chloronaphthalene	91-58-7	5.0
24.	Dimethyl phthalate	131-11-3	5.0
25.	Acenaphthylene	208-96-8	5.0
26.	Acenaphthene	83-32-9	5.0
27.	2,4-Dinitrophenol	51-28-5	10
28.	4-Nitrophenol	100-02-7	10
29.	2,4-Dinitrotoluene	121-14-2	5.0
30.	2,6-Dinitrotoluene	606-20-2	5.0
31.	Diethylphthalate	84-66-2	5.0
32.	4-Chlorophenyl phenyl ether	7005-72-3	5.0
33.	Fluorene	86-73-7	5.0
34.	4,6-Dinitro-2-methylphenol	534-52-1	10
35.	N-nitroso diphenylamine	86-30-6	5.0
36.	4-Bromophenyl phenyl ether	101-55-3	5.0
37.	Hexachlorobenzene	118-74-1	5.0
38.	Pentachlorophenol	87-86-5	5.0
39.	Phenanthrene	85-01-8	5.0
40.	Anthracene	120-12-7	5.0
41.	Di-n-butyl phthalate	84-74-2	5.0
42.	Fluoranthene	206-44-0	5.0
43.	Benzidine	92-87-5	50
44.	Pyrene	129-00-0	5.0
45.	Butyl benzyl phthalate	85-68-7	5.0

40CFR Part 136 Parameters
Target Compound List (TCL) and
Contract Required Quantitation Limit (CRQL) (Continued)

	Parameter	CAS Number	Contract Required Quantitation Level (µg/L)
Semivolatile Organics (Method 625) (Continued)			
46.	3,3'-Dichlorobenzidine	91-94-1	5.0
47.	Benz[a]anthracene	56-55-3	5.0
48.	bis(2-ethylhexyl)phthalate	117-81-7	5.0
49.	Chrysene	218-01-9	5.0
50.	Di-n-octyl phthalate	117-84-0	5.0
51.	Benzo[b]fluoranthene	205-99-2	5.0
52.	Benzo[k]fluoranthene	207-08-9	5.0
53.	Benzo[a]pyrene	50-32-8	5.0
54.	Indeno[1,2,3-cd]pyrene	193-39-5	5.0
55.	Dibenz[a,h]anthracene	53-70-3	5.0
56.	Benzo[g,h,i]perylene	191-24-2	5.0
Pesticides/PCBs (Method 608)			
1.	alpha-BHC	319-84-6	0.01
2.	beta-BHC	319-85-7	0.01
3.	delta-BHC	319-86-8	0.01
4.	gamma-BHC (Lindane)	58-89-9	0.01
5.	Heptachlor	76-44-8	0.01
6.	Aldrin	309-00-2	0.01
7.	Heptachlor epoxide	1024-57-3	0.01
8.	Endosulfan I	959-98-8	0.01
9.	Dieldrin	60-57-1	0.02
10.	4,4'-DDE	72-55-9	0.02

40CFR Part 136 Parameters
Target Compound List (TCL) and
Contract Required Quantitation Limit (CRQL) (Continued)

	Parameter	CAS Number	Contract Required Quantitation Level (µg/L)
Pesticides/PCBs (Method 608) (Continued)			
11.	Endrin	72-20-8	0.02
12.	Endosulfan II	33213-65-9	0.02
13.	4,4'-DDD	72-54-8	0.02
14.	Endrin aldehyde	7421-93-4	0.02
15.	Endosulfan sulfate	1031-07-8	0.02
16.	4,4'-DDT	50-29-3	0.02
17.	Chlordane	57-74-9	0.01
18.	Toxaphene	8001-35-2	1.0
19.	AROCLOR-1016	12674-11-2	0.20
20.	AROCLOR-1221	11104-28-2	0.40
21.	AROCLOR-1232	11141-16-5	0.20
22.	AROCLOR-1242	53469-21-9	0.20
23.	AROCLOR-1248	12672-29-6	0.20
24.	AROCLOR-1254	11097-69-1	0.20
25.	AROCLOR-1260	11096-82-5	0.20
Other Pesticides/Herbicides (Various methods)			
1.	Ametryn	834-12-8	2.0
2.	Aminocarb	2032-59-9	1.0
3.	Atraton	1610-17-9	2.0
4.	Atrazine	1912-24-9	2.0
5.	Azinphos methyl	86-50-0	1.0
6.	Barban	101-27-9	0.5
7.	Captan	133-06-2	1.0

40CFR Part 136 Parameters
Target Compound List (TCL) and
Contract Required Quantitation Limit (CRQL) (Continued)

	Parameter	CAS Number	Contract Required Quantitation Level (µg/L)
Other Pesticides/Herbicides (Various methods) (Continued)			
8.	Carbaryl	63-25-2	1.0
9.	Chlorpropham	101-21-3	1.0
10.	2,4-Dichlorophenoxy acetic acid (2,4-D)	94-75-2	2.0
11.	Demeton-O	298-03-3	1.0
12.	Demeton-S	126-75-0	1.0
13.	Diazinon	333-41-5	1.0
14.	Dicamba	1918-00-9	2.0
15.	Dichloran	102-30-7	1.0
16.	Disulfoton	298-04-4	2.0
17.	Diuron	330-54-1	1.0
18.	Fenuron	101-42-8	0.5
19.	Fenuron-TCA	4482-55-7	1.0
20.	Linuron	330-55-2	1.0
21.	Malathion	121-75-5	1.0
22.	Methiocarb	2032-65-7	2.0
23.	Methoxychlor	72-43-5	0.5
24.	Mexacarbate	315-18-4	1.0
25.	Mirex	2385-85-5	1.0
26.	Monuron	150-68-5	0.5
27.	Monuron-TCA	140-41-0	1.0
28.	Neburon	555-37-3	1.0
29.	Parathion ethyl	56-38-2	1.0

40CFR Part 136 Parameters
Target Compound List (TCL) and
Contract Required Quantitation Limit (CRQL) (Continued)

	Parameter	CAS Number	Contract Required Quantitation Level (µg/L)
Other Pesticides/Herbicides (Various methods) (Continued)			
30.	Parathion methyl	298-00-0	1.0
31.	Pentachloronitrobenzene (PCNB)	82-68-8	1.0
32.	Prometon	1610-18-0	2.0
33.	Prometryn	7287-19-6	2.0
34.	Propazine	139-40-2	2.0
35.	Propham	122-42-9	2.0
36.	Propoxur	114-26-1	1.0
37.	Secbumeton	26259-45-0	2.0
38.	Siduron	1982-49-6	0.5
39.	Simazine	122-34-9	2.0
40.	Strobane	8001-50-1	1.0
41.	Swep	1918-18-9	2.0
42.	2,4,5-Trichlorophenoxyacetic acid (2,4,5-T)	93-76-5	2.0
43	(2,4,5-Trichlorophenoxy)-propionic acid (2,4,5-TP; Silvex)	93-72-1	2.0
44.	Terbuthylazine	5915-41-3	2.0
45.	Trifluraline	1582-09-8	1.0
Dioxin			
1.	2,3,7,8-Tetrachlorodibenzo-p-dioxin (2,3,7,8-TCDD)	1746-01-6	0.01 (ng/L)

Resource Conservation and Recovery Act (RCRA) Parameters
RCRA Target Compound List (TCL) and
Contract Required Quantitation Limit (CRQL)

	Parameter	CAS Number	Contract Required Quantitation Level (µg/L)
A.	Ignitability (°C or °F)	N/A	N/A
B.	Corrosivity (pH units)	N/A	N/A
C.	Reactivity	--	--
1.	Total Releasable Cyanide as HCN	N/A	100000
2.	Total Releasable Sulfide as H_2S	N/A	100000
D.	Extraction Procedure Toxicity (EP Tox) (concentrations in extract)	--	--
1.	Arsenic	7440-38-2	1000
2.	Barium	7440-39-3	10000
3.	Cadmium	7440-43-9	100
4.	Total Chromium	7440-47-3	1000
5.	Lead	7439-92-1	1000
6.	Mercury	7439-97-6	50
7.	Selenium	7782-49-2	100
8.	Silver	7440-22-4	1000
9.	gamma-BHC (Lindane)	58-89-9	100
10.	2,4-Dichlorophenoxyacetic acid (2,4-D)	94-75-2	1000
11.	Endrin	72-20-8	5
12.	Methoxychlor	72-43-5	1000
13.	2,4,5-Trichlorophenoxy-propionic acid (2,4,5-TP; Silvex)	93-72-1	100
14.	Toxaphene	8001-35-2	100

Resource Conservation and Recovery Act (RCRA) Parameters
RCRA Target Compound List (TCL) and
Contract Required Quantitation Limit (CRQL) (Continued)

	Parameter	CAS Number	Contract Required Quantitation Level (µg/L)
E.	Toxicity Characteristic Leaching Procedure (TCLP) (concentrations in extract) (Continued)	--	--
TCLP Metals (Continued)			
1.	Arsenic	7440-38-2	1000
2.	Barium	7440-39-3	10000
3.	Cadmium	7440-43-9	100
4.	Total Chromium	7440-47-3	1000
5.	Lead	7439-92-1	1000
6.	Mercury	7439-97-6	50
7.	Selenium	7782-49-2	100
8.	Silver	7440-22-4	1000
TCLP Volatiles (ZHE)			
1.	Benzene	71-43-2	10
2.	2-Butanone (Methylethylketone)	78-93-3	10
3.	Carbon tetrachloride	56-23-5	10
4.	Chlorobenzene	108-90-7	10
5.	Chloroform	67-66-3	10
6.	1,2-Dichloroethane	107-06-2	10
7.	1,1-Dichloroethylene	75-35-4	10
8.	Tetrachloroethylene	127-18-4	10
9.	Trichloroethylene	79-01-6	10
10.	Vinyl chloride	75-01-4	10
TCLP Semivolatiles			
1.	1,4-Dichlorobenzene	106-46-7	10
2.	2,4-Dinitrotoluene	121-14-2	10

Resource Conservation and Recovery Act (RCRA) Parameters
RCRA Target Compound List (TCL) and
Contract Required Quantitation Limit (CRQL) (Continued)

	Parameter	CAS Number	Contract Required Quantitation Level (µg/L)
E.	Toxicity Characteristic Leaching Procedure (TCLP) (concentrations in extract) (Continued)	--	--
TCLP Semivolatiles (Continued)			
3.	Hexachlorobenzene	118-74-1	10
4.	Hexachlorobutadiene	87-68-3	10
5.	Hexachloroethane	67-72-1	100
6.	2-Methylphenol (o-Cresol)	95-48-7	10
7.	3-Methylphenol (m-Cresol)	108-39-4	10
8.	4-Methylphenol (p-Cresol)	106-44-5	10
9.	Nitrobenzene	98-95-3	10
10.	Pentachlorophenol	87-86-5	5
11.	Pyridine	110-86-1	100
12.	2,4,5-Trichlorophenol	95-95-4	10
13.	2,4,6-Trichlorophenol	88-06-2	10
TCLP Pesticides			
1.	gamma-BHC (Lindane)	58-89-9	10
2.	Chlordane	57-74-9	10
3.	2,4-Dichlorophenoxyacetic acid (2,4-D)	94-75-7	100
4.	Endrin	72-20-8	0.5
5.	Heptachlor	76-44-8	0.5
6.	Heptachlor epoxide	1024-57-3	0.5
7.	Methoxychlor	72-43-5	100
8.	2,4,5-Trichlorophenoxy-propionic acid (2,4,5-TP; Silvex)	93-76-5	10
9.	Toxaphene	8001-35-2	10

Resource Conservation and Recovery Act (RCRA) Parameters
RCRA Target Compound List (TCL) and
Contract Required Quantitation Limit (CRQL) (Continued)

	Parameter	CAS Number	Contract Required Quantitation Level	
			Low Level Water (µg/L)	Low/Medium Level Solids (µg/Kg)
F.	Appendix IX Substances	--	--	--
Volatiles				
1.	Acetone	67-64-1	10	10
2.	Acetonitrile	75-05-8	100	100
3.	Acrolein	107-02-8	5	5
4.	Acrylonitrile	107-13-1	5	5
5.	Benzene	71-43-2	5	5
6.	Bromodichloromethane	75-27-4	5	5
7.	Bromoform	75-25-2	5	5
8.	Bromomethane	74-83-9	10	10
9.	2-Butanone (Methyl ethyl ketone)	78-93-3	10	10
10.	Carbon disulfide	75-15-0	5	5
11.	Carbon tetrachloride	56-23-5	5	5
12.	Chlorobenzene	108-90-7	5	5
13.	2-Chloro-1,3-butadiene	126-99-8	5	5
14.	Chloroethane	75-00-3	10	10
15.	Chloroform	67-66-3	5	5
16.	Chloromethane	74-87-3	10	10
17.	3-Chloropropene	107-05-1	100	100
18.	Dibromochloromethane	124-48-1	5	5
19.	1,2-Dibromo-3-chloropropane	96-12-8	5	5
20.	1,2-Dibromoethane	106-93-4	5	5
21.	Dibromomethane	74-95-3	5	5

Resource Conservation and Recovery Act (RCRA) Parameters
RCRA Target Compound List (TCL) and
Contract Required Quantitation Limit (CRQL) (Continued)

	Parameter	CAS Number	Contract Required Quantitation Level	
			Low Level Water (µg/L)	Low/Medium Level Solids (µg/Kg)
F.	Appendix IX Substances (Continued)	--	--	--
Volatiles (Continued)				
22.	trans-1,4-Dichloro-2-butene	110-57-6	5	5
23.	Dichlorodifluoromethane	75-71-8	5	5
24.	1,1-Dichloroethane	75-34-3	5	5
25.	1,2-Dichloroethane	107-06-2	5	5
26.	1,1-Dichloroethylene	75-35-4	5	5
27.	trans-1,2-Dichloroethylene	156-60-5	5	5
28.	Dichloromethane	75-09-2	5	5
29.	1,2-Dichloropropane	78-87-5	5	5
30.	cis-1,3-Dichloropropane	10061-01-5	5	5
31.	trans-1,3-Dichloropropane	10061-02-6	5	5
32.	1,4-Dioxane	123-91-1	150	150
33.	Ethylbenzene	100-41-4	5	5
34.	Ethylmethacrylate	97-63-2	5	5
35.	2-Hexanone	591-78-6	10	10
36.	Iodomethane	74-88-4	5	5
37.	Methacrylonitrile	126-98-7	5	5
38.	Methylmethacrylate	80-62-6	5	5
39.	4-Methyl-2-pentanone (Methyl iso-butyl ketone)	108-10-1	10	10
40.	2-Methyl-1-propanol (Iso-butyl alcohol)	78-83-1	50	50

Resource Conservation and Recovery Act (RCRA) Parameters
RCRA Target Compound List (TCL) and
Contract Required Quantitation Limit (CRQL) (Continued)

	Parameter	CAS Number	Contract Required Quantitation Level	
			Low Level Water (µg/L)	Low/Medium Level Solids (µg/Kg)
F.	Appendix IX Substances (Continued)	--	--	--
Volatiles (Continued)				
41.	Pentachloroethane	76-01-7	5	5
42.	2-Picoline	109-06-8	5	5
43	Propionitrile	107-12-0	5	5
44.	Pyridine	110-86-1	5	5
45.	Styrene	100-42-5	5	5
46.	1,1,1,2-Tetrachloroethane	630-20-6	5	5
47.	1,1,2,2-Tetrachloroethane	79-34-5	5	5
48.	Tetrachloroethylene	127-18-4	5	5
49.	Toluene	108-88-3	5	5
50.	1,1,1-Trichloroethane	71-55-6	5	5
51.	1,1,2-Trichloroethane	79-00-5	5	5
52.	Trichloroethylene	79-01-6	5	5
53.	Trichlorofluoromethane	75-69-4	5	5
54.	1,2,3-Trichloropropane	96-18-4	5	5
55.	Vinyl acetate	108-05-4	5	5
56.	Vinyl chloride	75-01-4	10	10
57.	Xylene (Total)	1330-20-7	5	5
Semivolatiles				
58.	Acenaphthene	83-32-9	10	330
59.	Acenaphthylene	208-96-8	10	330

Resource Conservation and Recovery Act (RCRA) Parameters
RCRA Target Compound List (TCL) and
Contract Required Quantitation Limit (CRQL) (Continued)

	Parameter	CAS Number	Contract Required Quantitation Level	
			Low Level Water (µg/L)	Low/Medium Level Solids (µg/Kg)
F.	Appendix IX Substances (Continued)	--	--	--
Semivolatiles (Continued)				
60.	Acetophenone	98-86-2	10	330
61.	2-Acetylaminofluorene	53-96-3	10	330
62.	4-Aminobiphenyl	92-67-1	10	330
63.	Aniline	62-53-3	10	330
64.	Anthracene	120-12-7	10	330
65.	Aramite	140-57-8	10	330
66.	Benz[a]anthracene	56-55-3	10	330
67.	Benzo[b]fluoranthene	205-99-2	10	330
68.	Benzo[k]fluoranthene	207-08-9	10	330
69.	Benzo[g.h,i]perylene	191-24-2	10	330
70.	Benzo[a]pyrene	50-32-8	10	330
71.	Benzyl alcohol	100-51-6	10	330
72.	Bis(2-chloroethoxy)methane	111-91-1	10	330
73.	Bis(2-chloroethyl)ether	111-44-4	10	330
74.	2,2'-oxybis(1-Chloropropane)	108-60-1	10	330
75.	Bis(2-ethylhexyl)phthalate	117-81-7	10	330
76.	4-Bromophenyl phenyl ether	101-55-3	10	330
77.	Butyl benzyl phthalate	85-68-7	10	330
78.	p-Chloroaniline	106-47-8	10	330
79.	Chlorobenzilate	510-15-6	10	330

Resource Conservation and Recovery Act (RCRA) Parameters
RCRA Target Compound List (TCL) and
Contract Required Quantitation Limit (CRQL) (Continued)

	Parameter	CAS Number	Contract Required Quantitation Level	
			Low Level Water (µg/L)	Low/Medium Level Solids (µg/Kg)
F.	Appendix IX Substances (Continued)	--	--	--
Semivolatiles (Continued)				
80.	4-chloro-3-methylphenol (p-chloro-m-cresol)	59-50-7	10	330
81.	2-Chloronaphthalene	91-58-7	10	330
82.	2-Chlorophenol	95-57-8	10	330
83.	4-Chlorophenyl phenyl ether	7005-72-3	10	330
84.	Chrysene	218-01-9	10	330
85.	Diallate	2303-16-4	10	330
86.	Dibenz[a,h]anthracene	53-70-3	10	330
87.	Dibenzofuran	132-64-9	10	330
88.	Di-n-butylphthalate	84-74-2	10	330
89.	1,2-Dichlorobenzene	95-50-1	10	330
90.	1,3-Dichlorobenzene	541-73-1	10	330
91.	1,4-Dichlorobenzene	106-46-7	10	330
92.	3,3'-Dichlorobenzidine	91-94-1	20	660
93.	2,4-Dichlorophenol	120-83-2	10	330
94.	2,6-Dichlorophenol	87-65-0	10	330
95.	Diethylphthlate	84-66-2	10	330
96.	O,O-Diethyl-0-2-pyrazinyl-phosphorothioate	297-97-2	10	330
97.	Dimethoate	60-51-5	10	330

Resource Conservation and Recovery Act (RCRA) Parameters
RCRA Target Compound List (TCL) and
Contract Required Quantitation Limit (CRQL) (Continued)

	Parameter	CAS Number	Contract Required Quantitation Level	
			Low Level Water (µg/L)	Low/Medium Level Solids (µg/Kg)
F.	Appendix IX Substances (Continued)	--	--	--
Semivolatiles (Continued)				
98.	p-(Dimethylamino)-azobenzene	60-11-7	10	330
99.	7,12-Dimethylbenz[a]-anthracene	57-97-6	10	330
100.	3,3'-Dimethylbenzidine	119-93-7	10	330
101.	a,a-Dimethylphenethylamine	122-09-8	10	330
102.	2,4-Dimethylphenol	105-67-9	10	330
103.	Dimethylphthlate	131-11-3	10	330
104.	1,3-Dinitrobenzene	99-65-0	10	330
105.	4,6-Dinitro-2-methylphenol	534-52-1	50	1700
106.	2,4-Dinitrophenol	51-28-5	50	1700
107.	2,4-Dinitrotoluene	121-14-2	10	330
108.	2,6-Dinitrotoluene	606-20-2	10	330
109.	Di-n-octylphthalate	117-84-0	10	330
110.	Diphenylamine	122-39-4	10	330
111.	Ethyl methanesulfonate	62-50-0	10	330
112.	Famphur	52-85-7	10	330
113.	Fluoranthene	206-44-0	10	330
114.	Fluorene	86-73-7	10	330
115.	Hexachlorobenzene	118-74-1	10	330
116.	Hexachlorobutadiene	87-68-3	10	330

Resource Conservation and Recovery Act (RCRA) Parameters
RCRA Target Compound List (TCL) and
Contract Required Quantitation Limit (CRQL) (Continued)

	Parameter	CAS Number	Contract Required Quantitation Level	
			Low Level Water (µg/L)	Low/Medium Level Solids (µg/Kg)
F.	Appendix IX Substances (Continued)	--	--	--
Semivolatiles (Continued)				
117.	Hexachlorocyclopentadiene	77-47-4	10	330
118.	Hexachlorodibenzo-p-dioxins (all isomers)	N/A	0.01	1.0
119.	Hexachlorodibenzofurans (all isomers)	N/A	0.01	1.0
120.	Hexachloroethane	67-72-1	10	330
121.	Hexachlorophene	70-30-4	10	330
122.	Hexachloropropene	1888-71-7	10	330
123.	Indeno[1,2,3-c,d]pyrene	193-39-5	10	330
124.	Isodrin	465-73-6	10	330
125.	Isophorone	78-59-1	10	330
126.	Isosafrole	120-58-1	10	330
127.	Kepone	143-50-0	10	330
128.	Methapyrilene	91-80-5	10	330
129.	3-Methylcholanthrene	56-49-5	10	330
130.	Methyl methane sulfonate	66-27-3	10	330
131.	2-Methylnaphthalene	91-57-6	10	330
132.	2-Methylphenol (o-Cresol)	95-48-7	10	330
133.	3-Methylphenol (m-Cresol)	108-39-4	10	330
134.	4-Methylphenol (p-Cresol)	106-44-5	10	330
135.	Naphthalene	91-20-3	10	330

Resource Conservation and Recovery Act (RCRA) Parameters
RCRA Target Compound List (TCL) and
Contract Required Quantitation Limit (CRQL) (Continued)

	Parameter	CAS Number	Contract Required Quantitation Level	
			Low Level Water (µg/L)	Low/Medium Level Solids (µg/Kg)
F.	Appendix IX Substances (Continued)	--	--	--
Semivolatiles (Continued)				
136.	1,4-Naphthoquinone	130-15-4	10	330
137.	1-Naphthylamine	134-32-7	10	330
138.	2-Naphthylamine	91-59-8	10	330
139.	2-Nitroaniline	88-74-4	50	1700
140.	3-Nitroaniline	99-09-2	50	1700
141.	4-Nitroaniline	100-01-6	50	1700
142.	Nitrobenzene	98-95-3	10	330
143.	2-Nitrophenol	88-75-5	10	330
144.	4-Nitrophenol	100-02-7	50	1700
145.	4-Nitroquinoline-1-oxide	56-57-5	10	330
146.	N-Nitrosodi-n-butylamine	924-16-3	10	330
147.	N-Nitrosodiethylamine	55-18-5	10	330
148.	N-Nitrosodimethylamine	62-75-9	10	330
149.	N-Nitrosodiphenylamine	86-30-6	10	330
150.	N-Nitrosodi-n-propylamine	621-24-7	10	330
151.	N-Nitrosomethylethylamine	10595-95-6	10	330
152.	N-Nitrosomorpholine	59-89-2	10	330
153.	N-Nitrosopiperidine	100-75-4	10	330
154.	N-Nitrosopyrolidine	930-55-2	10	330
155.	5-Nitro-o-toluidine	99-55-8	10	330
156.	Parathion	56-38-2	10	330

Resource Conservation and Recovery Act (RCRA) Parameters
RCRA Target Compound List (TCL) and
Contract Required Quantitation Limit (CRQL) (Continued)

	Parameter	CAS Number	Contract Required Quantitation Level	
			Low Level Water (µg/L)	Low/Medium Level Solids (µg/Kg)
F.	Appendix IX Substances (Continued)	--	--	--
Semivolatiles (Continued)				
157.	Pentachlorobenzene	608-93-5	10	330
158.	Pentachlorodibenzo-p-dioxins (all isomers)	N/A	0.01	1.0
159.	Pentachlorodibenzofurans (all isomers)	N/A	0.01	1.0
160.	Pentachloronitrobenzene	82-68-8	10	330
161.	Pentachlorophenol	87-86-5	50	1700
162.	Phenacetin	62-44-2	10	330
163.	Phenanthrene	85-01-8	10	330
164.	Phenol	108-95-2	10	330
165.	p-Phenylenediamine	106-50-3	10	330
166.	Pronamide	23950-58-5	10	330
167.	Pyrene	129-00-0	10	330
168.	Safrole	94-59-7	10	330
169.	1,2,4,5-Tetrachlorobenzene	95-94-3	10	330
170.	2,3,7,8-Tetrachloro-dibenzo-p-dioxin (2,3,7,8-TCDD)	1746-01-6	0.005	0.5
171.	Tetrachlorodibenzo-p-dioxins (all isomers)	N/A	0.01	1.0
172.	Tetrachlorodibenzofurans (all isomers)	N/A	0.01	1.0
173.	2,3,4,6-Tetrachlorophenol	58-90-2	10	330
174.	Tetraethyldithiopyrophos-phate	3689-24-5	10	330

Resource Conservation and Recovery Act (RCRA) Parameters
RCRA Target Compound List (TCL) and
Contract Required Quantitation Limit (CRQL) (Continued)

	Parameter	CAS Number	Contract Required Quantitation Level	
			Low Level Water (µg/L)	Low/Medium Level Solids (µg/Kg)
F.	Appendix IX Substances (Continued)	--	--	--
Semivolatiles (Continued)				
175.	o-Toluidine	95-53-4	10	330
176.	1,2,4-Trichlorobenzene	120-82-1	10	330
177.	2,4,5-Trichlorophenol	95-95-4	10	330
178.	2,4,6-Trichlorophenol	88-06-2	10	330
179.	O,O,O-Triethylphosphoro-thioate	126-68-1	10	330
180.	1,3,5-Trinitrobenzene	99-35-4	10	330
Pesticides/Herbicides/PCBs				
181.	Aldrin	309-00-2	0.05	8.0
182.	AROCLOR-1016	12674-11-2	1.0	33.0
183.	AROCLOR-1221	11104-28-2	1.0	33.0
184.	AROCLOR-1232	11141-16-5	1.0	33.0
185.	AROCLOR-1242	53469-21-9	1.0	33.0
186.	AROCLOR-1248	12672-29-6	1.0	33.0
187.	AROCLOR-1254	11097-69-1	1.0	33.0
188.	AROCLOR-1260	11096-82-5	1.0	33.0
189.	alpha-BHC	319-84-6	0.05	8.0
190.	beta-BHC	319-85-7	0.05	8.0
191.	delta-BHC	319-86-8	0.05	8.0
192.	gamma-BHC (Lindane)	58-89-9	0.05	8.0
193.	2-sec-Butyl-4,6-dinitrophenol (Dinoseb; DNBP)	88-85-7	1.0	160

Resource Conservation and Recovery Act (RCRA) Parameters
RCRA Target Compound List (TCL) and
Contract Required Quantitation Limit (CRQL) (Continued)

	Parameter	CAS Number	Contract Required Quantitation Level	
			Low Level Water (µg/L)	Low/Medium Level Solids (µg/Kg)
F.	Appendix IX Substances (Continued)	--	--	--
Pesticides/Herbicides/PCBs (Continued)				
194.	Chlordane (Total)	57-74-9	0.5	80
195.	2,4-Dichlorophenoxyacetic acid (2,4-D)	94-75-7	10	800
196.	4,4'-DDD	72-54-8	0.10	16
197.	4,4'-DDE	72-55-9	0.10	16
198.	4,4'-DDT	50-29-3	0.10	16
199.	Dieldrin	60-57-1	0.10	16
200.	Disulfoton	298-04-4	2.0	320
201.	Endosulfan I	959-98-8	0.10	16
202.	Endosulfan II	33213-65-9	0.10	16
203.	Endosulfan sulfate	1031-07-8	0.10	16
204.	Endrin	72-20-8	0.10	16
205.	Endrin aldehyde	7421-93-4	0.20	32
206.	Heptachlor	76-44-8	0.05	8.0
207.	Heptachlor epoxide	1024-57-3	0.05	8.0
208.	Methoxychlor	72-43-5	0.5	80
209.	Methyl parathion	298-00-0	0.5	80
210.	Phorate	298-02-2	2.0	320
211.	(2,4,5-Trichlorophenoxy) propanoic acid (2,4,5-TP; Silvex)	93-72-1	2.0	320
212.	2,4,5-Trichlorophenoxyacetic acid (2,4,5-T)	93-76-5	2.0	320

Resource Conservation and Recovery Act (RCRA) Parameters
RCRA Target Compound List (TCL) and
Contract Required Quantitation Limit (CRQL) (Continued)

	Parameter	CAS Number	Contract Required Quantitation Level	
			Low Level Water (µg/L)	Low/Medium Level Solids (µg/Kg)
F.	Appendix IX Substances (Continued)	--	--	--
Pesticides/Herbicides/PCBs (Continued)				
213.	Toxaphene	8001-35-2	1.0	160
Inorganics				
214.	Antimony	7440-36-0	60	6000
215.	Arsenic	7440-38-2	10	1000
216.	Barium	7440-39-3	200	20000
217.	Beryllium	7440-41-7	5.0	500
218.	Cadmium	7440-43-9	5.0	500
219.	Chromium	7440-47-3	10	1000
220.	Cobalt	7440-48-4	50	5000
221.	Copper	7440-50-8	25	2500
222.	Cyanide	57-12-5	40	4000
223.	Lead	7439-92-1	5.0	500
224.	Mercury	7439-97-6	0.2	20
225.	Nickel	7440-02-0	40	4000
226.	Selenium	7782-49-2	5.0	500
227.	Silver	7440-22-4	10	1000
228.	Sulfide	18496-25-8	10000	--
229.	Thallium	7440-28-0	10	1000
230.	Tin	7440-31-5	40	4000
231.	Vanadium	7440-62-2	50	5000
232.	Zinc	7440-66-6	20	2000

Resource Conservation and Recovery Act (RCRA) Parameters
RCRA Target Compound List (TCL) and
Contract Required Quantitation Limit (CRQL) (Continued)

	Parameter	CAS Number	Contract Required Quantitation Level	
			Low Level Water (µg/L)	Low/Medium Level Solids (µg/Kg)
G.	Volatile Organics (Method 8240)	--	--	--
1.	Acetone	67-64-1	10	10
2.	Benzene	71-43-2	10	10
3.	Bromodichloromethane	75-27-4	10	10
4.	Bromoform	75-25-2	10	10
5.	Bromomethane	74-83-9	10	10
6.	2-Butanone (Methyl ethyl ketone)	78-93-3	10	10
7.	Carbon disulfide	75-15-0	10	10
8.	Carbon tetrachloride	56-23-5	10	10
9.	Chlorobenzene	108-90-7	10	10
10.	Chloroethane	75-00-3	10	10
11.	2-Chloroethyl vinyl ether	110-75-8	10	10
12.	Chloroform	67-66-3	10	10
13.	Chloromethane	74-87-3	10	10
14.	Dibromochloromethane	124-48-1	10	10
15.	1,2-Dichlorobenzene	95-50-1	10	10
16.	1,3-Dichlorobenzene	541-73-1	10	10
17.	1,4-Dichlorobenzene	106-46-7	10	10
18.	1,1-Dichloroethane	75-34-3	10	10
19.	1,2-Dichloroethane	107-06-2	10	10
20.	1,1-Dichloroethene	75-35-4	10	10
21.	1,2-Dichloroethene (Total)	540-59-0	10	10
22.	Dichloromethane	75-09-2	10	10

Resource Conservation and Recovery Act (RCRA) Parameters
RCRA Target Compound List (TCL) and
Contract Required Quantitation Limit (CRQL) (Continued)

	Parameter	CAS Number	Contract Required Quantitation Level	
			Low Level Water (µg/L)	Low/Medium Level Solids (µg/Kg)
G.	Volatile Organics (Method 8240) (Continued)	--	--	--
23.	1,2-Dichloropropane	78-87-5	10	10
24.	cis-1,3-Dichloropropene	10061-01-5	10	10
25.	trans-1,3-Dichloropropene	10061-02-6	10	10
26.	Ethylbenzene	100-41-4	10	10
27.	2-Hexanone	591-78-6	10	10
28.	4-Methyl-2-pentanone (Methyl iso-butyl ketone)	108-10-1	10	10
29.	Styrene	100-42-5	10	10
30.	1,1,2,2-Tetrachloroethane	79-34-5	10	10
31.	Tetrachloroethylene	127-18-4	10	10
32.	Toluene	108-88-3	10	10
33.	1,1,1-Trichloroethane	71-55-6	10	10
34.	1,1,2-Trichloroethane	79-00-5	10	10
35.	Trichloroethene	79-01-6	10	10
36.	Vinyl acetate	108-05-4	10	10
37.	Vinyl chloride	75-01-4	10	10
38.	Xylenes (Total)	1330-20-7	10	10
Method 8240 Supplemental Compound List				
1.	Acetonitrile	75-05-8	100	100
2.	Acrolein	107-02-8	100	100
3.	Acrylonitrile	107-13-1	100	100

Resource Conservation and Recovery Act (RCRA) Parameters
RCRA Target Compound List (TCL) and
Contract Required Quantitation Limit (CRQL) (Continued)

	Parameter	CAS Number	Contract Required Quantitation Level	
			Low Level Water (µg/L)	Low/Medium Level Solids (µg/Kg)
G.	Volatile Organics (Method 8240) (Continued)	--	--	--
Method 8240 Supplemental Compound List (Continued)				
4.	Allyl alcohol	107-18-6	100	100
5.	Allyl chloride (3-Chloropropene)	107-05-1	10	10
6.	Benzyl chloride	100-44-7	100	100
7.	Bromoactone	598-31-2	100	100
8.	Bromochlormethane	74-97-5	10	10
9.	2-Chloroethanol	107-07-3	100	100
10.	Cloroprene (2-Chloro-1,3-butadiene)	126-99-8	10	10
11.	3-Chloropropionitrile	542-76-7	100	100
12.	1,2-Dibromo-3-chloropropane	96-12-8	100	100
13.	1,2-Dibromoethane	106-93-4	10	10
14.	Dibromomethane	74-95-3	10	10
15.	1,4-Dichloro-2-butene	110-57-6	100	100
16.	Dichlorodifluoromethane	75-71-8	10	10
17.	1,3-Dichloro-2-propanol	96-23-1	100	100
18.	1,2,3,4-Diepoxybutane	1464-53-5	100	100
19.	1,4-Dioxane	123-91-1	100	100
20.	Epichlorohydrin	106-89-8	100	100
21.	Ethanol	64-17-5	100	100
22.	Ethylene oxide	75-21-8	100	100

Resource Conservation and Recovery Act (RCRA) Parameters
RCRA Target Compound List (TCL) and
Contract Required Quantitation Limit (CRQL) (Continued)

	Parameter	CAS Number	Contract Required Quantitation Level	
			Low Level Water (µg/L)	Low/Medium Level Solids (µg/Kg)
G.	Volatile Organics (Method 8240) (Continued)	--	--	--
Method 8240 Supplemental Compound List (Continued)				
23.	Ethylmethacrylate	97-63-2	10	10
24.	2-Hydroxypropionitrile	591-78-6	100	100
25.	Iodomethane (Methyl iodide)	74-88-4	10	10
26.	Isobutyl alcohol (2-Methyl-1-propanol)	78-83-1	100	100
27.	Malononitrile	126-98-7	100	100
28.	Methacrylonitrile	126-98-7	100	100
29.	Methylmethacrylate	80-62-6	10	10
30.	Pentachloroethane	76-01-7	10	10
31.	2-Picoline	109-06-8	100	100
32.	Propargyl alcohol	107-19-7	100	100
33.	ß-Propiolacetone	57-57-8	100	100
34.	Propionitrile	107-12-0	100	100
35.	n-Propylamine	107-10-8	100	100
36.	Pyridine	110-86-1	100	100
37.	1,1,1,2-Tetrachloroethane	630-20-6	10	10
38.	Trichlorofluoromethane	75-69-4	10	10
39.	1,2,3-Trichloropropane	96-18-4	10	10
H.	Low Concentration Volatile Organics (Method 8260)	--	--	--
1.	Acetone	67-64-1	5	10
2.	Acrylonitrile	107-13-1	10	100

Resource Conservation and Recovery Act (RCRA) Parameters
RCRA Target Compound List (TCL) and
Contract Required Quantitation Limit (CRQL) (Continued)

	Parameter	CAS Number	Contract Required Quantitation Level	
			Low Level Water (µg/L)	Low/Medium Level Solids (µg/Kg)
H.	Low Concentration Volatile Organics (Method 8260) (Continued)	--	--	--
3.	Benzene	71-43-2	1	10
4.	Bromochlormethane	74-97-5	1	10
5.	Bromodichloromethane	75-27-4	1	10
6.	Bromoform	75-25-2	1	10
7.	Bromomethane (Methyl bromide)	74-83-9	1	10
8.	2-Butanone (Methyl ethyl ketone)	78-93-3	5	10
9.	Carbon disulfide	75-15-0	1	10
10.	Carbon tetrachloride	56-23-5	1	10
11.	Chlorobenzene	108-90-7	1	10
12.	Chloroethane	75-00-3	1	10
13.	2-Chloroethyl vinyl ether	110-75-8	1	10
14.	Chloroform	67-66-3	1	10
15.	Chloromethane (Methyl chloride)	74-87-3	1	10
16.	Dibromochloromethane	124-48-1	1	10
17.	1,2-Dibromo-3-chloropropane	96-12-8	1	100
18.	1,2-Dibromoethane	106-93-4	1	10
19.	1,2-Dichlorobenzene	95-50-1	1	10
20.	1,3-Dichlorobenzene	541-73-1	1	10
21.	1,4-Dichlorobenzene	106-46-7	1	10

Resource Conservation and Recovery Act (RCRA) Parameters
RCRA Target Compound List (TCL) and
Contract Required Quantitation Limit (CRQL) (Continued)

	Parameter	CAS Number	Contract Required Quantitation Level	
			Low Level Water (µg/L)	Low/Medium Level Solids (µg/Kg)
H.	Low Concentration Volatile Organics (Method 8260) (Continued)	--	--	--
22.	trans-1,4-Dichloro-2-butene	110-57-6	1	100
23.	1,1-Dichloroethane	75-34-3	1	10
24.	1,2-Dichloroethane	107-06-2	1	10
25.	1,1-Dichloroethene	75-35-4	1	10
26.	cis-1,2-Dichloroethene	156-59-2	1	10
27.	trans-1,2-Dichloroethene	156-60-5	1	10
28.	Dichloromethane (Methylene chloride)	75-09-2	2	10
29.	1,2-Dichloropropane	78-87-5	1	10
30.	cis-1,3-Dichloropropene	10061-01-5	1	10
31.	trans-1,3-Dichloropropene	10061-02-6	1	10
32.	Ethylbenzene	100-41-4	1	10
33.	2-Hexanone	591-78-6	5	10
34.	Iodomethane (Methyl iodide)	74-88-4	1	10
35.	4-Methyl-2-pentanone (Methyl iso-butyl ketone)	108-10-1	5	10
36.	Styrene	100-42-5	1	10
37.	1,1,1,2-Tetrachloroethane	630-20-6	1	10
38.	1,1,2,2-Tetrachloroethane	79-34-5	1	10
39.	Tetrachloroethene	127-18-4	1	10
40.	Toluene	108-88-3	1	10

Resource Conservation and Recovery Act (RCRA) Parameters
RCRA Target Compound List (TCL) and
Contract Required Quantitation Limit (CRQL) (Continued)

	Parameter	CAS Number	Contract Required Quantitation Level	
			Low Level Water (µg/L)	Low/Medium Level Solids (µg/Kg)
H.	Low Concentration Volatile Organics (Method 8260) (Continued)	--	--	--
41.	1,1,1-Trichloroethane	71-55-6	1	10
42.	1,1,2-Trichloroethane	79-00-5	1	10
43.	Trichloroethene	79-01-6	1	10
44.	Trichlorofluoromethane	75-69-4	1	10
45.	1,2,3-Trichloropropane	96-18-4	1	10
46.	Vinyl acetate	108-05-4	1	10
47.	Vinyl chloride	75-01-4	1	10
48.	Xylenes (Total)	1330-20-7	1	10
Method 8260 Supplemental Compound List				
1.	Acetonitrile	75-05-8	100	100
2.	Acrolein	107-02-8	100	100
3.	Allyl chloride (3-Chloro-propene)	107-05-1	10	10
4.	Bromobenzene	108-86-1	1	10
5.	n-Butylbenzene	104-51-8	1	10
6.	sec-Butylbenzene	135-98-8	1	10
7.	tert-Butylbenzene	98-06-6	1	10
8.	Cloroprene (2-Chloro-1,3-butadiene)	126-99-8	10	10
9.	2-Chlorotoluene	95-49-8	1	10
10.	4-Chlorotoluene	106-43-4	1	10

Resource Conservation and Recovery Act (RCRA) Parameters
RCRA Target Compound List (TCL) and
Contract Required Quantitation Limit (CRQL) (Continued)

	Parameter	CAS Number	Contract Required Quantitation Level	
			Low Level Water (µg/L)	Low/Medium Level Solids (µg/Kg)
H.	Low Concentration Volatile Organics (Method 8260) (Continued)	--	--	--
Method 8260 Supplemental Compound List (Continued)				
11.	Dibromomethane	74-95-3	1	10
12.	Dichlorodifluoromethane	75-71-8	1	10
13.	1,3-Dichloropropane	142-28-9	1	10
14.	2,2-Dichloropropane	594-20-7	1	10
15.	1,1-Dichloropropene	563-58-6	1	10
16.	Ethylmethacrylate	97-63-2	10	10
17.	Hexachlorobutadiene	87-68-3	1	10
18.	Hexachloroethane	67-72-1	1	10
19.	Isobutyl alcohol (2-Methyl-1-propanol)	78-83-1	100	100
20.	Isodrin	465-73-6	10	10
21.	Isopropylbenzene	98-82-8	1	10
22.	p-Isopropyltoluene	99-87-6	1	10
23.	Methacrylonitrile	126-98-7	100	100
24.	Methylmethacrylate	80-62-6	10	10
25.	Naphthalene	91-20-3	1	10
26.	Propionitrile	107-12-0	100	100
27.	n-Propylbenzene	103-65-1	1	10
28.	1,2,3-Trichlorobenzene	87-61-6	1	10
29.	1,2,4-Trichlorobenzene	120-82-1	1	10

Resource Conservation and Recovery Act (RCRA) Parameters
RCRA Target Compound List (TCL) and
Contract Required Quantitation Limit (CRQL) (Continued)

	Parameter	CAS Number	Contract Required Quantitation Level	
			Low Level Water (µg/L)	Low/Medium Level Solids (µg/Kg)
H.	Low Concentration Volatile Organics (Method 8260) (Continued)	--	--	--
Method 8260 Supplemental Compound List (Continued)				
30.	1,2,4-Trimethylbenzene	95-63-6	1	10
31.	1,3,5-Trimethylbenzene	108-67-8	1	10
I.	Semivolatile Organics (Method 8270)	--	--	--
1.	Acenaphthene	83-32-9	10	330
2.	Acenaphthylene	208-96-8	10	330
3.	Anthracene	120-12-7	10	330
4.	Benz[a]anthracene	56-55-3	10	330
5.	Benzo[b]fluoranthene	205-99-2	10	330
6.	Benzo[k]fluoranthene	207-08-9	10	330
7.	Benzo[g.h,i]perylene	191-24-2	10	330
8.	Benzo[a]pyrene	50-32-8	10	330
9.	Benzyl alcohol	100-51-6	10	330
10.	Benzyl butyl phthalate	85-68-7	10	330
11.	Bis(2-chloroethoxy)methane	111-91-1	10	330
12.	Bis(2-chloroethyl)ether	111-44-4	10	330
13.	Bis(2-ethylhexyl)phthalate	117-81-7	10	330
14.	4-Bromophenyl phenyl ether	101-55-3	10	330
15.	4-Chloroaniline	106-47-8	10	330
16.	4-Chloro-3-methylphenol (p-Chloro-m-cresol)	59-50-7	10	330

Resource Conservation and Recovery Act (RCRA) Parameters
RCRA Target Compound List (TCL) and
Contract Required Quantitation Limit (CRQL) (Continued)

	Parameter	CAS Number	Contract Required Quantitation Level	
			Low Level Water (µg/L)	Low/Medium Level Solids (µg/Kg)
I.	Semivolatile Organics (Method 8270) (Continued)	--	--	--
17.	2-Chloronaphthalene	91-58-7	10	330
18.	2-Chlorophenol	95-57-8	10	330
19.	4-Chlorophenyl phenyl ether	7005-72-3	10	330
20.	Chrysene	218-01-9	10	330
21.	Dibenz[a,h]anthracene	53-70-3	10	330
22.	Dibenzofuran	132-64-9	10	330
23.	Di-n-butyl phthalate	84-74-2	10	330
24.	1,2-Dichlorobenzene	95-50-1	10	330
25.	1,3-Dichlorobenzene	541-73-1	10	330
26.	1,4-Dichlorobenzene	106-46-7	10	330
27.	3,3'-Dichlorobenzidine	91-94-1	20	660
28.	2,4-Dichlorophenol	120-83-2	10	330
29.	Diethyl phthlate	84-66-2	10	330
30.	2,4-Dimethylphenol	105-67-9	10	330
31.	Dimethyl phthlate	131-11-3	10	330
32.	4,6-Dinitro-2-methylphenol	534-52-1	25	800
33.	2,4-Dinitrophenol	51-28-5	25	800
34.	2,4-Dinitrotoluene	121-14-2	10	330
35.	2,6-Dinitrotoluene	606-20-2	10	330
36.	Di-n-octylphthalate	117-84-0	10	330
37.	Fluoranthene	206-44-0	10	330
38.	Fluorene	86-73-7	10	330

Resource Conservation and Recovery Act (RCRA) Parameters
RCRA Target Compound List (TCL) and
Contract Required Quantitation Limit (CRQL) (Continued)

	Parameter	CAS Number	Contract Required Quantitation Level	
			Low Level Water (µg/L)	Low/Medium Level Solids (µg/Kg)
I.	Semivolatile Organics (Method 8270) (Continued)	--	--	--
39.	Hexachlorobenzene	118-74-1	10	330
40.	Hexachlorobutadiene	87-68-3	10	330
41.	Hexachlorocyclopentadiene	77-47-4	10	330
42.	Hexachloroethane	67-72-1	10	330
43.	Indeno[1,2,3-c,d]pyrene	193-39-5	10	330
44.	Isophorone	78-59-1	10	330
45.	2-Methylnaphthalene	91-57-6	10	330
46.	2-Methylphenol (o-Cresol)	95-48-7	10	330
47.	4-Methylphenol (p-Cresol)	106-44-5	10	330
48.	Naphthalene	91-20-3	10	330
49.	2-Nitroaniline	88-74-4	25	800
50.	3-Nitroaniline	99-09-2	25	800
51.	4-Nitroaniline	100-01-6	25	800
52.	Nitrobenzene	98-95-3	10	330
53.	2-Nitrophenol	88-75-5	10	330
54.	4-Nitrophenol	100-02-7	25	800
55.	N-Nitrosodimethylamine	62-75-9	10	330
56.	N-Nitrosodiphenylamine	86-30-6	10	330
57.	N-Nitrosodi-n-propylamine	621-24-7	10	330
58.	2,2'-oxybis(1-Chloropropane) (Bis(2-chloroisopropyl)ether)	108-60-1	10	330
59.	Pentachlorophenol	87-86-5	25	800

Resource Conservation and Recovery Act (RCRA) Parameters
RCRA Target Compound List (TCL) and
Contract Required Quantitation Limit (CRQL) (Continued)

	Parameter	CAS Number	Contract Required Quantitation Level	
			Low Level Water (µg/L)	Low/Medium Level Solids (µg/Kg)
I.	Semivolatile Organics (Method 8270) (Continued)	--	--	--
60.	Phenanthrene	85-01-8	10	330
61.	Phenol	108-95-2	10	330
62.	Pyrene	129-00-0	10	330
63.	1,2,4-Trichlorobenzene	120-82-1	10	330
64.	2,4,5-Trichlorophenol	95-95-4	10	330
65	2,4,6-Trichlorophenol	88-06-2	10	330
Method 8270 Supplemental Compound List				
1.	Acetophenone	98-86-2	10	330
2.	2-Acetylaminofluorene (2-AAF)	53-96-3	20	660
3.	Acetyl-2-thiourea	591-08-2	1000	33000
4.	2-Aminoanthraquinone	117-79-3	20	660
5.	Aminoazobenzene	60-09-3	10	330
6.	4-Aminobiphenyl	92-67-1	20	660
7.	Anilazine	101-05-3	100	3300
8.	Aniline	62-53-3	10	330
9.	o-Anisidine	90-04-0	10	330
10.	Aramite	140-57-8	20	660
11.	Azinphos methyl	86-50-0	100	3300
12.	Barban	101-27-9	200	6600
13.	Benzidine	92-87-5	1000	33000
14.	Benzoic Acid	65-85-0	50	1700

Resource Conservation and Recovery Act (RCRA) Parameters
RCRA Target Compound List (TCL) and
Contract Required Quantitation Limit (CRQL) (Continued)

	Parameter	CAS Number	Contract Required Quantitation Level	
			Low Level Water (µg/L)	Low/Medium Level Solids (µg/Kg)
I.	Semivolatile Organics (Method 8270) (Continued)	--	--	--
Method 8270 Supplemental Compound List (Continued)				
15.	p-Benzoquinone	106-51-4	10	330
16.	Bromoxynil	1689-84-5	10	330
17.	Captafol	2425-06-1	20	660
18.	Captan	133-06-2	50	1700
19.	Carbaryl	63-25-2	10	330
20.	Carbazole	86-74-8	10	330
21.	Carbofuran	1563-66-2	10	330
22.	Carbophenothion	786-19-6	10	330
23.	Chlorfenvinphos	470-90-6	20	660
24.	Chlorobenzilate	510-15-6	10	330
25.	5-Chloro-2-methylaniline	95-79-4	10	330
26.	3-(Chloromethyl)pyridine hydrochloride	6959-48-4	100	3300
27.	1-Chloronaphthalene	90-13-1	10	330
28.	Coumaphos	56-72-4	40	1300
29.	p-Cresidine	120-71-8	10	330
30.	Crotoxyphos	7700-17-6	20	660
31.	2-Cyclohexyl-4,6-dinitrophenol	131-89-5	100	3300
32.	Demeton-O	298-03-3	10	330
33.	Demeton-S	126-75-0	10	330
34.	Diallate	2303-16-4	10	330

Resource Conservation and Recovery Act (RCRA) Parameters
RCRA Target Compound List (TCL) and
Contract Required Quantitation Limit (CRQL) (Continued)

	Parameter	CAS Number	Contract Required Quantitation Level	
			Low Level Water (µg/L)	Low/Medium Level Solids (µg/Kg)
I.	Semivolatile Organics (Method 8270) (Continued)	--	--	--
Method 8270 Supplemental Compound List (Continued)				
35.	2,4-Diaminotoluene	95-80-7	20	660
36.	Dibenz[a,j]acridine	224-42-0	10	330
37.	Dibenzo[a,e]pyrene	192-65-4	10	330
38.	Dichlone	117-80-6	100	3300
39.	2,6-Dichlorophenol	87-65-0	10	330
40.	Dichlorovos	62-73-7	10	330
41.	Dicrotophos	141-66-2	10	330
42.	Diethylstilbestrol	56-53-1	20	660
43.	O,O-Diethyl-0-2-pyrazinyl-phosphorothioate	297-97-2	10	330
44.	Diethyl sulfate	64-67-5	100	3300
45.	Dimethoate	60-51-5	10	330
46.	3,3'-Dimethoxybenzidine	119-90-4	100	3300
47.	p-(Dimethylamino)-azobenzene	60-11-7	10	330
48.	7,12-Dimethylbenz[a]-anthracene	57-97-6	10	330
49.	3,3'-Dimethylbenzidine	119-93-7	10	330
50.	a,a-Dimethylphenethylamine	122-09-8	10	330
51.	1,2-Dinotrobenzene	528-29-0	40	1300
52.	1,3-Dinitrobenzene	99-65-0	20	660
53.	1,4-Dintrobenzene	100-25-4	40	1300

Resource Conservation and Recovery Act (RCRA) Parameters
RCRA Target Compound List (TCL) and
Contract Required Quantitation Limit (CRQL) (Continued)

	Parameter	CAS Number	Contract Required Quantitation Level	
			Low Level Water (µg/L)	Low/Medium Level Solids (µg/Kg)
I.	Semivolatile Organics (Method 8270) (Continued)	--	--	--
Method 8270 Supplemental Compound List (Continued)				
54.	Dinocap	39300-45-3	100	3300
55.	Dinoseb	88-85-7	20	660
56.	Diphenylamine	122-39-4	10	330
57.	5,5-Diphenylhydantoin	57-41-0	20	660
58.	1,2-Diphenylhydrazine	122-66-7	100	3300
59.	Disulfoton	298-04-4	10	330
60.	Ethion	563-12-2	10	330
61.	Ethoxy-4-nitrophenoxypheny-phosphine sulfide (EPN)	2104-64-5	10	330
62.	Ethyl carbamate	51-79-6	50	1700
63.	Ethyl methanesulfonate	62-50-0	20	660
64.	Famphur	52-85-7	20	660
65.	Fensulfothion	115-90-2	40	1300
66.	Fenthion	55-38-9	10	330
67.	Fluchloralin	33245-39-5	20	660
68.	Hexachlorophene	70-30-4	50	1700
69.	Hexachloropropene	1888-71-7	10	330
70.	Hexamethylphosphoramide	680-31-9	20	660
71.	Hydroquinone	123-31-9	100	3300
72.	Isodrin	465-73-6	20	660
73.	Isosafrole	120-58-1	10	330
74.	Kepone	143-50-0	20	660

Resource Conservation and Recovery Act (RCRA) Parameters
RCRA Target Compound List (TCL) and
Contract Required Quantitation Limit (CRQL) (Continued)

	Parameter	CAS Number	Contract Required Quantitation Level	
			Low Level Water (µg/L)	Low/Medium Level Solids (µg/Kg)
I.	Semivolatile Organics (Method 8270) (Continued)	--	--	--
Method 8270 Supplemental Compound List (Continued)				
75.	Leptophos	21609-90-5	10	330
76.	Malathion	121-75-5	50	1700
77.	Maleic anhydride	108-31-6	100	3300
78.	Mestranol	72-33-3	20	660
79.	Methapyrilene	91-80-5	100	3300
80.	3-Methylcholanthrene	56-49-5	10	330
81.	4,4'-Methylenebis(2-chloro-aniline)	101-14-4	100	3300
82.	Methyl methanesulfonate	66-27-3	10	330
83.	Methyl parathion	298-00-0	10	330
84.	3-Methylphenol (m-Cresol)	108-39-4	10	330
85.	Mevinphos	7786-34-7	10	330
86.	Mexacarbate	315-18-4	20	660
87.	Monocrotophos	6923-22-4	40	1300
88.	Naled	300-76-5	20	660
89.	1,4-Naphthoquinone	130-15-4	10	330
90.	1-Naphthylamine	134-32-7	10	330
91.	2-Naphthylamine	91-59-8	10	330
92.	Nicotine	54-11-5	20	660
93.	5-Nitroacenaphthene	602-87-9	10	330
94.	5-Nitro-o-anisidine	99-59-2	10	330

Resource Conservation and Recovery Act (RCRA) Parameters
RCRA Target Compound List (TCL) and
Contract Required Quantitation Limit (CRQL) (Continued)

	Parameter	CAS Number	Contract Required Quantitation Level	
			Low Level Water (µg/L)	Low/Medium Level Solids (µg/Kg)
I.	Semivolatile Organics (Method 8270) (Continued)	--	--	--
Method 8270 Supplemental Compound List (Continued)				
95.	4-Nitrobiphenyl	92-93-3	10	330
96.	Nitrofen	1836-75-5	20	660
97.	4-Nitroquinoline-1-oxide	56-57-5	40	1300
98.	N-Nitrosodi-n-butylamine	924-16-3	10	330
99.	N-Nitrosodiethylamine	55-18-5	20	660
100.	N-Nitrosomethylethylamine	10595-95-6	10	330
101.	N-Nitrosomorpholine	59-89-2	10	330
102.	N-Nitrosopiperidine	100-75-4	20	660
103.	N-Nitrosopyrrolidine	930-55-2	40	1300
104.	5-Nitro-o-toluidine	99-55-8	10	330
105.	Octamethyl pyrophosphoramide	152-16-9	200	6600
106.	4,4'-Oxydianiline	101-80-4	20	660
107.	Parathion	56-38-2	10	330
108.	Pentachlorobenzene	608-93-5	10	330
109.	Pentachloronitrobenzene	82-68-8	20	660
110.	Phenacetin	62-44-2	20	660
111.	Phenobarbital	50-06-6	10	330
112.	p-Phenylenediamine	106-50-3	10	330
113.	Phorate	298-02-2	10	330
114.	Phosalone	2310-17-0	100	3300

Resource Conservation and Recovery Act (RCRA) Parameters
RCRA Target Compound List (TCL) and
Contract Required Quantitation Limit (CRQL) (Continued)

	Parameter	CAS Number	Contract Required Quantitation Level	
			Low Level Water (µg/L)	Low/Medium Level Solids (µg/Kg)
I.	Semivolatile Organics (Method 8270) (Continued)	--	--	--
Method 8270 Supplemental Compound List (Continued)				
115.	Phosmet	732-11-6	40	1300
116.	Phosphamidon	13171-21-6	100	3300
117.	Phthalic anhydride	85-44-9	100	3300
118.	2-Picoline	109-06-8	100	3300
119.	Piperonyl sulfoxide	120-62-7	100	3300
120.	Pronamide	23950-58-5	10	330
121.	Propylthiouracil	51-52-5	100	3300
122.	Pyridine	110-86-1	100	3300
123.	Resorcinol	108-46-3	100	3300
124.	Safrole	94-59-7	10	330
125.	Strychnine	60-41-3	40	1300
126.	Sulfallate	95-06-7	10	330
127.	Terbufos	13071-79-9	20	660
128.	1,2,4,5-Tetrachlorobenzene	95-94-3	10	330
129.	2,3,4,6-Tetrachlorophenol	58-90-2	10	330
130.	Tetrachlorvinphos (Stirophos)	961-11-5	20	660
131.	Tetraethyldithiopyrophosphate	3689-24-5	10	330
132.	Tetraethyl pyrophosphate	107-49-3	40	1300
133.	Thionazine	297-97-2	20	660
134.	Thiophenol (Benzenethiol)	108-98-5	20	660

Resource Conservation and Recovery Act (RCRA) Parameters
RCRA Target Compound List (TCL) and
Contract Required Quantitation Limit (CRQL) (Continued)

	Parameter	CAS Number	Contract Required Quantitation Level	
			Low Level Water (µg/L)	Low/Medium Level Solids (µg/Kg)
I.	Semivolatile Organics (Method 8270) (Continued)	--	--	--
135.	Toluene diisocyanate	584-84-9	100	3300
136.	o-Toluidine	95-53-4	10	330
137.	Trifluralin	1582-09-8	10	330
138.	2,4,5-Trimethylaniline	137-17-7	10	330
139.	Trimethyl phosphate	512-56-1	10	330
140.	O,O,O-Triethylphosphorothioate	126-68-1	10	330
141.	1,2,3-Trinitrobenzene	99-35-4	10	330
142.	Tris(2,3-dibromopropyl)-phosphate	126-72-7	200	6600
143.	Tri-p-tolyl phosphate	78-32-0	10	330
J.	Pesticides/Aroclors (Method 8080)	--	--	--
1.	Aldrin	309-00-2	0.05	8.0
2.	AROCLOR-1016	12674-11-2	0.5	80
3.	AROCLOR-1221	11104-28-2	0.5	80
4.	AROCLOR-1232	11141-16-5	0.5	80
5.	AROCLOR-1242	53469-21-9	0.5	80
6.	AROCLOR-1248	12672-29-6	0.5	80
7.	AROCLOR-1254	11097-69-1	1.0	160
8.	AROCLOR-1260	11096-82-5	1.0	160
9.	alpha-BHC	319-84-6	0.05	8.0

Resource Conservation and Recovery Act (RCRA) Parameters
RCRA Target Compound List (TCL) and
Contract Required Quantitation Limit (CRQL) (Continued)

	Parameter	CAS Number	Contract Required Quantitation Level	
			Low Level Water (µg/L)	Low/Medium Level Solids (µg/Kg)
J.	Pesticides/Aroclors (Method 8080) (Continued)	--	--	--
10.	beta-BHC	319-85-7	0.05	8.0
11.	delta-BHC	319-86-8	0.05	8.0
12.	gamma-BHC (Lindane)	58-89-9	0.05	8.0
13.	Chlordane (Total)	57-74-9	0.5	80
14.	4,4'-DDD	72-54-8	0.10	16
15.	4,4'-DDE	72-55-9	0.10	16
16.	4,4'-DDT	50-29-3	0.10	16
17.	Dieldrin	60-57-1	0.10	16
18.	Endosulfan I	959-98-8	0.10	16
19.	Endosulfan II	33213-65-9	0.10	16
20.	Endosulfan sulfate	1031-07-8	0.10	16
21.	Endrin	72-20-8	0.10	16
22.	Endrin aldehyde	7421-93-4	0.20	32
23.	Heptachlor	76-44-8	0.05	8.0
24.	Heptachlor epoxide	1024-57-3	0.05	8.0
25.	Methoxychlor	72-43-5	0.5	8.0
26.	Toxaphene	8001-35-2	1.0	160

PART IV – NYSDEC DIVISION OF SOLID AND HAZARDOUS MATERIALS 6 NYCRR PART 360 PARAMETERS

6 NYCRR Part 360 Parameters
Target Compound List (TCL) and
Contract Required Quantitation Limit (CRQL)

	Parameter[1]	CAS Number[2]	Contract Required Quantitation Level (µg/L)
Routine Parameters			
Leachate Indicators:			
1.	Total Kjeldahl Nitrogen, as N	7727-37-9	60
2.	Ammonia, as N	7664-41-7	50
3.	Nitrate-Nitrite	14797-55-8 /14797-65-0	100
4.	Chemical Oxygen Demand (COD)	N/A	1000
5.	Biochemical Oxygen Demand (BOD_5)	N/A	2000
6.	Total Organic Carbon	N/A	2000
7.	Total Dissolved Solids (TDS)	N/A	10000
8.	Sulfate	14808-79-8	5000
9.	Total Alkalinity as $CaCO_3$	N/A	6000
10.	Total Phenols	64743-03-9	10
11.	Chloride	16887-00-6	5000
12.	Bromide	24959-67-9	2000
13.	Total Hardness as $CaCO_3$	N/A	20000
Inorganic Parameters:			
1.	Cadmium, Total	7440-43-9	5
2.	Calcium, Total	7440-70-2	40
3.	Iron, Total	7439-89-6	100
4.	Lead, Total	7439-92-1	5
5.	Magnesium, Total	7439-95-4	5
6.	Manganese, Total	7439-96-5	15
7.	Potassium, Total	7440-09-7	40
8.	Sodium, Total	7440-23-5	10

6 NYCRR Part 360 Parameters
Target Compound List (TCL) and
Contract Required Quantitation Limit (CRQL) (Continued)

	Parameter[1]	CAS Number[2]	Contract Required Quantitation Level (µg/L)
Baseline Parameters			
Leachate Indicators:			
1.	Total Kjeldahl Nitrogen, as N	7727-37-9	60
2.	Ammonia, as N	7664-41-7	50
3.	Nitrate-Nitrite	14797-55-8 /14797-65-0	100
4.	Chemical Oxygen Demand (COD)	N/A	1000
5.	Biochemical Oxygen Demand (BOD_5)	N/A	2000
6.	Total Organic Carbon	N/A	2000
7.	Total Dissolved Solids (TDS)	N/A	10000
8.	Sulfate	14808-79-8	5000
9.	Total Alkalinity as $CaCO_3$	N/A	6000
10.	Total Phenols	64743-03-9	10
11.	Chloride	16887-00-6	5000
12.	Bromide	24959-67-9	2000
13.	Total Hardness as $CaCO_3$	N/A	20000
14.	Color	N/A	80
Inorganic Parameters:			
1.	Aluminum, Total	7429-90-5	10
2.	Antimony, Total	7440-36-0	30
3.	Arsenic, Total	7440-38-2	10
4.	Barium, Total	7440-39-3	20
5.	Beryllium, Total	7440-41-7	5
6.	Boron, Total	7440-42-8	20
7.	Cadmium, Total	7440-43-9	5
8.	Calcium, Total	7440-70-2	40

6 NYCRR Part 360 Parameters
Target Compound List (TCL) and
Contract Required Quantitation Limit (CRQL) (Continued)

	Parameter[1]	CAS Number[2]	Contract Required Quantitation Level (µg/L)
Baseline Parameters (Continued)			
Inorganic Parameters (Continued):			
9.	Chromium, Total	7440-47-3	10
10.	Chromium, Hexavalent[3]	18540-29-9	30
11.	Cobalt, Total	7440-48-4	10
12.	Copper, Total	7440-50-8	10
13.	Cyanide, Total	57-12-5	10
14.	Iron, Total	7439-89-6	100
15.	Lead, Total	7439-92-1	5
16.	Magnesium, Total	7439-95-4	5
17.	Manganese, Total	7439-96-5	15
18.	Mercury, Total	7439-97-6	0.2
19.	Nickel, Total	7440-02-0	40
20.	Potassium, Total	7440-09-7	40
21.	Selenium, Total	7782-49-2	5
22.	Silver, Total	7440-22-4	10
23.	Sodium, Total	7440-23-5	10
24.	Thallium, Total	7440-28-0	10
25.	Vanadium, Total	7440-62-2	40
26.	Zinc, Total	7440-66-6	20
Organic Parameters:			
1.	Acetone	67-64-1	5
2.	Acrylonitrile	107-13-1	10
3.	Benzene	71-43-2	1
4.	Bromochlormethane	74-97-5	1

6 NYCRR Part 360 Parameters
Target Compound List (TCL) and
Contract Required Quantitation Limit (CRQL) (Continued)

	Parameter[1]	CAS Number[2]	Contract Required Quantitation Level (µg/L)
Baseline Parameters (Continued)			
Organic Parameters (Continued):			
5.	Bromodichloromethane	75-27-4	1
6.	Bromoform	75-25-2	1
7.	Bromomethane	74-83-9	1
8.	2-Butanone	78-93-3	5
9.	Carbon disulfide	75-15-0	1
10.	Carbon tetrachloride	56-23-5	1
11.	Chlorobenzene	108-90-7	1
12.	Chloroethane	75-00-3	1
13.	Chloroform	67-66-3	1
14.	Chloromethane	74-87-3	1
15.	Dibromochloromethane	124-48-1	1
16.	1,2-Dibromo-3-chloro-propane	96-12-8	1
17.	1,2-Dibromoethane	106-93-4	1
18.	1,2-Dichlorobenzene	95-50-1	1
19.	1,3-Dichlorobenzene	541-73-1	1
20.	1,4-Dichlorobenzene	106-46-7	1
21.	trans-1,4-Dichloro-2-butene	110-57-6	10
22.	1,1-Dichloroethane	75-34-3	1
23.	1,2-Dichloroethane	107-06-2	1
24.	1,1-Dichloroethene	75-35-4	1
25.	cis-1,2-Dichloroethene	156-59-2	1
26.	trans-1,2-Dichloroethene	156-60-5	1
27.	Dichloromethane (Methylene chloride)	75-09-2	2

6 NYCRR Part 360 Parameters
Target Compound List (TCL) and
Contract Required Quantitation Limit (CRQL) (Continued)

	Parameter[1]	CAS Number[2]	Contract Required Quantitation Level (µg/L)
Baseline Parameters (Continued)			
Organic Parameters (Continued):			
28.	1,2-Dichloropropane	78-87-5	1
29.	cis-1,3-Dichloro-propene	10061-01-5	1
30.	trans-1,3-Dichloro-propene	10061-02-6	1
31.	Ethylbenzene	100-41-4	1
32.	2-Hexanone	591-78-6	5
33.	Iodomethane	74-88-4	1
34.	4-Methyl-2-pentanone	108-10-1	5
35.	Styrene	100-42-5	1
36.	1,1,1,2-Tetrachloroethane	630-20-6	1
37.	1,1,2,2-Tetrachloroethane	79-34-5	1
38.	Tetrachloroethene	127-18-4	1
39.	Toluene	108-88-3	1
40.	1,1,1-Trichloroethane	71-55-6	1
41.	1,1,2-Trichloroethane	79-00-5	1
42.	Trichloroethene	79-01-6	1
43.	Trichlorofluoromethane	75-69-4	1
44.	1,2,3-Trichloropropane	96-18-4	1
45.	Vinyl acetate	108-05-4	1
46.	Vinyl chloride	75-01-4	1
47.	Xylenes (Total)[4]	1330-20-7	1

6 NYCRR Part 360 Parameters
Target Compound List (TCL) and
Contract Required Quantitation Limit (CRQL) (Continued)

	Parameter[1]	CAS Number[2]	Contract Required Quantitation Level (µg/L)
Expanded Parameters			
Leachate Indicators:			
1.	Total Kjeldahl Nitrogen, as N	7727-37-9	60
2.	Ammonia, as N	7664-41-7	50
3.	Nitrate-Nitrite	14797-55-8 /14797-65-0	100
4.	Chemical Oxygen Demand (COD)	N/A	1000
5.	Biochemical Oxygen Demand (BOD_5)	N/A	2000
6.	Total Organic Carbon	N/A	2000
7.	Total Dissolved Solids (TDS)	N/A	10000
8.	Sulfate	14808-79-8	5000
9.	Total Alkalinity as $CaCO_3$	N/A	6000
10.	Total Phenols	64743-03-9	10
11.	Chloride	16887-00-6	5000
12.	Bromide	24959-67-9	2000
13.	Total Hardness as $CaCO_3$	N/A	20000
14.	Color	N/A	80
Inorganic Parameters:			
1.	Aluminum, Total	7429-90-5	10
2.	Antimony, Total	7440-36-0	30
3.	Arsenic, Total	7440-38-2	10
4.	Barium, Total	7440-39-3	20
5.	Beryllium, Total	7440-41-7	5
6.	Boron, Total	7440-42-8	20
7.	Cadmium, Total	7440-43-9	5

6 NYCRR Part 360 Parameters
Target Compound List (TCL) and
Contract Required Quantitation Limit (CRQL) (Continued)

	Parameter[1]	CAS Number[2]	Contract Required Quantitation Level (µg/L)
Expanded Parameters (Continued)			
Inorganic Parameters (Continued):			
8.	Calcium, Total	7440-70-2	40
9.	Chromium, Total	7440-47-3	10
10.	Chromium, Hexavalent[3]	18540-29-9	30
11.	Cobalt, Total	7440-48-4	10
12.	Copper, Total	7440-50-8	10
13.	Cyanide, Total	57-12-5	10
14.	Iron, Total	7439-89-6	100
15.	Lead, Total	7439-92-1	5
16.	Magnesium, Total	7439-95-4	5
17.	Manganese, Total	7439-96-5	15
18.	Mercury, Total	7439-97-6	0.2
19.	Nickel, Total	7440-02-0	40
20.	Potassium, Total	7440-09-7	40
21.	Selenium, Total	7782-49-2	5
22.	Silver, Total	7440-22-4	10
23.	Sodium, Total	7440-23-5	10
24.	Thallium, Total	7440-28-0	10
25.	Vanadium, Total	7440-62-2	40
26.	Zinc, Total	7440-66-6	20
Organic Parameters:			
1.	Acenaphthene	83-32-9	10
2.	Acenaphthylene	208-96-8	10
3.	Acetone	67-64-1	5

6 NYCRR Part 360 Parameters
Target Compound List (TCL) and
Contract Required Quantitation Limit (CRQL) (Continued)

	Parameter[1]	CAS Number[2]	Contract Required Quantitation Level (µg/L)
Expanded Parameters (Continued)			
Organic Parameters (Continued):			
4.	Acetonitrile	75-05-8	100
5.	Acetophenone	98-86-2	10
6.	2-Acetylaminofluorene	53-96-3	10
7.	Acrolein	107-02-8	100
8.	Acrylonitrile	107-13-1	10
9.	Aldrin	309-00-2	0.05
10.	Allyl chloride	107-05-1	10
11.	4-Aminobiphenyl	92-67-1	20
12.	Anthracene	120-12-7	10
13.	Benzene	71-43-2	1
14.	Benzo[a]anthracene	56-55-3	10
15.	Benzo[b]fluoranthene	205-99-2	10
16.	Benzo[k]fluoranthene	207-08-9	10
17.	Benzo[ghi]perylene	191-24-2	10
18.	Benzo[a]pyrene	50-32-8	10
19.	Benzyl alcohol	100-51-6	10
20.	alpha-BHC	319-84-6	0.05
21.	beta-BHC	319-85-7	0.05
22.	delta-BHC	319-86-8	0.05
23.	gamma-BHC	58-89-9	0.05
24.	Bis(2-chloroethoxy)methane	111-91-1	10
25.	Bis(2-chloroethyl) ether	111-44-4	10
26.	Bis-(2-chloro-1-methyl- ethyl) ether	108-60-1	10

6 NYCRR Part 360 Parameters
Target Compound List (TCL) and
Contract Required Quantitation Limit (CRQL) (Continued)

	Parameter[1]	CAS Number[2]	Contract Required Quantitation Level (µg/L)
Expanded Parameters (Continued)			
Organic Parameters (Continued):			
27.	Bis(2-ethylhexyl)phthalate	117-81-7	10
28.	Bromochlormethane	74-97-5	1
29.	Bromodichloromethane	75-27-4	1
30.	Bromoform	75-25-2	1
31.	Bromomethane	74-83-9	1
32.	2-Butanone	78-93-3	5
33.	4-Bromophenyl phenyl ether	101-55-3	10
34.	Butyl benzyl phthalate	85-68-7	10
35.	Carbon disulfide	75-15-0	1
36.	Carbon tetrachloride	56-23-5	1
37.	Chlordane (Total)	See Note 5	0.5
38.	p-Chloroaniline	106-47-8	10
39.	Chlorobenzene	108-90-7	1
40.	Chlorobenzilate	510-15-6	10
41.	p-Chloro-m-cresol	59-50-7	10
42.	Chloroethane	75-00-3	1
43.	Chloroform	67-66-3	1
44.	Chloromethane	74-87-3	1
45.	2-Chloronaphthalene	91-58-7	10
46.	2-Chlorophenol	95-57-8	10
47.	4-Chlorophenyl phenyl ether	7005-72-3	10
48.	Chloroprene	126-99-8	10
49.	Chrysene	218-01-9	10

6 NYCRR Part 360 Parameters
Target Compound List (TCL) and
Contract Required Quantitation Limit (CRQL) (Continued)

	Parameter[1]	CAS Number[2]	Contract Required Quantitation Level (µg/L)
Expanded Parameters (Continued)			
Organic Parameters (Continued):			
50.	m-Cresol	108-39-4	10
51.	o-Cresol	95-48-7	10
52.	p-Cresol	106-44-5	10
53.	2,4-Dichlorophenoxyacetic acid	94-75-7	10
54.	4,4'-DDD	72-54-8	0.10
55.	4,4'-DDE	72-55-9	0.10
56.	4,4'-DDT	50-29-3	0.10
57.	Diallate	2303-16-4	10
58.	Dibenz[a,h]anthracene	53-70-3	10
59.	Dibenzofuran	132-64-9	10
60.	Dibromochloromethane	124-48-1	1
61.	1,2-Dibromo-3-chloro-propane	96-12-8	1
62.	1,2-Dibromoethane	106-93-4	1
63.	Di-n-butyl phthalate	84-74-2	10
64.	1,2-Dichlorobenzene	95-50-1	1
65.	1,3-Dichlorobenzene	541-73-1	1
66.	1,4-Dichlorobenzene	106-46-7	1
67.	3,3'-Dichlorobenzidine	91-94-1	20
68.	trans-1,4-Dichloro-2-butene	110-57-6	10
69.	Dichlorodifluoromethane	75-71-8	10
70.	1,1-Dichloroethane	75-34-3	1
71.	1,2-Dichloroethane	107-06-2	1
72.	1,1-Dichloroethene	75-35-4	1

6 NYCRR Part 360 Parameters
Target Compound List (TCL) and
Contract Required Quantitation Limit (CRQL) (Continued)

	Parameter[1]	CAS Number[2]	Contract Required Quantitation Level (µg/L)
Expanded Parameters (Continued)			
Organic Parameters (Continued):			
73.	cis-1,2-Dichloroethene	156-59-2	1
74.	trans-1,2-Dichloroethene	156-60-5	1
75.	2,4-Dichlorophenol	120-83-2	10
76.	2,6-Dichlorophenol	87-65-0	10
77.	Dichloromethane (Methylene chloride)	75-09-2	2
78.	1,2-Dichloropropane	78-87-5	1
79.	1,3-Dichloropropane	142-28-9	1
80.	2,2-Dichloropropane	594-20-7	1
81.	1,1-Dichloropropene	563-58-6	1
82.	cis-1,3-Dichloro-propene	10061-01-5	1
83.	trans-1,3-Dichloro-propene	10061-02-6	1
84.	Dieldrin	60-57-1	0.10
85.	Diethyl phthalate	84-66-2	10
86.	0,0-Diethyl 0-2-pyrazinyl phosphorothioate	297-97-2	10
87.	Dimethoate	60-51-5	10
88.	p-(Dimethylamino)azo- benzene	60-11-7	10
89.	7,12-Dimethylbenz[a]- anthracene	57-97-6	10
90.	3,3'-Dimethylbenzidine	119-93-7	10
91.	2,4-Dimethylphenol	105-67-9	10
92.	Dimethyl phthalate	131-11-3	10
93.	m-Dinitrobenzene	99-65-0	20
94.	4,6-Dinitro-o-cresol	534-52-1	25

6 NYCRR Part 360 Parameters
Target Compound List (TCL) and
Contract Required Quantitation Limit (CRQL) (Continued)

	Parameter[1]	CAS Number[2]	Contract Required Quantitation Level (µg/L)
Expanded Parameters (Continued)			
Organic Parameters (Continued):			
95.	2,4-Dinitrophenol	51-28-5	25
96.	2,4-Dinitrotoluene	121-14-2	10
97.	2,6-Dinitrotoluene	606-20-2	10
98.	Dinoseb	88-85-7	20
99.	Di-n-octyl phthalate	117-84-0	10
100.	Diphenylamine	122-39-4	10
101.	Disulfoton	298-04-4	10
102.	Endosulfan I	959-98-8	0.10
103.	Endosulfan II	33213-65-9	0.10
104.	Endosulfan sulfate	1031-07-8	0.10
105.	Endrin	72-20-8	0.10
106.	Endrin aldehyde	7421-93-4	0.20
107.	Ethylbenzene	100-41-4	1
108.	Ethyl methacrylate	97-63-2	10
109.	Ethyl methanesulfonate	62-50-0	20
110.	Famphur	52-85-7	20
111.	Fluoranthene	206-44-0	10
112.	Fluorene	86-73-7	10
113.	Heptachlor	76-44-8	0.05
114.	Heptachlor epoxide	1024-57-3	0.05
115.	Hexachlorobenzene	118-74-1	10
116.	Hexachlorobutadiene	87-68-3	10
117.	Hexachlorocyclopentadiene	77-47-4	10

6 NYCRR Part 360 Parameters
Target Compound List (TCL) and
Contract Required Quantitation Limit (CRQL) (Continued)

	Parameter[1]	CAS Number[2]	Contract Required Quantitation Level (µg/L)
Expanded Parameters (Continued)			
Organic Parameters (Continued):			
118.	Hexachloroethane	67-72-1	10
119.	Hexachloropropene	1888-71-7	10
120.	2-Hexanone	591-78-6	5
121.	Iodomethane	74-88-4	1
122.	Indeno(1,2,3-cd)pyrene	193-39-5	10
123.	Isobutyl alcohol	78-83-1	100
124.	Isodrin	465-73-6	20
125.	Isophorone	78-59-1	10
126.	Isosafrole	120-58-1	10
127.	Kepone	143-50-0	20
128.	Methacrylonitrile	126-98-7	100
129.	Methapyrilene	91-80-5	100
130.	Methoxychlor	72-43-5	0.05
131.	3-Methylcholanthrene	56-49-5	10
132.	Methyl methacrylate	80-62-6	10
133.	Methyl methanesulfonate	66-27-3	10
134.	2-Methylnaphthalene	91-57-6	10
135.	Methyl parathion	298-00-0	10
136.	4-Methyl-2-pentanone	108-10-1	5
137.	Naphthalene	91-20-3	10
138.	1,4-Naphthoquinone	130-15-4	10
139.	1-Naphthylamine	134-32-7	10
140.	2-Naphthylamine	91-59-8	10
141.	2-Nitroaniline	88-74-4	25

6 NYCRR Part 360 Parameters
Target Compound List (TCL) and
Contract Required Quantitation Limit (CRQL) (Continued)

	Parameter[1]	CAS Number[2]	Contract Required Quantitation Level (µg/L)
Expanded Parameters (Continued)			
Organic Parameters (Continued):			
142.	3-Nitroaniline	99-09-2	25
143.	4-Nitroaniline	100-01-6	25
144.	Nitrobenzene	98-95-3	10
145.	2-Nitrophenol	88-75-5	10
146.	4-Nitrophenol	100-02-7	25
147.	N-Nitrosodi-n-butylamine	924-16-3	10
148.	N-Nitrosodiethylamine	55-18-5	20
149.	N-Nitrosodimethylamine	62-75-9	10
150.	N-Nitrosodiphenylamine	86-30-6	10
151.	N-Nitrosodipropylamine	621-64-7	10
152.	N-Nitrosomethylethalamine	10595-95-6	10
153.	N-Nitrosopiperidine	100-75-4	20
154.	N-Nitrosopyrrolidine	930-55-2	40
155.	5-Nitro-o-toluidine	99-55-8	10
156.	Parathion	56-38-2	10
157.	Pentachlorobenzene	608-93-5	10
158.	Pentachloronitrobenzene	82-68-8	20
159.	Pentachlorophenol	87-86-5	25
160.	Phenacetin	62-44-2	20
161.	Phenanthrene	85-01-8	10
162.	Phenol	108-95-2	10
163.	p-Phenylenediamine	106-50-3	10
164.	Phorate	298-02-2	10
165.	Polychlorinated biphenyls (PCB's)	See note 6	20

6 NYCRR Part 360 Parameters
Target Compound List (TCL) and
Contract Required Quantitation Limit (CRQL) (Continued)

	Parameter[1]	CAS Number[2]	Contract Required Quantitation Level (µg/L)
Expanded Parameters (Continued)			
Organic Parameters (Continued):			
166.	Polychlorinated dibenzo-p-dioxins (PCDD's)	See note 7	0.01
167.	Polychlorinated dibenzo-furans (PCDF's)	See note 8	0.01
168.	Pronamide	23950-58-5	10
169.	Propionitrile	107-12-0	100
170.	Pyrene	129-00-0	10
171.	Safrole	94-59-7	10
172.	Silvex	93-72-1	2.0
173.	Styrene	100-42-5	1
174.	2,4,5-trichlorophenoxyacetic acid	93-76-5	2.0
175.	1,2,4,5-Tetrachlorobenzene	95-94-3	10
176.	2,3,7,8-Tetrachlorodibenzo-p-dioxin	1746-01-6	0.005
177.	1,1,1,2-Tetrachloroethane	630-20-6	1
178.	1,1,2,2-Tetrachloroethane	79-34-5	1
179.	Tetrachloroethene	127-18-4	1
180.	2,3,4,6-Tetrachlorophenol	58-90-2	10
181.	Toluene	108-88-3	1
182.	o-Toluidine	95-53-4	10
183.	Toxaphene (Total)	See note 9	1.0
184.	1,2,4-Trichlorobenzene	120-82-1	1
185.	1,1,1-Trichloroethane	71-55-6	1
186.	1,1,2-Trichloroethane	79-00-5	1
187.	Trichloroethene	79-01-6	1

6 NYCRR Part 360 Parameters
Target Compound List (TCL) and
Contract Required Quantitation Limit (CRQL) (Continued)

	Parameter[1]	CAS Number[2]	Contract Required Quantitation Level (µg/L)
Expanded Parameters (Continued)			
Organic Parameters (Continued):			
188.	Trichlorofluoromethane	75-69-4	1
189.	2,4,5-Trichlorophenol	95-95-4	10
190.	2,4,6-Trichlorophenol	88-06-2	10
191.	1,2,3-Trichloropropane	96-18-4	1
192.	0,0,0-Triethyl phosphorothioate	126-68-1	10
193.	sym-Trinitrobenzene	99-35-4	10
194.	Vinyl acetate	108-05-4	1
195.	Vinyl chloride	75-01-4	1
196.	Xylenes (Total)[4]	1330-20-7	1

Part 360 Notes

[1]Parameter names are those widely used in government regulations, scientific publications, and commerce; synonyms exist for many chemicals.

[2]Chemical Abstracts Service registry number given is for the basic element or ion being measured. Where the parameter being measured is a "Total", all species in the groundwater that contain this element or ion are measured and included in the reported total.

[3]The department may waive the requirement to analyze Hexavalent Chromium provided that Total and Hexavalent and Trivalent Chromium values do not exceed 0.05 mg/l.

[4]Xylene (Total): This parameter should include a total of the following: o-xylene (CAS RN 96-47-6), m-xylene (CAS RN 108-38-3), p-xylene (CAS RN 106-42-3), and unspecified xylenes (dimethylbenzenes) (CAS RN 1330-20-7).

[5]Chlordane (Total): This parameter should include a total of the following: alpha-chlordane (CAS RN 5103-71-9), beta-chlordane (CAS RN 5103-74-2), gamma-chlordane (CAS RN 5566-34-7), and constituents of chlordane (CAS RN 57-74-9 and CAS RN 12789-03-6).

Part 360 Notes (Continued)

[6]Polychlorinated biphenyls (Totals - CAS RN 1336-36-3): This parameter should include a total of the following: congener chemicals, including constituents of Aroclor 1016 (CAS RN 12674-11-2), Aroclor 1221 (CAS RN 11104-28-2), Aroclor 1232 (CAS RN 11141-16-5), Aroclor 1242 (CAS RN 53469-21-9), Aroclor 1248 (CAS RN 12672-29-6), Aroclor 1254 (CAS RN 11097-69-1), and Aroclor 1260 (CAS RN 11096-82-5).

[7]Polychlorinated dibenzo-p-dioxins (Total): This parameter should include a total of the following: congener chemicals, including tetrachlorodibenzo-p-dioxins (see also 2,3,7,8-TCDD), pentachlorodibenzo-p-dioxins, and hexachlorodibenzo-p-dioxins. The PQL shown is an average value for PCDD congeners. Upon request of the applicant, the department may waive the requirement to analyze for dioxins, where appropriate.

[8]Polychlorinated dibenzofurans (Total): This parameter should include a total of the following: congener chemicals, including tetrachlrodibenzofurans, pentachlorodibenzofurans, and hexachlorodibenzofurans. The PQL shown is an average value for PCDF congeners. Upon request of the applicant, the department may waive the requirement to analyze for furans, where appropriate.

[9]Toxaphene (Total): This parameter should include a total of the following: congener chemicals contained in technical toxaphene (CAS RN 8001-35-2), i.e., chlorinated camphene.

Part V – NYSDEC Division of Fish, Wildlife, and Marine Resources Analytical Parameters and Contract Required Detection Limits

Contract Required Detection Limits for Organic Parameters in Tissue and Soil/Sediment

Analyte Group	Detection Limit (ng/g)[1]
Polychlorinated Biphenyls (PCBs) by Aroclor	10
DDT and Metabolites	2.0
Chlordane and Metabolites (except Oxychlordane)	2.0
Oxychlordane	5.0
Hexachlorobenzene	2.0
Hexachlorocyclohexane (HCH) Isomers	5.0
Endrin	2.0
Dieldrin	5.0
Mirex	2.0
Heptachlor Epoxide	2.0
Endosulphans	5.0
PCB Congeners (209 List)	0.1 [2]
PCB Congeners (WHO List)	0.1
PCB Congeners (Selected)	0.1
PCB Congeners (IUPAC 77, 126, and 169)	0.05
Chlorinated Dioxins and Furans (PCDD/Fs)	0.001
Brominated Dioxins and Furans (PBDD/Fs)	0.001
Polybrominated Diphenyl Ethers (PBDEs)	0.001
Polycyclic Aromatic Hydrocarbons (PAHs)	1.0
Toxaphene (Total)	10

[1] For all analytes except “Toxaphene (Total)”, the detection limit listed represents the detection limit required for each compound (or Aroclor) in the listed “Analyte Group”.

[2] The detection limit for IUPAC congeners 1, 2, and 3 is 1.0 ng/g.

Contract Required Detection Limits for Inorganic Parameters in Tissue and Soil/Sediment

Analyte	CAS Number	Detection Limit (ng/g)
Mercury (Hg)	7439-97-6	10
Methyl Mercury	22967-92-6	1.0
Cadmium (Cd)	7440-43-9	2.0
Lead (Pb)	7439-92-1	10
Arsenic (As)	7440-38-2	10
Selenium (Se)	7782-49-2	10

Contract Required Detection Limits for Miscellaneous Parameters in Tissue and Soil/Sediment

Parameter	Detection Limit (%)
Lipid Content (tissue only)	0.01
Moisture Content	0.01

APPENDIX B: TESTAMERICA LABORATORIES, INC. STANDARD OPERATING PROCEDURES

Lab SOP 01a

Volatile Organics By GC/MS (Method EPA 8260C), Revision 5, 12/11/2015, TestAmerica Laboratories, Inc.

Title: SW846 Method 8260C, Volatile Organic Compounds by Gas Chromatography/Mass Spectrometry (GC/MS)

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Approvals (Signature/Date):

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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.

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
2-Chloroethyl vinyl ether	110-75-8	Styrene	100-42-5

COMPOUND	CAS#	COMPOUND	CAS#
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 Automatic Sampler/4560 concentrator;
- Archon 5100A Automatic sampler/ OI Analytical 4660 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 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 5890/Agilent 6890/7890 equipped with temperature programming capability.

6.1.7 GC column: 75M long x 0.53mm ID, J&W DB-624 capillary column with 3um 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 (5971/5972/Agilent 5973/5975): 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: glass jet separator with fused silica transfer lines heated to 180°C or capillary direct.

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*	2000 ppm	Restek	567645
8260 List 1 / Std #3 Gases – (SS)*	2000 ppm	Restek	567645 sec
8260 List 1 / Std #1 MegaMix*	1000-50000 ppm	Restek	567641
8260 List 1 / Std #1 MegaMix (SS)*	1000-50000 ppm	Restek	567641 sec
8260 List 1 / Std #2 Ketones	10000 ppm	Restek	567642
8260 List 1 / Std #2 Ketones * (SS)	10000 ppm	Restek	567642 sec
8260 List 1 / Std #5 Acrolein	10000 ppm	Restek	568720
8260 List 1 / Std #5 Acrolein (SS)	10000 ppm	Restek	568720 sec
8260 List 1 /Std #4 2 CEVE	2000 ppm	Restek	567643
8260 List 1 /Std #4 2 CEVE (SS)	2000 ppm	Restek	567643 sec
8260 List 1 /Std #6 Vinyl Acetate	4000 ppm	Restek	567646
8260 List 1 /Std #6 Vinyl Acetate (SS)	4000 ppm	Restek	567646 Sec
8260 List 2 / Std #1 Additions	2000-50000ppm	Restek	568722
8260 List 2 / Std #1 Additions (SS)	2000-50000 ppm	Restek	568722 sec
8260 List 3 / Std #1 Polar Additions	2000-100000ppm	Restek	568723
8260 List 3 / Std #1 Polar Additions (SS)	2000-100000 ppm	Restek	568723 sec
Edison VOA Standard #1	2000-4000 ppm	Restek	568019-FL
Edison VOA Standard #1 (SS)	2000-4000 ppm	Restek	568019-SL
Edison VOA Standard #2	2000-10000 ppm	Restek	568713-FL
Edison VOA Standard #2 (SS)	2000-10000 ppm	Restek	568713-SL
Edison VOA Standard #3	2000 ppm	Restek	568021-FL
Edison VOA Standard #3 (SS)	2000 ppm	Restek	568021-SL
Edison VOA Standard #4	40000-50000 ppm	Restek	568022-fl
Edison VOA Standard #4 (SS)	40000-50000 ppm	Restek	568022-sl
1,4-Dioxane	Neat	Supelco	
Propenes *	1000/2000ppm	Supelco	NA
Propenes * (SS)	1000/2000ppm	Supelco	NA
1,4-Dioxane	1000 ppm	Absolute	70373
Freon 114	1000 ppm	Absolute	71232

(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) (25 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 2500 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)	100ul	
1,4-Dioxane-d8	10000 ppm	450ul	

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 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	Concentration	Volume of Standard Added to 50 ml of Reagent Water (ul)	Final Concentration (ug/L)
8260SIM Mix1	50ppm	0.5	0.50
1,4-Dioxane	500ppm (varied)	2	20

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 50 ml volumetric flasks filled with organic free reagent water. Once prepped the solution is poured into a 40 ml VOA vial and loaded onto the purge and trap autosampler

Standard Solution	Concentration	Volume of Standard Added to 50 ml of Reagent Water (ul)	Final Concentration (ug/L)
8260 Mix1	50ppm	0.5	0.50
1,4-Dioxane	500ppm	2	20

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, either 1,2-Dichloroethane-d4 or Dibromofluoromethane 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, 0.05, 0.10, 0.50, 1.0 and 2.0 ppb; 1,4-Dioxane at 2, 5, 10, 20, 30, 40 ppb. 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 (50 ug/ml and 0.50ug/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
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

Minimum Relative Response Factor	
Common Target Analytes	**Minimum RF**
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 $\geq$ 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

Poor Performers	
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.

10.3. 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):

$$\% D = \frac{RRF_c - \overline{RRF_i}}{\overline{RRF_i}} X\ 100$$

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 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	2000ppm	500ppm	20mL 15mL TV/M
Gas Mix Li	Gas (Li)	Restek	567645	500 uL	2000ppm	50ppm	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	2000ppm	500ppm	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	2000ppm	50ppm	40ml 31ml TV/M
Acrolein	AC	Restek	82402	1.0ml	20000ppm	500ppm	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	2000ppm	500ppm	10mL
8260 Mix 3	Mix 3 (Hi)	Restek	568723 568021-fl	2.5ml 2.5ml	2000ppm	500ppm	10ml 5ml TV/M
1,4-Dioxane	1,4-Dioxane	Supelco	360481	483.6ul	Neat	50000ppm	10ml/9.52TVM
1,4-Dioxane	1,4-Dioxane	Supelco	NA	100ul	50000ppm	500ppm	10ml/9.90TVM
Propenes*	Propenes	Supelco	21240202	NA	1000/2000 ppm	NA	NA
Propenes*	Propenes	Supelco	21240202	1ml	1000/2000 ppm	50ppm (varied)	20ml/ 19ml
Gas SS	Gas SS	Restek	567645.sec	1ml	2000ppm	50ppm	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	2000ppm	50 ppm	40 ml 30 ml TV/M
Acrolein SS	AC SS	Restek	568720.sec	1 ml	20000ppm	500 ppm	40 ml 39 ml TV/M
Propenes SS	Propenes ss	Supelco		1 ml	1000/2000ppm	50/100 ppm	40 ml 39 ml TV/M
8260Mix 1 SIM SS	SIM MIX1 SS	Supelco	5S-02111	50ul	2000ppm	10ppm	10ml 9.95 TV/M
1,4-Dioxane (SS)	1,4-Dioxane	Absolute	70373	1ml	1000ppm	500ppm	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)					
Mix 1 (SIM) (10ppm)	1ul (0.02 ppb)	0.5ul (0.05 ppb)	10 ul (0.1 ppb)	2.5 ul (0.5 ppb)	5 ul (1 ppb)	10 ul (2 ppb)
1,4-Dioxane (500ppm)	0.4 ul (0.4 ppb)	1 ul (5 ppb)	1 ul (10 ppb)	2 ul (20 ppb)	3 ul (30 ppb)	4 ul (40 ppb)
Final Vol. (reagent water)	**500ml**	**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 50 ml of Reagent Water (ul)	Final Concentration (ug/L)
8260 SS (Second source)	50ppm	0.5	0.50
1,4-Dioxane SS	500ppm	2	20

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

TABLE 7 Characteristic Ions of Volatile Organic Compounds		
1-Propene	41	42
2,2-Dichloropropane	77	97
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

TABLE 7 Characteristic Ions of Volatile Organic Compounds		
Chlortrifluoroethene	116	118
cis-1,3-Dichloropropene	75	77
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

TABLE 7 Characteristic Ions of Volatile Organic Compounds		
P-Isopropyltoluene`	119	134, 91
Propyl Acetate	43	61, 73
Propionitrile	54	52, 54
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

Lab SOP 01b

Closed System Purge and Trap and Extraction for Volatile Organics in Soil (Method EPA 5035A), Revision 9, 11/27/2012, TestAmerica Laboratories, Inc.

Title: Closed System Purge and Trap and Extraction for Volatile Organics in Soil , SW846 Method 5035A

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Approvals (Signature/Date):

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1.0 Scope and Application

1.1 Analytes, Matrix(s), and Reporting Limits

1.1.1 This method describes a closed system purge and trap procedure for the analysis of volatile organic compounds in soils, sediments and solid wastes.

1.1.2 This method is designed for use on samples containing low levels of volatile compounds (0.5 to 200ug/kg), and requires the use of hermetically sealed sample vials, which limits a samples exposure to the atmosphere, thereby minimizing the loss of volatile compounds.

1.1.3 Method 5035A can be used for most volatile organic compounds that have boiling points below 200°C and are insoluble or slightly soluble in water. Water-soluble compounds can be included in this technique, but will have higher quantitation limits due to poor purging efficiency.

1.1.4 Method 5035A also includes sample collection and preparation procedures for medium and high concentration soil samples (i.e., >200 ug/kg). Medium and high level soil samples are prepared either by field preservation of a soil sample in methanol or by creating a methanol extract from a soil sample that was collected as bulk soil.

1.1.5 The preparation steps described for medium and high level samples will be followed by the aqueous purge and trap procedure detailed in TestAmerica Edison SOP No. ED-MSV-001(*Purge and Trap for Aqueous Samples, SW846 Method 5030, current revision*).

1.1.6 The preparation and sample introduction steps described for low-level soils will be followed by analysis using TestAmerica Edison SOP Nos. ED-GCV-006 (*Gasoline Range Organics using GC FID Method SW846 8015, current revision*) or ED-MSV-005 (*SW846 Method 8260B Volatile Organic Compounds by Gas Chromatography/Mass Spectrometry, current revision*) as applicable.

1.1.7 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 **Low Level Soils:** Volatile organic compounds are determined by collecting a 5g-soil sample with a specially designed sampling device (ex., En Core® Sampler) in which the sample is stored without headspace. Upon receipt and within 48 hours of sampling, the soil is placed in a pre-weighed vial containing 5 mL of reagent water and a stir bar. The vial containing the soil is then frozen extending the hold time to 14 days. The same samples may be received from the field in a 40 mL vial with 5 mL water (ex., Terra Core® Sampler). Immediately prior to analysis, the soil is thawed, reagent water, surrogate spiking solution and/or internal standard

spiking solution are added to the vial by the automatic sampler, without opening the vial to the atmosphere. The sample vial is heated and purged with helium while being magnetically stirred. The volatile components travel through a transfer line to a sorbent trap. When purging is complete, the trap is heated and backflushed with helium to desorb the sample components on to a GC column for separation and analysis by the appropriate determinative method.

2.2 **Medium Level Soils:** Medium level soils are soils that were originally intended to be analyzed by a low level soil method, but show contaminants >200ug/kg when screened. A methanol extract is prepared by extracting 5g of soil with 10mL of methanol. The volatiles are effectively transferred from the soil to the methanol. A portion of the methanol extract is introduced into the GC or GC/MS system by using the purge and trap Method SW846 5030 followed by the appropriate determinative method.

2.2.1 Samples may also arrive from the field as 5 mL or 10 mL methanol preserved samples. These are considered to be medium level prepared samples.

2.3 **High Level Soils:** The high level soil method acts as a combined preservation and extraction technique. 25mL of methanol /surrogate solution is placed in each VOA vial and weighed prior to field sampling. Approximately 10g of soil is added to each vial in the field. Upon returning to the lab, the vial is weighed again, and the difference between the two weights is recorded as the initial weight of sample. As with the medium level method, the volatiles are effectively transferred to the methanol. A portion of the methanol extract is introduced into the GC or GC/MS system by using the purge and trap Method SW846 5030 followed by the appropriate determinative method.

2.4 **Waste Dilution:** Organic waste samples are prepared by extracting 1g of sample with 10mL of methanol / surrogate solution. A portion of the methanol extract is introduced into the GC or GC/MS system by using the purge and trap Method SW846 5030 followed by the appropriate determinative method.

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 located 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 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 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.

4.9 Potential cross-contamination of samples stored in lab refrigerators is monitored by preparation, analysis and evaluation of storage blanks. 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.

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.

5.2 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 Automatic Sampler/4560 concentrator;
- Archon 5100A Automatic sampler/ OI Analytical 4560 concentrator;
- EST Centurion Autosampler/ EST Encon concentrator;
- Archon Autosampler/EST Encon concentrator.

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 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 5890/Agilent 6890/7890 equipped with temperature programming capability.

6.1.7 GC column: reference the applicable determinative method for column specifics.

6.1.8 Detector: Flame Ionization Detector or Mass Spectrometer (HP5971/5972/Agilent 5973). Reference the applicable determinative method for detector specifics.

6.1.9 Data system: HP Chemstation II for data acquisition and HP UNIX based TARGET software for data processing.

6.2 Equipment and Supplies

6.2.1 Freezer: Capable of holding a temperature of -7°C to -20°C.

6.2.2 Refrigerator: Capable of holding a temperature of 4°C ±2°C

6.2.3 Top loading analytical balance.

6.2.4 Portable field balance. Capable of weighing to 0.01g.

6.2.5 Microsyringes: 10 uL to 1000 uL.

6.2.6 Syringes: 5mL, 10mL, and 30mL gas-tight.

6.2.7 Volumetric flasks: Class “A” glassware, 10mL , 50mL , and 100 mL.

6.2.8 VOA vials: 40 mL glass with PTFE –faced septum.

6.2.9 En Core® Sampler. Designed to take a 5g-soil sample. Sealed to prevent loss of volatiles.

6.2.10 Vials: 2mL amber glass with screw cap with Teflon-faced septa.

6.2.11 Spatula: Narrow, stainless steel.

6.2.12 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.

7.1.2 Methanol. Purge and trap grade, purchased from JT Baker. (Cat # 9077-02)

7.1.2.1 Each lot of methanol and hydrochloric 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*).

7.2 Standards

7.2.1 Calibration standard preparation and use is described in detail in each of the determinative methods.

7.2.2 Method 8260 Methanol/Surrogate solution (2.5 ppm) is prepared using purge and trap grade methanol and neat a,a,a-Triflurotoluene solution. Initially a primary solution is prepared at 2500 ppm:

Standard Name	Vendor	Catalog #	Volume added	Concentration of Stock Std.	Concentration of Standard	Total Volume Volume of MeOH
1°Surrogate Mix: 4-Bromofluorobenzene Toluene-d8 1,2-Dichloroethane-d4	Sigma Aldrich	 B67201 151998 396540	 1585 ul 2678 ul 1932 ul	Neat	2500ppm	1000 ml

The 2500 ppm solution is used to prepare a 2.5 ppm Method 8260 Methanol/Surrogate working solution by diluting 1.0 ml of 2500 ppm stock to 1000ml with purge and trap grade methanol.

7.2.3 Method 8015 (GRO) Methanol/Surrogate solution (1.25 ppm) is prepared by diluting 0.5ml of the 2500 ppm stock (see Section 7.1.4) to 1000 ml with purge and trap grade methanol.

8.0 Sample Collection, Preservation, Shipment and Storage

8.1 Low Level Soils

Sample containers used to collect low-level soils are 40 mL VOA vials with PTFE-faced septa (closed system purge and trap), En Core® Samplers, Terra Core® kits or bulk soil.

8.1.1 For the 40 mL VOA vial (closed system purge and trap) collection procedure, add 1 clean magnetic stir bar and 5 mL of reagent water to each vial.

8.1.1.1. Cap the vial and weigh to the nearest 0.01g. Record this as the tare weight.

8.1.1.2. Collect approximately 5g of soil using an appropriate sampling device and add 5g to each of 3 vials for low level analysis.

8.1.1.3. Collect a fourth aliquot for field methanol preservation into a pre-weighed vial containing 10 mL of methanol.

8.1.1.4. Closed system purge and trap collection vials are re-weighed to determine actual sample weight. Record the sample weight on the sample vial.

8.1.1.5. Within 48 hours of sampling the collection vials are placed into a freezer (on their side to prevent breakage) at -7°C to -20°C. Freezing in this manner extends the holding time to 14 days from sampling.

8.1.2 For En Core® Sampler collection, collect up to 3 En Core® Sampler for low level preservation via freezing and a fourth for medium level preservation with methanol (Section 8.2 details the medium level preservation procedures for En Core® samplers). The low level preservation procedures are:

8.1.2.1. The En Core® device is received at the lab sealed without headspace with a cap and viton o-rings.

8.1.2.2. For each En Core® requiring low level preservation, add 5ml of reagent water and a magnetic stir bar to a 40 ml vial. Cap the vial.

8.1.2.3. Weigh each vial to the nearest 0.01g. Record this as the tare weight.

8.1.2.4. Transfer the contents of each En Core® to separate preweighed vials within 48 hours of sampling.

8.1.2.5. Reweigh the vial. Record the weight on each sample vial.

8.1.2.6. The vials are placed into a freezer (on their side to prevent breakage) at -7°C to -20°C. Freezing in this manner extends the holding time to 14 days from sampling.

8.1.2.7. A bulk soil sample must also be collected for the purposes of percent moisture analysis for dry weight determination.

8.1.2.8. Vials are removed from the freezer the day of analysis for thawing.

8.1.2.9. Stir bars may be reused after the vial is analyzed. Retrieve the stir bar from each vial using the magnetic stir bar retriever, rinse the stir bar with methanol and bake at 105°C for 2 hours.

8.1.3 Terra Core® Kits. This option requires samples to be field preserved:

8.1.3.1. The sample kit typically includes two pre-weighed 40 mL vials containing 5 mL of water with a small magnet stir bar and one or two pre-weighed 40 mL vials containing pre-measured and recorded amount of methanol.

8.1.3.2. Approximately 5g of sample is placed into each vial by the field sampler.

8.1.3.3. The vials containing the samples are re-weighed in the lab and the net sample weight is recorded for each vial. The vials containing the sample and water are frozen up until the time of analysis.

8.1.3.4. The methanol preserved samples are considered to be medium level prepared samples and do not require freezing but they must be refrigerated at 4°C until time of analysis.

8.1.3.5. A bulk soil sample must also be collected for the purposes of percent moisture analysis for dry weight determination.

8.1.3.6. Stir bars may be reused after the vial is analyzed. Retrieve the stir bar from each vial using the magnetic stir bar retriever, rinse the stir bar with methanol and bake at 105°C for 2 hours.

8.1.4 Bulk soil. Typically consists of soil collected in 8-oz or 16-oz glass jars. At client request bulk soil samples can be sub-sampled, preserved and analyzed for volatile organics. Soils collected for low level VOA analysis as bulk soil in jars will be preserved as described above in Section 8.1.1. Documentation of preservation is recorded in the Soil Preservation Logbook.

8.2 Medium Level Soils

8.2.1 Medium level refers to any soil sample extracted in methanol not preserved in the field. The fourth Encore sample collected (as described in Section 8.1.2 above) will be prepared as a medium level soil in case the analysis of a low level preserved aliquot exhibits concentrations of target compounds exceeding 200 ug/kg.

8.2.1.1. Prepare the medium level soil by adding 5 g of soil or Encore to 10 mL of Methanol / Surrogate solution (either the 8260 solution as described in Section 7.1.4 ***or*** the 8015 solution as described in Section 7.1.5. depending upon the method to be analyzed). All medium level extracts shall be prepared within 48 hours of collection. The weight of soil used to prepare the extract is recorded on the sample vial.

8.2.1.2. Since methanol extract results tend to increase with time as the sample contact time increases, a minimum contact time of one day is allowed ***or*** the soil is sonicated for 20 minutes.

8.2.1.3. Any soil requiring medium level preservation but collected out of compliance with method 5035A (bulk soil without preservative or not in an Encore device) will be prepared in the same manner as in section 8.2.1.1. Documentation will be noted in the soil preservation log.

8.3 High Level Soils

8.3.1 High level soils are field collected using laboratory prepared 40 ml VOA vials (with PTFE-faced septa) containing 25 mL of the Methanol/Surrogate solution (either the 8260 solution as described in Section 7.1.4 ***or*** the 8015 solution as described in Section 7.1.5. depending upon the method to be analyzed).

8.3.2 The vials containing the Methanol/Surrogate solution are weighed using a barcode reader and an analytical balance. The bottle ID and weight (Weight 1) are recorded in the methanol-tracking program.

8.3.3 The containers are issued to the field as required.

8.3.4 Field samplers are directed to weigh 10 g of sample using a field balance. They must carefully add the 10 g sample to the sample vial containing the Methanol/Surrogate solution, taking care not to spill any of the solution from the vial.

8.3.5 Field samplers are directed to wipe the neck of the vial carefully to remove any soil around the threads. This helps to ensure a good seal once the vial is capped. Field samplers are further directed to cap the vial, shake gently, dispersing the soil into the methanol. An additional aliquot of soil without any methanol or preservative must be collected for use in determining percent moisture (i.e., for dry weight determination).

8.3.6 Upon the return to the laboratory, the sample vials (now containing both the Methanol/Surrogate solution and approximately 10g of soil) are reweighed using the barcode reader and analytical balance. Record this weight (Weight 2) in the methanol tracking system. Calculate the initial weight of soil by subtracting Weight 1 from Weight 2.

8.3.7 All samples for volatile analysis are protected from light and stored at 4°C±2°C in an area free of organic solvents. No standards or solvents are stored in the sample refrigerators or in the surrounding area.

8.3.8 All samples preserved in this manner must be analyzed within 14 days of collection.

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 Reagent water blanks are analyzed to ensure that reagents and/or sample dilutions are free of interferences.

9.3 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.

9.4 Potential cross-contamination of samples stored in lab refrigerators is monitored by preparation, analysis and evaluation of storage blanks. 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.

9.5 An initial demonstration of accuracy and precision is performed for each determinative method. This demonstration is done for each sample introduction technique.

10.0 Procedure

10.1 This section provides guidance for the analysis of low level soils using a closed system purge and trap procedure. Reference the SOPs for the determinative methods for additional details as applicable: TestAmerica Edison SOP Nos. ED-GCV-006 (*Gasoline Range Organics using GC FID Method SW846 8015, current revision*) or ED-MSV-005 (*SW846 Method 8260B Volatile Organic Compounds by Gas Chromatography/Mass Spectrometry, current revision*).

10.2 Instrument operating parameters are set at the beginning of a method of analysis and remain constant throughout the entire analytical procedure. Again, reference the SOP for the applicable determinative method for specifics. Sample operating conditions are listed here:

Example Instrument Operating Parameters

Purge and trap unit	
Purge Time	11 minutes
Dry Purge	1 Minutes
Purge Gas	Helium
Purge Flow	40-45 ml/min
Purge Temp	Water - Ambient; Solids - 40^{0}C
Trapping Temp	Ambient, <30^{0}C
Desorb Time	1-2 Minutes
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
Run Time	30 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 All samples are screened prior to analysis, using the procedure outlined in TestAmerica Edison SOP No. ED-GCV-001, *Screening for Volatile Organics, Static Headspace with GC FID SW846 Method 5021(modified),* current revision. In addition to reducing excessive instrument contamination and/or extensive instrument clean up, screen data provides the analyst with an approximate dilution factor and is used to determine which sample preparation technique should be used for a particular sample. If the estimated concentration from the screening procedure shows concentrations below 200 ug/kg, follow the low concentration closed system purge and trap method. If the screen result shows concentrations >200 ug/kg, follow the medium level procedure.

10.4 Low Level Soil Procedure

10.4.1 Low level soils are field collected as described in Section 8.1 using 40 mL VOA vials with PTFE-faced septa (closed system purge and trap), En Core® Samplers, Terra Core® kits or bulk soil.

10.4.2 Set up the purge and trap autosampler (Archon 5100A or EST Centurion, see Section 6.1) for the closed system method for low-level soils. The purge and trap autosampler will add 5 mL reagent water, and 1uL of the internal standard and/or surrogate spiking solution to the sample vials. The heating block will heat each sample to 40° C and hold for 0.5 minutes before purging begins.

10.4.3 Tuning, calibration, and method blank analysis:

- Tuning and calibration is accomplished as detailed in the SOP for the applicable determinative method.

- The volume of water used when purging a calibration standard must be the same as the volume of water purged during a sample analysis. Since the low-level soils have 5 mL water added at vial preparation plus the 5 mL of reagent water added by the purge and trap autosampler prior to purging, a total volume of 10 mL is required for calibration standards.

- Internal standard (if required by the determinative method) is added to each calibration standard by the purge and trap autosampler, in the same manner it will be added to the samples.

- After successful calibration, analyze a method blank. The method blank must meet all criteria in the determinative method. A successful method blank shows that the system is free of interferences and sample analysis may begin.

10.5 Medium Level Soil Procedure

10.5.1 Medium level soils are typically field collected using laboratory prepared 40 ml VOA vials (with PTFE-faced septa) containing 25 mL of the Methanol/Surrogate solution (either the 8260 solution as described in Section 7.1.4 ***or*** the 8015 solution as described in Section 7.1.5 depending upon the method to be analyzed).

10.5.2 When preparing a medium level soil from a bulk soil sample, do not decant any supernatant liquids. Quickly mix the contents of the sample container with a narrow spatula. Weigh out 5g of soil into a 40ml vial, add 10ml of Methanol/Surrogate mix solution (either the 8260 solution as described in Section 7.1.4 ***or*** the 8015 solution as described in Section 7.1.5 depending upon the method to be analyzed) and cap the container and proceed with Section 10.5.6.

10.5.3 For samples collected in the Encore sampling device, break open one end and push the aliquot of soil out into a vial. Add 10ml of the Methanol/Surrogate mix solution (either the 8260 solution as described in Section 7.1.4 ***or*** the 8015 solution as described in Section 7.1.5 depending upon the method to be analyzed) and cap the vial and proceed with Section 10.5.6.

10.5.4 For Method 8260 samples collected as Terra Cores® in 5 mL or 10 mL methanol add 5 uL and 10 uL of the 2500 ppm 8260 Methanol/Surrogate mix (see Section 7.1.4) respectively to extract and proceed with Section 10.5.6.

10.5.5 For Method 8015 (GRO) samples collected as Terra Cores® in 5 mL or 10 mL methanol add 2.5 uL and 5 uL of the Methanol/Surrogate Mix surrogate solution (see Section 7.1.5) respectively and proceed with Section 10.5.6.

10.5.6 Cap and shake the extract for 2 minutes. Allow soil to settle.

10.5.7 Transfer 2 mL of the extract to a 2 mL amber glass vial for storage with minimal headspace.

10.5.8 Refrigerate at 4°C until the time of analysis.

10.5.9 Analyze a portion of this extract using Method 5030 purge and trap for aqueous samples (TestAmerica Edison SOP No. ED-MSV-001, *Purge and Trap for Aqueous Samples, SW846 Method 5030, current revision*). The appropriate dilution factor is calculated from the low-level analysis.

10.6 High Level Soil Procedure

10.6.1 High level soils have been preserved in the field by sampling either 10 g of soil into vials containing 25 mL Methanol/Surrogate mix solution (either the 8260 solution as described in Section 7.1.4 ***or*** the 8015 solution as described in Section 7.1.5 depending upon the method to be analyzed) or 5 g of soil into vials containing 10 mL Methanol/Surrogate mix solution (again, either the 8260 solution as described in Section 7.1.4 ***or*** the 8015 solution as described in Section 7.1.5 depending upon the method to be analyzed).

10.6.2 Screen high level soils by Method 5021 to determine the proper dilution factor prior to analysis (see TestAmerica Edison SOP No. ED-GCV-001, *Screening for Volatile Organics,Static Headspace with GC FID SW846 Method 5021(modified),* current revision.)

10.6.3 Transfer 2 ml of the extract to a 2 mL amber glass vial for storage with minimal headspace.

10.6.4 Analyze a portion of this extract using Method 5030 purge and trap for aqueous samples.

10.7 Sample Analysis

10.7.1 Remove sample vials from storage and allow thawing to ambient temperature before analysis. Shake the vial gently to ensure that the contents move freely within the vial. Place the sample vials in the autosampler tray.

10.7.2 Schedule the autosamplers to run the samples using the low level soil method. 5 mL of reagent water plus 1 uL of internal standard/ surrogate spiking solution is added to the sample vial directly through the septum.

10.7.3 Prior to purging, the sample is heated to 40°C for 0.5 minutes.

10.7.4 Purge the sample with helium for 11 minutes while agitating with magnetic stirring.

10.7.5 The purgeable compounds will flow out of the vial through a glass lined transfer line to a sorbent trap.

10.7.6 Desorb the contents of the trap on to the GC column by rapidly heating the trap to 260 °C and backflushing it with helium.

10.7.7 Begin the GC temperature program and data acquisition.

10.7.8 Re-condition the trap after desorb by baking at 260° C for 8-12 minutes.

10.7.9 Perform quantitative and qualitative analysis using TARGET software. Evaluate data according to each determinative method.

10.7.10 If a re-analysis is required, perform the re-analysis using the second frozen sample vial.

10.7.11 If a dilution is required, analyze the methanol preserved aliquot by Method 5030.

10.7.12 Technical acceptance criteria for sample analysis. (Note: these are general guidelines; please see the SOP for the determinative method for more specific acceptance criteria)

- The samples must be analyzed on a GC or GC/MS system meeting the initial calibration, continuing calibration and blank technical acceptance criteria.
- The sample must be analyzed within the required holding time.
- The sample must have an associated method blank meeting the blank technical acceptance criteria.
- The percent recovery of each of the system monitoring compounds in the sample must be within the acceptance windows.
- 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.

10.7.13 Corrective Action for Sample Analysis (Note: these are general guidelines; please see the SOP for the determinative method for more specific corrective actions):

- Samples must meet technical acceptance criteria before reporting data.

- Corrective action for failure to meet instrument performance checks, initial, continuing calibration and method blanks must be completed prior to sample analysis.

- Corrective action for system monitoring compounds and internal standard compounds that fail to meet acceptance criteria must be completed prior to sample analysis.

- If any of the system monitoring compounds and internal standard compounds fail to meet acceptance criteria, 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.

- Check the preparation of the internal standards and system monitoring compounds for concentration and expiration.

- Verify that the instrument is operating correctly.

- 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 Corrective Action Reports will be initiated in order to provide for investigation and follow-up.

11.0 Calculations / Data Reduction

11.1 Methanol volume correction for soil moisture content (medium/high level methods):

$$Vt\ (\mu L\ solvent/water) = [\ ml\ of\ solvent + \frac{(\%moisture \times g\ of\ sample)}{100}] \times 1000\ uL/mL$$

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 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* (latest 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 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.

- 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 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, December 1996.

15.2 TestAmerica Edison Document No. ED-QA-LQM, *Laboratory Quality Manual*, most current revision.

15.3 TestAmerica Edison SOP No. ED-GCV-001, *Screening for Volatile Organics,Static Headspace with GC FID SW846 Method 5021(modified),* current revision.

15.4 TestAmerica Edison SOP No. ED-MSV-001, *Purge and Trap for Aqueous Samples, SW846 Method 5030, current revision*.

15.5 TestAmerica Edison SOP Nos. ED-GCV-006, *Gasoline Range Organics using GC FID Method SW846 8015, current revision.*

15.6 TestAmerica Edison SOP No ED-MSV-005, *SW846 Method 8260B Volatile Organic Compounds by Gas Chromatography/Mass Spectrometry, current revision.*

15.7 United States Environmental Protection Agency, *Method 8000C: Determinative Chromatographic Determinations*, Test Methods for Evaluating Solid Wastes, SW846 Third Edition, Volume 1B: Laboratory Manual, Physical/Chemical Methods, Revision 3, March 2003

15.8 TestAmerica Edison SOP No. ED-SPM-004, *Sample Storage & Handling Procedures for Mitigation of Sample and Laboratory Contamination,* current revision.

15.9 TestAmerica Corporate Environmental Health and Safety Manual (CW-E-M-001)

15.10 TestAmerica SOP No. ED-GEN-022, *Training* ,latest revision.

15.11 TestAmerica Edison SOPs Nos. ED-SPM-007, *Disposal of Samples and Associated Laboratory Waste, current revision.*

15.12 TestAmerica Edison SOP No. ED-SPM-008, *Laboratory Waste Disposal Procedures,* current revision.

16.0 Method Modifications:

16.1 N/A

17.0 Attachments

17.1 N/A

18.0 Revision History

- Revision 9, effective date: 11/27/2012
  - Updated referenced Lab Quality Manual section and appendix numbers as appropriate

- Revision 8, effective date: 12/6/2010
  - Section 3: revised to reference new location for definitions.
  - Sections 8.2.1.4 and 8.3.9 referencing methanol solvent adjustment for solid samples with significant moisture content (>10%) was removed.

- Revision 7, effective date: 11/4/2008:
  - Revised SOP format in accordance with TestAmerica Corporate Quality SOP No. CW-QS-002 (Rev 0) Writing a Standard Operating Procedure (SOP).
  - Updated the SOP to comply with SW846 Method 5035A (was 5035). Retitled SOP accordingly.
  - Section 1.1.5: Added reference to TestAmerica Edison SOP No. ED-MSV-001(*Purge and Trap for Aqueous Samples, SW846 Method 5030, current revision*).
  - Section 1.1.6: Added references to TestAmerica Edison SOP Nos. ED-GCV-006 (*Gasoline Range Organics using GC FID Method SW846 8015, current revision*) and ED-MSV-005 (*SW846 Method 8260B Volatile Organic Compounds by Gas Chromatography/Mass Spectrometry, current revision*)
  - Section 1.1.7: Added reference to Quality Assurance Manual for method modifications.
  - Section 3: revised to reference new location for definitions.

- Section 4: revised to include a reference to storage blanks and the applicable SOP: TestAmerica Edison SOP No. ED-SPM-004, *Sample Storage & Handling Procedures for Mitigation of Sample and Laboratory Contamination*, current revision.
- Section 5 (Safety): Revised to include most up to date corporate health and safety references and information.
- Section 6: Updated with current instrumentation and configurations.
- Section 7.0: added details of the solvent testing and approval program.
- Section 7.2: Updated standards sources and catalog numbers.
- Section 8: Updated with additional details for each type of preservation/analysis (low level, medium level, high level). Added used of Terra Core® Sampling Kits (Section 8.1.3).
- Section 8: Updated to include requirement to adjust the methanol volume (medium/high level) for soil moisture content.
- Section 9.0: Added details of storage blanks and reference to TestAmerica Edison SOP No. ED-SPM-004, *Sample Storage & Handling Procedures for Mitigation of Sample and Laboratory Contamination,* current revision..
- Section 10: Revised and expanded to include instrument operating conditions and specific sample prep details for low level, medium and high level preps.
- Section 11: Deleted reference to SOP for Organic Calculations. Added statement that calculations can now be found in the applicable determinative methods.
- Section12.0: added reference to Training SOP.
- Section 13.0: Revised to include most up to date company policy on Pollution Control as well as to include corporate health and safety references and information.
- Section 14.0: Revised to include most up to date company policy on Waste Management as well as to include corporate health and safety references and information.
- References: Expanded to include more specific SOP references.
- Revision history: updated.

Lab SOP 02

Preparation and Analysis of Diesel Range Organics and Mineral Spirits in Soil and Water Samples by EPA Method 3510C (Separatory Funnel), 3546 (Microwave), and 8015B (GC/FID), Revision 13, 8/17/2016, TestAmerica Laboratories, Inc.

Title: Preparation and Analysis of Diesel Range Organics and Mineral Spirits in Soil and Water Samples by SW846 Methods 3510C (Separatory Funnel), 3546 (Microwave) and 8015B (GC/FID)

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Approvals (Signature/Date):

Catalina Dalangin	8/17/16 Date	Dan Helfrich	8/17/16 Date
SVOA GC Manager		Health & Safety Manager	
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Quality Assurance Manager		Laboratory Director	

Hemex Patel — 8/17/16 Date
Organic Extractions Manager

Facility Distribution No. Electronic **Distributed To:** Facility Intranet

1.0 Scope and Application

1.1 Analytes, Matrix(s), and Reporting Limits

This SOP describes TestAmerica Edison's procedure for the preparation and analysis for Diesel Range Organics (DRO) in the range of C10 through C28 and C10 through C44 in soil and water samples. This method may also be used to determine Mineral Spirits (area sum) in soil and water samples. Water samples are prepared using SW846 Method 3510C separatory funnel extraction, with either full volume or reduced volume (RVE/LVI). Soil samples are prepared using SW846 3546 (microwave extraction). The SW 3510C and SW 3546 prep are detailed in this SOP. The determinative analytical method is SW846 Method 8015B.

Analyte	Matrix	Reporting Limit
DRO (C10-C28)	Solid	6.7 mg/kg
DRO (C10-C28)	Aqueous	0.10 mg/L
DRO (C10-C44)	Solid	6.7 mg/kg
DRO (C10-C44)	Aqueous	0.10 mg/L
Mineral Spirits	Solid	3.3 mg/kg
Mineral Spirits	Aqueous	0.05 mg/L

Note: For a complete listing of current Method Detections Limits (MDLs) and Reporting Limits (RLs) please refer to the current active 8015B_DRO method limit group in the TestAmerica LIMS (TALS) database.

1.2 On occasion clients may request modifications to this SOP. These modifications are handled following the procedures outlined in Sections 7 (Review of Work) and 19 (Test Methods and Method Validation) in the Quality Assurance Manual.

2.0 Summary of Method

Diesel Range Organic (DRO) compounds in the ranges of C10-C28 and C10-C44 are extracted from soil and/or water samples with Methylene Chloride. All samples are spiked with ortho-terphenyl surrogate prior to extraction. The sample extract is analyzed utilizing GC/FID instrumentation. The concentration of DRO is determined using a summation of the area response (i.e., all resolved peaks and the unresolved "envelope" in the time window established during initial calibration). This time window starts at the time of initial rise of peak C10 and ends at the time of final elution of peak C28 or C44 (as applicable) . Sample results are quantified and reported as "Diesel Range Organics (C10-C28)" or "Diesel Range Organics (C10-C44)". The concentration of Mineral spirits is determined using a summation of the area response within the time window established by the time of the rise of the first peak to the time of elution of the final peak of the mineral spirits product Virgin 105 supplied by Safety-Kleen.

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

Solvents, reagents glassware, and other sample prep hardware may yield artifacts to sample analysis. All these materials must be demonstrated to be free from interferences under the conditions of the analysis by analyzing method blanks. All glassware must be washed with hot soapy water and then rinsed once with 50:50 Methylene Chloride/Acetone followed by two rinsings with Methylene Chloride to reduce any contaminants.

Soap residue, which results in a basic pH on the surface of glassware, may cause analyte degradation.

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

Nitrile gloves should be used when performing this extraction. Latex and vinyl gloves provide no significant protection against the organic solvents used in this SOP, and should not be used.

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 gas chromatograph contains zones that have elevated temperatures. The analyst needs to be aware of the locations of those zones, and must cool them to room temperature prior to working on them.

There are areas of high voltage in the gas chromatograph. Depending on the type of work involved, either turn the power to the instrument off, or disconnect it from its source of power.

The block that the extraction beaker sits is hot enough to burn unprotected skin. Use caution when removing extraction beakers that haven't had time to cool and use appropriate protective equipment (e.g., insulated gloves or tongs).

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.

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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.
Acetone	Flammable	1000 ppm-TWA	Inhalation of vapors irritates the respiratory tract. May cause coughing, dizziness, dullness, and headache.
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 Agilent Technologies Gas Chromatograph (Series 6890 or 5890) with Flame Ionization Detector (FID), splitless injection port and Electronic Pressure Control (EPC), or equivalent;

6.1.2 Dual tower Agilent Technologies 7673 Autosampler, or equivalent;

6.1.3 Restek Capillary GC Column: 15meter x 0.32mm ID x 0.15 um df capillary column. (Restek Rtx-Mineral Oil Part Number: 18074 or equivalent)

6.1.4 Agilent Chemstation Data System: The Chemstation system is utilized for automation of runs and acquisition of data.

6.1.5 TestAmerica Chrom chromatography data processing software package. Chrom is used for post acquisition GC data processing, reporting and storage and is fully integrated with TestAmerica's LIMs (TALS).

6.1.6 Hydrogen Generator, Parker Hannifin Corporation or equivalent (part number H2PEMPD-510-100).

6.2 Supplies

6.2.1 GC Supplies

6.2.1.1 4mm splitless sleeve (Restek Catalog # 11868-773 or equivalent) - This liner/glass wool combination provides many functions. The glass wool serves as a heat sink rapidly vaporizing solvent and samples resulting in higher response factors. The liner also protects the column head from accumulation of high boiling residuals and particulates.

6.2.1.2 0.8mm ID, Gold Inlet Seal (Restek Catalog # 21241 or 21318 or equivalent)

6.2.1.3 Snoop Leak Detector solution or equivalent.

6.2.2 Organic Prep Supplies (Aqueous)

- 250 ml Erlenmeyer Flask, AMK Glass ERL-0252 or equivalent
- 2000 ml Separatory Funnel (500ml if LVI), AMK Glass SFC-0095 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
- 100 ml Graduated Cylinder
- Pasteur 5¾" Disposable Pipettes, Fisher 13-678-20B or equivalent
- Kuderna Danish Flask (500 ml), TEC Glass TG7-01 or equivalent
- Separatory funnel rotator, APR Machine or equivalent
- Analytical Evaporator (N-Evap) Organomation
- Centrifuge, Varifuge F; Hereaus Sepatech
- 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
- Six Position Steam Bath, Fisher 15-496 or equivalent
- Muffle Oven, Thermolyne 6000 or equivalent

6.2.3 Organic Prep Supplies (Soil)

- Weigh boats
- 10, 50 and 200 mL Concentrator tubes.
- Vials, - 1ml, 4ml, and 10ml with PTFE lined snapcaps.
- Glass syringes, 25ul, 50ul, 100ul, 500ul, 1000ul
- Pastuer pipets
- Glass Buchner funnels (stainless steel funnels may be substituted) – Fisher Scientific.
- 500 ml Kuderna-Danish (KD) flask
- Stainless steal spatulas
- Glass wool- Contaminant-free (silane treated or oven baked at 400^{O}C)
- 75mL Teflon Express vessels with stopper and cap (CEM Corp.)
- Glass fiber filter paper - #1 185mm (Whatman)
- Weigh boats
- Tongue depressors, wood
- Electronic Balance, capable of weighing to 0.01g.
- N-Evap Analytical Evaporator
- Fisher 8 Position Steam Bath
- MARS 5 microwave oven with temperature sensor (CEM Corp) capable of sensing temperature within 2.5℃ and adjusting micr owave field output within 2 sec of sensing.
- MARS 40 position carousel (CEM Corp)
- Visiprep DL column holder

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, pesticide grade (BA9254-03 or equivalent)

7.1.2 Acetone, pesticide grade (J.T. Baker BA9264-03)

7.1.3 Sodium sulfate crystals (Mallinckrodt MA8024-06 or equivalent). Sodium sulfate crystals must be baked at 400°C for four hours prior to use.

7.1.4 De-ionized, organic free reagent water

7.1.5 Concentrated Sulfuric acid

7.2 Standards

Note: Stock standards are typically purchased from Supelco, Inc. or Restek. Standards of similar quality from other suppliers may be substituted as required

7.2.1 **Retention Time (RT) Marker Stock Standard:** The RT Marker standard is Supelco TPH Mix 3 (Supelco Catalog Number 861394-U) which contains seventeen (17) individual hydrocarbons in the range of C6 thru C44 at a concentration of 1,000 ug/ml (ppm) each in carbon disulfide (see Table 1 below). Retention time markers are evaluated as discussed in Section 9.2.3.1 below.

Table 1
RT Marker Standard
Individual Components and Concentrations
Supelco TPH Mix 3 (Cat # 861394-U)

Number of Carbons	Compound	Concentration (ppm)
C6	hexane	1000
C7	n-heptane	1000
C8	n-octane	1000
C9	n-nonane	1000
C10	n-decane	1000
C11	n-undecane	1000
C12	n-dodecane	1000
C14	n-tetradecane	1000
C16	n-hexadecane	1000
C18	n-ocatadecane	1000
C20	n-eicosane	1000
C24	n-tetracosane	1000
C28	n-octacosane	1000
C32	n-dotriacontane	1000
C36	n-hexatriacontane	1000
C40	n-tetracontane	1000
C44	n-tetratetracontane	1000

7.2.2 **Calibration and Surrogate Stock Standards:** Calibration standards for DRO are prepared using Restek's Diesel Fuel #2 Composite

Standard (Restek Catalog No. 31259) and Supelco's O-Terphenyl (OTP) (Supelco Catalog No. 4-7580U) which is the surrogate. Calibration standards for Mineral Spirits analysis are prepared using neat Virgin 105 supplied by Safety-Kleen. The stock standards are detailed in Table 2.

Table 2			
Calibration Standard Sources			
Standard Name	**CAS #**	**Source**	**Concentration**
O-Terphenyl (Surrogate) OTP	84-15-1	Supelco Cat. # 4-7580U	10,000 ug/ml
Diesel Fuel #2 Composite Standard	68334-30-5	Restek Cat. # 31259	50,000 ug/ml
Mineral Spirits Blend	8030-30-6	Restek Cat. # 31261	50,000 ug/ml
Virgin 105 (Mineral Spirits)	9072-35-9	Safety-Kleen	50,000 ug/ml*

* 5 grams of Virgin 105 (Mineral Spirit neat standard) into 100 ml of MeCl2

7.2.3 Individual Calibration Standards:

- **DRO:** Five levels of calibration standards are prepared using the Diesel Fuel #2 Composite Standard and the OTP surrogate standard (see Table 2.

- **Mineral Spirits**: Five levels of calibration standards are prepared using the Virgin 105 (Mineral Spirits) and the OTP surrogate standard (see Table 2).

7.2.3.1 The first step is to prepare Level 5 as follows (using either #2 Diesel or Mineral Spirits as appropriate):

Level 5 Calibration Standard Prep				
Final Concentration of #2 Diesel Fuel/Mineral Spirits	**Final Concentration of OTP (Surrogate)**	**Volume of OTP Surrogate Stock used (10,000 ug/ml)**	**Volume of Diesel Fuel #2 Stock/ Mineral Spirits used (50,000 ug/ml)**	**Final Volume in Methylene Chloride**
5000 ug/ml	200 ug/ml	200 uL	1000 uL	10 ml

7.2.3.2 Levels 1-4 are made by way of serial dilutions in volumetric flasks with methylene chloride:

Level 4 is a 2x dilution of Level 5
Level 3 is a 5x dilution of Level 5
Level 2 is a 10x dilution of Level 5
Level 1 is a 10x dilution of Level 2

7.2.3.3 When prepared as detailed above the final analyte and surrogate concentrations of the five initial calibration standards are:

Table 3
Initial Calibration Standard Concentration

Calibration Standard Level	#2 Diesel Fuel Concentration /Mineral Spirits (ug/ml)	OTP (Surrogate) (ug/ml)
1	50	4
2	500	20
3	1000 (mid point)	40
4	2500	100
5	5000	200

7.2.4 **Initial Calibration Verification (ICV) Standard:** An Initial Calibration Verification (ICV) standard is prepared and analyzed for DRO or Mineral Spirits (as appropriate):

- **DRO:** Prepare the ICV using a verified separate lot of the Diesel Fuel #2 Composite Standard (see Table 2). Dilute 200 ul Diesel Fuel #2 Composite Standard to a final volume of 10 ml methylene chloride for a final ICV concentration of 1000 ug/ml (i.e., the same concentration as the midpoint of the calibration).

- **Mineral Spirits:** Prepare the ICV using the Restek Mineral Spirits Blend (see Table 2). Dilute 200 ul of the Mineral Spirits Blend to a final volume of 10 ml methylene chloride for a final ICV concentration of 1000 ug/ml (i.e., the same concentration as the midpoint of the calibration).

7.2.5 **DRO Spiking Solution:** Prepare a 2000 ug/mL DRO Spiking Solution by diluting 4 ml of 50,000 ug/ml Diesel Fuel #2 Composite Standard (Restek Cat. No. 31259) to a final volume of 100 mL methylene chloride using volumetric glassware. This solution is used to spike the MS, MSD, LCS and (if necessary) the LCSD as described in Section 10.1 below.

7.2.6 **Mineral Spirits Spiking Solution:** Prepare 2000ug/ml of Mineral Spirits Spiking Solution by diluting 4ml of 50,000ug/ml Virgin 105 Mineral Spirits (see Table 2) to a final volume of 100mL methylene chloride using volumetric glassware. This solution is used to spike the MS, MSD, LCS and (if necessary) the LCSD as described in Section 10.1 below.

7.2.7 **Surrogate Spiking Solution:** Prepare a 20 ug/mL Surrogate Spiking Solution by diluting 1ml of 10,000 ug/mL O-Terphenyl (OTP) (Supelco Cat. No. 4-7580U) to a final volume of 500mL acetone using volumetric glassware. The solution is used to spike in all samples and associated QC as described in Section 10.1.1below

7.2.8 Standards Prep Documentation: All standards preparation information must be documented in full within the TALS Reagent Module.

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
Water	Glass	1000ml/250 ml	Cool 4±2C	7 Days to prep/40 days to analysis	SW846 Method 8000
Soil	Glass	30 grams	Cool 4 ± 2°C	14 Days to prep/40 days to analysis	SW846 Method 8000

9.0 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	< Rpt. Limit
Laboratory Control Sample aka Blank Spike (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 or Blank Spike 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 designated for MS/MSD is randomly selected by the extraction lab unless specifically requested by a client
[3] Analytical and QC samples (MB, LCS/LCSD, MS/MSD)
[4] Statistical control limits are updated annually.

9.1.1 **Method Blank:** A method blank is a volume of reagent water or a clean matrix that is carried through the entire analytical procedure. The purpose

of the method blank is to determine the levels of contamination (if any) associated with the extraction and analysis of samples.

9.1.1.1 A method blank (spiked with OTP surrogate) must be extracted for every 20 samples (or less), per matrix, per day.

9.1.1.2 For the method blank to be considered acceptable, it must contain less than the reporting limit of DRO. If the method blank does not meet this criterion, then all associated samples must be re-extracted and re-analyzed

9.1.2 **Laboratory Control Sample (LCS)/Laboratory Control Sample Duplicate (LCSD):** also referred to as Blank Spike (BS)/Blank Spike Duplicate (BSD). A Laboratory Control Sample (LCS) is 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. An LCS is analyzed at a frequency of one set per 20 samples. Additionally, an LSCD is analyzed only when insufficient client sample volume is available for the Matrix Spike (MS)/Matrix Spike Duplicate (MSD) or when requested by the client/project/contract.

9.1.2.1 Prepare the appropriate LCS (or LCS/LCSD) spiking solution as applicable and as detailed in Section 7.2.5 (DRO Spiking Solution) or 7.2.6 (Mineral Spirits Spiking Solution). Use the working standards detailed in Section 7.2.2.

9.1.2.2 The LCS data is used to assess performance if the MS/MSD recoveries fall outside of established limits. The recoveries of the LCS must fall within the lab generated acceptance criteria (refer to the active TALS method limit group database for current limits). If the MS/MSD recovery results fall outside the laboratory generated limits, the LCS recovery is evaluated. If LCS recovery is within limits the poor MS/MSD recoveries can be attributed to matrix interference. If the LCS recovery results are outside QC limits, first the LCS extract is re-analyzed and if it is still outside the limits the entire QC batch must be re-extracted and re-analyzed with an acceptable LCS.

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9.1.3 **Matrix Spike (MS)/Matrix Spike Duplicate (MSD):** A Matrix Spike (MS)/Matrix Spike Duplicate (MSD) pair is prepared and analyzed with every 20 client samples of the same matrix.

9.1.3.1 Prepare the appropriate MS/MSD spike solution as applicable and as detailed in Section 7.2.5 (DRO Spiking Solution) or 7.2.6 (Mineral Spirits Spiking Solution). Use the working standards detailed in Section 7.2.2.

9.1.3.2 The percent recovery of the MS/MSD must be calculated and compared against the lab generated limits (refer to the active TALS method limit group database for current limits).

9.1.3.3 If, after, analysis, the recovery the MS/MSD is outside the listed recovery limits, follow the procedures detailed in Section 9.1.2.2.

9.1.4 **Surrogate Recovery:** Surrogate standard (see Sections 7.2.7) is added to all client samples and QC samples prior to extraction (see Sections 10.1.1 and 10.1.2). The percent recovery of the surrogate standards in each sample and QC sample must be calculated and compared against the lab generated limits (refer to the active TALS method limit group database for current limits). (Note: these criteria do not apply when a required sample dilution causes unacceptable surrogate recoveries). If after analysis the recovery of surrogate is outside the listed recovery limits, follow the procedure outlined below:

If, after, analysis, the recovery the surrogate is outside the listed recovery limits follow the procedure outlined below:

9.1.4.1 If a dilution was performed on the extract, remake the dilution and re-analyze.

9.1.4.2 Verify the final volume of the extract is correct and re-inject the extract.

9.1.4.3 If the surrogate fails after re-analysis, re-extract and re-analyze the sample (if sample volume permits).

9.2 Instrument QC

9.2.1 Initial Calibration Range and Initial Calibration Verification

9.2.1.1. **Initial Calibration:** A five point initial calibration range is analyzed prior to the analysis of any samples. The initial calibration standards are prepared as detailed in Section 7.2.3. The initial calibration analyzed and evaluated as described in Section 9.2.3.

9.2.1.2. **Initial Calibration Verification (ICV):** An Initial Calibration Verification (ICV) standard is analyzed immediately after the Initial Calibration Range and before any samples are analyzed. The ICV is prepared as detailed in Section 7.2.4. The ICV must be from a source (or lot) separate from the standards used in the Initial Calibration Range. The ICV is evaluated as detailed in Section 9.2.3.3.

9.2.2 Continuing Calibration Verification (CCV): A mid-point Continuing Calibration Verification (CCV) standard is analyzed every 12 hours or after every 10 environmental samples (whichever is more frequent) and at the end of each analytical sequence. The CCV consists of the 1000 ug/ml midpoint standard analyzed with the initial calibration range (see Section 7.2.3 for details on preparation of this standard). The CCV is evaluated as detailed in Section 9.2.3.4.

9.2.3 Calibration Acceptance Summary

9.2.3.1 Retention Time Windows: Retention time (RT) windows must be established every 24 hours to compensate for minor shifts in absolute retention times as a result of sample loading and normal chromatographic variability as well as to determine the start/end of a given carbon range: C10-C28 ,C10-C44 and Mineral Spirits. All gas chromatographs used for analysis of DRO and Mineral Spirits at TestAmerica Edison are equipped with electronic pressure control (EPC). The use of EPC results in little retention time variability between analyses. Accordingly, retention time variability for the purpose of retention time window determination for standards analysis is extremely small. The default retention time window option must therefore be employed as follows to accommodate the excellent precision of EPC equipped systems.

9.2.3.1.1 Initially (and when necessary thereafter) establish retention time windows by making 3 injections of the 1000 ug/ml calibration standard (which contains 40 ug/ml of o-terphenyl surrogate) over the course of a 72 hour period. Record the retention times for o-terphenyl in each of the 3 injections. Calculate the standard deviation of the three absolute retention times of o-terphenyl. The retention time window is defined as ± 3 times the standard deviation of the absolute retention time. (If, as may be the case, the standard deviation is zero, use ± 0.1 minutes as the default retention time window).

9.2.3.1.2 Retention times must be monitored for each continuing calibration standard, method blank, environmental sample and QC sample. The RT for the o-terphenyl surrogate in the continuing calibration standard must not vary more than 0.1 minutes from the RT of the OTP from the initial calibration. If the RT of the OTP fails to meet this criterion in the continuing calibration standard a new 5-point calibration must be analyzed.

9.2.3.1.3 The RT of the o-terphenyl surrogate in blanks, samples, and QC samples must not vary more than 0.1 minutes from the RT of the o-terphenyl surrogate in the continuing calibration verification (CCV) standard. If the

RT of the o-terphenyl surrogate fails to meet this criterion the samples must be re-analyzed.

9.2.3.1.4 Analyze the Retention Time (RT) Marker Stock Standard (Section 7.2.1) every 24 hours to establish the retention times of the following individual components: C10, C28, C44 and Mineral Spirits. These retention times are used to determine the integration start and stop times for the target carbon ranges C10-C28, C10-C44 and Mineral Spirits.

9.2.3.2 Initial Calibration Range Acceptance Criteria: External standard calibration is employed for this method. After analysis of the 5 point calibration range calculate the response factor for each level by tabulating the total area response of the standard against the amount injected:

$$RF = \frac{\text{Total area of C10-C28 carbon range*/Mineral Spirits**}}{\text{Standard amount}}$$

*Total area is defined as the time window from the initial rise of C10 peak to the end of the elution of C28 peak (area does not include area of peak for ortho-Terphenyl surrogate)

** Total Area is defined as the time window from the initial rise to the end of the of the elution of Mineral Spirits Virgin 105 (does not include area of peak for ortho- Terphenyl surrogate)

9.2.3.2.1. Calculate the % Relative Standard Deviation (% RSD) of the response factors (RF). If the %RSD of the response factors over the working range is $\leq$ 20%, linearity can be assumed and an Initial Calibration Verification (ICV) standard may be analyzed (see Section 9.2.3.3 for ICV evaluation). Employ the same procedure to determine the RF for the OTP (o-Terphenyl) surrogate standard in the ICAL standards.

9.2.3.3 Initial Calibration Verification (ICV): An ICV must be analyzed immediately after the Initial Calibration Range. The ICV consists of a second source (or verified separate lot) standard (see Section 7.2.4 for prep instructions) at the midpoint of the Initial Calibration Range analyzed at the frequency specified in Section 9.2.1. The calculated concentration of the ICV check must meet the criteria of ± 15%D of the expected value. Should the %D exceed 15% the analyst must take corrective action (check the standard solution, perform instrument maintenance, etc.) and re-inject the ICV. If the %D still exceeds 15% after a single ICV reinjection, a new Initial Calibration Range must be analyzed.

9.2.3.4 Continuing Calibration Verification (CCV): A CCV consisting of a standard prepared at 1000 ug/ml (ppm) is analyzed every 12 hours or after every 10 sample injections (whichever is more

frequent). The CCV must meet the criteria of ± 15%D of the expected value. Should the %D exceed 15% the analyst should take corrective action (check standard solution, perform instrument maintenance, etc.) and re-inject the CCV. If the %D still exceeds 15% after a single CCV reinjection, a new Initial Calibration Range must be analyzed.

Instrument QC Summary: Method # 8015B: DRO				
Step	**Standards**	**Type**	**Control Limit**	**Frequency**
Initial Calibration Range	100, 500, 1000, 2500 and 5000 ug/mL	*Average response factor or 1st order linear regression*	*For average RF: <20% RSD. For linear egression: r >0.95*	*As required. Initially and when ICV or CCV do not meet requirement.*
ICV (Initial Calibration Verification)	1000 ppm (2nd source or verified separate lot)	*Average RF*	*±15% Difference*	*Once after each initial calibration*
CCV (Continuing Calibration Verification)	1000 ppm	*Average RF*	*±15% Difference*	*Every 12 hours or 10 samples, whichever is more frequent*

10.0 Procedure

10.1 Sample Preparation

10.1.1 Aqueous Sample Preparation

10.1.1.1 Rinse 2000-ml/500ml separatory funnels and 250-ml Erlenmeyer flasks 3 times with Acetone. Place the funnels on the rotator.

10.1.1.2 Place a small amount of glass wool into a 100-mm funnel and fill with pre-baked sodium sulfate crystals. Rinse 3 times with methylene chloride. Also rinse the outside of the funnel stem 3 times with methylene chloride, as it 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.

10.1.1.3 Mark sample numbers on the separatory funnels with red wax pencil.

10.1.1.4 Make up hang tags with the following information and place on a 250ml Erlenmeyer flask: Fraction (DRO), Lab Sample ID number, Date of extraction

10.1.1.5 Place the 100 ml funnel containing rinsed sodium sulfate crystals onto the 250ml Erlenmeyer flask.

10.1.1.6 Mark the fluid level on sample bottles with a black magic marker.

10.1.1.7 Rinse out a 1000 ml graduated cylinder 2 to 3 times with deionized water .Obtain 1000 ml/250ml of deionized organic free water for the Method Blank and the LCS from the Millipore filtering apparatus located in the Organic Extractions laboratory.

10.1.1.8 Pour each sample into its corresponding separatory funnel.

10.1.1.9 Rinse syringes 8 to 10 times with Methylene Chloride.

10.1.1.10 Add 1ml /250ul of the Surrogate Standard solution (see Section 7.2.7) to each separatory funnel.

10.1.1.11 If extracting QC samples (MS, MSD, and/or LCS/LCSD), add 1ml of the DRO Spiking Solution (Sec. 7.2.5) or Mineral Spirits Spiking Solution (Sec. 7.2.6) to the corresponding separatory funnel. (When spiking, ensure that all bubbles are out of the syringe. In addition, hold the syringe just above the level of the liquid when adding the spike. Don't touch the tip of the syringe to the liquid or the side of the separatory funnel).

10.1.1.12 Add 60/20ml of Methylene Chloride to each sample bottle.

10.1.1.13 Swirl the bottle and add the 60-ml/20ml of Methylene Chloride to its corresponding separatory funnel.

10.1.1.14 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.

10.1.1.15 Allow the aqueous sample to separate from the Methylene Chloride (the Methylene Chloride layer is the bottom layer; the water layer is on top).

10.1.1.16 Drain Methylene Chloride (the bottom layer) from the separatory funnel into the funnel/Erlenmeyer flask apparatus.

10.1.1.17 Repeat steps 10.1.1.13 through 10.1.1.16 twice, adding the Methylene Chloride directly to separatory funnel, rather than rinsing the sample container as in 10.1.1.12

10.1.1.18 After the last of the three Methylene Chloride aliquots has been transferred to the Erlenmeyer flask, the water sample remaining in each separatory funnel may be discarded.

10.1.1.19 Retrieve the empty sample bottles that were marked with a black magic marker. Fill with tap water up to the black water level line made on the bottle per 10.1.1.6. Pour the water into a 1000 graduated cylinder. Record the volume on the Organic Extraction Data Sheet in the 'Initial Sample Volume' field.

10.1.1.20 Assemble a KD/Snyder column/concentration tube apparatus for each sample. Rinse each KD concentration tube apparatus and a Snyder column three times with Acetone.

10.1.1.21 Pour the Methylene Chloride from each Erlenmeyer flask into a separate KD/ concentration tube apparatus. Rinse each Erlenmeyer flask twice with Methylene Chloride and pour both rinsates into the corresponding KD apparatus. Transfer the hang tag from the Erlenmeyer flask to the correct corresponding KD apparatus.

10.1.1.22 Attach the Snyder column to the top of the KD apparatus.

10.1.1.23 Concentrate the sample to approximately 5-ml in Steam Bath.

10.1.1.24 Remove the Snyder column from the top of the KD flask. Remove 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.

10.1.1.25 <u>If an emulsion forms during extraction:</u> Rinse a centrifuge tube well with Methylene Chloride. Drain the Methylene Chloride layer (i.e., the bottom layer) from the separatory funnel into the tube. Centrifuge for 3 to 5 minutes at 2000 rpm. (Ensure that the centrifuge is balanced before starting. The levels of the samples directly opposite one another in the centrifuge should be approximately equal.) After the centrifuging process is completed, there will be two layers: water on top and the Methylene Chloride with the desired sample extract on the bottom. Using a 1 ml disposable pipette transfer the Methylene Chloride (bottom layer) from the centrifuge tube to the corresponding Erlenmeyer flask. Take care not to transfer any of the top layer. The top layer that remains is poured back into the 2000 ml separatory funnel with the rest of the original sample.

10.1.2 Solid Sample Preparation

10.1.1 15 grams of soil sample (DRO) is weighed out into a 75mL Teflon microwave vessel, the weight is recorded to two significant figures.

10.1.1.1 Where practical samples should be air dried and ground prior to extraction. Drying should be performed in a hood to avoid contamination.

10.1.1.2 Samples shall be mixed with sodium sulfate prior to extraction.

10.1.2 Decant and discard any water layer on a sediment sample. Discard any foreign objects such as rocks, leaves and sticks. Mix sample thoroughly prior to subsampling.

10.1.3 For laboratory blanks and control spikes, an equal portion of clean sand is prepped along with samples.

10.1.4 All samples, blanks, and spikes are fortified with 1ml surrogates. All matrix spikes and lab control spikes are fortified with 1ml spiking solution. See section 7.2. **NOTE:** When spiking the samples, it is critical to remove all bubbles from the syringe.

10.1.5 Add 30 ml of methylene chloride to each sample vessel depending upon the fraction:

10.1.6 A stopper is placed over the opening along with the vessel cap. The vessel is then tightened using the MARS capping wrench. This capping wrench tightens the cap to the appropriate torque.

10.1.7 The sample vessels are placed into the 40-position carousel. Each position has an insulator sleeve that the vessel slides into – slide the vessel into the appropriate sleeve – and press firmly down to ensure a complete connection.

10.1.8 Place the carousel (with samples and vessels) into the microwave unit.

Start Run: (load method – using key pad on face of unit)
Press: Home
Press Select: load method
Press Select: from user directory
Press Select: 3546 Microwave – Xpress method
Press Start.

Microwave Operating Parameters				
Power Max	**Power %**	**Oven Ramp**	**Degrees Celsius**	**Hold Time**
1200W	100	10.0 minutes	110	10.0 minutes
1600W[1]	100	10.0 minutes	130	10.0 minutes
1600W[2]	100	20.0 minutes	115	10.0 minutes

[1] NJDEP Method EPH 10/09 samples only

[2] For DRO,pesticide,PCB and BNA samples only

10.1.9 A 5-minute cool down will close out the run.

10.1.10 Remove vessels from carousel and allow cooling (either at room

temperature or in a cold water bath).

10.1.11 When cool down is complete, remove carousel from microwave.

10.1.12 Pre-wash a funnel filled with sodium sulfate with three successive washings of 30 ml acetone. Pour the extract carefully through the sodium sulfate funnel (to remove any moisture) into the 200mL concentrator tube. Rinse the 75mL vessel with 10 mL methylene chloride three times to ensure quantitative transfer.

10.1.13 Set the water temperature in the concentrator to 35℃ and adjust the pressure to around 5psi of nitrogen.

10.1.14 Place the tube into the apparatus and slowly raise the pressure to 20psi. If splashing occurs, start with a lower pressure and raise it when the solvent gets lower.

10.1.15 Concentrate to just under the 1mL mark on the nipple of the tube. Remove from the bath and transfer to an auto sampler vial with a glass Pasteur pipette. See Attachment 1.

10.1.16 Add a few drops of clean methylene chloride to the tube, rinsing the narrow part and add to the vial to adjust to 1mL and return to concentrator set at 35℃.

10.1.17 Total concentration time should be approximately 20 minutes for methylene chloride. If a sample takes a longer than usual amount of time to concentrate or if the extract becomes viscous the final volume should be higher.

10.1.18 The extract is now ready for the appropriate analysis.

10.1 Analytical Procedure

10.2.1 Instrument Setup: Prior to calibration and analysis the instrumentation detailed in section 6.1 must be setup with the following operation conditions:

10.2.1.1 Injection System: A splitless injection port with electronic pressure control (EPC) is used. Fifty seconds after sample injection, the purge valve is turned on to facilitate the sweeping of any remaining residual solvent/sample from the injection port. The EPC is used in the ramp pressure mode. The ramp constant pressure program is setup as described in Attachment 3 - DRO Temperature Program.

10.2.1.1.1 The injection port liner used is a 4mm splitless sleeve (Restek Catalog No. 20782-211.5). This liner/glass wool combination performs several functions. The glass wool serves as a heat sink rapidly vaporizing solvent and

samples resulting in higher response factors. The liner also protects the column head from accumulation of high boiling residuals and particulates.

10.2.1.1.2 Regular maintenance is performed on the injection port. Prior to performing maintenance the injection port, oven and detector temperatures are lowered to ambient prior to "cracking" the system. This is so as to introduce a minimum of damaging oxygen molecules into the system.

10.2.1.1.3 After the system has cooled, the old liner is removed. The injection port should be checked for particulate residues and cleaned as needed. A flashlight is usually required for this. After a new liner has been prepared it is placed into the injection port. A graphite seal is placed around the liner. The edges of the seal must be flat, not knife-edged, and free of nicks or burrs. If any of these conditions are not met, the graphite seal must be replaced as well. The graphite seal is critical to proper operation of the injection port. If in doubt, replace it.

10.2.1.1.4 The locking ring on the top of the injection port should be turned, with the wrench, about 1/8 turn past finger tight. The septum nut should never be tightened more than finger tight. After the injection port is reassembled, all column nuts inside the oven should be checked for leaks using Snoop (Supelco) or another suitable leak tester.

10.2.1.1.5 The septa should be changed each time the injection port is opened. Another routine maintenance operation to improve column performance is the removal of the first 3 cm of the column. Document all such column maintenance in the instrument maintenance log.

10.2.1.1.6 Once the signal from both detectors has stabilized, it is time to re-heat the zones. The zones should be heated in the order of detectors, oven and then injectors. This is to ensure that volatilized contaminants do not condense on the column or detector.

10.2.1.2 **Oven**: Temperature programming is employed to achieve higher resolution of compounds and shorter run times than could be accomplished using isothermal methods. The oven temperature program and pressure ramping for DRO and Mineral Spirits analysis is employed for all columns as detailed in Attachment 8 – DRO/Mineral Spirits Temperature Program.

10.2.2 **Calibration:** Refer to Section 9.2 for instrument calibration requirements.

10.2.3 Sample Analysis

10.2.3.1 Once all Initial and Continuing Calibration criteria are satisfied, analysis of samples may proceed.

10.2.3.2 Analyze all method blanks, samples and associated QC samples, remembering to analyze the CCV after every 10 samples (or every 12 hours) and at the end of the sequence.

10.2.3.3 An idealized analytical sequence is presented in the table below:

Idealized Analytical Sequence with Initial Calibration	
1. RT Marker (run every 24 hours) 2. Instrument Blank 3. Initial Calibration Level - 1 4. Initial Calibration Level - 2 5. Initial Calibration Level - 3 6. Initial Calibration Level - 4 7. Initial Calibration Level - 5 8. ICV 9. Instrument Blank 10. CCV 11 thru 20. Ten samples*	21. Instrument Blank 22. CCV 23. thru 32. Ten samples 33. Instrument Blank 34. CCV 35-44. Ten samples 45. Instrument Blank 46. CCV (end of sequence)

*May be up to 10 samples or 12 hours from the start of the calibration verification standards

10.2.3.4 If the sample concentration exceeds that of the high point of the calibration range, the extract must be diluted and re-analyzed within range. NOTE: Methylene chloride blanks should also be analyzed after a sample suspected to be highly concentrated to prevent carryover.

10.2.3.5 Retention times must be monitored for each Continuing Calibration Verification (CCV) standard, method blank, sample and associated QC sample. The retention time for the OTP surrogate must meet the specifications detailed in Section 9.2.3.1.

10.2.3.6 The acceptability of the calibration must be verified after every 10 samples (or every 12 hours) and at the end of each analytical sequence by analysis and evaluation of the Continuing Calibration Verification (CCV) standard. If the % difference is $\leq$15% from the range, sample analysis may

begin. If the response varies by more than 15%, a new calibration curve must be analyzed. For more details on the CCV acceptability see Section 9.2.3.4.

10.2.4 Routine GC Maintenance

10.2.4.1 During the course of calibration and analysis it may be necessary to perform routine maintenance activities on the GC injection port system and the analytical column. This section details the procedures for routine maintenance which may include:

- Replacement of the injection port liner;
- Cleaning of the injection port;
- Replacement of the septum;
- Replacement of the gold inlet seal;

10.2.4.2 Cool the inlet. This is best accomplished by reducing the inlet temperature to 40C. Leaving the inlet on allows the injection port fan to continue to operate thus, aiding in cooling the injection port. It is also helpful to lower the oven temperature to 40C. These steps can be performed from the GC front panel or the Chemstation software.

10.2.4.3 If an autosampler is in place, remove tower, tray and top cover.

10.2.4.4 While wearing appropriate safety apparel, remove the weldment assembly that covers the GC liner.

10.2.4.5 Turn the GC oven off and open the door. Loosen and remove the GC column nut from the inlet. Place a septa over the injection port end of the column, so as NOT introduce O2 to the column. Remove the insulator and the gray reducing nut that houses the gold seal and washer from the bottom of the inlet.

10.2.4.6 The injection port should be checked for particulate residues and cleaned as needed (use a brush, methylene chloride, methanol and a Kimwipe as needed).

10.2.4.7 Prepare a new liner and place it into the injection port. A graphite seal is placed around the liner. The edges of the seal must be flat, not knife-edged, and free of nicks or burrs. If any of these conditions are not met, the graphite seal must be replaced as well. The graphite seal is critical to proper operation of the injection port. If in doubt, replace it

10.2.4.8 Replace the septum and gold seal.

10.2.4.9 The locking ring on the top of the injection port should be turned, with the wrench, about 1/8 turn past finger tight. The septum nut should never be tightened more than finger tight.

10.2.4.10 If required, clip 3cm from the injection port end of the column and reinstall the column. All column nuts inside the oven should be checked for leaks using Snoop or another suitable leak tester.

10.2.4.11 The injection port and oven zones should be heated in the order of detectors, oven and then injectors. This is to ensure that volatilized contaminants do not condense on the column or detector.

10.2.4.12 Document all maintenance activity in the appropriate Instrument Maintenance Logbook.

11.0. Calculations / Data Reduction

11.1 Calculate the concentration of the DRO organics in the sample using a summation of peak response for all chromatographic peaks in the time window within which the target analytes elute. Use the following equations:

11.1.1 Aqueous Samples:

DRO C10-C28 :

$$\text{Concentration mg/L} = \frac{(Ax)\ (Vt)\ (D)}{(RF)\ (Vs)}$$

Where: Ax = sum of area for DRO (C10-C28) range organics in the sample
RF = response factor from initial calibration standard
Vt = final volume of extract (ml)
Vs = initial volume of sample extract (L)
D = dilution factor

DRO C10-C44 :

$$\text{Concentration mg/L} = \frac{(Ax)\ (Vt)\ (D)}{(RF)\ (Vs)}$$

Where: Ax = sum of area for DRO (C10-C44) range organics in the sample
RF = response factor from initial calibration standard
Vt = final volume of extract (ml)
Vs = initial volume of sample extract (L)
D = dilution factor

Mineral Spirits :

$$\text{Concentration mg/L} = (Ax)\ (Vt)\ (D)$$

(RF) (Vs)

Where: Ax = sum of area for Mineral Spirits in the sample
RF = response factor from initial calibration standard
Vt = final volume of extract (ml)
Vs = initial volume of sample extract (L)
D = dilution factor

11.1.2 Solid samples

DRO C10-C28 :

$$\text{Concentration mg/kg.} = \frac{(Ax)\ (Vt)\ (D)}{(RF)\ (Vs)\ (Dw)}$$

Where: Ax = sum of area for DRO (C10-C28) range organics in the sample
RF = response factor from initial calibration standard
Vt = final volume of extract (ml)
Vs = initial weight of sample extract (mg)
D = dilution factor
Dw = Dry weight correction factor $\frac{(100 - \%\ \text{moisture})}{100}$

DRO C10-C44 :

$$\text{Concentration mg/kg.} = \frac{(Ax)\ (Vt)\ (D)}{(RF)\ (Vs)\ (Dw)}$$

Where: Ax = sum of area for DRO (C10-C44) range organics in the sample
RF = response factor from initial calibration standard
Vt = final volume of extract (ml)
Vs = initial weight of sample extract (mg)
D = dilution factor
Dw = Dry weight correction factor $\frac{(100 - \%\ \text{moisture})}{100}$

Mineral Spirits :

$$\text{Concentration mg/kg.} = \frac{(Ax)\ (Vt)\ (D)}{(RF)\ (Vs)\ (Dw)}$$

Where: Ax = sum of area for Mineral Spirits in the sample
RF = response factor from initial calibration standard
Vt = final volume of extract (ml)
Vs = initial weight of sample extract (mg)
D = dilution factor

$$Dw = \text{Dry weight correction factor } \frac{(100 - \%\ \text{moisture})}{100}$$

NOTE: All dry weight corrections are made in LIMS at the time the final report is prepared.

11.1. Accuracy:

$$\text{ICV / CCV, LCS \% Recovery} = \frac{\text{observed concentration}}{\text{known concentration}} \times 100$$

$$\text{MS \% Recovery} = \frac{(\text{spiked sample}) - (\text{unspiked sample})}{\text{spiked concentration}} \times 100$$

11.2. Precision (RPD):

$$\text{Matrix Duplicate (MD)} = \frac{|\text{orig. sample value} - \text{dup. sample value}|}{[(\text{orig. sample value} + \text{dup. sample value})/2]} \times 100$$

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.

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

15.0 References / Cross-References

15.1 United States Environmental Protection Agency, "Method SW8015B, Non-Halogenated Organics , Test Methods for Evaluating Solid Wastes, SW846 Third Edition, Volume 1B: Laboratory Manual, Physical/Chemical Methods, Revision 3, December 1996.

15.2 United States Environmental Protection Agency, "Method SW8000B, Determinative Chomatographic Determinations.", Test Methods for Evaluating Solid Wastes, SW846 Third Edition, Volume 1B: Laboratory Manual, Physical/Chemical Methods, Revision 3, December 1996.

15.3 TestAmerica Edison Document No. ED-QA-LQM, *Laboratory Quality Manual*, most current revision.

15.4 TestAmerica Edison Work Instruction No. EDS-WI-019, *EPA Method 8015B DROs 8-44 Current QC Limits*, current revision.

15.5 TestAmerica Corporate Quality SOP No. CA-Q-S-001 (*Solvent & Acid Lot Testing Approval*)

15.6 TestAmerica Edison SOP No. ED-GEN-023 (*Bulk Solvent Testing and Approval*), urrent revision.

15.7 TestAmerica Environmental Health and Safety Manual, CW-E-M-001.

15.8 TestAmerica SOP No. ED-GEN-022, Training, current revision.

15.9 TestAmerica Edison SOP No. ED-GEN-008, Standard Operating Procedure for reparation, Purity and Storage of Reagents and Standards, current revision

15.10 TestAmerica Edison SOP No. ED-SPM-007 (*Disposal of Samples and Associated Laboratory Waste,* current revision.

15.11 TestAmerica Edison SOP No. ED-SPM-008 (*Laboratory Waste Disposal Procedures,* current revision.

15.12 TestAmerica Edison SOP No. ED-ORP-044, *Procedures for Microwave Extraction of Soilds,* current revision.

16.0 Method Modifications:

N/A

17.0 Attachments

- **Attachment 1:**

Example Certificate of Analysis TPH Mix 3 Supelco Catalog # 861394-U

- **Attachment 2:**

Example Certificate of Analysis, Diesel Fuel #2 Composite Standard
Restek Catalog # 31259

- **Attachment 3:**

Example Chromatogram, Diesel Fuel #2 Composite Standard Restek Catalog # 31259

- **Attachment 4:**

Example Certificate of Analysis, Mineral Spirits Standard Restek Catalog # 31261

- **Attachment 5:**

Example of Chromatogram , Mineral Spirits Standard Restek Catalog # 31261

- **Attachment 6:**

Chain of Custody, Virgin 105 Solvent provided by Safety-Kleen Systems, Inc

- **Attachment 7 :**

Example of Chromatogram, Virgin 105 Solvent provided by Safety-Kleen Systems, Inc

- **Attachment 8:**

GC Temperature Program

18.0 Revision History

Revision 13, 17 August, 2016

- Revised title to remove SW846 3541 as a prep method.
- Updated section 1.1: removed reference to SW3541 and added microwave instructions to this SOP.
- Section 6.1.3: Revised analytical column used (now Restek Rtx-Mineral Oil)
- Section 6.2.2: added glassware required for LVI prep.
- Section 6.2.3: replaced soxtherm equipment with microwave equipment.
- Section 8.0: added option for 250ml amber glass container.
- Section 10.1: replaced full volume aqueous prep instructions with LVI aqueous prep instructions. Replaced soxtherm soil prep instructions with microwave soil prep instructions.
- Section 15: removed SW3541 from list of references.

Revision 12, July 7, 2014

- Section 1.1: Revised the Reporting Limit (RL) for Mineral Spirits (Soils RL is now 3.3 mg/kg; Aqueous RL is now 0.05 mg/L)..
- Section 7.2.3.2: Revised the preparation instructions for the low level standard (Level 1) such that is supports the newly revised RLs.
- Section 7.2.3.3: Table 3: revised the concentration of #2 Diesel Fuel/Mineral Spirits (ug/ml) to 50.

Revision 11, April 10, 2014

- Revised title of SOP to include analysis for Mineral Spirits.
- Throughout document: revisions to included analysis for and quantitation of Mineral Spirits.
- Section 6.1.3: updated and added additional detail concerning the analytical column (Restek Rtx-5ms Part Number: 565647 or equivalent).
- Added Section 6.1.6: added following equipment necessitated by the change of carrier gas from helium to hydrogen: Hydrogen Generator, Parker Hannifin Corporation or equivalent (part number H2PEMPD-510-100)

- Section 7.2.2 and Table 2: revised to include reference to stock Mineral Spirits standard as well as prep instructions. Added reference to Mineral Spirits in subsequent tables/text as necessary.
- Added new Section 7.2.6 which describes the preparation of a Mineral Spirits Spiking Solution. Subsequent sections renumbered accordingly.
- Sections 9.1.2 and 9.1.3: revised to include a reference to Mineral Spirits Spiking Solution.
- Section 9.2.3.1 revised to include requirement to establish RT windows every 24 hours.
- Sections 10.1.11 revised to include QC spiking instructions for Mineral Spirits.
- Added new Section 10.1.2.8 which describes soil QC spiking instructions for both DRO and Mineral Spirits. Subsequent sections renumbered accordingly.
- Section 11: added calculations (water and solid) for Mineral Spirits.
- Updated Temperature Program attachment and renamed it to Attachment 8.
- Added new Attachments 4 thru 7 (related to Mineral Spirits standards).

Revision 10, June 11, 2013

- This SOP (ED-GCS-009) which has historically covered analysis of SW8015C DRO C10-C44 is, effective with this revision, being combined with SOP No. ED-GCS-008 which covered analysis of SW8015C DRO C10-C28. SOP No. ED-GCS-008 has been retired. The SOP has been re-titled to reflect the change and revisions have been made throughout the document to combine the procedures as required.
- Revised throughout to update Lab Quality Manual section references.
- Section 1.1: updated RLs as needed.
- Section 4.0: Removed references to phthalate ester contaimination. Clarified baseline subtraction language.
- Section 6.1.5: Deleted references to Target data processing software. Replaced with references to Chrom software.
- Section 7.2.1: Added details for new RT Marker Standard. All other subsection numbers adjusted accordingly.
- Section 7.2.3: Revised to include details of the new calibration stock standard (#2 Diesel Fuel Composite Standard) and individual calibration standard prep.
- Section 7.2.4: updated to include a new source for the ICV.
- Section 7.2.5: updated to include a new source and prep instructions for the DRO Spiking Solution.
- Section 7.2.6: added to include preparation instructions for the Surrogate Spiking Solution.
- Section 7.2.7: revised the standards preparation documentation section to replace the standard preparation logbook with the TALS Reagent Module.
- Section 9.2.1.2: added a reference to the SOP section describing ICV evaluation procedures.
- Section 9.2.2: updated to include a reference to the new CCV standard (midpoint of ICAL detailed in Section 7.2.3).
- Sections 9.2.3.: renamed this section ‘Initial Calibration Range Acceptance Criteria’. Updated the carbon range with which the RF is calculated (now C10-C28).
- Section 9.2.3.1: Revised standard concentration to 1000 ug/ml (was 3250 ug/ml). Added discussion of establishing integration start/end times for C10-C28 and C10-

C44. Added Section 9.2.3.1.4 which discusses daily analysis of RT Marker standard.
- Section 9.2.3.2 and 9.2.3.3: updated to include new standards information. Revised Instrument QC Summary Table to reflect current calibration levels.
- Section 10.2.3.3: updated the Idealized Analytical Sequence with Initial Calibration table.
- Section 11.1: Expanded to include calculations for both ranges (C10-C28 and C10-C44).
- Section 15, References: updated with Microwave prep SOP.
- Section 17: updated all Attachments.

Revision 9, August 31, 2011
- Section 1.1: Added note identifying location of current MDLs/RLs in TALS database.
- Section 3.0: revised to identify current location of 'Definitions'.
- Deleted Section 6.1.2.1.1: deleted. Text did not apply to GC supplies.
- Section 7.1: added note detailing solvent approval procedures.
- Section 7.2.3.1: Revised table to include final concentration of OTP surrogate in each calibration standard.
- Section 9.1.2.2: removed obsolete reference to work instruction and replaced with reference to TALS Method Limit Group.
- Section 9.1.3.2: removed obsolete reference to work instruction and replaced with reference to TALS Method Limit Group.
- Section 9.1.3.2: removed obsolete reference to work instruction and replaced with reference to TALS Method Limit Group.
- Section 9.2.2.1: corrected reference to Section number detailing the prep of the initial calibration standards. Added language describing the determination and treatment of area response due to normal column bleed.
- Section 9.2.2: Removed in appropriate reference to DFTPP tuning standard. Added text correcting the frequency of CCV analysis.
- Added Sections 9.2.3.1.1 through 9.2.3.1.3 which provide more detail on the establishment and monitoring of Retention Time Windows.
- Deleted Section 10.1.2.1 which is not applicable to soils prep. Deleted text: "Rinse 2000-ml separatory funnels and 250-ml Erlenmeyer flasks 3 times with Acetone. Place the funnels on the rotator."
- Expanded Section 10.2.1 to include more details on Instrument Setup.
- Section 10.2.2: deleted all text and included a reference to Section 9.2 for instrument calibration requirements.
- Section 11.1: Expanded to include details on both soil and water calculations.
- Added Attachment 1:Example Standard Chromatogram, TPH Mix 3 Supelco Catalog # 861394-U
- Added Attachment 2: Example Certificate of Analysis, TPH Mix 3 Supelco Catalog # 861394-U
- Added Attachment 3:DRO Temperature Program

Revision 8, December 2, 2008:

- Updated SOP into TestAmerica format in order to meet the requirements of Corporate Quality SOP # CW-Q-S-002 (Rev. O): *Writing a Standard Operating Procedure (SOP).*

- Revised SOP Title as follows: “Preparation and Analysis of Diesel Range Organics (DRO C10-C44) in Soil and Water Samples by SW846 Methods 3510C (Separatory Funnel), 3541 (Automated Soxhlet Extraction) and 8015B (GC/FID)”

- Revised Section 1.0 (Scope and Application) to include reporting limit (RL) info for both solid and aqueous matrices. Additionally, this section was revised to include details on the procedures required when clients request modifications to this SOP.

- Revised Section 3.0 (Definitions) to reflect the change in location for TestAmerica Edison’s standard definitions (from SOP No. ED-GEN-018 to the lab Quality Assurance Manual).

- Added additional detail to Section 6.0 (Instrumentation & Supplies), Section 7.0 (Reagents & Standards) & Section 8.0 (Sample Collection, Preservation, Shipment & Storage).

- Added the requirement of an Initial Calibration Verification from a source independent of the initial calibration standards (see Section 9.2, Instrument QC).

- Substantially revised the method GC conditions (see Section 10.2.1)

- Added additional detail to the Soxtherm operating conditions (see Section 10.1.2, Solid Sample Preparation).

- Included Routine GC Maintenance in separate section (see Section 10.2.4) and added additional detail.

- Updated Section 15.0 (References/Cross-References) to include all of the applicable methods, the Lab Quality Manual and TestAmerica Edison’s Training SOP.

- Added this Revision History Section 18.

Attachment 1
Example Standard Chromatogram
TPH Mix 3
Supelco Catalog # 861394-U

Certificate of Analysis

DESCRIPTION: TPH Mix 3

CATALOG NO.: 861394-U MFG DATE: Jan-2012

LOT NO.: LB90337 EXPIRATION DATE: Jan-2015

SOLVENT: CARBON DISULFIDE

ANALYTE (1)	CAS NUMBER	PERCENT PURITY(2)	WEIGHT(3) CONCENTRATION	ANALYTICAL(4) CONCENTRATION		STD DEV	SUPELCO LOT NO
HEXANE	110-54-3	99.9	1000	1005	+/-	22.0	LB84384
N-DECANE	124-18-5	99.9	1000	979	+/-	40.3	LB79197
N-DODECANE	112-40-3	99.8	1000	1001	+/-	48.9	LB78955
N-DOTRIACONTANE	544-85-4	98.9	1000	961	+/-	43.3	LB76972
N-EICOSANE	112-95-8	99.9	1000	971	+/-	43.2	LB79677
N-HEPTANE	142-82-5	99.5	1000	988	+/-	51.5	LB67552
N-HEXADECANE	544-76-3	99.5	1000	961	+/-	42.6	LB87053
N-HEXATRIACONTANE	630-06-8	99.9	1000	997	+/-	44.8	LB84585
N-NONANE	111-84-2	99.9	1000	971	+/-	40.9	LB41696
N-OCTACOSANE	630-02-4	99.9	1000	978	+/-	44.2	LB85517
N-OCTADECANE	593-45-3	98.1	1000	949	+/-	42.1	LB77591
N-OCTANE	111-65-9	99.4	1000	957	+/-	28.2	LB63797
N-TETRACONTANE	4181-95-7	98.5	1000	956	+/-	42.2	LB81306
N-TETRACOSANE	646-31-1	99.9	1000	969	+/-	43.6	LB75056
N-TETRADECANE	629-59-4	99.7	1000	977	+/-	43.1	LB80294
N-TETRATETRACONTANE	7098-22-8	97.1	1000	966	+/-	43.4	LB71768
N-UNDECANE	1120-21-4	99.9	1000	995	+/-	45.2	LB78475

(1) Listed in alphabetical order.
(2) Determined by capillary GC-FID, unless otherwise noted.
(3) NIST traceable weights are used to verify balance calibration with the preparation of each lot. Concentration of analyte in solution is ug/ml +/- 0.5%, uncertainty based upon balance and Class A volumetric glassware. Weights are corrected for analytes less than 98% pure.
(4) Determined by chromatographic analysis against an independently prepared reference lot. Mean of replicate injections.

Elwood Doughty
Quality Control Supervisor

Supelco warrants that its products conform to the information contained in this publication. Purchaser must determine the suitability of the product for its particular use. Please see the latest catalog or order invoice and packing slip for additional terms and conditions of sale.

SUPELCO Analytical
595 North Harrison Road
Bellefonte, PA 16823-0048 USA
Phone (814) 359-3441

Attachment 2
Example Certificate of Analysis
Diesel Fuel #2 Composite Standard
Restek Catalog # 31259

RESTEK

110 Benner Circle
Bellefonte, PA 16823-8812
Tel: (800)356-1688
Fax: (814)353-1309

www.restek.com

ACCREDITED ISO Guide 34 Accredited Reference Material Producer Certificate #3222.01

ilac-MRA

ACCREDITED ISO/IEC 17025 Accredited Testing Laboratory Certificate #3222.02

Certificate of Analysis

FOR LABORATORY USE ONLY-READ MSDS PRIOR TO USE.

This Reference Material is intended for Laboratory Use Only as a standard for the qualitative and/or quantitative determination of the analyte(s) listed.

Catalog No. : 31259 **Lot No.:** A092394

Description : Diesel Fuel #2 Composite Standard

Diesel Fuel #2 Std 50,000µg/mL, Methylene Chloride, 5mL/ampul

Container Size : 5 mL **Pkg Amt:** > 5 mL

Expiration Date : January 2020 **Storage:** 25°C nominal

CERTIFIED VALUES

Elution Order	Compound	Grav. Conc. (weight/volume)	Expanded Uncertainty (95% C.L.; K=2)		
1	Diesel Fuel #2 Composite	50,005.2 µg/mL	+/- 290.7193	µg/mL	Gravimetric
	CAS # 68334-30-5		+/- 2,016.5678	µg/mL	Unstressed
	Purity ----%		+/- 2,068.3786	µg/mL	Stressed

Solvent: Methylene Chloride
CAS # 75-09-2
Purity 99%

Page 2 of 6

Attachment 3
Example Chromatogram
Diesel Fuel #2 Composite Standard
Restek Catalog # 31259

Column:
30m x .25mm x .25um
Rtx-5 (cat.#10223)

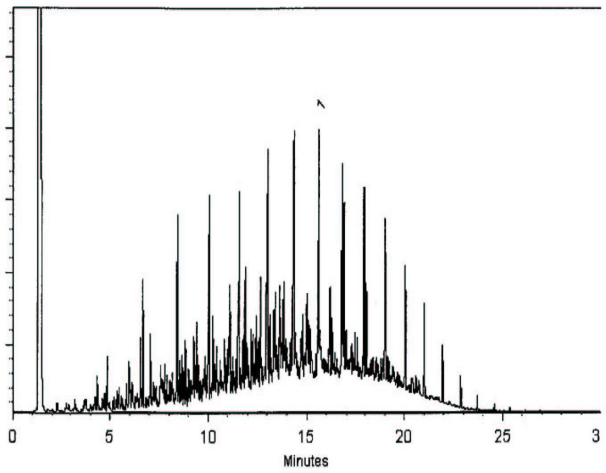
Carrier Gas:
hydrogen-constant pressure 10 psi.

Temp. Program:
40°C (hold 2 min.) to 330°C
@ 10°C/min. (hold 10 min.)

Inj. Temp:
250°C

Det. Temp:
330°C

Det. Type:
FID



Jennifer L. Pollino - QC Analyst

Date Passed: 13-Dec-2012 **Balance:** 1128342314

Manufactured under Restek's ISO 9001:2008
Registered Quality System
Certificate #FM 80397

Attachment 4:
Example Certificate of Analysis, Mineral Spirits Standard Restek Catalog # 31261

RESTEK® CERTIFIED REFERENCE MATERIAL

110 Benner Circle
Bellefonte, PA 16823-8812
Tel: (800)356-1688
Fax: (814)353-1309

www.restek.com

ACCREDITED
ISO Guide 34 Accredited Reference Material Producer Certificate #3212.01

ilac-MRA

ACCREDITED
ISO/IEC 17025 Accredited Testing Laboratory Certificate #3212.02

Certificate of Analysis

FOR LABORATORY USE ONLY-READ SDS PRIOR TO USE.

This Reference Material is intended for Laboratory Use Only as a standard for the qualitative and/or quantitative determination of the analyte(s) listed.

Catalog No.: 31261 **Lot No.:** A098768

Description: Mineral Spirits standard

Mineral Spirits Std 50,000µg/mL, Methylene Chloride, 5mL/ampul

Container Size: 5 mL **Pkg Amt:** > 5 mL

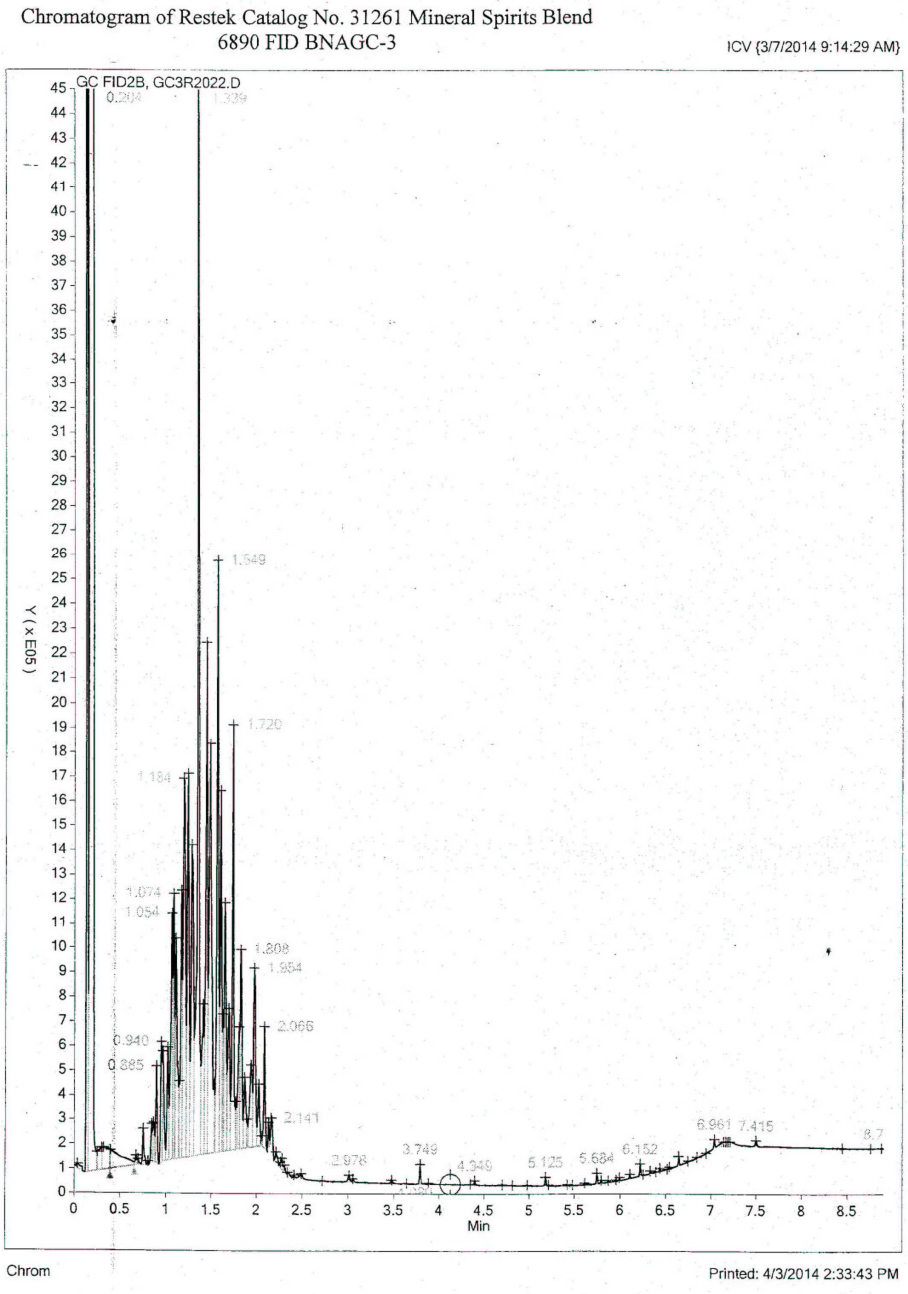
Expiration Date: November 30, 2020 **Storage:** 25°C nominal

CERTIFIED VALUES

Elution Order	Compound	Grav. Conc. (weight/volume)	Expanded Uncertainty (95% C.L.; K=2)	
1	Mineral Spirits Blend CAS # 8030-30-6 (Lot A083767) Purity ---%	50,002.0 µg/mL	+/- 292.7727 µg/mL +/- 1,996.1078 µg/mL +/- 2,048.4295 µg/mL	Gravimetric Unstressed Stressed

Solvent: Methylene Chloride
CAS # 75-09-2
Purity 99%

Attachment 5:
Example of Chromatogram , Mineral Spirits Standard Restek Catalog # 31261



Attachment 6:
Virgin 105 Solvent provided by Safety-Kleen Systems, Inc

Safety-Kleen

Chain Of Custody

Safety-Kleen Systems, Inc.
Telephone: 847.468.6713
Fax: 847.468.6674

Branch: Please complete and fax to the EHS Manager or NCWS for approval.

Branch Information

Branch Name: Safety-Kleen	Branch Number: 2-118-08	Branch Fax Number:	(631) 842-1524
Branch Address: 60 Seabro Ave	N. Amityville, NY 11701	Branch General Manager:	George Paul
Branch Phone Number:	(631) 842-6311	Branch EHS Manager:	Terri Cowans

Sample Information

	SAMPLE ID #	INCIDENT NUMBER	PICKUP DATE	Customer Name	CONTAINER SIZE	COMMENTS
1	Virgin 105 Solvent		1/29/2014	Safety-Kleen	32 oz	
2						
3						
4						
5						
6						
7						
8						
9						
10						

ANALYSIS REQUEST (PLACE CHECKS BY TESTS REQUIRED)

___ pH	___ Flash Point	___ Ash %	___ Full Prequal
___ VOC's	___ PCB's	___ Water%	
___ HVOC's	___ Metals	___ BTU	___ Other (List in Additional Information)

Additional Information:
Sample sent per Stephen Fleming and Joseph Basile

ANALYTICAL APPROVAL (EHS OR NCWS USE ONLY)

PRINT NAME	Signature	DATE	TIME

SAMPLE TRANSFER RECORD

RELINQUISHED BY	DATE	TIME	RECEIVED BY	DATE	TIME
Kevin K Cowans	1/29/14	10:00 Am			

LAB USE ONLY

TEMPERATURE WHEN RECEIVED_____ °C

SAMPLE KIT OPENED AND CHECKED IN BY_____ AT____:____ ON___/___/___

C.O.C. SEALS SIGNED, DATED, AND INTACT ON ALL SAMPLES JARS? YES_____ NO_____ IF NO, EXPLAIN: _____

Attachment 7 :
Example of Chromatogram, Virgin 105 Solvent provided by Safety-Kleen Systems, Inc

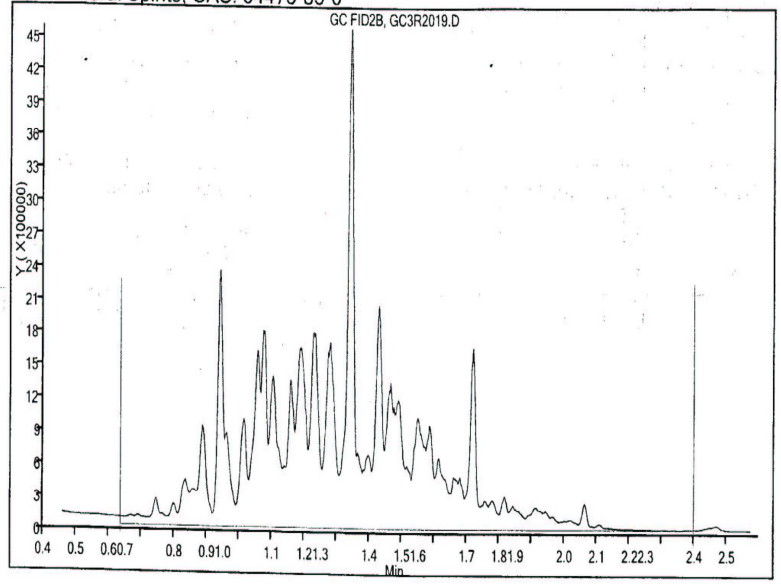
Report Date: 27-Mar-2014 11:24:48 Chrom Revision: 2.2 12-Mar-2014 11:19:24

Preliminary Report

TestAmerica Edison

Data File: \\EDICHROM\ChromData\CBNAGC3\20140306-10530.b\GC3R2019.D
Injection Date: 07-Mar-2014 08:29:04 Instrument ID: CBNAGC3
Lims ID: STD3
Client ID:
Operator ID: 615 ALS Bottle#: 57 Worklist Smp#: 4
Injection Vol: 1.0 ul Dil. Factor: 1.0000
Method: MS3R Limit Group: GC 8015C DRO ICAL
Column: Detector GC FID2B

A 2 Mineral Spirits, CAS: 64475-85-0



Attachment 8
GC Temperature Program

Method: C:\HPCHEM\1\METHODS\DRO.M of 20-Mar-14 6:26:39 AM

(Mineral/DRO GC3)

Data Acquisition: on

Standard Data Analysis: on

Customized Data Analysis: on
Macro Name: macro "ccopy.mac", go

Save GLP Data: off

Post-Run Cmd/Macro: off

Save Method with Data: off

Injection Source and Location

Injection Source: GC Injector

Injection Location: Dual

6890 GC METHOD

OVEN
Initial temp: 45 'C (On)
Initial time: 0.50 min
Ramps:

#	Rate	Final temp	Final time
1	45.00	150	0.00
2	45.00	190	0.30
3	60.00	360	2.00
4	0.0	(Off)	

Post temp: 50 'C
Post time: 0.00 min
Run time: 8.86 min
Maximum temp: 360 'C
Equilibration time: 0.10 min

FRONT INLET (SPLIT/SPLITLESS)
Mode: Splitless
Initial temp: 335 'C (On)
Pressure: 40.00 psi (On)
Purge flow: 30.0 mL/min
Purge time: 0.00 min
Total flow: 61.5 mL/min
Gas saver: Off
Gas type: Hydrogen

BACK INLET (SPLIT/SPLITLESS)
Mode: Splitless
Initial temp: 335 'C (On)
Pressure: 40.00 psi (On)
Purge flow: 30.0 mL/min
Purge time: 0.00 min
Total flow: 63.2 mL/min
Gas saver: Off
Gas type: Hydrogen

COLUMN 1
Capillary Column
Model Number: RESTEK 565647
dro-eph
Max temperature: 360 'C
Nominal length: 15.0 m
Nominal diameter: 250.00 um
Nominal film thickness: 0.50 um
Mode: constant pressure
Pressure: 40.00 psi
Nominal initial flow: 25.0 mL/min
Average velocity: 348 cm/sec
Inlet: Front Inlet
Outlet: Front Detector
Outlet pressure: ambient

COLUMN 2
Capillary Column
Model Number: RESTEK 565647
dro-eph
Max temperature: 360 'C
Nominal length: 15.0 m
Nominal diameter: 250.00 um
Nominal film thickness: 0.50 um
Mode: constant pressure
Pressure: 40.00 psi
Nominal initial flow: 25.0 mL/min
Average velocity: 348 cm/sec
Inlet: Back Inlet
Outlet: Back Detector
Outlet pressure: ambient

BNAGC3 24-Mar-14 7:45:13 AM 615

Page 2 of 6

Attachment 8 (cont)
GC Temperature Program

Method: C:\HPCHEM\1\METHODS\DRO.M of 20-Mar-14 6:26:39 AM

FRONT DETECTOR (FID)
- Temperature: 360 'C (On)
- Hydrogen flow: 10.0 mL/min (On)
- Air flow: 450.0 mL/min (On)
- Mode: Constant makeup flow
- Makeup flow: 5.0 mL/min (On)
- Makeup Gas Type: Helium
- Flame: On
- Electrometer: On
- Lit offset: 2.0

BACK DETECTOR (FID)
- Temperature: 360 'C (On)
- Hydrogen flow: 10.0 mL/min (On)
- Air flow: 450.0 mL/min (On)
- Mode: Constant makeup flow
- Makeup flow: 5.0 mL/min (On)
- Makeup Gas Type: Helium
- Flame: On
- Electrometer: On
- Lit offset: 2.0

SIGNAL 1
- Data rate: 20 Hz
- Type: front detector
- Save Data: On
- Start Save Time: 0.80 min
- Stop Save Time: 8.86 min
- Zero: 0.0 (Off)
- Range: 0
- Fast Peaks: Off
- Attenuation: 0

SIGNAL 2
- Data rate: 20 Hz
- Type: back detector
- Save Data: Off
- Zero: 0.0 (Off)
- Range: 0
- Fast Peaks: Off
- Attenuation: 0

COLUMN COMP 1
- Derive from front detector

COLUMN COMP 2
- Derive from back detector

POST RUN
- Post Time: 0.00 min

TIME TABLE

Time	Specifier	Parameter & Setpoint

GC Injector

Front Injector:

Sample Washes	1
Sample Pumps	5
Injection Volume	1.0 microliters
Syringe Size	10.0 microliters
PostInj Solvent A Washes	5
PostInj Solvent B Washes	5
Viscosity Delay	0 seconds
Plunger Speed	Fast
PreInjection Dwell	0.00 minutes
PostInjection Dwell	0.00 minutes

Back Injector:

Sample Washes	1
Sample Pumps	5
Injection Volume	1.0 microliters
Syringe Size	10.0 microliters
PostInj Solvent A Washes	5
PostInj Solvent B Washes	5
Viscosity Delay	0 seconds
Plunger Speed	Fast
PreInjection Dwell	0.00 minutes
PostInjection Dwell	0.00 minutes

BNAGC3 24-Mar-14 7:45:13 AM 615

Page 3 of 6

Lab SOP 03

Analysis of Alkalinity in Water, Wastewater and Soil by Manual Titration or Auto-Titrator, Standard Method 2320 B-11, Revision 11, 1/27/2015, TestAmerica Laboratories, Inc.

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	01/27/2015 Date	Kene' Kasperek	01/27/2015 Date
Department Manager		Health & Safety Manager /Coordinator	
Carl Armbruster	01/27/2015 Date	Mark Acierno	01/27/2015 Date
Quality Assurance Manager		Laboratory Director	

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^oC) 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 Environmental Resources Associates (ERA) 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 100 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 analytical logbook.

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, proceed to the next step. For all other forms of alkalinity proceed to 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. This will be equal to P (or the phenolphthalein alkalinity) in the calculations. Then 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 Titration	Hydroxide as CaCO3	Carbonate as CaCO3	Bicarbonate 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 the drop down menu by “look in” at the top and choose “c:” drive and choose “export data” folder. 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. FCO2 for carbon dioxide and for Alkalinity select all of the following: 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 Wet Chem Data Review checklist (WI# EDS-WI-008) during the first level review. The batch is second level reviewed and the checklist is filed in 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 Edison Work Instruction # EDS-WI-008, Wet Chemistry 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 11, dated 27 January 2015
  - o Sec. 9.1 & 9.1.3: Clarified the QC frequency of Matrix Duplicate to daily or with each batch of 20 or fewer samples.
  - o 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.

Lab SOP 04a

Anions by Ion Chromatography using EPA Method 300.0, 9056A, and SM4110B, Revision 5, 3/16/2016, TestAmerica Laboratories, Inc.

Anions by Ion Chromatography using EPA Method 300.0, SW846 9056A and SM 4110 B

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Approvals (Signature/Date):

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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 10 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

6.1.1.2 Guard column to protect the separator column from fouling by particles. Metrosep RP 2 Guard Cat # 6.1011.030

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

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: Dilute 2.8 ml of concentrated sulfuric acid to 4L 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 10 ml Class A volumetric flasks. Bring to the final volume of 10 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	40 ul	100 ul	500 ul	1 ml	1.5 ml	2 ml
Final Volume with Reagent Water	10 ml	10 ml	10 ml	10 ml	10 ml	10 ml	10 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) is prepared by adding 500 ul of Multi-Element IC standard describe in Section 7.6 into 10.0 ml Class A volumetric flasks. Bring to the final volume of 10ml 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.

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 1.0 grams of Ottawa sand into a scintillation vial. Pipette 9.5 ml of analyte-free reagent water into the vial. Add 500ul 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 1 gram of the sample into a scintillation vial. Pipette 9.5 ml of analyte-free reagent water into the vial. Add 500 ul 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 +/- 10 %.

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 1 gram of soil sample to 10 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):

$$\text{Matrix Duplicate (MD)} = \frac{|\text{orig. sample value - dup. sample value}|}{[(\text{orig. sample value + dup. sample value})/2]} \times 100$$

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 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

Controlled by

Lab ID	Worklist ID	Sample Type	Cal Lvl	Inj Date/Time	File Name	Vial	Dil Factor	Client ID	Fract	pH	Std Lot	STA Lot# Comments
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

Ok Eluent_00005 + Suppressor regenerant Solution_00005

Worklist Run Log: 11/26/2013 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

IX 9/20/2013

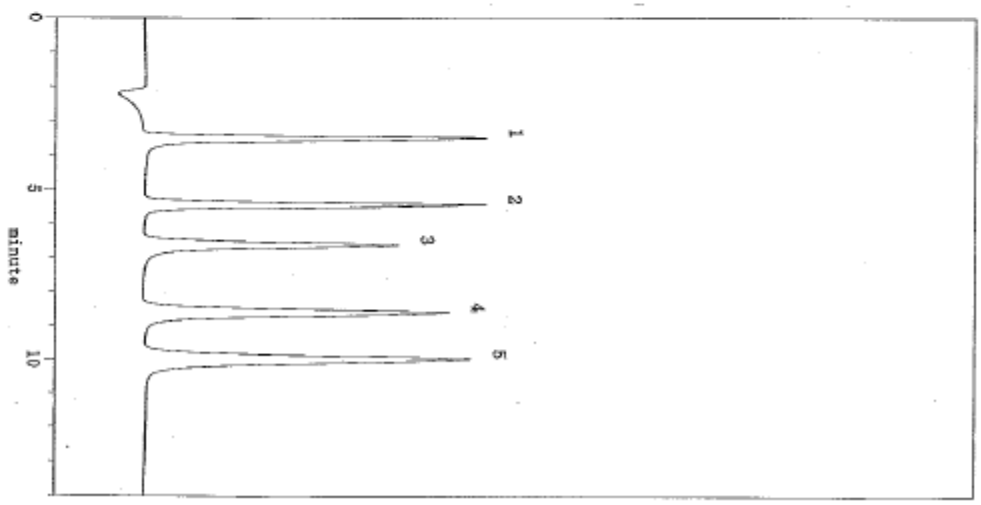
CERTIFICATE OF ANALYSIS

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



Lab SOP 04b

Analysis of Nitrate and Nitrite in Water, Wastewater, and Soil-Automated by EPA 353.2 and SM 4500-NO3F, Revision 7, 9/8/2015, TestAmerica Laboratories, Inc.

Title: Analysis of Nitrate and Nitrite in Water, Wastewater, and Soil-Automated by EPA 353.2 and SM 4500-NO3F

Once printed, this is considered an uncontrolled document

Approvals (Signature/Date):

Jasmine Parillo — 09/08/2015	Kene' Kasperek — 09/08/2015
Jasmine Parillo — Date	Kene' Kasperek — Date
Department Manager	Health & Safety Manager /Coordinator
Carl Armbruster — 09/08/2015	Mark Acierno — 09/08/2015
Carl Armbruster — Date	Mark Acierno — Date
Quality Assurance Manager	Laboratory Director

Facility Distribution No. Electronic **Distributed To:** Facility Intranet

1.0 Scope and Application

1.1 Analytes, Matrix(s), and Reporting Limits

1.1.1 Methods EPA 353.2, SM 4500-$N0_3$F and QuikChem Method 10-107-04-1-C are applicable to the analysis of Nitrate and Nitrite in waters, wastewaters and soils.

1.1.2 This method can be used to determine nitrate or as a sum of both forms of nitrogen from 0.02-2.0 mg/l as NO_3 and for nitrite from 0.01 - 1.0 mg/L as NO_2. The laboratory's reporting limit is 0.10 mg N/L as NO_3 and NO_2.

1.1.3 On occasion clients may request modifications to this SOP. These modifications are handled following the procedures outlined in Sections 7 (Review of Work Request) and 19 (Test Methods and Method Validation) of TestAmerica Edison's Quality Assurance Manual (ED-QA-LQM).

2.0 Summary of Method

2.1 Nitrate and nitrite are analyzed using an automated reduction and subsequent colorimetric measurement for quantitation. Nitrate is reduced to nitrite by passage of the sample through a copperized cadmium column. The nitrite (reduced nitrate plus original nitrite) is then determined by diazotizing with sulfanilamide followed by coupling with N-(1-naphthyl) ethylenediamine dihydrochloride. The resulting water-soluble dye has a magenta color that is read at 520 nm. Nitrite alone can be determined by removing the cadmium column.

2.2 Prior to colorimetric measurement Nitrate and nitrite in soils are extracted using the ASTM Leachate method (ASTM D3987). For detailed procedure of the ASTM leachate, see TestAmerica Edison SOP No. ED-ORP-041 ASTM LEACHATE, Procedure for the Extraction of Solid Waste with Water.

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 Residual chlorine can interfere by oxidizing the cadmium column.

4.2 Low results might be obtained for samples that contain high concentrations of iron, copper or other metals. In this method, EDTA is added to the buffer to reduce this interference.

4.3 Sample turbidity may interfere. Filter samples if sample has particulates, color, or is turbid prior to analysis. Use a 0.45 um filter syringe.

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

None

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
Ammonium Hydroxide	Corrosive Poison	50 ppm-TWA	Vapors and mists cause irritation to the respiratory tract. Causes irritation and burns to the skin and eyes.
Phosphoric Acid	Corrosive	1 Mg/M3 TWA	Inhalation is not an expected hazard unless misted or heated to high temperatures. May cause redness, pain, and severe skin burns. May cause redness, pain, blurred vision, eye burns, and permanent eye damage.
Potassium Nitrate	Oxidizer	None	Causes irritation to the respiratory tract, skin and eyes. Symptoms may include coughing, shortness of breath. Symptoms include redness, itching, and pain.
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

- Flow injection analysis equipment-designed to deliver and react sample and reagents in the required order and ratios. Lachat QuikChem 8000.

- Autosampler
- Multichannel proportioning pump
- Reaction unit and manifold
- Colorimetric detector, 540nm
- Two state switching valve
- Data system

- Analytical balance, capable of accurately weighing to the nearest 0.0001g.
- pH meter

6.2. Supplies

- Cd-Cu Reduction Column
- Class A volumetric flasks – varying volumes
- Calibrated pipettors – Eppendorf or equivalent
- 0.45 um filter syringe

7. Reagents and Standards

7.1. Reagents

7.1.1. 15 N NaOH: Slowly add 150g to 250 ml deionized water. CAUTION: The solution is very hot! Cool solution and store in a plastic bottle at room temperature. Stable for 6 months.

7.1.2. Ammonium Chloride buffer, pH 8.5: In a 1L beaker, dissolve 85.0g ammonium chloride (Aldrich-NH_4Cl) and 1.0g disodium tetraacetic acid dihydrate ($Na_2EDTA*2H_2O$) in about 800ml deionized water. Dilute to mark and invert to mix. Adjust the pH to 8.5 with 15N NaOH. Stable for 6 months. Store at room temperature.

7.1.3. Sulfanilamide color reagent: In a 1 L beaker containing about 600 ml of deionized water, add 100 ml of 85% phosphoric acid (H_3PO_4), 40.0 g sulfanilamide, and 1.0 g N-(1-naphthyl) ethylenediamine dihydrochloride (NED). Stir with stir bar for about 30 minutes until dissolved. Transfer to a 1L volumetric flask, dilute to mark and mix. Stable for 1 month. Store at room temperature in a brown bottle and keep in the dark when not in use.

7.2. Standards

7.2.1. Primary Nitrate Stock Standard, 1000ppm: Purchased commercially,

ERACal, Catalog # 052. Follow manufacturer's expiration date. Store at 4ºC.

7.2.2. Secondary Nitrate Stock Standard, 1000ppm: Purchased commercially, Accuspec, Catalog # 250-220-520. Follow manufacturer's expiration date. Store at 4ºC.

7.2.3. Primary Nitrite Stock Standard, 1000ppm: Purchased commercially, ERACal, Catalog # 053. Follow manufacturer's expiration date. Store at 4ºC.

7.2.4. Secondary Nitrite Stock Standard, 1000ppm: Purchased commercially, Accuspec, Catalog # 250-220-550. Follow manufacturer's expiration date. Store at 4ºC.

7.2.5. Primary Mixed Nitrate/Nitrite Working Solution, 20.0 mg N/L as NO_3- and NO_2-: In a 100 ml volumetric flask, add 1 ml of Primary Nitrate Stock Standard (Sec 7.2.1) and 1ml of Primary Nitrite Stock Standard (Sec 7.2.3). Dilute to mark with deionized water and mix. Prepare weekly. Store at 4ºC.

7.2.6. Secondary Mixed Nitrate/Nitrite working solution, 20.0 mg N/L as NO_3- and NO_2-: In a 100 ml volumetric flask, add 1 ml of Secondary Nitrate Stock Standard (Sec 7.2.2) and 1ml of Secondary Nitrite Stock Standard (Sec 7.2.4). Dilute to mark with deionized water and mix. Prepare weekly. Store at 4ºC.

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
Water	Plastic or glass	100 ml	Cool 4 ± 2°C	48 hours	40 CFR Part 136.3
Water	Plastic or glass	100 ml	Cool 4 ± 2°C, H_2SO_4 to pH<2[1]	28 days[1]	40 CFR Part 136.3

[1] For Nitrate plus Nitrite analysis only.

9. Quality Control

9.1. **Sample QC** - The following quality control samples are prepared daily 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. Use deionized water for the method blank. The results must be below the reporting limit if not, all samples associated with the method blank must be re-analyzed.

9.1.2. Laboratory Control Sample: The LCS is obtained from an outside vendor (ERA) and is used to measure method performance on the matrix being analyzed. There is a separate LCS for Nitrate and Nitrite. The recoveries must be within vendor specified QC limits. If the result of the LCS is not within the acceptance limits, all samples requiring that particular analyte must be re-analyzed.

9.1.3. Matrix Spike/Matrix Spike Duplicate: Two portions of the same sample are each spiked with 2.5 ml of the 20ppm Primary Mixed Nitrate/Nitrite Working Solution (Sec. 7.2.5) to 50ml of sample. The true value of Nitrate is 0.5 ppm, true value of the Nitrite is 0.5ppm, and the true value of Nitrate-Nitrite is 1.0 ppm. The recovery and relative percent difference must be within laboratory generated control limits. One matrix spike and matrix spike duplicate must be analyzed daily with each batch of samples or one in every 20 samples, whichever is more frequent.

9.2. Instrument QC

9.2.1. Initial Calibration Verification (ICV): The ICV is a mid-point calibration verification and is analyzed immediately after analyzing the calibration standards. It is prepared from a different source than the calibration standards. The ICV is prepared by adding 2.5 ml of the 20ppm Secondary Mixed Nitrate-Nitrite Working Solution (Sec. 7.2.6) to a final volume of 50ml with deionized water. The true value of the NO_3 is 0.5ppm, true value of the NO_2 is 0.5ppm, and the true value of the NO_3 -NO_2 is 1.0 ppm. Acceptance criteria for the ICV are 90-110 % of the true value.

9.2.2. Continuing Calibration Verification (CCV): The CCV is analyzed after every 10 samples or less and at the end of the analytical run. It is prepared the same way as ICV. Acceptance criteria for the CCV are 90-110% of the true value. If the result is not within the acceptance limits, all samples following the last acceptable CCV must be re-analyzed.

9.2.3. Initial Calibration Blank (ICB): An initial calibration blank must be analyzed immediately after analyzing the calibration standards. Use deionized water for the ICB. The concentration of the ICB must be below the reporting limit.

9.2.4. Continuing Calibration Blank (CCB): A continuing calibration blank is analyzed after every 10 samples or less and at the end of the analytical run. Use deionized water for the CCB. The concentration of the CCB must be below the reporting limit. If the result of the CCB is not below the reporting limit, all samples following the last acceptable CCB must be re-analyzed.

10. Procedure

10.1. Sample Preparation

10.1.1. If the sample pH is below 5 or above 9, adjust to between 5 and 9 with either NaOH or H_2SO_4.

10.1.2. Filter samples if sample has particulates, color, or is turbid prior to analysis. Use a 0.45 um filter syringe.

10.1.3. Soil samples must be extracted prior to analysis. Extract samples utilizing the ASTM D3987 procedure described in SOP# ED-ORP-041 (ASTM LEACHATE, Procedure for the Extraction of Solid Waste with Water).

10.2. Calibration

10.2.1. Prepare Nitrate/Nitrite standards weekly using 20 mg/L Primary Mixed Nitrate/Nitrite working solution (Sec 7.2.5) in 100 ml of deionized water. A calibration curve must be analyzed daily. The correlation coefficient must be 0.995 or greater.

Volume of Standard Solution (ml)	Concentration of NO_3-NO_2 (ppm)	Concentration of NO_2 (ppm)
0	0	0
0.50	0.10	0.050
2.5	0.50	0.25
5.0	1.0	0.50
7.5	1.5	0.75
10.0	2.0	1.0

10.2.2. 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.

10.3. Sample Analysis

10.3.1. Follow manifold scheme of this method as described in QuikChem Automated Ion Analyzer Methods Manual. Place all reagent lines in deionized water, turn pump on and check for flow rate and for leaks.

10.3.2. Remove reagent lines from deionized water and place into appropriate reagent. Let reagent run through for approximately 5 minutes before putting the column on line to prevent introducing air into the column.

10.3.3. Set up tray table with all appropriate QC checks (includes method blank, LCS, matrix spike, and matrix spike duplicate). **Note**: Samples which are analyzed with dilution must be typed in the sequence with the dilution factors entered next to the sample (ex. 460-5255-a-1 @20).

10.3.4. Place standards and samples into the autosampler cups. The following analytical sequence must be used:

Calibration
ICV
ICB
Method Blank
LCS
8 samples
CCV1
CCB1
10 samples
CCV2
CCB2
Repeat until run is complete
CCV3
CCB3

10.3.5. The column should be on line for approximately 5 minutes before running the tray. This allows the baseline to stabilize.

10.3.6. Run tray.

10.3.7. Make all appropriate dilutions for samples which exceeded the calibration 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 aqueous samples:

Concentration = mg/L

Concentration (mg/L) from instrument is imported to TALS.
Samples with dilution factor are calculated in TALS for final results.

11.4. Final results calculation in solid samples

Concentration = mg/kg = C x D

Where:
C= final concentration in mg/L
D= Dilution factor, 20 (solid samples extracted via Method ASTM D3987 uses a standard 20 x Dilution Attenuation Factor (DAF)).

NOTE: All dry weight corrections are made in TALS at the time the final report is prepared.

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 in 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 in 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 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 special waste streams associated with 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 4500 NO_3 F.

15.2. Methods for Chemical Analysis of Water and Wastewaters, EMSL-Cincinnati, EPA/600/4-79-020, March 1983 and 1979; Test Method 353.2.

15.3. QuikChem Method 10-107-04-1-C, EPA Test Method -353.2, Standard Methods, 18th edition, Test Method- 4500-NO3 F

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 Edison SOP No. ED-SPM-008, *Laboratory Waste Disposal Procedures*, current revision.

15.7. TestAmerica Edison SOP No ED-ORP-041, ASTM LEACHATE, Procedure for the Extraction of Solid Waste with Water, most current revision.

15.8. QuikChem Automated Ion Analyzer Methods Manual, Lachat instruments.

15.9. TestAmerica Edison Work Instruction # EDS-WI-008, Wetchem Data Review Checklist, most current revision.

15.10. Lachat instruments, cadmium column installation instructions.

15.11. TestAmerica Calibration Spreadsheet, Work Instruction No. CA-Q-WI-013, (8/10/2011).

15.12. TestAmerica Edison Wet Chemistry Initial Calibration Data Review) EDS-WI-122, most current revision.

16.0. Method Modifications:

Item	Method	Modification
Sec. 7.1.2	SM 4500-NO3 F; EPA 353.2	Preparation of Ammonium Chloride Buffer follows QuikChem Method 10-107-04-1-C using 1.0g EDTA instead of 0.1g as mentioned in SM 4500 NO3F and EPA 353.2
Sec. 7.1.3	SM 4500-NO3 F; EPA 353.2	Preparation of Sulfanilamide Color Reagent follows QuikChem Method 10-107-04-1-C; using 1.0g NED instead of 2.0g as mentioned in SM4500 NO3F and EPA 353.2

17.0. Attachments

N/A

18.0. Revision History

- Revision 7, dated 08 September 2015
  - Sec. 10.2.2: 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 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 to include ERA as the source for the LCS.
  - Sec 15.10 & 15.11: Added applicable references WI# CA-Q-WI-013 and EDS-WI-122.

- Revision 6, dated 30 July 2013

- Sec 1 and 12: Updated LQM section references to reflect the most current LQM revision.
- Sec.9.1.2: Changed criteria for LCS from 85-115% to vendor specified QC limits to reflect actual laboratory practices.
- Sec. 9.2.1: Removed efficiency check as instrument QC; the column efficiency is verified via the LCS; subsequent sections adjusted accordingly.

- Revision 5, dated 07 November 2011
  - Sec. 9.1.1 & 9.1.2: Added corrective action procedure if MB and LCS are outside of the acceptance limits.
  - Sec. 9.2.1: Added procedure for cadmium column efficiency check following manufacturer's instructions.
  - Sec. 9.2.3 & 9.2.5: Added procedure if CCV and CCB are not within acceptance limits.
  - Sec 10.1.2: Section added; syringe filter added to list of supplies. Subsequent sections adjusted accordingly.
  - Sec. 10.2.1: Added frequency for preparing and analyzing calibration standards. Removed calibration standard (0.05/0.025 ppm) to reflect actual laboratory practices.
  - Sec 15.10: Added reference.
  - Sec. 16.0: Added method modifications.

- Revision 4, dated 15 September 2009
  - Revised SOP format in accordance with TestAmerica Corporate Quality SOP No. CW-QS-002 (Rev 0) Writing a Standard Operating Procedure (SOP).
  - Sec 2.2: Added extraction of soil samples via ASTM method in Method summary section and Sample Summary section.
  - Sec 4.1 & 4.2: Added interference
  - Section 7: Added storage information
  - Section 7.2: Revised source of NO3 and NO2 stock standards to commercially purchased pre-made stocks; also revised stock concentration from 2000 mg N/L to 1000 mg N/L.
  - Section 7.2.5: Changed the amount of each stock that is added from 500ul to 1ml to reflect the revised stock concentration.
  - Section 7.2.6: Added Secondary Mixed Nitrate/Nitrite Working Solution.
  - Section 11.4: Added final results calculation in solid samples.
  - Section 11.5: Revised new data reduction procedures in accordance with new TALS.
  - Section 15: Added applicable references.

Lab SOP 05

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 QuickChem Method 10-107-06-1-C, Revision 10, 3/4/2016, TestAmerica Laboratories, Inc.

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

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Approvals (Signature/Date):

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Jasmine Parillo — Date	Dan Helfrich — Date
Department Manager	Health & Safety Manager /Coordinator
Carl Armbruster — 03/04/2016	Mark Acierno — 03/04/2016
Carl Armbruster — Date	Mark Acierno — Date
Quality Assurance Manager	Laboratory Director

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- Thermo Orion 320
- 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.

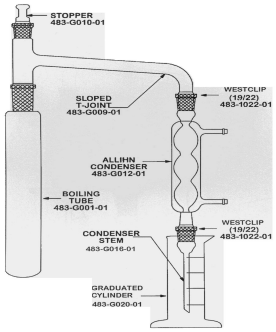


Figure 1: EASY-Dist Ammonia Distillation Apparatus

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 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 (19^{th}/20^{th} Edition)
  - Section 1.1 and throughout document: Revised to reflect added method reference for SM 4500 NH3-B+G (19^{th}/20^{th} Edition)
  - Section 15.0: Revised to reflect added method reference for SM 4500 NH3-B+G (19^{th}/20^{th} 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.

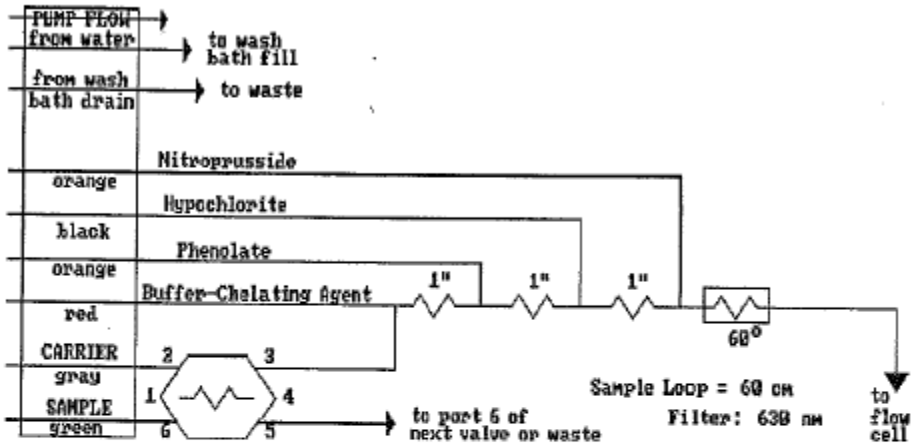
- Revision 4, dated May 30, 2008

  - 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

Lab SOP 06

Sample Preparation and Calculation for Dissolved Gas Analysis in Water Samples Using a GC Headspace Equilibration Technique Method RSK 175 Modified, Revision 13a, 10/31/2016, TestAmerica Laboratories, Inc.

SOP Number/Revision No.: RSK 175 / NV04-89.13 **Effective Date: 10/31/2016**
Last Mod. Date: 8/31/2016
SOP Title: **Sample Preparation And Calculation For Dissolved Gas Analysis In Water Sample Using A GC Headspace Equilibration Technique Method RSK 175 Modified**

CONTROLLED DISTRIBUTION
ISSUED TO: \\TAFS\Lab\Nashville\Public\QA\SOPs, 04P

Revision Number with Mod ID: **13a**

The following SOP change is in effect as of the stated date. This form will remain attached to the referenced SOP until such a time that the SOP is updated, approved, and redistributed, at which time it will become part of the historical SOP record. ***Append this form to the front of the SOP copy.***

1. Reason for SOP Change:

☐ Typographical Corrections (Non-Technical) – Re-Training Not Required.

☐ Typographical Corrections (Technical – Define) – Analyst acknowledgement of corrections is required.

■ Procedural Changes (Define Below) – Re-Training Required.

☐ Other

2. Summary of Procedure Change: Delete crossed out text; add highlighted text.

Section 6.2, Supplies

- VOA vials with ~~Teflon™~~/Butyl ~~silicone~~ septa/ Teflon™-lined, commercial.

Section 8.0, Sample Collection, Preservation, Shipment and Storage

Matrix	Container	Preservation*	Holding Time
Water	VOA vials with ~~Teflon™~~/Butyl septa/Teflon™-lined ~~septa~~ screw caps	Add 3 to 4 drops of 1:1 HCl or H_2SO_4 to pH < 2; cool to 0-6°C.	14 days from time of collection
		No preservation	7 days from time of collection

Section 9.2, Instrument QC

QC Check	Frequency	Acceptance Criteria[1]	Corrective Action[2]
Minimum five-point initial calibration for all target analytes	Initial calibration prior to sample analysis. Perform instrument re-calibration once per month minimum. Use weighted calibration models.	RSD of CF ≤ 15 ~~20~~%. Linear least squares regression $r^2 \geq 0.990$, r ≥ 0.995. Re-calculation of lowest point must be within 30% true or re-calibrate.	Correct problem then repeat initial calibration
Initial calibration verification (ICV), second source.	Immediately following initial calibration	All target analytes within 15 ~~20~~% of expected value.	Correct problem then repeat initial calibration

Initial Calibration Blank (ICB)	Immediately after ICV.	< RL	Correct problem, repeat.
Continuing Calibration Verification (CCV)	At the beginning and the end of the analysis sequence or every 12 hours, at the minimum, not to exceed 20 samples.	± 15 ~~20~~% of expected value and within the RT Window	Make a fresh CCV and run; if it still fails, correct problem, re-calibrate, and re-analyze all samples since last successful CCV. If the CCV is high and the sample is ND, it is acceptable to report.

- **Initial Calibration Verification (ICV)**: The ICV is nominally Butane, Ethane, Ethene, Isobutane, Methane, and Propane. Prepare in the same manner as a level 5 ~~4~~ initial calibration standard.
- **Initial Calibration Blank (ICB)**: The ICB is reagent water.
- **The Continuing Calibration Verification Standard (CCV) is prepared from the primary source standard**.
  - Prepare a standard with nominally Butane, Ethane, Ethene, Isobutane, Methane, and Propane. Prepare in the same manner as a level 5 ~~4~~ initial calibration standard.

Section 18.0, Revision History

- ~~Add a lower level calibration standard.~~

10/10/16	10/10/16
Department Manager Approval Date	Department Supervisor Date
10/10/16	10/7/16
Quality Assurance Approval Date	Technical Director Approval Date

Title: SAMPLE PREPARATION AND CALCULATION FOR DISSOLVED GAS ANALYSIS IN WATER SAMPLE USING A GC HEADSPACE EQUILIBRATION TECHNIQUE METHOD RSK 175 MODIFIED

Approvals (Signature/Date)			
Lauren Gillins Department Supervisor	7/22/16 Date		
James Jacobs Department Manager	8/16/16 Date	Aidan Casey Health & Safety Coordinator	7/27/16 Date
Donovin Mulvaney Quality Assurance Manager	7/25/16 Date	Michael H. Dunn Technical Director	8/7/16 Date

1.0 Scope and Application

1.1 Analyte, Matrices: This method describes a procedure for the determination of dissolved gases, Butane, Ethane, Ethene, Isobutane, Methane, and Propane, in groundwater, non-potable water, and potable water.

1.2 Reporting Limit (RL): The RL water is 5 µg/L for Ethane, Ethene, Methane, and Propane. The RL is 10 µg/L for Isobutane (synonym: 2-Methylpropane) and Butane,.

1.3 This method is used by, or under the supervision of, analysts experienced in the use of gas chromatographs. The analysts are skilled in the interpretation of gas chromatograms and their use as a quantitative tool.

1.4 If for any reason a part of this method cannot be followed, seek the guidance of the Department Supervisor, Department Manager, or the Technical Director. All abnormalities must be noted in the Laboratory Information Management System (LIMS).

2.0 Summary of Method

A water sample is collected in a VOA vial and capped using a Teflon™-faced septum. A headspace is prepared by displacing a specified sample volume. A sample is taken of the generated headspace and injected onto a gas chromatographic column where the gaseous components are separated and detected by flame ionization detector. The concentration of the gas in the headspace and the sample volume allows the concentration of dissolved gas in the original water sample to be determined.

3.0 Definitions

See the definitions in the Quality Assurance Manual. Also, refer to Controlled Document TestAmerica Nashville – Acronyms, Keywords, and Definitions QAF-45.

4.0 Interferences

The flame ionization detector (FID) is a non-selective detector. There is a potential for non-target compounds present in samples to interfere with this analysis.

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 document does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of the method to follow appropriate safety, waste disposal and health practices under the assumption that all samples and reagents are potentially hazardous. Safety glasses, gloves, lab coats, and closed-toe, nonabsorbent shoes are a minimum.

5.1 Specific Safety Concerns or Requirements:

- The gas chromatograph contains zones that have elevated temperatures. The injector and detector heating zones must be cooled to near room temperature prior to working on them.
- There are areas of high voltage in both the gas chromatograph and autosampler. Depending on the type of work involved, either turn the power to the instrument off, or disconnect it from its source of power.
- The headspace analyzer contains pressurized vessels which should be de-pressurized prior to service.

5.2 Primary Materials Used: There are no materials used in this method that have a serious or significant hazard rating. 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 SDS for each material before using it for the first time or when there are major changes to the SDS.

6.0 Equipment and Supplies

6.1 Instrumentation

- **Gas Chromatograph** (Hewlett-Packard or equivalent) complete with temperature programmable gas chromatograph. The data station (EZChrom/Chrom) is capable of storing and reintegrating chromatographic data and is capable of determining peak areas using a forced baseline projection.
  - Column: 30 m x 0.53 mm x 20 µm film thickness, RT U Plot, or equivalent.
  - Detector: Flame Ionization Detector (FID).
  - Suggested Operating Conditions

	Ethane, Ethene, Methane, Propane only	Butane, Ethane, Ethene, Isobutane, Methane, Propane
Carrier gas	Hydrogen	
Flowrate	20 mL/minute	
Injector temperature	150°C	
Detector temperature	250°C	
Temperature Program	60°C isothermal	
Initial Temperature		60°C
Initial Time		1.0 minute
Rate		30°C/minute
Final Temperature		130°C
Final Time		0.5 minute
These conditions are recommended as a starting point and may need to be changed for optimization. See the instrument maintenance logbook for any changes to these conditions.		

6.2 Supplies

- Microsyringes, gas-tight, various volumes, Hamilton brand or equivalent.
- VOA vials with Teflon™/silicone septa, commercial.
- Compressed gas, Hydrogen, Helium, or Nitrogen, commercial, high purity.

7.0 Reagents and Standards

7.1 Reagent grade chemicals are used in all tests. Unless otherwise, indicated, it is intended that all reagents shall conform to the specifications of the Committee on Analytical Reagents of the Analytical Reagents of the American Chemical Society, where such specifications are available. Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination.

7.2 Reagent water, analyte-free.

7.3 Butane, Ethane, Ethene, Isobutane, Methane, and Propane are purchased in a certified, custom mix nominally at 1% (10,000 ± 2% ppmv) in Helium. Standards should be replaced after 12 months or sooner, if comparison with check standards indicates a problem.

- The primary standard is Air Liquide, custom mixture (7000-44, MN700417), or equivalent, 1% each compound.
- The secondary standard is the same as the primary but from a different lot.

7.4 Surrogate Standard: Acetylene, purchased as a certified standard 1% (10,000 ppmv) in Nitrogen from Air Liquide. 875 µL of surrogate standard is added to all samples.

7.5 See SOP Reagent and Standard Purchase, Preparation, Control, Documentation / NV08-214 for shelf-life and storage requirements for reagents and standards. Also, refer to LIMS.

8.0 Sample Collection, Preservation, Shipment and Storage

Matrix	Container	Preservation*	Holding Time
Water	VOA vials with Teflon™-	Add 3 to 4 drops of 1:1 HCl or	14 days from time

Matrix	Container	Preservation*	Holding Time
	lined septa screw caps	H_2SO_4 to pH < 2; cool to 0-6°C.	of collection
		No preservation	7 days from time of collection

*An unpreserved sample is acceptable.

9.0 Quality Control

The laboratory maintains a formal quality assurance program and records to document the quality of the data generated.

9.1 Sample QC

The following QC is run **every batch of no more than 20 samples:**			
QC Check	**Frequency**	**Acceptance Criteria**	**Corrective Action[1]**
Method blank	Each batch	≤ RL	Correct problem then re-prep[3] and analyze method blank and all samples processed with the contaminated blank. If target > 10 blank, report but qualify. OK to report ND samples.
LCS for all analytes.	One each batch.	See LIMS.	Re-prep[2] and analyze the LCS and all samples in the affected analytical batch. If high and samples are ND, report.
Matrix Spike/Matrix Spike Duplicate	One each batch.	See LIMS.	Report.
Surrogate	Every sample, spiked sample, standard, and method blank	See LIMS.	Check system, re-analyze[3], may qualify. If % recovery is high and the sample is ND, it is acceptable to report.

1 – This is a summary of the acceptance criteria.
2 -- All abnormalities must be noted in LIMS.
3 – If unable to re-prep the samples because of insufficient sample volume or holding time has expired, place a comment in LIMS.

- Analyze all laboratory method blanks and QC samples under the same conditions as that used for samples.
- **Method Blank:** Use reagent water as the blank matrix.
- **Laboratory Control Sample:** The LCS consists of an aliquot of reagent water spiked with the standard. The LCS is nominally Butane, Ethane, Ethene, Isobutane, Methane, Propane. Prepare in the same manner as a level 4 initial calibration standard.
- **Matrix Spike/Matrix Spike Duplicate:** The MS/MSD is prepared from the same standard and spiked at the same concentrations as the ICV and LCS.
- **Surrogate Standards:** The analyst monitors both the performance of the analytical system and the effectiveness of the method in dealing with each sample matrix by spiking each client and QC sample, standard, and blank with the surrogate compound (875 µL of the surrogate standard/sample) which is not expected to be affected by method interferences.
- **pH Check:** The analyst must document the pH of each sample by checking with wide-range pH paper. The pH check is performed after sample analysis. Record the pH in the LIMS.

9.2 Instrument QC

QC Check	Frequency	Acceptance Criteria[1]	Corrective Action[2]
Minimum five-point initial calibration for all target analytes	Initial calibration prior to sample analysis. Perform instrument re-calibration once per month minimum. Use weighted calibration models.	RSD of CF ≤ 20%. Linear least squares regression $r^2 \geq 0.990$, $r \geq 0.995$. Re-calculation of lowest point must be within 30% true or re-calibrate.	Correct problem then repeat initial calibration
Initial calibration verification (ICV), second source.	Immediately following initial calibration	All target analytes within 20% of expected value.	Correct problem then repeat initial calibration
Initial Calibration Blank (ICB)	Immediately after ICV.	< RL	Correct problem, repeat.
Continuing Calibration Verification (CCV)	At the beginning and the end of the analysis sequence or every 12 hours, at the minimum, not to exceed 20 samples.	± 20% of expected value and within the RT Window	Make a fresh CCV and run; if it still fails, correct problem, re-calibrate, and re-analyze all samples since last successful CCV. If the CCV is high and the sample is ND, it is acceptable to report.
Continuing Calibration Blank	After each CCV	< RL	Correct problem, repeat. If the blank is positive and the samples are ND, it is acceptable to report.
Retention time window calculated for each analyte	System set-up or major instrument maintenance. Update the mid-RTW at the start of the run or daily.	Each analyte of the LCS and CCV must be within the established RTW.	Correct the problem and re-process or re-analyze samples. For questions, see the supervisor or technical manager.
Methane RL verification (MRLREC in LIMS)	Minimum daily when in use	70-130%	Re-evaluate MDL standard used and MDL; see technical manager.

1 – This is a summary of the acceptance criteria.
2 -- All abnormalities must be noted in LIMS.

- **Calibration standards:** See Section 10.
- **Initial Calibration Verification (ICV)**: The ICV is nominally Butane, Ethane, Ethene, Isobutane, Methane, and Propane. Prepare in the same manner as a level 4 initial calibration standard.
- **Initial Calibration Blank (ICB)**: The ICB is reagent water.
- **The Continuing Calibration Verification Standard (CCV) is prepared from the primary source standard**.
  - Prepare a standard with nominally Butane, Ethane, Ethene, Isobutane, Methane, and Propane. Prepare in the same manner as a level 4 initial calibration standard.
- **Continuing Calibration Blank (CCB):** The CCB is reagent water.
- **Retention Time Windows**: The analytes are distinguished on the basis of the retention time.
  - Before establishing retention time windows, make sure that the chromatographic system is functioning reliably and that the operating parameters have been optimized for the target analytes and surrogates in the sample matrix to be analyzed.
  - Establish the retention time windows. Inject standards over a minimum 72-hour period. Calculate 3 x standard deviation of the absolute retention time to set the RT window width. The default window is ± 0.03 minute.
- **Report Limit Verification (RLV) of Methane:** Use 20 µL for a 6.2 µg/L standard. (The LIMS synonym for this standard is MRLREC, method RL recovery.)

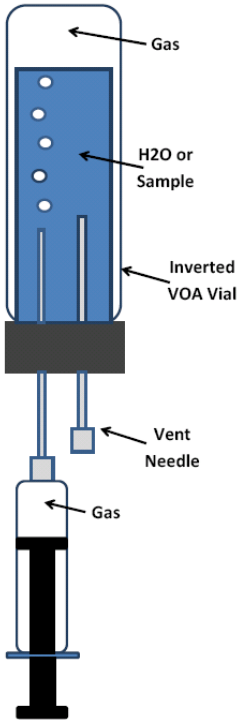
10.0 Procedure

10.1 Sample Preparation

Matrix	Sample Size
Water	21 mL

1	Remove samples from the refrigerator and allow them to come to room temperature.
2	Invert and insert a vent needle into the VOA vial through the septum.
3	While inverted, slowly add 875 µL of the surrogate standard to each vial. If needed, slowly add 875 µL spike to the LCS, MS/MSD using a gas-tight syringe.
4	Insert needle on a 25 mL syringe through the septum and slowly inject 20.125 mL[a] of Helium or Nitrogen with the VOA vial inverted. Shake well for at least two minutes to equilibrate headspace. Store the VOA vial inverted.

[a] A total of 21.0 mL of headspace is developed in the VOA vial. For example, if the sample will be spiked with 0.875 mL of standard material, then add 20.125 mL of Helium or Nitrogen.



10.2 Calibration and Daily Continuing Calibration Verification: Refer to SOP Calibration Curves and Selection of Calibration Points / CA-Q-P-003, and Acceptable Manual Integration Practices / CA-Q-S-002. See Section 11 for equations. Vendor software and LIMS perform the calculation.

Prepare a minimum of five calibration standards with 21.0 mL reagent water and 21.0 mL headspace in the VOA vial:

LEVEL	µL Stock Injected	Concentration of Target (µg/L)						
		METHANE	ETHENE	ETHANE	PROPANE	ACETYLENE	ISOBUTANE	BUTANE
1	4.5	1.4	2.5	2.5	3.8	2.3	5	5
2	9	2.8	4.9	4.9	7.7	4.6	10	10
3	90	27.28	49	49	77	46	100	100
4	175	55	96	103	150	89	198	198
5	875	273	478	512	750	444	991	991
6	1750	547	957	1025	1500	888	1982	1982

Extending the calibration range, either up or down, is allowed as long as the standards remain within the linear range of the instrument. The low standard must be at or below the report limit.

10.3 GC Analysis

1	Using the initial opening standard, set the absolute retention time to the midpoint of the window for that day.
2	If the response exceeds the linear range of the system, dilute the sample and reanalyze. It is recommended that samples be diluted so that all peaks are on scale. Overlapping peaks are not always evident when peaks are off scale.
3	The concentration in a sample is determined by using the calibration curve or the calibration factor. The calculation is performed by vendor software. See Section 11.
4	Peak areas measured from blanks are **not** subtracted from sample peak areas.

Dilution Required	Volume, Using a Gas-Tight Syringe with Sideport Needle, 100 µL injection
1	100 µL
2	50 µL
5	20 µL
10	10 µL
20	2 mL Headspace to empty VOA vial
40	1 Headspace to empty VOA vial
80	0.5 Headspace to empty VOA vial
160	0.25 Headspace to empty VOA vial
400	0.1 Headspace to empty VOA vial
800	0.05 Headspace to empty VOA vial

10.4 Recommended Analysis Batch Sequence*

1	Initial Calibration, as needed
2	ICV, immediately after initial calibration
3	ICB
4	CCV
5	LCS
6	Method Blank
7	Matrix Spike
8	Matrix Spike Duplicate
9	Samples 1-10
10	CCV
11	CCB
12	Samples 11-20
13	CCV
14	CCB

11.0 Calculations / Data Reduction

11.1 Accuracy

$$\% \text{ Recovery} = \frac{\text{Measured concentration} \times 100}{\text{Known concentration}}$$

11.2 Precision (RPD)

RPD = Absolute value (orig. sample value - dup. sample value) x 100
(Orig. sample value + dup. sample value)/2

11.3 % Difference, % Drift

% Difference = $\frac{(CF_v) - (\text{Avg. CF}) \times 100}{(\text{Avg. CF})}$

CF_v = CF from verification standard
Avg. CF = Average CF from Initial Calibration.

% Drift = $\frac{(\text{Result - True Value}) \times 100}{\text{True Value}}$

11.4 Calibration Factor

CF = $\frac{\text{Area of compound}}{\text{Concentration of compound}}$

11.5 Mean Calibration Factor, Standard Deviation, Relative Standard Deviation

$$CF_{mean} = \frac{\sum_{i=1}^{n} CF_i}{n} \qquad SD = \frac{\sum_{i=1}^{n} (CF_i - CF_{mean})}{n-1}$$

$$RSD = \frac{SD \times 100}{CF_{mean}}$$

11.6 Linear Calibration Using a Least Squares Regression: If RSD of the response factors is greater than 20% over the calibration range, then linearity through the origin cannot be assumed. If this is the case, the analyst employs a regression equation.

This is most easily achieved by performing a first-order linear regression of the instrument response versus the concentration of the standards. Make certain that the instrument response is treated as the dependent variable (y) and the concentration as the independent variable (x). This is a statistical requirement and is not simply a graphical convention.

The regression produces the slope and intercept terms for a linear equation in the form:

$y = ax + b$

y = instrument response (peak area)
a = slope of the line
x = concentration of the calibration standard
b = the intercept

The mathematics used in least squares regression has a tendency to favor numbers of larger value over numbers of smaller value. Thus the regression curves generated tend to fit points at the upper calibration levels better than points at the lower calibration levels. To compensate for this, a weighting factor which reduces this tendency is used. Examples of acceptable weighting factors which place more emphasis on numbers of smaller value are:

$w = 1/x$ or $w = 1/x^2$

The analyst should not force the line through the origin, but have the intercept calculated from the data points. The use of a linear regression may not be used as a rationale for reporting results below the calibration range demonstrated by the analysis of the standards. The regression calculation generates 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 0.995 or $r^2 \geq 0.990$.

11.7 Coefficient of Determination

$$r^2 = \frac{\left(\sum xy\right)^2}{\sum x^2 \sum y^2}$$

Correlation Coefficient

$$r = \frac{\left(\sum xy\right)}{\sqrt{\sum x^2 \sum y^2}}$$

y = Response
x = Concentration

11.8 Sample Concentration

C = $(A/CF_{avg}) \times D$

C = Concentration of target analyte in sample (µg/L)
A = Peak area of analyte
CF_{avg} = Average calibration factor for an analyte from the calibration
D = Dilution factor

For linear regression, Instrument Readout = (Instrument concentration) (dilution)

- Example Calculation (Based on 22°C at 754 mm Hg, Molar equivalent 0.04099)

For 1000 µL of 10,000 ppmv stock standard:

$$\frac{10{,}000\ \mu L\ CH_4}{L\ He} \times \frac{1\ L\ CH_4}{1{,}000{,}000\ \mu L\ CH_4} \times \frac{0.0409\ moles\ gas}{1\ L\ gas} \times \frac{16\ g\ CH_4}{1\ mole\ CH_4} \times \frac{0.001\ L\ He}{0.021\ L\ H_2O}$$

$$\times \frac{1{,}000{,}000\ \mu g\ CH_4}{1\ g\ CH_4} = \frac{6.54}{0.021} = \frac{312\ \mu g\ CH_4}{L\ H_2O}$$

- $ppmV = \frac{\mu g/L \times 24.47}{MW}$ $\qquad \mu g/L = \frac{ppmV \times MW}{24.47}$

ppmV = parts per million by volume

µg/L = micrograms per liter
24.47 = molal gas constant at standard temperature and pressure
MW = molecular weight of analyte, g/mole

- Example Using 1% Methane, i. e., 10,000 ppmV

$$\mu g/L = \frac{10000 \times 16}{24.47} = 6538\ \mu g/L \text{ or } ng/mL$$

Preparation of Standard

$$\frac{6538\ ng/mL \times mL\ gas\ standard}{mL\ water\ standard}$$

Example: $\frac{6538\ ng/mL \times 1\ mL}{21\ mL} = 311\ ng/mL$ (or µg/L)

Note: Must keep headspace and sample volume constant.

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 SOP Detection Limits / CA-Q-S-006. 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 Capability: The laboratory demonstrates initial proficiency by generating data of acceptable accuracy and precision for target analyses in a clean matrix. The laboratory also repeats the operation whenever new staff is trained or significant changes in instrumentation are made and on an annual basis thereafter. See TestAmerica-Nashville's QA Manual and SOP Training / NV08-199 for information on how to accomplish this demonstration.

12.3 Training Requirements: Demonstration of Capability is performed initially when learning the method and annually thereafter. Four Laboratory Control Samples resulting in an average % recovery within the control limits and a precision less than the quality control maximum are required.

12.4 Proficiency Testing Studies: The laboratory participates in formal proficiency testing (PT) studies, where solutions of unknown concentrations are analyzed and the performance of all participants is compared. See the QA department for the results of recent PT studies.

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 managed, stored, and disposed of in accordance with all federal and state laws and regulations. Waste description rules and land disposal restrictions are followed. Waste disposal procedures are incorporated by reference to the QA Manual and SOP Waste Disposal / NV10-83.

14.2 Wastestreams Produced by the Method:

- Purged samples are discharged into the sanitary sewer.

15.0 References / Cross References

15.1 Method RSK-175 - Kampbell, D.H., J.T. Wilson, S.A. Vandegrift, Dissolved Oxygen and Methane in Water by a GC Headspace Equilibration Technique, International Journal of Environmental Analytical Chemistry, Volume 36, pp. 249-257, 1994.

15.2 TestAmerica Nashville's Quality Assurance Manual.

15.3 Corporate Environmental Health and Safety Manual (CW-E-M-001).

15.4 SOPs: Acceptable Manual Integration Practices / CA-Q-S-002, Calibration Curves and Selection of Calibration Points / CA-Q-P-003, Waste Disposal / NV10-83, Training Procedures for Technical Staff / NV08-199, Detection Limits / CA-Q-S-006, Reagent and Standard Purchase / NV08-214.

15.5 Controlled Document: TestAmerica Nashville – Acronyms, Keywords, and Definitions QAF-45.

16.0 Method Modifications

None.

17.0 Attachments

Molecular Weight and Retention Times of Target Compounds

Compounds in Elution Order	Molecular Weight	Retention Time (minutes)
Methane	16.04	1.05
Ethene	28.05	1.37
Ethane	30.07	1.47
Acetylene	26.04	1.68
Propane	44.11	2.78
Isobutane	58.12	2.82
Butane	58.12	3.16

18.0 Revision History

- Revision 6, dated 31 March 2010
  - Integration for TestAmerica and STL operations.
  - Removal of 3810 reference.
  - Addition of Section 14.2 and QAF-45.
- Revision 7, dated 31 January 2011
  - Remove autosampler references.
  - Update temperature program used.
  - The secondary standard is a different lot from the primary standard.
  - Addition of 8000C information on weighting factors in linear calibration section.
  - Simplification of equations.
- Revision 8, dated 30 September 2011
  - Organizational changes.
  - Addition of non-potable and potable water matrices.
  - Addition of Isobutane and Butane.
  - Update concentrations of Methane, Ethane, Ethene, Propane.
  - Change to the Level 7 concentration for example calculation for Methane in Section 11.
- Revision 9, dated 15 December 2011
  - Addition of Department Supervisor.
  - Addition of Hexane and Pentane information.

- Revision 10, dated 31 May 2012
  - Change low standard control limits to ± 20%.
  - Clarify Sections 11.6, Linear Calibration Using a Least Squares Regression, and 11.8, Sample Concentration.
- Revision 11, dated 31 July 2014
  - Organizational changes.
  - Revised reporting limit for Isobutane.
  - Added amendments 10a and 10b.
  - Modified amendment 10b regarding transferring and shaking steps to prepare the samples. Changed calibration standard concentrations. Added CCV to beginning of analytical sequence. Improved example calculations.
- Revision 12, dated 6 March 2015
  - Organizational changes.
  - Removed the use of headspace vials.
  - Sample preparation in VOA vials.
  - Removed target analytes Pentane and Hexane
- Revision 13, dated 31 August 2016
  - Organizational changes.
  - Incorporation of change forms
    - 12a: change spike amount of Acetylene; clarify sample preparation steps; correct low volume requirement for the dilution table.
    - 12b: change frequency for 5-point calibration and Methane RL in Section 9.2. Establish % recovery for RL verification.
    - 12c: add a holding time criterion for unpreserved samples.
  - Update SOP references.
  - Add a lower level calibration standard.

Lab SOP 07

TOC, Analysis of Total Organic Carbon in Aqueous Samples by EPA Method 5310B, Revision 10, 8/30/2016, TestAmerica Laboratories, Inc.

Title: TOC, Analysis of Total Organic Carbon in Aqueous Samples By Method No. EPA 5310B

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Approvals (Signature/Date):

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Carl Armbruster Date	Mark Acierno Date
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Facility Distribution No. Electronic **Distributed To:** Facility Intranet

1.0 Scope and Application

1.1. This SOP describes the procedure to measure the organic carbon in drinking, surface and saline waters, domestic and industrial wastes using Standard Methods, Test Method 5310B.

1.2. The carbonaceous analyzer measures all of the carbon in a sample. The manner of preliminary treatment of the sample and instrument settings defines the types of carbon being measured. Forms of carbon that are measured by this method are:

- Soluble, nonvolatile organic carbon such as sugars
- Soluble, volatile organic carbons such as mercaptans
- Insoluble, partially volatile carbons such as oils
- Insoluble particulate carbonaceous matter such as cellulose filters
- Soluble or insoluble carbonaceous matter adsorbed or insoluble organic suspended matter such as oily matter adsorbed on silt particles.

1.3. This procedure is applicable to homogeneous samples that can be injected by means of a microliter syringe.

1.4. The method detection limit is 1.0 mg/l total organic carbon. Samples with total organic carbon concentrations higher than 100 mg/l must be diluted first with deionized water.

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 19 (Test Methods and Method Validation) of TestAmerica Edison's Quality Assurance Manual (ED-QA-LQM).

2.0 Summary of Method

2.1. Organic carbon in a sample is converted to carbon dioxide by catalytic combustion. The CO_2 formed can be measured directly by a non-dispersive infrared gas analyzer. The NDIR outputs a detection signal that generates a peak whose area is proportional to the TOC concentration of the sample. Dissolved organic carbon is filtered through a 0.45um filter and preserved prior to sample analysis.

2.2. Inorganic Carbon is decomposed to become CO2 when a sample is introduced into an IC reactor vessel where a carrier gas is flowing. The IC concentration is determined similar to TOC run.

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. Carbonate and bicarbonate interference. Remove by acidification and purging sample with nitrogen.

4.2. Acidification and purging can result in the loss of volatile organic substances.

4.3. Homogeneity of samples can cause a problem if the sample cannot be readily injected into the instrument.

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 auto sampler has a probe that is sharp; use caution not to stick yourself.

The furnace is very hot and can cause severe burns if touched.

5.2. Primary Materials Used

The following is a list of the materials used in this method, which have a serious or significant hazard rating. **Note: This list does not include all materials used in the method. The table contains a summary of the primary hazards listed in the MSDS for each of the materials listed in the table.** A complete list of materials used in the method can be found in the reagents and materials section. Employees must review the information in the MSDS for each material before using it for the first time or when there are major changes to the MSDS.

Material (1)	Hazards	Exposure Limit (2)	Signs and symptoms of exposure
Phosphoric Acid	Corrosive	1 Mg/M3 TWA	Inhalation is not an expected hazard unless misted or heated to high temperatures. May cause redness, pain, and severe skin burns. May cause redness, pain, blurred vision, eye burns, and permanent eye damage.

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 – 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

- Shimadzu TOC-V-CSH/CPN
- Shimadzu TOC-L
- Autosampler: ASI-V, SAI-L
- Analytical Balance
- Printer

6.2. Supplies

- Scintillation Vials
- Air, Ultra Grade 50 psig
- Eppendorf Pipettes, varying volumes
- Whatman syringe filters & 0.45 um syringe filters

7.0. Reagents and Standards

7.1. Reagents

7.1.1. Sulfuric Acid, conc.: Reagent grade.

7.2. Standards

7.2.1. Potassium Hydrogen Phthalate, KHP, stock (2000 mg/l TOC): Partially fill a 100-ml amber flask with deionized water and dissolve 0.426 g of potassium hydrogen phthalate (reagent grade). Bring to volume with deionized water. Store at 4 ° C. Prepare every six months.

7.2.2. Prepare a fresh 100 mg/l KHP standard solution by diluting 5 ml of 2000 mg/L stock solution (section 7.2.1) to a final volume of 100 ml with deionized water. Also prepare a 10 mg/l and a 1.0 mg/l KHP standard solution by diluting 10 ml and 1.0 ml of the 100 mg/l solution to a final volume of 100 ml with deionized water, respectively. Acidify all standard solutions and a 0-ppm standard with conc. H_2SO_4 to a pH of 2. Store at 4°C. Prepare every three months.

7.2.3. Potassium Hydrogen Phthalate, KHP, secondary stock (2000 mg/l TOC): prepared similar to section 7.2.1 but taken from a different manufacturer or lot number. Store at 4°C. Prepare every six months.

7.2.4. Inorganic Carbon stock solution (2000 mg/L): Dissolve 0.70 g of Sodium bicarbonate, $NaHCO_3$ and 0.882 g of Sodium Carbonate, Na_2CO_3 (heated for 1 hour at 285℃ and cooled) in deionized water in 100 ml volumetric flask. Bring the volume to mark with DI water. Do not acidify, prepare every six months. Standard solution is used for TIC determination.
Prepare running standard solutions at 0, 1.0 ppm, 10.0 ppm and 100 ppm concentrations.

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[1]	Holding Time	Reference
Waters	Polyethylene, glass	50 mLs	H_2SO_4 or HCl to pH < 2; Cool 4 ±2°C	28 Days	40 CFR Part 136.3

[1] Samples for TIC determination should be taken from the unpreserved sample.

Note: Samples for dissolved organic carbon must 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.

9.0. Quality Control

9.1. **Sample QC** - The following quality control samples are prepared with each batch of 10 samples. This does not include MS, MSD, Blank, and Laboratory Control Sample. Samples can be put into a batch for up to 2 weeks after the initial date; that is the date the first sample was analyzed.

9.1.1. Method/ Preparation Blank: For every QC batch, prepare a Method Blank that consists of deionized water which is pH adjusted to ≤ 2.0 and is carried through the entire analytical procedure. This will identify any system/ process contamination. The results of this analysis must fall below the reporting limit.

9.1.2. Filtering Blank: If Dissolved Organic Carbon (DOC) is to be determined, analyze a blank sample which has been filtered through a 0.45 um-pore-diameter filter.

9.1.3. Matrix Spike and Matrix Spike Duplicate: Two portions of the same sample (matrix spike and matrix spike duplicate) are spiked with 50ppm (1.25 ml of 2000 ppm stock (section 7.2.1) is spiked into 50 ml volumetric flask and brought up to the final volume with the sample aliquot). The recovery for MS/MSD and RPD must be within laboratory generated control limits. See Section 11.0 to calculate recoveries.

9.1.4. Laboratory Control Sample (LCSRM) TOC/TIC mix: The LCS is analyzed daily and is used to measure method performance on the matrix being analyzed. The TOC LCS is obtained from an outside vendor and the TIC LCS is prepared in-house. To monitor the efficiency of the instrument's purging process (removal of inorganic carbon) a 50 mg/L TIC standard is added to the TOC LCS standard.

- In a 1000 ml volumetric flask prepare TOC LCS following manufacturer's instructions and at a concentration that falls within the middle range of the calibration curve. Spike the solution with 50 mg/L TIC by adding 25ml of the 2000ppm TIC standard (Sec. 7.2.4) and bring the volume to mark with DI water.

- The LCS recovery must be within vendor specified QC limits. If LCS recovery is high, it is possible that the purging process was incomplete. Prepare a new LCS and re-analyze. If results are still outside of the acceptance limits, perform corrective action. Check the sparge flow and verify that the sparge tube is not clogged, if necessary perform instrument maintenance. Analysis should not continue until acceptable LCS result is obtained.

9.2. **Instrument QC**

9.2.1. Initial Calibration Verification (ICV), 50 mg/L: The curve must be verified by analyzing an Initial Calibration Verification solution immediately after the

calibration curve. The ICV is approximately at mid-range standard (50 mg/L) and is prepared by adding 2.5 ml of the KHP, secondary source, (Sec. 7.2.3) into a 100 ml volumetric flask and brought to the mark 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), 50 mg/L: The validity of the calibration curve must be verified periodically during the analysis. A Continuing Calibration Verification is prepared the same way as ICV (see section 9.2.1). The solution must be analyzed following every ten samples; "samples" include matrix spike, matrix spike duplicate, laboratory blank, Laboratory Control Sample (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 (CCV), a calibration blank must be analyzed. Use deionized water which is pH adjusted to ≤ 2.0. The results of this analysis must fall below the reporting limit.

10.0. Procedure

10.1. Equipment preparation:

10.1.1. Instrument:TOC-V-CSH/CPN and ASI-V

10.1.2.1. Turn on power of TOC-VCSH and ASI-V. Turn Air, Ultra Zero pressure up to 50 psig.

10.1.2.2. Allow instrument to stabilize for about 30 minutes until screen shows “Ready” (combustion temperature at 680℃).

10.1.2.3. On the computer desktop click on “TOC-Control V” icon. This will open the ‘Control V Sample Table’ window, click File then New.

10.1.2.4. Select “Sample Run.” This will bring up a system selection screen. For TOC determination, choose system “TOC-V CSH,” click “ok” and “save.” For TIC, select system “TOC-V W/ ASI.”

10.2. Calibration

10.2.1. Instrument: TOC-V-CSH/CPN and ASI-V

10.2.1.1. Prepare the standards as described in section 7.2.2 and begin setting up the instrument.

10.2.1.2. Select 'Calibration Curve' to run new calibration.

10.2.1.3. Click 'Next' twice and select 'NPOC' in the analysis box.

10.2.1.4. Uncheck 'Zero shift' box to disable, and check 'Multiple injection' box.

10.2.1.5. Enter file name in the format: HVYYMMDD (i.e. HV080403).

10.2.1.6. Click 'Next' and set the following conditions:

- Number of injections: 4
- Sparge time: 10 minutes

10.2.1.7. Enter the calibration concentration points in mg/L (0, 1.0, 10.0, and 100).

10.2.1.8. Click 'Next' twice then click 'Finish.'

10.2.1.9. From the drop down menu, click on 'File' then 'New.'

10.2.1.10. Select 'Sample Run' and click 'ok' twice.

10.2.1.11. Click on 'Save' to save the calibration curve.

10.2.1.12. Click on the #1 position (row 1). From the drop down menu, select 'Insert,' and 'Calibration curve.'

10.2.1.13. Double click on the calibration curve file just created.

10.2.1.14. Enter the position of each of the vials (i.e. 1-4), click 'ok.'

10.2.1.15. From the taskbar, click the 'Connect' (lightning) icon, then select 'Use Settings on PC.'

10.2.1.16. From the taskbar, click 'Start,' (traffic light icon), then click 'Standby' and 'ok.'

10.2.1.17. Uncheck 'External acid addition' box to disable, and then click 'Start.'

10.2.1.18. After completion of measurement, check calibration and linearity of range, correlation coefficient should be $\geq$ 0.997.

10.2.2. Calibration standards are prepared and analyzed every 3 months or when QC requirements fail, whichever is first.

10.3. Sample Analysis:

10.3.1. Instrument: TOC-V-CSH/CPN and ASI-V

10.3.1.1. Check that sample pH is less than 2 or acidify with H_2SO_4. Pour well-mixed samples into scintillation vials and position them in the autosampler. Be sure to fill all the vials between initial sample and final sample in the turntable without leaving any empty positions.

10.3.1.2. Follow Section 10.1.2.1-10.1.2.4 (Equipment preparation). Select 'Sample' and '2008 Data;' folder. The 2008 Data folder stores the data for the particular year.

10.3.1.3. The system will create a filename for the analysis (i.e. TOC-2008_04_03_14_41_49_0.t32). Click 'Save' to save the analysis on this file.

10.3.1.4. When the excel Table opens up, click 'Connect' icon and select 'Settings on PC.'

10.3.1.5. From the drop-down menu, select 'Insert' and 'Auto Generate.'

10.3.1.6. From the message box, click 'Method.' Select the appropriate method (i.e. 5310B).

10.3.1.7. Click 'Next,' then enter the No. of Samples and Start vial position.

10.3.1.8. Click 'Next' twice, then click 'Finish' and 'ok.'

10.3.1.9. From the taskbar, click the 'Start' icon then click 'Standby' and 'ok.'

10.3.1.10. Uncheck the 'External Acid addition,' and click 'Start.'

10.3.1.11. Take reading of sample from two separate aliquots and record the average of two results. Dilute samples that are over the calibration range and record dilution in TALS; denote dilution with @ symbol.

10.3.1.12. Repeat injection until consecutive measurements are obtained that is within 10% RPD.

Note: Certain projects (i.e. DOW samples) will require four injections where the third and fourth injections are averaged. Check that the RPD of the third and fourth results are within 10%, if not, repeat injection. Refer to TALS method *SM5310_TOC_B* when processing DOW samples.

10.3.1.13. For TIC DETERMINATION:

Ensure that there is sufficient amount of 25% H3PO4 into the IC

reagent container.

If IC reagent does not flow into the vessel, run the program for REGENERATION OF IC SOLUTION on MAINTENANCE SCREEN. Also, check the joint of IC reagent container for any leakage.

Follow section 10.3.2 for analysis.

10.3.1.14. For DOC Determination:

- If samples are lab filtered, the unpreserved sample must be filtered through a 0.45um filter.
  Note: Whatman 0.45um syringe filter must be rinsed with deionized water prior to use.
- Collect the required volume of filtrate and immediately acidify the filtrate with H2SO4 to a pH of <2.

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 on instrument (mg/L)
B = dilution factor

11.4. Data Reduction

11.4.1. Instrument data is imported to TALS by following these steps:

11.4.1.1. Once the run is complete, highlight all samples in the sample table.

11.4.1.2. Go to 'File' then from the drop down menu, select 'ASCII export options,' then click 'Misc' tab and uncheck 'Export Strings in Quotation Marks.' Click 'Ok.'

11.4.1.3. Go to 'File' then from the drop down menu, select 'ASCII export.' This opens up a save window, choose 'C:/TALS Export.' Then click 'Open.' Enter the file name and click 'Save.'

11.4.1.4. From the desktop, choose 'TALS export' icon. Browse for the file by clicking on the '…' button and select the file, click 'Open.' Next, use the drop down menu to choose the analyte (i.e. TOC, TIC, or DOC). Then hit 'Transfer file.'

11.4.1.5. The data has now been transferred to TALS.

11.4.2 Record special notes and observations in the 'worksheet' tab (i.e. sample appearance and notes on why samples were rejected).

11.4.3 Record reagent information in the prep batch information (this can be viewed in the 'batch information' page).

11.4.4 All raw data is attached as a pdf file. The raw data includes the instrument report and calibration curve.

11.4.5 Analyst must fill out the Wet Chem Data Review Checklist (WI# EDS-WI-008) during the first level review. After the second level review, the checklist is filed in the 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. The following waste streams are produced when this method is carried out:

Waste TOC Acid: This material contains sulfuric acid is collected in 5 gallon poly containers at satellite accumulation and will be submitted for elementary neutralization using 50 % sodium hydroxide (Siedler Chemical SC-1824-03) and 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

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 5310B.

15.2. Standard Methods for the Examination of Water and Wastewater , 22nd Edition, American Public Health Association, Washington, DC 2011, Editorial Revision 2011, SM 5310B-11.

15.3. Shimadzu TOC-5000/ASI-5000 Systems Manual.

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 Edison SOP No. ED-SPM-008, *Laboratory Waste Disposal Procedures*, current revision.

15.7. TestAmerica Corporate Environmental Health and Safety Manual, Document No. CW-E-M-001, most current revision.

15.8. TestAmerica Edison Work Instruction # EDS-WI-008, Wetchem Data Review Checklist, most current revision.

16.0. Method Modifications:

None

17.0. Attachments

None

18.0. Revision History

- Revision 10, dated 30, August 2016
  - Sec 10.3.1.12: Added reproducibility criteria (10% RPD) at sample injection; added special instructions when analyzing and processing DOW samples. Subsequent sections adjusted accordingly.
- Revision 9, dated 02, October 2014
  - Sec. 9.1.4: Revised LCS to TOC/TIC mix; the addition of inorganic carbon in the TOC LCS standard will monitor the efficiency of the instrument's purging process.
  - Sec 9.1: Revised batch QC frequency from 1/20 to 1/10 samples.
  - Sec 15.2 Added reference to Standard Methods Editorial revision 2011; subsequent sections adjusted accordingly.
- Revision 8, dated 08 October 2012
  - Added Sec 1.5; updated LQM reference in Sec 12 to reflect the most current LQM revision.
  - Deleted all references to TOC-5000 and ASI-5000 (i.e. equipment list, calibration procedure); sections adjusted where applicable.
  - Sec 6.1: Added TOC instrument Shimadzu TOC-L and autosampler SAI-L to list of equipment.
  - Sec 10.2.2 (previously sec 10.2.3): Revised preparation frequency for the calibration standards from 6 months to 3 months.
  - Sec 10.3.1.13 (previously Sec. 10.3.2.13): Added note that syringe filters must be

rinsed with DI prior to use.

- Revision 7, dated 08/03/2010
  - Section 3: Updated the LQM reference for the list of definitions.
  - Sec. 9.2.3: Revised to include ICB; added acidified deionized water.
  - Added dissolved organic procedures in Sections 2.1, 6.2, 8.0 and 10.3.2.13.
  - Sec 11.4: Revised data reduction procedures in accordance with new TALS.
  - Sections 13 and 14: Sections updated to reflect the most current TestAmerica Corporate Quality SOP No. CW-QS-002 (Writing a Standard Operating Procedure (SOP)).
  - Sec. 15: Added applicable references.

- Revision 6, dated 04/07/2008
  - Updated to format as per TestAmerica SOP format.
  - Modified sample sparge time from 2 minutes to 10 minutes to comply with method 5310B, (Section 10.1.1.4).
  - Deleted instrument condition for soil; not applicable.
  - Added secondary stock solution, (section 7.2.3).
  - Added Filtering blank in sample QC when analyzing DOC (section 9.1.2).
  - Added procedure for the Calibration and Sample analysis for instrument TOC-V-CSH/CPN and ASI-V, (sections 10.2.2 and 10.3.2).

- Revision 5, dated 11/12/07
  - Section 1.1. Deleted reference to Method 415.1; USEPA Method Update Rule has removed the method from use.
  - Section 4. Apparatus and Materials. Added new TOC instrument and autosampler:
    4.3.ShimadzuTOC-V-CSH/CPN
    4.4. Autosampler ASI-V
  - Section 9. Procedure.
    Section 9.2. Added equipment preparation procedure for the Shimadzu TOC-V-CSH/CPN. Subsequent sections adjusted accordingly.
    Section 9.5. Added sample analysis procedure for the Shimadzu TOC-V-CSH/CPN. Subsequent sections adjusted accordingly
  - Section 15.1 References. Deleted EPA 600/4-79-020, Test Method 415.1 reference as a result of the Method Update Rule.

Lab SOP 08

Method SM4500-S2D and SM4500-S2B: Sulfide (Colorimetric, Methylene blue), Revision 8a, 11/2/2015, TestAmerica Laboratories, Inc.

TestAmerica
THE LEADER IN ENVIRONMENTAL TESTING

Nashville
Standard Operating Procedure (SOP)
Change Form

SOP Number/Revision No.: **SM4500-S2 D / NV07-180.8**	Effective Date: **11/2/2015**

Last Mod. Date: 8/31/15

SOP Title: **Method SM4500-S2 D and SM4500-S2 B: Sulfide (Colorimetric, Methylene blue)**

CONTROLLED DISTRIBUTION

ISSUED TO: \\TAFS\Lab\Nashville\Public\QA\SOPs, 07

Revision Number with Mod ID: **8a**

The following SOP change is in effect as of the stated date. This form will remain attached to the referenced SOP until such a time that the SOP is updated, approved, and redistributed, at which time it will become part of the historical SOP record. ***Append this form to the front of the SOP copy.***

1. Reason for SOP Change:

☐ Typographical Corrections (Non-Technical) – Re-Training Not Required.

☐ Typographical Corrections (Technical – Define) – Analyst acknowledgement of corrections is required.

■ Procedural Changes (Define Below) – Re-Training Required.

☐ Other

2. Summary of Procedure Change: Add highlighted, italicized text, delete crossed-out text.

9.2 Instrument QC:

Quality Controls	Frequency	Control Limit	Corrective Action
Initial calibration (minimum five standards)	Initial ~~daily~~ calibration prior to sample analysis, *as needed.*	$r^2 \geq 0.990$, $r \geq 0.995$ for linear regression	Correct problem then repeat initial calibration

Section 10.3, Calibration, Manual method, step 2:

For the manual method	
1	Transfer 10.0 mL sample to a centrifuge tube using a wide-tip pipet. If the sample has been preserved with Zinc acetate, shake vigorously before taking the sub-sample.
2	Add 0.7 mL Amine-sulfuric acid reagent and 0.2 mL (4 drops) $FeCl_3$ solution to the centrifuge tube. Mix immediately by inverting slowly, only once. (Excessive mixing causes low results by loss of H_2S as a gas before it has had time to react*). Wait 3-5 minutes and add 2 mL Diammonium hydrogen phosphate solution. Wait 3 to 15 minutes to make the determination.*
3	To the blank, add 0.5 mL (13 drops) 1+1 H_2SO_4 and 0.2 mL (4 drops) $FeCL_3$ solution. Mix. The presence of S^{2-} is indicated by the appearance of blue color. Color development usually is complete in about 1 minute, but a longer time often is required for fading out of the initial pink color. Wait 3 to 5 minutes, and make color comparisons. If Zinc acetate was used, wait at least 10 minutes before making a determination.

Sessily Overton-May 11/2/15	
Department Manager Approval Date	
 10/2/15	 10/2/15
Quality Assurance Approval Date	Technical Director Date

Title: SULFIDE (COLORIMETRIC, METHYLENE BLUE) METHODS SM4500-S^{-2} D AND SM4500-S^{-2} B

Approvals (Signature/Date)			
		Wm Ryan Fitzwater	8/21/15
		Ryan Fitzwater Health & Safety Manager / Coordinator Laboratory Director	Date
Sessily Overton-Gray	8/25/15	Michael H. Dunn	8/3/15
Sessily Overton-Gray Department Manager	Date	Michael H. Dunn Technical Director Interim QA Manager	Date

1.0 Scope and Application

1.1 Analyte, Matrices: The method is applicable to the measurement of total and soluble sulfides in drinking, surface, and saline waters, domestic and industrial wastes.

1.2 Reporting Limits: The RL is nominally 0.1 mg/L.

If for any reason a part of this method cannot be followed, seek the guidance of the Department Supervisor, Department Manager, or the Technical Director. All abnormalities must be noted in the Laboratory Information Management System (LIMS).

2.0 Summary of Method

Sulfide reacts with N, N-dimethyl-p-phenylenediamine (p-aminodimethyl aniline) in the presence of Ferric chloride to produce methylene blue, a dye which is measured at a wavelength 640- 660 ηm

3.0 Definitions

See TestAmerica Nashville's Quality Assurance Manual for laboratory definitions. Also, refer to Controlled Document QAF-45, TestAmerica Nashville Acronyms, Keywords, and Definitions.

4.0 Interferences

4.1 Samples must be taken with a minimum of aeration. Sulfide may be volatilized by aeration and any oxygen inadvertently added to the sample may convert the sulfide to an unmeasurable form.

4.2 Color and turbidity may interfere with photometric readings.

4.3 Eliminate interferences due to sulfite, thiosulfate, iodide, and many other soluble substances, but not ferrocyanide by first precipitating Zinc sulfide (ZnS), removing the supernatant, and replacing it with reagent water.

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 document 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 SDS 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 SDS for each material before using it for the first time or when there are major changes to the SDS.

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.
Aluminum chloride	Reactive	2 mg/m^3 TWA	Do not allow water to get into the container because of violent reaction. Minimize dust generation and accumulation. Contents may develop pressure upon prolonged storage. Do not et in eyes, on skin, or on clothing. Do not ingest or inhale. Do not allow contact with water. Use only in a chemical fume hood. Store in a cool, dry, dark place.
Ferric chloride	Corrosive	1 mg/m^3 as Fe TWA	Extremely destructive to tissues of the mucous membranes and upper respiratory tract. Symptoms may include burning sensation, coughing, wheezing, laryngitis, shortness of breath, headache, nausea and vomiting. Swallowing can cause severe burns of the mouth, throat, and stomach. Can cause sore throat, vomiting, diarrhea. Low toxicity in small quantities but larger doses (30 mg/kg) may cause nausea, vomiting and diarrhea. Pink urine discoloration is a strong indicator of iron poisoning. Liver damage, coma and death may follow, sometimes delayed as long as three days. Can cause blurred vision, redness, pain and severe tissue burns. Repeated ingestion may cause liver damage. Prolonged exposure to the eyes may cause discoloration.
Sulfuric acid	Corrosive Oxidizer Dehydrator Poison Carcinogen	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.
n, n-Dimethyl-p-phenylene-diamine	Toxic Irritant	LD50 25 mg/kg	May be fatal if inhaled. Causes respiratory tract irritation. Toxic if absorbed through skin. Causes skin irritation. Causes eye irritation. May be harmful if swallowed.
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

- Lachat Model 8000 or 8500, flow-injection analyzer, with pump and autosampler.
- Filter photometer, with filter providing transmittance at 660 ηm.
- UV/VIS spectrophotometer, Hach DR6000, or equivalent.

6.2 Supplies

- Volumetric flask, 100-mL, 500-mL, 1-L, Class A.
- Centrifuge tubes.
- Laboratory glassware, Class A.
- Stir plate with stirring bar.

7.0 Reagents and Standards

7.1 **Reagent water**, analyte-free.

7.2 Hydrochloric acid, 6N: Prepare by diluting 500 mL concentrated HCl with 500 mL reagent

water. Add acid to water. A commercial solution is acceptable.

7.3 For the automated (Lachat) method:

7.3.1 **Stock n, n-Dimethyl-p-phenylenediamine Reagent:** In a 500-mL, Class A, volumetric flask, dissolve 1.00 g n,n-Dimethyl-p-phenylenediamine dihydrochloride (Aldrich 21,923-1 or equivalent) in about 450 mL 6 M Hydrochloric acid. Dilute to volume with 6 M HCl. If the prepared reagent appears dark, discard and obtain a fresh supply of the diamine reagent. Store in the dark. A commercial product is acceptable.

7.3.2 **Working Diamine / HCl Reagent**: To a 1-liter volumetric flask, add about 750 mL reagent water and 200.0 mL of reagent described in 7.2. Mix briefly, dilute to volume with reagent water, and invert 3 times.

7.3.3 **Ferric chloride solution, 0.1 N:** In a 500-mL, Class A, volumetric flask, dissolve 13.5 g Ferric chloride hexahydrate in about 450 mL 6 M HCl. Dilute to volume with 6 M HCl and invert 3 times.

7.4 For the manual method:

7.4.1 **Amine-sulfuric acid stock solution:** Dissolve 27 g n,n-Dimethyl-p-phenylenediamine oxalate in an iced mixture of 50 mL concentrated H_2SO_4 and 20 mL reagent water. Use of 1:1 or 50% acid is acceptable. Cool and dilute to 100 mL with reagent water. Smaller proportional volumes are acceptable.

7.4.2 **Amine-sulfuric acid reagent:** Dilute 2.5 mL of the stock solution with 97.5 mL 1:1 H_2SO_4. Store in a dark glass bottle.

7.4.3 **Ferric chloride solution:** Dissolve 100 g $FeCL_3{\cdot}6H_2O$ in 40 mL reagent water.

7.4.4 **Sulfuric acid solution:** 1:1 H_2SO_4 or 50%.

7.4.5 **Diammonium hydrogen phosphate solution:** Dissolve 40 g $(NH_4)_2HPO_4$ in 100 mL reagent water.

7.5 Primary Working Stock Standard: In a 500-mL volumetric flask, dissolve 3.75 g Sodium sulfide nonahydrate in about 450 mL reagent water. Dilute to volume and invert 3 times. Standardize this solution daily using iodometric titration. (See SOP SM4500-S^{-2} F / NV07-32.) The concentration is about 1000 µg/mL. Actual concentration may vary slightly based on daily standardization.

7.6 Zinc acetate solution, 2M: Preserve client samples with 0.2 mL 2M zinc acetate solution per 100 mL sample.

7.7 See SOP Standard and Reagent Purchase, Preparation, Control, Documentation / NV08-214 for shelf-life and storage requirements.

8.0 Sample Collection, Preservation, Shipment and Storage

Matrix	Sample Container	Min. Sample Size	Preservation	Holding Time	Reference
Water	HDPE or glass	250 mL	Cool 4 ± 2°C, Total sulfide: Zinc acetate, NaOH to pH >9. Soluble sulfide, add only NaOH.	As soon as possible but no more than 7 days	40 CFR Part 136.3

9.0 Quality Control

Refer to TestAmerica Nashville's QA Manual for specific quality control (QC) procedures. The laboratory maintains a formal quality assurance program and records to document the quality of the data generated.

9.1 Sample QC

The following quality control samples are prepared with **each batch of 20 or fewer samples.**			
Quality Controls	**Frequency**	**Control Limit**	**Corrective Action**
Method Blank	1 per batch	< RL	Re-prep, rerun the samples that are < 10 times the blank result.
Laboratory Control Sample (LCS)*	1 per batch	See LIMS.	If %R is low, re-prep, rerun. If high, report non-detect samples, re-prep and reanalyze any positive samples.
Matrix Spike (MS)	1 per batch	See LIMS.	Report. Qualify as needed.
Matrix Spike Duplicate (MSD)	1 per batch	See LIMS.	Report.

*AZ, MA, and TX require LCSDs.

- **Method blank**: Use reagent water.
- **Laboratory Control Sample (LCS)**: Prepare from **the primary source**, a commercial source at 1000 µg/mL. Dilute 10 µL of 1000 µg/mL stock to 10 mL reagent water for a 1.0 µg/mL control.
- **Matrix Spike / Matrix Spike Duplicate (MS/MSD)**: Run one **matrix spike/matrix spike duplicate** per batch. Add 10 µL of 1000 µg/mL primary source standard to a 10-mL sample. Both the matrix spike and matrix spike duplicate are brought through the entire sample preparation and analytical process. Verification is required to ensure that neither a reducing condition nor chemical interference is affecting color development.

9.2 Instrument QC:

Quality Controls	**Frequency**	**Control Limit**	**Corrective Action**
Initial calibration (minimum five standards)	Initial daily calibration prior to sample analysis.	$r^2 \geq 0.990$, $r \geq 0.995$ for linear regression	Correct problem then repeat initial calibration
Initial Calibration Verification Sample (ICV), second source (may use LCS)	Immediately after calibration	90-110% recovery	Recalibrate
Continuing Calibration Verification Sample (CCV)	Every 10 samples	90-110% recovery	Correct, recalibrate, rerun samples. If high and samples are ND, it is acceptable to report the results.
Continuing Calibration Blank (CCB)	After each CCV	< RL	Clean, repeat samples. If samples are ≥ 10X blank, it is acceptable to report the results.

- **Initial Calibration**: See Section 10.2.
- **Initial Calibration Verification (ICV)**: from a 2nd source, as for LCS, run immediately after calibration
- **Continuing Calibration Verification Sample (CCV)**: The CCV is a mid-level standard**.** Samples must be bracketed by acceptable CCVs. Dilute 25 µL of the 1000 µg/mL primary source to 10 mL for a 2.5 µg/mL standard.
- **Continuing Calibration Blank**: Use reagent water.

10.0 Procedure

10.1 Sample Preparation: See SOP Sample Homogenization, Sub-sampling, and Compositing / NV08-229. Only mix samples that have had Zinc acetate preservation.

Matrix	Sample Size
Water	50.0 mL for total or soluble sulfide

- If using the Lachat method and samples are preserved with Zinc acetate, all samples and QC must be distilled.

Separation of Soluble and Insoluble Sulfides (if soluble sulfide is requested): To measure soluble sulfide, first remove insoluble matter by producing an Aluminum hydroxide floc that is settled, leaving a clear supernatant for analysis.	
1	To a 100-mL glass bottle, add 0.2 mL (nominally 4 drops) 6N NaOH unless the sample is already preserved to pH ≥ 9.
2	Fill the bottle with sample and immediately add 0.2 mL (4 drops) $AlCl_3$ solution.
3	Stopper the bottle with no air under the stopper. Rotate back and forth about a transverse axis vigorously for 1 minute or longer to flocculate the contents. Vary the volumes of these added chemicals to get good clarification without using excessively large amounts and to produce a pH of 8.5 to 9.0.
4	Let the floc settle until reasonably clear supernatant can be drawn off. With proper flocculation, this may take 5 to 15 minutes. Do not wait longer than necessary.
5	Decant and analyze the supernatant immediately.

10.2 Pretreatment for Total Sulfide

1	Eliminate interferences due to sulfite, thiosulfate, iodide, and many other soluble substances, but not ferrocyanide by precipitating ZnS using Zinc acetate, removing the supernatant, and replacing it with reagent water.
2	Shake to re-suspend the precipitate, and quickly withdraw a sample. The sample precipitate must remain suspended.

10.3 Calibration: Refer to SOP Calibration Curves and Selection of Calibration Points / CA-Q-P-003. See Section 11 for equations. Calculations are performed by vendor software and LIMS.

For the Lachat

1 Prepare a series of 6 standards by pipetting suitable volumes of working standard with a final volume of each standard being 10.0 mL. The low standard must be at or below the reporting limit (RL). Also, prepare a blank.

mL Standard Solution	Final Volume (mL)	Concentration (µg/mL)
1 mL of 0.5 µg/mL	10.0	0.05
1 mL of 1 µg/mL	10.0	0.1
1 mL of 2.5 µg/mL	10.0	0.25
1 mL of 5 µg/mL	10.0	0.5
1 mL of 10 µg/mL	10.0	1.0
2.5 mL of 10 µg/mL	10.0	2.5

2 Place calibration standards in test tubes and start the method. Obtain absorbance values for the standard curve.

3	Prepare a standard curve by plotting absorbance of standard versus the sulfide concentration. The correlation coefficient (r^2) must be ≥ 0.990 (r ≥ 0.995) or re-calibrate.
4	The verification of linearity must use a minimum of a blank and five standards. If a quadratic fit is selected for the curve, a minimum of a blank and six standards must be analyzed. Each standard above the report limit must be within 10% of its known value, and the standard at the report limit must be within 30% of its known value, or linearity must be re-established.
For the manual method	
1	Transfer 10.0 mL sample to a centrifuge tube using a wide-tip pipet. If the sample has been preserved with Zinc acetate, shake vigorously before taking the sub-sample.
2	Add 0.7 mL Amine-sulfuric acid reagent and 0.2 mL (4 drops) $FeCl_3$ solution to the centrifuge tube. Mix immediately by inverting slowly, only once. (Excessive mixing causes low results by loss of H_2S as a gas before it has had time to react)
3	To the blank, add 0.5 mL (13 drops) 1+1 H_2SO_4 and 0.2 mL (4 drops) $FeCL_3$ solution. Mix. The presence of S^{2-} is indicated by the appearance of blue color. Color development usually is complete in about 1 minute, but a longer time often is required for fading out of the initial pink color. Wait 3 to 5 minutes, and make color comparisons. If Zinc acetate was used, wait at least 10 minutes before making a determination.

10.4 Sample Analysis

1	See SOP Lachat / NV07-39 for operation.
2	Read the sulfide concentration from the calibration curve and calculate the result as in Section 11.
3	If the concentration is above the upper calibration standard, dilute with reagent water and re-analyze immediately.

10.5 Example Analysis Queue / Sequence*

1	Initial Calibration
2	ICV
3	Method Blank
4	LCS
5	Sample 1
6	LCS
7	MS
8	MSD
9	Samples 2-10
10	CCV
11	CCB
12	Samples 11-20
13	CCV
14	CCB

*May be up to 20 samples.

11.0 Calculations / Data Reduction

11.1 Accuracy: Not applicable.

11.2 Precision

$$RPD = \frac{\text{Absolute value (orig. sample value - dup. sample value)} \times 100}{\text{(Orig. sample value + dup. sample value)/2}}$$

11.3 Linear Regression using a least squares regression

This is most easily achieved by performing a linear regression of the instrument response versus the concentration of the standards. Make certain that the instrument response is treated as the dependent variable (y) and the concentration as the independent variable (x). This is a statistical requirement and is not simply a graphical convention.

The regression produces the slope and intercept terms for a linear equation in the form:

$y = ax + b$

y = instrument response
a = slope of the line
x = concentration of the calibration standard
b = the intercept

The analyst should not force the line through the origin, but have the intercept calculated from the five data points. Otherwise, the problems noted with the RSD value will occur, i.e., a line through the origin will not meet the QC specifications. In addition, do not include the origin (0, 0) as a sixth calibration point. The use of a linear regression may not be used as a rationale form reporting results below the calibration range demonstrated by the analysis of the standards. 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 0.995 or $r^2 \geq 0.990$.

11.4 Coefficient of Determination **Correlation Coefficient**

$$r^2 = \frac{\left(\sum xy\right)^2}{\sum x^2 \sum y^2} \qquad r = \frac{\left(\sum xy\right)}{\sqrt{\sum x^2 \sum y^2}}$$

y = Response
x = Concentration

11.5 Result Calculation

Obtain the concentration directly from the calibration curve to determine the concentration:

Concentration (mg/L) = (Concentration (µg/mL), instrument) x Dilution

Dilution =1, if no dilution

12.0 <u>Method Performance</u>

12.1 Method Detection Limit Study (MDL): The method detection limit (MDL) is the lowest concentration that can be detected for a given analytical method and sample matrix with 99% confidence that the analyte is present. The MDL is determined according to the laboratory's MDL procedure in SOP Detection Limits / CA-Q-S-006. 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 Capability: The laboratory demonstrates initial proficiency by generating data of acceptable accuracy and precision for target analyses in a clean matrix. The

laboratory also repeats the operation whenever new staff is trained or significant changes in instrumentation are made and on an annual basis thereafter. See TestAmerica Nashville's QA Manual and SOP Training / NV08-199 for information on how to accomplish this demonstration.

12.3 Training Requirements: Demonstration of Capability is performed initially when learning the method and annually thereafter. Four Laboratory Control Samples resulting in an average % recovery within the control limits and a precision less than the quality control maximum are required.

12.4 Proficiency Testing Studies: The laboratory participates in formal proficiency testing (PT) studies, where solutions of unknown concentrations are analyzed and the performance of all participants is compared. See the QA department for the results of recent PT studies.

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 the QA Manual and SOP Waste Disposal / NV10-83.

14.2 Wastestreams Produced by the Method:

- Titrated samples are taken to waste disposal for neutralization and discharge to the sanitary sewer.

15.0 References / Cross-References

15.1 Method SM4500-S^{-2} D, B, and C - 2000, Standard Methods for the Examination of Water and Wastewater, on-line edition, 2011 editorial revisions.

15.2 Lachat Method 10-116-29-1-A.

15.3 TestAmerica Nashville's Quality Assurance Manual.

15.4 Corporate Environmental Health and Safety Manual (CW-E-M-001).

15.5 SOPs: Waste Disposal / NV10-83, Training Procedures for Technical Staff / NV08-199, Balance Calibration / NV08-213, Detection Limits / CA-Q-S-006, SM4500-S^{-2} F / NV07-32, Calibration Curves and Selection of Calibration Points / CA-Q-P-003, Standard and Reagent Purchase, Preparation, Control, Documentation / NV08-214, Lachat / NV07-39, Sample Homogenization, Sub-sampling, and Compositing / NV08-229.

15.6 Controlled Document: QAF-45, TestAmerica Nashville – Acronyms, Keywords, and Definitions.

16.0 Method Modifications

Item	Modification
1	Analyzed by Lachat method, SM4500-S^{-2} I / Lachat / NV07-39.

17.0 Attachment

None.

18.0 Revision History

- Revision 4, dated 15 May 2009
  - Integration for TestAmerica and STL operations.
- Revision 5, dated 30 November 2010
  - Addition of QAF-45, Section 14.2, and soluble sulfide separation and preservation
  - Remove reference to EPA 376.2 (MUR).
- Revision 6, dated 30 September 2011
  - Organizational changes.
  - Addition of the pretreatment step to remove interferences and concentrate the sulfide.
  - Addition of SOP Calibration Curves (General).
- Revision 7, dated 4 October 2013
  - Organizational changes.
  - Specify that $r^2 \geq 0.990$.
  - Add Konelab.
  - OK no longer limits batches to 10 samples.
  - Add the Konelab instrument and re-define LCS as the primary standard.
- Revision 8, dated 31 August 2015
  - Organizational changes.
  - Add the need for LCSDs for AZ, MA, and TX samples.
  - Updated SOP references.
  - Remove the Konelab instrument. Add the manual method.
  - Add Zinc acetate and preparation and instructions when samples are preserved with Zn acetate.
  - Change the number of calibration standards.

Lab SOP 09

Analysis of Carbon Dioxide in Water and Wastewater by Standard Method 4500 CO_2D, Revision 6, 4/23/2014, TestAmerica Laboratories, Inc.

Title: Analysis of Carbon Dioxide in Water and Wastewater by Standard Method 4500 CO_2D

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1.0 Scope and Application

1.1 Analytes, Matrix(s), and Reporting Limits

1.1.1 Method 4500 CO_2 D is the procedure for determining Carbon dioxide in water through alkalinity calculation. The method is applicable when the sample's total alkalinity is due almost entirely to hydroxides, carbonates, or bicarbonates and the total dissolved solids are not greater than 500 mg/L. The laboratory's reporting limit is 5 mg/l for waters.

1.1.2 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

Carbon Dioxide is calculated from the sample pH and total alkalinity. Carbon Dioxide can be analyzed either manually or by automation (PC-titrate). For alkalinity procedures, refer to TestAmerica SOP No. ED-WET-039, *Analysis of Alkalinity in Water, Wastewater and Soil by Manual Titration or Auto-Titrator, Standard Method 2320B*. A method blank and duplicate are analyzed with each batch of 20 samples or less.

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 Dissolved gas will affect the CO2 value; prevent undo agitation or exposure to atmosphere as much as possible.

4.2 Soaps, oily matter, suspended solids or precipitates may coat the electrode; clean electrode occasionally. Allow electrode to come to equilibrium between additions of titrant. Do not filter, dilute or alter the 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 De-hydrator	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.			

6.0 Equipment and Supplies

6.1. Instrumentation

- MAN-TECH PC-Titration Plus
- pH meter

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)
- 500ml Erlenmeyer flask +watchglass (for standardization)

7. Reagents and Standards

7.1. Reagents

7.1.1. 0.05N Sodium Carbonate Solution: Place 0.625g Na_2CO_3 (dried 4 hours at 250ºC) in a 250 ml volumetric flask and dilute to the mark with deionized water. Solution is good for one week; refrigerate until ready to use.

7.2. Standards

7.2.1. 0.10N H_2SO_4: Dilute 3 ml of concentrated H_2SO_4 with de-ionized water to 1000 ml final volume. This reagent is stable for 6 months and should be stored at room temperature. The solution should be standardized every 2 weeks. Standardize against Na_2CO_3 solution as follows:

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 the flask with a watch glass and boil for 3 minutes.

7.2.1.4. Cool and continue titrating to pH 4.5.

7.2.1.5. Titrant concentration (N)= $\frac{2.50 \times A}{53.00 \times B}$

Where: A= volume of Na_2CO_3 (ml)
B= volume of titrant (ml)

7.2.2. 0.020N H_2SO_4: Purchased commercially. For stability and storage information, refer to manufacturer's instructions. The solution should be standardized every 3 months. Standardize against Na_2CO_3 solution as follows:

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= volume of Na_2CO_3 (ml)
B= volume of 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	Reference
Water	Glass-limit headspace	50 mls	Cool 4 ±2°C	Analyze immediately	Standard Methods 4500 CO_2D

9. Quality Control

9.1. Sample QC - The following quality control samples are prepared with each batch of 20 samples or less.

9.1.1. Method Blank: A blank must be analyzed each time samples are analyzed. Use deionized water for the blank and the results must be below the reporting limit. If the result is greater than the reporting limit, all samples associated with the MB must be re-analyzed.

9.1.2. 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.

9.2. Instrument QC

None

10. Procedure

10.1. Sample Preparation

N/A

10.2. Calibration

10.2.1. If the sample is to be analyzed by the PC-titrator, calibrate the instrument daily by following the calibration procedures in TestAmerica SOP No. ED-WET-039, *Analysis of Alkalinity in Water, Wastewater and Soil by Manual Titration or Auto-Titrator, Standard Method 2320B.*

10.3. Sample Analysis

10.3.1. Rinse all glassware well with deionized water.

10.3.2. Determine sample pH and total alkalinity using TestAmerica SOP No. ED-WET-060, *Analysis of pH for Waters and Drinking water Measured Electrochemically,EPA 150.1, SM 4500 H +B, SW846 9040B;* and ED-WET-039*, Analysis of Alkalinity in Water, Wastewater and Soil by Manual Titration or Auto-Titrator, Standard Method 2320B,* current revision .

10.3.3. For samples which are analyzed by manual titration, calculate the Carbon Dioxide following the calculation procedure in sections 11.2 and 11.3.

10.3.4. For samples which are analyzed by the PC-titrator, CO2 results are automatically calculated. Import data to TALS, see Sec. 11.4.

11.0. Calculations / Data Reduction

11.1. Precision (RPD):

$$\text{Matrix Duplicate (MD)} = \frac{|\text{orig. sample value - dup. sample value}|}{[(\text{orig. sample value + dup. sample value})/2]} \times 100$$

11.2. Free Carbon Dioxide:

$$\text{mg } CO_2/L = 2.0 \times B \times 10^{(6-pH)}$$

Where:
$$B = \text{Bicarbonate Alkalinity} = \frac{T - 5.0 \times 10^{(pH-10)}}{1 + 0.94 \times 10^{(pH-10)}}$$

Where:
T= Total Alkalinity, mg $CaCO_3/L$

11.3. Total Carbon Dioxide:

$$\text{mg total } CO_2/L = A + 0.44(2B + C)$$

Where:
A=mg free CO_2/L
B=Bicarbonate Alkalinity
C=Carbonate Alkalinity= $0.94 \times B \times 10^{(pH-10)}$ (Where B= Bicarbonate alkalinity)

11.4. Exporting Data from PC titrate to TALS

11.4.1. From the main menu, click “reporting” then click “prepare and/or print shazam reports.”

11.4.2. Go to “File,” then “open report.” In “reports” folder, select “water analysis historical data report. srw.” Click “open.”

11.4.3. 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.

11.4.4. Click “preview report” tab at the top to display the chosen data.

11.4.5. Click “file” then “export.” Click on “…” box next to file name box. Go to drop down menu by “look in” at the top and choose “c:” drive then choose “export data” folder. Type in the file name in the box at the bottom, then click “open.”

11.4.6. Use the drop down menu under “file type” to select “FixedFieldASCII File(*.txt).” Click “ok.”

11.4.7. Go to “shortcut to mantechFT.exe” icon on the desktop. Click “…” button next to the raw data file box. Choose the appropriate file and click “open.” On the left side, choose the appropriate analyte from “available analytes” box (i.e.

FCO2 for carbon dioxide and for Alkalintiy, select all of the following: talk, bcarb, carb, hydrx).

11.4.8. Click “transfer file”. The data will now be in TALS.

11.5. Data Reduction

11.5.1. All reagent information is recorded on the “Batch Information” page. Use the “Worksheet” tab if additional pages are necessary.

11.5.2. Record special notes and observations on the “Worksheet” tab (i.e. sample appearance and notes on why samples were rejected or diluted).

11.5.3. All raw data is attached as a pdf file. The raw data includes the instrument report and calibration curve.

11.5.4. The analyst must fill out the Wet Chem Data Review checklist (WI# EDS-WI-008) during the first level review. The batch is second level reviewed and the checklist is filed in the 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. 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 , 22nd Edition, American Public Health Association, Washington, DC 2011, Editorial Revision 2011, SM 4500-CO_2 D.

15.2. Standard Methods for the Examination of Water and Wastewater , 22nd Edition, American Public Health Association, Washington, DC 2011, Editorial Revision 2011, SM2320B-11.

15.3. TestAmerica Edison SOP ED-WET-060, Analysis of pH for Waters and Drinking water Measured Electrochemically,EPA 150.1, SM 4500 H +B, SW846 9040B, most current revision.

15.4. TestAmerica Edison SOP ED-WET-039, Analysis of Alkalinity in Water, Wastewater and Soil by Manual Titration or Auto-Titrator, Standard Method 2320B, most current revision.

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, Wetchem Data Review Checklist, most current revision.

16.0. Method Modifications:

N/A

17.0. Attachments

None

18.0. Revision History

- Revision 6, dated 23 April 2014
  - Sec 9.1: Revised to include the number of samples per batch.
  - Sec 15: Updated method reference to SM 2320B-11; added the Standard method reference 4500-CO_2 D-11.

- Revision 5, dated 03 April 2012
  - Sec 1.1.2 and 12: Revised LQM references to reflect the most current LQM revision.
  - Sec. 6.2: Added Erlenmeyer flask and watchglass to list of supplies.
  - Sec. 9.1.1: Added corrective action procedure when MB is greater than the reporting limit.

- Revision 4, 01 April 2010
  - Section 1: Added reporting limit for CO2.
  - Section 2.1: Revised to reference the Alkalinity SOP; deleted reference to the LCS since an LCS is not reported for CO2.
  - Section 4:2: Interference added.
  - Section 5.2: Added safety information for sulfuric acid.
  - Section 6.0: Added equipment and supplies used for PC titrate and manual titration.
  - Section 7: Added reagents, standards and standardization procedure.
  - Section 9.0: Removed LCS as QC sample, LCS is not analyzed for carbon dioxide.
  - Section 10.0: Added the procedures for calibrating the PC titrate.
  - Section 11.4: Added the procedure for exporting data from the PC titrate to TALS.
  - Section 11.5: Added data reduction information.
  - Section 14.2: Added waste stream generated when using the PC titrate.
  - Section 15: Added applicable references.
  - Added this revision history.

Lab SOP 10

Analysis of All Forms of Phosphorous in Water and Soil by Standard Methods 4500P, B5+E, Revision 7, 5/16/2016, TestAmerica Laboratories, Inc.

Title: Analysis of All Forms of Phosphorus in Water and Soil by Standard Methods 4500P, B5 + E

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Facility Distribution No. Electronic **Distributed To:** Facility Intranet

1.0 Scope and Application

1.1 Analytes, Matrix(s), and Reporting Limits

1.1.1 This SOP is applicable to the determination of phosphorus in waters and soils by Standard Methods 4500P, B5 + E. The laboratory's reporting limit for waters is 0.030 mg/l and the reporting limit for soils is 1.5 mg/kg.

1.1.2 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 digested in persulfate solution and gently boiled on a hotplate for 30 to 40 minutes. After digestion, a combined reagent added to the sample forms a blue complex proportional to the amount of ortho-phosphate present. Total phosphorus may be determined by converting all forms of phosphorus to ortho-phosphate by digestion. Total inorganic phosphorus may be determined by digestion with sulfuric acid only. Total organic phosphorus may be calculated by difference.

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 High iron concentrations can cause the precipitation of and subsequent loss of phosphorus.

4.2 Arsenate is determined similarly to phosphorus and should be considered when present in concentrations higher than 0.1 mg/l.

4.3 Hexavalent chromium and nitrite interfere to give low biased results if greater than 1 mg/l.

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

None

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
Potassium Persulfate	Oxidizer	None	Causes irritation to the respiratory tract. Symptoms may include coughing, shortness of breath. Causes irritation to skin and eyes. Symptoms include redness, itching, and pain. May cause dermatitis, burns, and moderate skin necrosis.
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.
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

- Spectrophotometer, 880 nm
- pH meter
- Hotplates

6.2. Supplies

- Erlenmeyer flasks, 125 ml: keep flasks for phosphorus digestion separate from others. Treat the flasks by rinsing with acid or with color indicator and then with deionized water.
- Eppendorf Pipettes, varying volumes
- Disposable Nalgene filters, 0.45 um
- Volumetric Flasks, varying volumes
- Class A graduated cylinder, 50 ml or 100 ml

7. Reagents and Standards

7.1. Reagents

7.1.1. Sulfuric Acid, conc.

7.1.2. Phenolphthalein Indicator

7.1.3. Sodium hydroxide solution, 10N: Dissolve 40.0 g of NaOH in deionized water and dilute to 100 ml. Stable for 6 months. Store at room temperature.

7.1.4. Ammonium persulfate solution: Dissolve 100 g of $(NH_4)_2 S_2O_8$ in 1000 ml of deionized water. Stable for 6 months. Store at 4ºC.

7.1.5. Antimony potassium tartrate solution: Dissolve 1.37 g K(SbO) $C_4H_4O_6 \bullet 1/2H_2O$ in deionized water and dilute to 500 ml. Store in an amber bottle at 4ºC. Stable for 6 months.

7.1.6. Ammonium molybdate solution: Dissolve 10 g $(NH_4)6\ Mo7O_{24}.4H_20$ in deionized water and dilute to 250 ml. Store in a plastic bottle at 4ºC. Stable for 6 months.

7.1.7. Ascorbic acid solution: Dissolve 4.40 g ascorbic acid in deionized water and dilute to 250 ml. Store at 4ºC. Solution is stable for 1 week.

7.1.8. Sulfuric acid, 5N: Dilute 35 ml of conc. H_2SO_4 with deionized water to 250 ml. Cool to room temperature with ice. Stable for 6 months. Store at room temperature.

7.1.9. Sulfuric acid, 11N: Dissolve 310 ml of conc. H_2SO_4 per 1 L DI water. Cool with ice to room temperature. Stable for 6 months. Store at room temperature.

7.1.10. Combined Reagent: Place 250 ml 5N H_2SO_4 in a 500-ml flask. Add 25 ml of tartrate solution. Swirl. Add 75 ml of molybdate solution. Swirl. Add 150 ml of ascorbic acid solution. Swirl. Stable for 4 hours.

7.2. Standards

7.2.1. Primary Phosphorus Stock Solution, ACS grade, 50 mg/l: Dissolve 0.2195 g KH_2PO_4 (dried at 105 °C) in deionized water and dilute to 1 liter. Solution is stable for six months. Store at 4ºC.

7.2.2. Secondary Phosphorus Stock Solution, ACS grade, 50 mg/l: Dissolve 0.2195 g KH_2PO_4 (dried at 105 °C) in deionized water and dilute to 1 liter. Solution is stable for six months. Store at 4ºC. Note: the KH_2PO_4 used for the secondary stock solution must be a different source than the KH_2PO_4 used for the primary stock solution.

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	HDPE	50 mLs	H_2SO_4, pH < 2; Cool 4 ±2°C	28 Days	40 CFR Part 136.3
Soils	Glass	1 grams	H_2SO_4, pH < 2; Cool 4 ± 2°C	28 Days	N/A

[1] Inclusive of digestion and analysis.

9. Quality Control

9.1. **Sample QC** - The following quality control samples are digested and analyzed daily with each batch of 20 samples or less whichever is more frequent.

9.1.1. **Method Blank**: Use deionized water for the blank. The blank results must be below the reporting limit, or the batch must be redigested and reanalyzed.

9.1.2. **Laboratory Control Sample (LCS):** The LCS is a whole volume quality control sample purchased from Environmental Resources Associates (ERA) and is used to measure method performance on the matrix being analyzed. The results must be within vendor specified QC limits or the samples associated with the LCS must be re-analyzed.

9.1.3. **Matrix Spike/Matrix Spike Duplicate (MS/MSD), 0.20 mg/L:** Two portions of the same sample (matrix spike and matrix spike duplicate) are spiked and brought through the entire sample preparation and analytical process. Prepare the MS/MSD by taking 0.20 ml of the primary stock solution (Sec. 7.2.1) brought up to a 50 ml final volume with sample. The recovery must be within laboratory generated limits.

9.2. **Instrument QC-** all instrument QC must be digested with the samples.

9.2.1. Initial Calibration Verification (ICV), 0.20 mg/l: The ICV is prepared and digested each time samples are digested. Prepare by taking 0.2 ml of the secondary stock solution (Sec. 7.2.2) and diluting it up to a 50ml final 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 re-calibrated, and the ICV re-analyzed.

9.2.2. Continuing Calibration Verification (CCV), 0.20 mg/l: The same solution that is used for the ICV is also used for the CCV. The CCV must be prepared and digested with the samples; analyze the CCV 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 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 CCV.

9.2.3. Initial Calibration Blank/Continuing Calibration Blank (ICB/CCB): Following each calibration verification, a calibration blank must be analyzed. The results of this analysis must be less than the reporting limit. If not, the problem must be corrected and the previous ten samples reanalyzed following the last good CCB.

10. Procedure

10.1. Sample Preparation

10.1.1. Place 50.0 ml of sample into 125 ml Erlenmeyer flask. For solid samples, add one gram of solid with 50 ml of deionized water into flask. Use only flasks pre-treated with 11 N Sulfuric Acid and rinsed with deionized water.

10.1.2. Add 1.0 ml 11N H_2SO_4 into the sample.

10.1.3. Add 5.0 ml of ammonium persulfate solution to sample.

10.1.4. Place beaker on hotplate under strong hood. When boiling starts, heat the sample down to approximately 10ml. Do not allow solution to boil to less than 10 ml. Hot plate may need to be turned down slightly to avoid "popping" of samples. Allow samples to cool. Note: Do not allow sample to go to dryness.

10.2. Calibration

10.2.1. Calibration standards are prepared and analyzed every 6 months or when QC requirements fail, whichever is first.

10.2.2. All standard solutions are digested and prepared in the same manner as the samples. Prepare standards in 50 ml volumetric flasks as indicated below.

Volume (uls) of Primary stock to add	Concentration (mg/l)
0	0
30	0.03
50	0.050
100	0.100
200	0.200
500	0.500

10.2.3. The correlation coefficient must be 0.995 or greater.

10.2.4. Use TestAmerica Calibration Spreadsheet CA-Q-WI-13 to back-calculate the concentration of each calibration point. The back-calculated and true concentrations should agree within 10%. Submit the calibration spreadsheet along with the ICAL checklist. Both documents will be scanned and filed after second level review.

10.3. Sample Analysis

10.3.1. Filter through 0.45 disposable filter if the sample has solids that have not dissolved.

10.3.2. Add a drop of aqueous phenolphthalein indicator. Slowly add 10N NaOH dropwise until the sample turns a faint pink color. If a red color develops adjust with 5N H_2SO_4 until the color is discharged.

10.3.3. Dilute the sample or filtrate to 50.0 ml with deionized water and pour back into flask. Save 25 ml of the extract in a separate cup for background and dilutions. Colorize samples in flasks not specimen cups.

10.3.4. Add 4.0 ml combined reagent in the 25 ml portion of the sample. Allow the sample to develop color for a total of 10-30 minutes. Read the absorbance at 880 nm on spectrophotometer. If sample develops color darker than highest standard use the remaining extract to make appropriate dilution up to 50 ml and re-colorize. Note: Be sure to read samples at consistent times. For the standards add 8.0 ml combined reagent to the 50 ml volume.

10.3.5. For samples containing color or turbidity, a background correction must be made. If 25 ml of the sample is used for background, add 2 ml 5N NaOH and 2 ml deionized water. Subtract the blank absorbance from the sample absorbance.

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 for aqueous samples:

Concentration mg P/L = A X B

Where:
A=concentration from the calibration curve in mg/l
B=dilution factor (if present)

11.4. Final results calculation for solid samples:

$$\text{Concentration mg P/Kg} = \frac{C \times D \times E}{F \times S}$$

Where:
C=concentration from the calibration curve in mg/l
D= dilution factor (if present)
E= final volume
F=weight of sample in grams
S = decimal equivalent of the percent solids (percent solids/100) *Note*: All dry weight corrections are made in TALS at the time the final report is prepared.

11.5. Data Reduction

11.5.1. Go to <calibration> then <calibration events> to see information about the calibration including the date the calibration was performed. Attach the calibration curve as a pdf file.

11.5.2. Record the ID number and the expiration date of the calibration curve on the "Batch Information" page.

11.5.3. Record reagent information in the prep batch information (this can be viewed in the "batch information" page).

11.5.4. Record special notes and observations on the "worksheet" tab (i.e. sample appearance and notes on why samples were rejected or diluted).

11.5.5. Attach raw data (pdf file) in the appropriate TALS batch. Raw data includes TALS raw data report, calibration curve, and dilution form (if applicable).

11.5.6. Analyst must fill out the Wet Chem Data Review checklist (WI # EDS-WI-008) during the first level review. The batch is second level reviewed and the checklist if filed in the 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 steams 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-P B5-11 and E-11.

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 Corporate Environmental Health and Safety Manual, Document No. 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. TestAmerica Work Instruction # CA-Q-WI-013, Calibration Spreadsheet, 10 Aug 2011.

16.0. Method Modifications:

N/A

17.0. Attachments

None

18.0. Revision History

- Revision 7, dated 16 May 2016
  - Sec. 9.1: Clarified batch QC frequency to daily or with each batch of 20 samples whichever is more frequent.
  - Sec. 9.1.2: Clarified the LCS source and acceptance limits used.
  - Sec. 11.5.5: Revised to list down which raw data should be attached in TALS analytical batch.

- Revision 6, dated 23 April 2014
  - Sec 10.2.4: Added requirements and criteria for back calculating each calibration points to comply with SM Rev 2011.
  - Sec 15.1: Updated Method reference to SM 4500-P B5-11 and E-11, Revision 2011.
  - Sec 15.7: Added reference CA-Q-WI-013.

- Revision 5, dated 02 April 2012
  - Sec 1.1.2 & 12: Revised LQM references to reflect the most current LQM revision.
  - Sec. 6.2: Added graduated cylinder to list of supplies
  - Sec. 7.1: Added Phenolphthalein Indicator to list of reagents
  - Sec 7.2.1 & 7.2.2: Revised the amount of KH2PO4 added to make the primary and secondary stock (from 0.2197 g to 0.2195 g) to comply with SM 4500-P E (not EPA 365.2).
  - Sec. 9.2.1 & 9.2.2: Added requirement that ICV and CCV should be prepared and digested with the samples.

- Revision 4, 01 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 365.2 and 365.3 due to Method

Update Rule; Added laboratory's reporting limit for soils and waters.

- Sec. 7: Added storage and stability information where appropriate
- Sec. 7.2.2: Added "Secondary Phosphorus Stock Solution" to standards list.
- Sec 9: Expanded Sample and Instrument QC to include QC concentration (mg/L) and spiking procedure.
- Sec.10.2.2: Revised correlation coefficient criteria limit from 0.997 to 0.995.
- Sec.11.5: Revised data reduction procedures in accordance with new TALS.
- Sec. 15: Added applicable references.
- Added this revision history.

Lab SOP 11a

Digestion of Water and Wastewater Samples for Analysis by ICP and ICP-MS, Method 3010A, Revision 10, 8/3/2016, TestAmerica Laboratories, Inc.

Title: Digestion of Water and Wastewater Samples for Analysis by ICP and ICP-MS, SW846 Method 3010A

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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

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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.

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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.

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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.

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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.

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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

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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

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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

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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

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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

Lab SOP 11b

Trace Metals Analysis for Water and Wastewater using Inductively Coupled Plasma Emission Spectroscopy by EPA Method 200.7 (Rev 4.4), Revision 9, 5/12/2015,
TestAmerica Laboratories, Inc.

Title: Trace Metals Analysis for Water and Wastewater using Inductively Coupled Plasma Emission Spectroscopy by EPA Method 200.7 (Rev 4.4)

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Approvals (Signature/Date):

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1.0 Scope and Application

1.1. Analytes, Matrix(s), and Reporting Limits

Method 200.7 (Rev 4.4) determines trace elements in water and waste water samples using inductively coupled plasma atomic emission spectrometry (ICP-AES). The method is applicable to all of the elements listed in Table 1. All matrices require some type of digestion/preparation step prior to an analysis on the instrument.

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
Analyte List and Method Reporting Limit

Element	Non-Potable Water (ug/L)	Potable Water (ug/L)
Aluminum	200	200
Barium	200	200
Beryllium	2	2
Boron	50	50
Cadmium	5	5
Calcium	5000	5000
Chromium	10	10
Cobalt	50	50
Copper	25	25
Iron	150	150
Manganese	15	15
Magnesium	5000	5000
Molybdenum	20	20
Nickel	40	40
Potassium	5000	5000
Silver	10	10
Sodium	5000	5000
Strontium	20	20
Tin	50	50
Titanium	20	20
Vanadium	50	50
Zinc	30	30
Antimony	10	n/a
Arsenic	5	n/a
Lead	5	n/a
Selenium	5	n/a
Thallium	10	n/a

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

An aliquot of a well mixed homogeneous aqueous sample is measured for sample processing. For total recoverable analysis of a drinking water sample or an aqueous sample containing undissolved material, the sample is prepared using the TestAmerica Edison SOP No. ED-MTP-001 (*Digestion of Aqueous Samples for Analysis by ICP and ICP-MS USEPA Method No(s) 200.2, 200.7 (Rev 4.4), 200.8*). For the determination of dissolved analytes in a filtered aqueous sample aliquot, the sample is made ready for analysis by the appropriate addition of nitric acid.

Following the sample preparation and 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 with photomultiplier tubes. 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 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. Drinking Water samples typically contain minimal interferences.

4.2. Most interference are eliminated or greatly reduced during the acid digestion of the sample matrix.

4.3. Spectral interferences encountered on the instrument are corrected for by using baseline correction points and by applying background correction factors.

4.4. 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.

4.5. Interelement Correction Factors are determined by analyzing single element solutions and recording the apparent analyte concentration.

4.6. All interfering elements must be analyzed at the same time as the elements of interest.

4.7. Physical interferences are effects associated with sample nebulization and transport. They are reduced or eliminated with the 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.

4.8. 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.

4.9. High salt concentrations can cause analyte signal suppressions and confuse interference tests. Dilute the sample if necessary. For the analysis of barium in samples having high sulfate, analysis should be completed as soon as possible after sample preparation.

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 Equipment

- 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

- Eppendorf & Fisher Pipettes, varying volumes
- Polypropylene tubes
- Argon supply - 99.99% (Liquid)
- Volumetric Flasks (Class A): 50 mL, 100 mL, 500 mL, 1000 mL

7.0 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. Reagent water, 5% HNO3: Add 1 liter of concentrated HNO3 to deionized water and bring to 20 liter volume with deionized water. Note: Always add acid to water. Record preparation in the TALS Reagent Module. Prepare every 12 months or refer to manufacturer's expiration date; store at room temperature. *Note*: This reagent water is required for the analysis of undigested samples.

7.1.5. Reagent water, 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 the TALS Reagent Module. Prepare every 12 months or refer to manufacturer's expiration date; store at room temperature. *Note*: This reagent water is required for the analysis of digested samples.

7.2. Standards

Storage requirements: all standards are stored at room temperature.
Note: Standard mixes containing Silver and HCl must be stored in amber containers or must be covered when not in use to minimize exposure to light.

Shelf-life: Stock standards – refer to manufacturer's instructions
Intermediate standards – 6 months
Working standards – 3 months
(Note: expiration date must not go beyond the expiration date of the source stock).

Concentration: see Attachment 2 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.

7.2.1. Stock Standards:

7.2.1.1. Calibration stock standards - CLPP-CAL-1, CLPP-CAL-3, STLNJ-CAL-1A, STLNJ-CAL-1B, STLNJ-CAL-3, Al, Cu, Ni, Fe, Si, Zn, As, Pb, Se, Tl each 10000 ppm; Sb 1,000ppm;; TANJ-STD-3, and TANJ-STD-4, purchased from Inorganic Ventures. See Attachment 2 for typical COA.

7.2.1.2. Calibration Verification Standards – Duo_CCV-int Solutions: A, B, C, and D; purchased from High-Purity Standards. See Attachment 2 for typical COA.

7.2.1.3. Interference Check Standards – CLPP-ICS-A; IV-7; IV-19; K, Na, Sr, Sn, each 10,000ppm, purchased from Inorganic Ventures Inc., Christiansburg, VA. See Attachment 2 for typical COA.

7.2.2. Calibration Standards (potable and non-potable):

7.2.2.1. **Cal 1:** add 100 ml of reagent water to a clean 1000 ml volumetric flask. Add 10 ml of ME_Cal1_INTBC (see 7.2.3.1.1). Bring to volume with reagent water.

7.2.2.1.1. **ME_Cal1_INTBC:** add 100 ml of reagent water to a clean 1000 ml volumetric flask. Using 10,000 ppm stock standards, add 0.05 ml each of As, Pb, Se, and 0.1 ml of Tl. Using 1,000ppm stock standard, add 1 ml of Sb. Bring to volume with reagent water.

7.2.2.2. **Cal 2:** add 100 ml of reagent water to a clean 1000 ml volumetric flask. Add 10 ml each of TANJ-STD-3, TANJ-STD-4, and ME_Cal2_INT. Add 0.02 ml of 10,000ppm Si. Bring to volume with reagent water.

7.2.2.2.1. **ME_Cal2_INT:** add 100 ml of reagent water to a clean 1000 ml volumetric flask. Using 10,000 ppm stock standards, add 0.05 ml of Pb; 0.1 ml each of As, Tl; 0.15 ml of Se. Using 1,000ppm stock standard, add 1 ml of Sb. Bring to volume with reagent water.

7.2.2.3. **Cal 3:** add 100 ml of reagent water to a clean 1000 ml volumetric flask. Add 1 ml each of CLPP-CAL-1, STLNJ-CAL-1A, and STLNJ-CAL-1B, 2 ml of STLNJ-CAL-2, 2.3 ml of 10000 ppm Aluminum, and 0.5 ml of CLPP-CAL-3. Add 0.2 ml of 10,000ppm Si. Bring to volume with reagent water.

7.2.2.4. **Cal 4:** add 100 ml of reagent water to a clean 1000 ml volumetric flask. Add 5 ml each of CLPP-CAL-1, STLNJ-CAL-1A, and STLNJ-CAL-1B, 10 ml of STLNJ-CAL-2, 11.5 ml of 10000 ppm Aluminum, and 2.5 ml of CLPP-CAL-3. Add 1 ml of 10,000ppm Si. Bring to volume with reagent water.

7.2.2.5. **Cal 5:** add 100 ml of reagent water to a clean 1000 ml volumetric flask. Add 10 ml each of CLPP-CAL-1, STLNJ-CAL-1A, and STLNJ-CAL-1B, 20 ml of STLNJ-CAL-2, 23 ml of 10000 ppm Aluminum, and 5 ml of CLPP-CAL-3. Add 2 ml of 10,000ppm Si. Bring to volume with reagent water.

7.2.3. Calibration Verification Standards:

7.2.3.1. Initial/Continuing Calibration Verification Standard (ICV/CCV): Add 100 mL of reagent water to a clean 1000 ml volumetric flask. Add 10 ml each of Duo_CCV-int Solutions: A, B, C, and D. Bring to volume with reagent water. A fresh solution should be prepared quarterly. Note: expiration date must not go beyond the expiration date of the source stock. See Attachment 1 for final concentrations.

7.2.4. Interference Check Standard A (ICSA): Add 100 mL of reagent water to a clean 1000 mL volumetric flask. Add 100 ml of CLPP-ICS-A. Bring to volume with reagent water. See Attachment 1 for final concentrations.

7.2.5. Interference Check Standard AB (ICSAB): Add 100 mL of reagent water to a clean 1000 mL volumetric flask. Add 100 ml CLPP-ICS-A, 1 ml each of IV-7, IV-19, and LCSW-III (see Sec 7.2.6.1), 0.9 ml each of 10000 ppm Na and 10000 ppm K. Bring to volume with reagent water. See Attachment 1 for final concentrations.

7.2.5.1. LCSW-III (potable/non-potable) – Add 100 mL of reagent water to a clean 200 mL volumetric flask. Add 18 ml of 10,000 ppm Na, 2 ml of 10,000ppm Sn and 2 ml of 10,000 ppm Sr. Bring to volume with reagent water.

7.2.6. Interferent-10 Standard (INT-10): Add the following standard solution in 1000 ml volumetric flask and bring to volume with reagent water.

Element	Concentration (ppm)	Volume to add (ml)
Titanium	10000	1
Manganese	10000	1
Chromium	10000	1
Strontium	10000	1
Tin	10000	1
Cobalt	10000	1
Nickel	10000	1
Vanadium	10000	0.5
Molybdenum	10000	0.5
Silicon	10000	1

7.2.7. Stock Spike Standards:

7.2.7.1. 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 2 for the standards certified concentrations and catalog numbers.

7.2.8. Working Spike Standards

7.2.8.1. 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.

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
Waters	Plastic, glass	500 ml	HNO_3 to pH < 2 prior to shipment; if not, acidify upon receipt in lab [3,4]	180 days	Method 200.7 Rev 4.4

[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 24hrs, verify ph<2).
[4] Samples may be stored at room temperature.

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
Method Blank (MB)	1 in 20 or fewer samples	< 2.2 times MDL or <10% sample concentration
Laboratory Control Sample (LCS)	1 in 20 or fewer samples	85-115%
Matrix Duplicate (DU)[1]	1 in 20 or fewer samples	If original sample and dup are

Quality Control	Frequency	Control Limit
		both ≥ 5X CRDL, then 20% RPD. If original sample and duplicate are less than the CRDL, the RPD is not calculated; otherwise ±CRDL.
Matrix Spike (MS)[1]	10% of the routine samples (1, if 10 samples or less; 2 if >10 but <20 samples)	70-130%
Matrix Spike Duplicate	When requested by client	70-130%; 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 (PDS)[1]	1 per batch of 20 or fewer samples	85-115%

[1] The sample for DU, MS, PDS is randomly selected, unless specifically requested by a client; Use the same environmental sample for the matrix spike and matrix duplicate sample whenever possible. If insufficient sample amount is available, another environmental sample may be used for the duplicate sample.

9.1.1. **Preparation Blank/Method Blank (MB):** Also known as laboratory reagent blank (LRB). One laboratory method/preparation blank will be analyzed with each batch of samples prepared together (not to exceed 20 samples). Preparation blank is used to identify possible contamination during acid digestion. Results must be less than 2.2 times the reporting limit or less than 10% of the determined analyte concentration for a sample. If any analyte concentration in the blank is above this control limit, the batch must be prepared again for the element in question and the samples reanalyzed.

9.1.2. **Laboratory Control Sample (LCS):** Also known as laboratory fortified blank (LFB). A laboratory control sample must be analyzed with each group of samples digested. The sample is spiked at levels indicated in Attachment 1. Results of the aqueous LCS must fall within ±15% of the true value. If not, all samples prepared in association with the LCS must be redigested and reanalyzed.

9.1.3. **Matrix Duplicate (DU):** Also known as replicate sample (LD1 and LD2). A duplicate is analyzed for each batch of samples. If original sample and duplicate are both ≥ CRDL, then 20% RPD. If original sample and duplicate are less than the CRDL, the RPD is not calculated; otherwise, ±CRDL.

9.1.4. **Matrix Spike (MS):** Also known as laboratory fortified matrix spike (LFM).

A matrix spike is prepared and analyzed for each batch of samples digested. The sample is spiked at levels indicated in Attachment 1. A recovery of 70-130% 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 and the LCS is within limits, the recovery problem is judged to be matrix related.

9.1.5. Serial Dilution (SD): A five fold serial dilution must be performed on one sample per batch. If the sample should contain analytes at a sufficiently high concentration; minimally a factor of 50 times above the instrument detection limit, the results must agree within 10% of the original determination. If not, a chemical or physical effect should be suspected.

9.1.6. Matrix Spike Duplicate (MSD): Also known as laboratory fortified matrix spike duplicate (LFMD). A matrix spike duplicate is prepared and analyzed for each batch of samples digested when requested by the client. The sample is spiked at levels indicated in Attachment 1. A recovery of 70-130% 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 and the LCS is within limits, the recovery problem is judged to be matrix related. If matrix spike sample and matrix spike duplicate are both $\geq$ CRDL, then 20% RPD. If matrix spike sample and matrix spike duplicate are less than the CRDL, the RPD is not calculated; otherwise, ±CRDL.

9.1.7. Post Digestion Spike (PDS): 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. 7.2.9.) and 9.80 ml of the sample digestate. If sample requires dilution, adjust the sample as appropriate (e.g. sample at 2X dilution: add 0.20 ml of the standard spike to 5 ml of sample digestate, and add 4.8 ml of the reagent water). Limits for post digestion spikes are 85-115% recovery.

9.2. Instrument QC

9.2.1. Initial Calibration Verification (ICV): Also known as the Quality Control Sample (QCS). Analyze the 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 results for the target elements in the initial calibration verification (ICV) must be within +/-5% 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): Also known as instrument performance check standard (IPC). Analyze the continuing calibration verification standard after every 10 analytical sample and at the end of the run. The results must be within 10% of the true value. See Sec 7.2.4

for the CCV standard preparation instructions.

9.2.3. **Initial and Continuing Calibration Blank (ICB/CCB):** ICB/CCB must be analyzed after the calibration curve, every 10 samples and at the end of the analytical run. The absolute value of the 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. **Interference Check solution A & B (ICSA/ICSAB):** Also known as spectral interference check solutions (SIC). The interelement background correction factors are verified at the beginning of each run. See Sec 7.2.5 and 7.2.6 for the standard preparation instructions.

9.2.4.1. Results for ICSA shall fall within ± 2 times the reporting limit of the analyte's true value (the true value shall be zero unless otherwise stated). Exception: Results for Al, Ca, Fe, and Mg, shall fall within 80-120% of the analyte's true value. If not, terminate the analysis, correct the problem and recalibrate the instrument. Reanalyze the samples since the last compliant ICSA. See Attachment 1 for final elemental concentration of ICSA.

9.2.4.2. Results for ICSAB solution shall fall within 80-120% of the true value for the analytes in the solution. If not, terminate the analysis, correct the problem and recalibrate the instrument. See Attachment 1 for the corresponding concentrations in ug/l.

9.2.5. **Interferent-10 (INT-10):** An INT-10 sample is analyzed for each analytical run. Results should fall within the control limit of +/-20% of the true value or +/-2 times the RL, whichever is greater; if not, problem must be identified and corrected. . See Sec 7.2.7 for the INT-10 standard preparation instructions.

10.0 Procedure

10.1. Set up the instrument according to the instrument's operating parameters.

10.1.1. TJA 6500 Duo ICP

10.1.1.1. 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 15.7 for reference). Allow the instrument to become thermally stable, minimally 30min. Perform a torch alignment by aspirating a 2 ppm Zn standard.

10.1.1.2. 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.

10.1.1.3. Interelement Correction factors must be verified semi-annually. Refer to the iTEVA Software Manual located in the Operator Manuals for instructions. Criteria for determining IEC's is an apparent positive or negative concentration for the analyte that falls outside of one reporting limit from zero

10.2. Calibration

10.2.1. Profile and calibrate the instrument according to the instrument manufacturer's instructions. The instrument is calibrated using five standards and a blank. Prepare the calibration standards as detailed in section 7.2.2.

10.2.2. The instrument must be calibrated daily or once every 24 hours and each time the instrument is set up. The correlation coefficient of the calibration curve must be ≥0.995. If not, the problem must be corrected, and the instrument must be recalibrated.

10.3. Instrument Performance Criteria

Prior to the analysis of any samples the following must be performed.

10.3.1. Background correction points must be determined during the initial set-up of the instrument. Refer to the specific instrument manual for instructions.

10.3.2. Interelement Correction factors must be determined semi-annually. Refer to the instrument manufacturer recommended procedures for instructions. Criteria for determining IEC's is an apparent positive or negative concentration for the analyte that falls within one reporting limit from zero.

10.3.3. The IDL for each analyte must be determined for each wavelength used on each instrument. The IDL must be determined when the instrument is initially set up for use and if the instrument is adjusted in any way that may affect the IDL. The IDL is determined by multiplying by 3 the average of the standard deviations obtained from the analysis of ten replicates of a reagent blank signal.

10.3.4. The MDL must be determined prior to the analysis of any samples. The MDL is determined using seven replicates of reagent water, spiked with all elements of interest that have been carried through the entire analytical procedure. MDL's must be re-determined yearly. The MDL may be analyzed on two or more non-consecutive days to provide a more appropriate estimate of the MDL. In this case calculate the MDL for each day and use an average of the values for each element.

10.3.5. Determine the linear dynamic range (LDR) of the instrument by analyzing a minimum of three succeedingly higher standards at the upper limit of the instrument. The analytically determined concentration of these standards must be within 10% of the true value. For elements validated in this manner you may report data up to 90% of that linear range before a dilution is required. The linear ranges should be redetermined semi-annually or if the instrument is significantly changed.

10.4. Sample Preparation

10.4.1. Wastewater and drinking water samples are digested prior to analysis per TestAmerica Edison SOP No. ED-MTP-001, Digestion of Aqueous Samples for Analysis by ICP and ICP-MS USEPA Method No. 200.2, 200.7 (Rev 4.4), 200.8.

10.5. Sample Analysis

10.5.1. 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. A minimum of three exposures for each standard, sample and blank is required. The average of the exposures is reported.

10.5.2. Flush the instrument between standards and sample using the calibration blank.

10.5.3. The following analytical run sequence is currently used for samples analyzed via the 200.7 protocol:

Instrument Calibration
ICV(QCS)
ICB
ICVL (for 6010C only)
ICSA(SIC)
ICSAB(SIC)
INT-10
7 Samples
CCV(IPC)
CCB
CCVL (for 6010C only)
10 Samples
CCV(IPC)
CCB
Repeat until run is complete
CCV(IPC)
CCB
CCVL (for 6010C only)

10.5.4. Dilute and reanalyze all samples for which the required analytes exceed 90% of the linear dynamic range as well as samples that contain high concentrations of an interfering element (all interfering elements must be analyzed at the same time as the elements of interest).

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 :

$$\text{Concentration = ug/ L} = \frac{C \times V1 \times D}{V2}$$

Where:
C= Element concentration from instrument (ug/L)
V1= Final volume of sample digested (in liters)
D= Dilution performed on sample)
V2= Initial volume of sample digested (in liters).

11.4. Data Reduction:

11.4.1. All data is recorded directly in TALS' Analyst Desktop II program.

11.4.2. Record standard preparations in TALS Reagent module.

11.4.3. 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.

11.4.4. Metals Data Review checklist (CA-Q-WI-043) 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. MDL is not applicable for this 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

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 U.S. Environmental Protection Agency, Determination of Metals and Trace elements in water and wastes by Inductively Coupled Plasma- Atomic Emission Spectrometry, Method 200.7, Rev. 4.4.

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-MTP-001, *Digestion of Aqueous Samples for Analysis by ICP and ICP-MS USEPA Method No(s) 200.2, 200.7 (Rev 4.4), 200.8,* most current revision.

15.6 Metals Data Review Checklist Work Instruction # EDS-WI-007, most current revision.

15.7 iCAP 6000 Series ICP-OES Spectrometer Operator Manuals (8499 400 90001), Thermo Electron Corporation, 2005.

16.0 Method Modifications:

Item	Method No.	Modification
1	200.7 (Rev 4.4)	A 2nd Source Standard is used for the CCV (IPC). ICV and CCV are prepared in the same manner and the acceptance limits will be in compliance with the method (± 5% for ICV and ± 10% for the CCV). IPC and CCV are analyzed daily. Method states that IPC should be prepared from the same source as the cal standards.
2	200.7 (Rev 4.4)	ICSA criteria for all non-spiked analytes will follow the same

Item	Method No.	Modification
		criteria: ± 2 times the RL of the analyte's true value. Section 7.13 of the method describes different criteria for different elements.
3	200.7 (Rev 4.4)	A pre-made Silver standard solution (in HNO3) is purchased commercially and stored in plastic containers as received. Standards are stored following vendor's instructions for storage and handling: *store and use at 20±4℃; stability: 1-10,000 ppm solutions chemically stable for years in 1-5% HNO3/LDPE container.* Section 7.8.25 of Method 200.7 describes preparing 1000 ug Ag stock standard solution using 1.000g Ag metal, heating and storing in amber bottle.

17.0 Attachments

Attachment 1: Working Standard Concentrations
Attachment 2: Certificate of Analysis for all multi element calibration and calibration verification standards and examples of typical interference standards.

18.0 Revision History

- Revision 9, dated 12 May 2015
  - Sec 1.1: Deleted the turbidity reference to drinking and dissolved water samples.
  - Sec 2.0: Deleted reference to CLP SOW ISM01.2 and instructions for direct analysis for drinking water samples.
  - Sec 7.1.4 & 7.1.5: Replaced ICP Reagent Dilution logbook with TALS Reagent Module.
  - Sec 7.2.1.1: Added As, Pb, Se, Tl, Sb to the list of stock standards
  - Sec 7.2.1.2: Removed standards QCP-CICV-1, QCP-CICV-3, STLNJ-QC-3, not applicable.
  - Sec 7.2.2: Removed preparation instructions for potable calibration standards
  - Sec 7.2.3: Revised to include calibration standards preparation instructions for both potable and non-potable cal stds.
  - Sec 7.2.4.1: Deleted ICV/CCV preparation instructions for potable cal standards.
  - Sec 7.2.8 & 7.2.9: Added stock spike stds and preparation instructions for the Working Spike standards.
  - Sec 9.1: Added Matrix Spike Duplicate (MSD) and Post Digestion Spike (PDS) to the list of sample QC.
  - Sec 9.1.6 & 9.1.7: Added information (i.e. QC limits) for MSD and PDS.
  - Sec 10.2: Removed reference for potable samples calibrated with three cal standards.
  - Sec 10.4.1 Deleted instructions for the direct analysis of drinking water; deleted reference to CLP SOP ISM01.2
  - Sec 10.5.3: Updated typical analytical run sequence (added ICVL and CCVL) to reflect actual laboratory practices.
  - Sec 11.4.4: Updated WI# for the Metals Data Review checklist.
  - Sec 15.5: Deleted reference to CLP SOW ISM01.2

- Attachment 1: Deleted table for Potable Water standards; updated the Non-Potable Water Table (Cal 1 and Cal 2) to include the standard concentrations for potable water.
- Attachment 2: Deleted COA's for QCP-CICV-1, QCP-CICV-3, STLNJ-QC-3; added COA's for Stock spike standards (Solutions A, B, C, and D (ICP-Spk), Part No. 4400-100419DD01).

- Revision 8, dated 07 December 2012

  - Sec 1 & 12: Updated LQM section reference to reflect the most current LQM revision.
  - Sec 6.1: Deleted TJA 61E Trace ICP from the list of equipment; not applicable.
  - Sec 7.2: Added storage requirements for standard mixes containing Ag and HCl.
  - Sec 7.2.1.1: Added Cu, Ni, Fe, Si, Zn to the list of Cal stock stds.
  - Sec 7.2.3.1: Added new Cal1 standard; subsequent sections adjusted accordingly. Added preparation procedures for the new Cal standard (Cal1) and its intermediate (ME_Cal1-int).
  - Sec 7.2.3.2 through 7.2.3.5: Added Si to Cal stds 2, 3, 4, and 5.
  - Sec 7.2.7: Added Si; deleted La and its reference to Trace 3.
  - Sec 9.2.5: Added control limits for INT-10 non-spiked analytes.
  - Sec 10: Deleted reference to TJA 61E Trace (Sec 10.1.2).
  - Sec 10.2.1: Revised the number of cal standards to five to reflect actual laboratory practices.
  - Sec 15: Deleted reference to TJA 61E Trace (Sec 15.5); subsequent sections adjusted accordingly.
  - Sec 16.0 (Item 3): Added method modification stating that std mixes preserved with HNO3 and purchased pre-made will be stored as per vendor's instructions (i.e. stored in LDPE containers).
  - Attachment 1 Potable: Removed the use of 2nd order curve fit for potable water ICAL.
  - Attachment 1 Non-Potable: Removed the use of 2nd order curve fit for non-potable water ICAL; added new Cal 1.

- Revision 7, dated 29 March 2011

  - Revised SOP to include Water and Wastewater analysis procedure.
  - Sec 1.1 Table 1: Added Reporting Limits for non-potable
  - Sec 2: Expanded method summary.
  - Sec 4.9: Added sulfate as an interference to barium.
  - Sec 6.1: Added the iCAP Duo instrument.
  - Sec 7.2.1: Added STLNJ-CAL-1A and STLNJ-CAL-1B to the calibration stock standards; added COA in attachment 2.
  - Sec 7.2.1.3: added Duo_CCV-int Solutions A, B, C & D to the CCV stock standards; added COA in attachment 2.
  - Sec 7.2.2: Revised the preparation procedure of the calibration standards used for wastewater analysis.
  - Sec 7.2.3: Added the preparation procedure of the calibration standards used for drinking water analysis.
  - Sec 7.2.3.2: Added the preparation procedure for the ICV/ CCV non-potable analysis.
  - Replaced INT-20 in various sections where applicable (such as sec 7.2.7, 9.2.6,

10.7.3) with INT-10 to reflect actual lab practices.
- Sec 7 & 9: Deleted Reporting Limit check standard (RepLim), standard not applicable; subsequent sections adjusted accordingly.
- Sec 10.1.1: Added TJA 6500 Duo's operating instructions.
- Sec 15.8: Added the iCAP 6000 Operator Manuals reference; Added reference for Method 200.7 Rev 4.4.
- Sec 18.0 Attachment 1: Revised working standard concentration for non-potable water; added new Table: *Working standard concentration for ICP elements potable water.*

- Revision 6, dated 15 December 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: Added reporting limits in Table 1.
  - Sec 6.3: Removed Nitrogen Gas on the list of supplies
  - Sec 7.2.2: Updated preparations for Cal Stds to reflect actual lab practices.
  - Sec 7.2.3: Updated preparations for Cal Ver. Stds to reflect actual lab practices.
  - Sec 7.2.6: Added RepLim Std to the list of Standards.
  - Sec 7.2.7: Added INT-20 Std to the list of Standards.
  - Sec 9.1& 9.2: Clarified the criteria for most of the QC samples (i.e. Method blank Matrix duplicate, ICSA/ICSB, INT-20 and Rep-Lim
  - Sec 9.2.2: Revised the CCV acceptance limits to 10% to comply with the method.
  - Sec 10.5.3: Revised analytical run sequence to reflect actual lab practices.
  - Sec 11.4: Added data reduction to comply with TALS.
  - Sec 15: Added applicable references.
  - Sec 16: Added Attachment 1 and 2
  - Sec 17: Added method modifications

Working Standard Concentrations for ICP Elements in ug/L
Potable & Non-Potable Water

Element	Cal 1	Cal 2	CAL3	CAL4 ICV/CCV	CAL5	ICSA	ICSAB	LCS & Matrix Spike
Al	n/a	200	25000	125000	250000	500000	500100	2000
Sb	10	20	200	1000	2000	n/a	100	500
As	5	15	500	2500	5000	n/a	100	2000
Ba	n/a	200	2000	10000	20000	n/a	100	2000
Be	n/a	2	200	1000	2000	n/a	100	50
B	n/a	50	200	1000	2000	n/a	100	50
Cd	n/a	4	250	1250	2500	500000	500100	20000
Ca	n/a	5000	25000	125000	250000	n/a	100	200
Cr	n/a	10	1000	5000	10000	n/a	100	500
Co	n/a	50	500	2500	5000	n/a	100	250
Cu	n/a	25	2500	12500	25000	200000	200100	1000
Fe	n/a	150	20000	100000	200000	n/a	100	500
Pb	5	10	1500	7500	15000	500000	500100	20000
Mn	n/a	15	1000	5000	10000	n/a	100	500
Mg	n/a	5000	25000	125000	250000	n/a	100	500
Mo	n/a	20	500	2500	5000	n/a	10000	20000
Ni	n/a	40	500	2500	5000	n/a	100	2000
K	n/a	5000	10000	50000	100000	n/a	100	50
Se	5	20	500	2500	5000	n/a	10000	20000
Ag	n/a	10	250	1250	2500	n/a	100	2000
Na	n/a	5000	25000	125000	250000	n/a	100	500
Sr	n/a	20	1000	5000	10000	n/a	100	500
Tl	10	20	500	2500	5000	n/a	100	500
Sn	n/a	50	200	1000	2000	n/a	100	500
Ti	n/a	20	2000	10000	20000	n/a	100	500
V	n/a	50	500	2500	5000	n/a	100	500
Zn	n/a	30	500	2500	5000	n/a	100	500
Si	n/a	n/a	2000	10000	20000	n/a	100	n/a

Attachment 2

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RSW 4 3/4/20 C.C.

CERTIFICATE OF ANALYSIS

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4873959
ID: ME_CAL_1_00012
Exp:06/01/11 Proc:000
CLPP-CAL-1

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2.0 **DESCRIPTION OF CRM**

	Stock Solution
Catalog No.:	CLPP-CAL-1
Lot Number:	**A2-MEB236013**
Matrix:	5% HNO3(abs)

5,000.00 µg/mL ea:
Ca, K, Mg, Na,
2,000.00 µg/mL ea:
Al, Ba,
1,000.00 µg/mL ea:
Fe,
500.00 µg/mL ea:
Co, Mn, Ni, V, Zn,
250.00 µg/mL ea:
Ag, Cu,
200.00 µg/mL ea:
Cr3,
50.00 µg/mL ea:
Be

3.0 **CERTIFIED VALUES AND UNCERTAINTIES**

ELEMENT	CERTIFIED VALUE	ELEMENT	CERTIFIED VALUE	ELEMENT	CERTIFIED VALUE
Aluminum, Al	2,004 ± 10 µg/mL	Barium, Ba	2,000 ± 3 µg/mL	Beryllium, Be	50.02 ± 0.10 µg/mL
Calcium, Ca	5,006 ± 13 µg/mL	Chromium+3, Cr3	200.0 ± 0.7 µg/mL	Cobalt, Co	500.0 ± 1.1 µg/mL
Copper, Cu	250.6 ± 0.5 µg/mL	Iron, Fe	1,000 ± 2 µg/mL	Magnesium, Mg	4,996 ± 17 µg/mL
Manganese, Mn	500.1 ± 1.1 µg/mL	Nickel, Ni	499.1 ± 1.7 µg/mL	Potassium, K	5,013 ± 9 µg/mL
Silver, Ag	250.0 ± 0.2 µg/mL	Sodium, Na	5,001 ± 23 µg/mL	Vanadium, V	500.4 ± 1.3 µg/mL
Zinc, Zn	500.2 ± 1.8 µg/mL				

Certified Density: 1.127 g/mL (measured at 22° C)

Rev'd 5/4/10
cc

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4R7065
ID: ME_CAL-2_00011
Exp: 4Q/H1/Year: CDC
CLPP-CAL-7, calibration s

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ACCREDITED Testing Laboratory Certificate 860.01

ACCREDITED Reference Material Producer Certificate 560.02

2.0 **DESCRIPTION OF CRM** Stock Solution

Catalog No.: CLPP-CAL-3

Lot Number: **D2-MEB324156**

Matrix: 7% HNO3(v/v)

1,000.00 µg/mL ea:
As, Pb, Se, Tl,

500.00 µg/mL ea:
Cd

3.0 **CERTIFIED VALUES AND UNCERTAINTIES**

ELEMENT	CERTIFIED VALUE	ELEMENT	CERTIFIED VALUE	ELEMENT	CERTIFIED VALUE
Arsenic, As	1,000 ± 3 µg/mL	Cadmium, Cd	500.0 ± 1.1 µg/mL	Lead, Pb	1,000 ± 2 µg/mL
Selenium, Se	1,000 ± 6 µg/mL	Thallium, Tl	1,000 ± 4 µg/mL		

Certified Density: 1.046 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.

$$\text{Certified Value } (\bar{x}) = \frac{\Sigma x_i}{n}$$

$$\text{Uncertainty } (\pm) = \frac{2[(\Sigma s_i)^2]^{1/2}}{(n)^{1/2}}$$

$(\bar{x})$ = mean
x_i = individual results
n = number of measurements
Σs_i = The summation of all significant estimated errors (Most common are the errors from instrumental measurement, weighing, dilution to volume and the fixed error reported on the NIST SRM certificate of analysis)

4.0 **TRACEBILITY TO NIST AND VALUES OBTAINED BY INDEPENDENT METHODS**

- "Property of the result of a measurement or the value of a standard whereby it can be related to stated references, usually national or international standards, through an unbroken chain of comparisons all having stated uncertainties." (ISO VIM, 2nd ed., 1993, definition 6.10)
- This product is Traceable to NIST via an unbroken chain of comparisons. The uncertainties for each certified value are reported, taking into account the SRM uncertainty error and the measurement, weighing and volume dilution errors. In rare cases where no NIST SRMs are available, the term 'in-house std.' is specified.

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442324
ID: IVE_NJ_CAL3_030312
Exp: 05/01/15 Prod: CDC
CLP CUSTOM CAL STANDARD

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2.0 **DESCRIPTION OF CRM**

Custom Solution
Catalog No.: STLNJ-CAL-3
Lot Number: **D2-MEB329039**
Matrix: tr. HF, 3% HNO3(v/v)

10,000.00 µg/mL ea:
Na,
2,500.00 µg/mL ea:
K,
100.00 µg/mL ea:
B, Mo, Sn, Sr, Ti

3.0 **CERTIFIED VALUES AND UNCERTAINTIES**

ELEMENT	CERTIFIED VALUE	ELEMENT	CERTIFIED VALUE	ELEMENT	CERTIFIED VALUE
Boron, B	99.9 ± 0.4 µg/mL	Molybdenum, Mo	99.9 ± 0.4 µg/mL	Potassium, K	2,504 ± 6 µg/mL
Sodium, Na	10,000.0 ± 90.0 µg/mL	Strontium, Sr	100.3 ± 0.3 µg/mL	Tin, Sn	100.1 ± 0.9 µg/mL
Titanium, Ti	99.8 ± 0.4 µg/mL				

Certified Density: 1.047 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.

$$\text{Certified Value } (\bar{x}) = \frac{\Sigma x_i}{n}$$

$$\text{Uncertainty } (\pm) = \frac{2[(\Sigma s_i)^2]^{1/2}}{(n)^{1/2}}$$

$(\bar{x})$ = mean
x_i = individual results
n = number of measurements
Σs_i = The summation of all significant estimated errors (Most common are the errors from instrumental measurement, weighing, dilution to volume and the fixed error reported on the NIST SRM certificate of analysis)

4.0 **TRACEBILITY TO NIST AND VALUES OBTAINED BY INDEPENDENT METHODS**

- "Property of the result of a measurement or the value of a standard whereby it can be related to stated references, usually national or international standards, through an unbroken chain of comparisons all having stated uncertainties." (ISO VIM, 2nd ed., 1993, definition 6.10)
- This product is Traceable to NIST via an unbroken chain of comparisons. The uncertainties for each certified value are reported, taking into account the SRM uncertainty error and the measurement, weighing and volume dilution errors. In rare cases where no NIST SRMs are available, the term 'In-house std.' is specified.

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2.0 **DESCRIPTION OF CRM** Custom Solution

Catalog No.: STLNJ-CAL-1A

Lot Number: **D2-MEB327085**

Matrix: 3% HNO3(v/v)

2,250.00 µg/mL ea:
Cu,

1,000.00 µg/mL ea:
Pb, Sr,

800.00 µg/mL ea:
Cr3,

500.00 µg/mL ea:
Mn,

200.00 µg/mL ea:
B,

150.00 µg/mL ea:
Be

3.0 **CERTIFIED VALUES AND UNCERTAINTIES**

ELEMENT	CERTIFIED VALUE	ELEMENT	CERTIFIED VALUE	ELEMENT	CERTIFIED VALUE
Beryllium, Be	150.6 ± 0.6 µg/mL	Boron, B	200.9 ± 0.8 µg/mL	Chromium+3, Cr3	797 ± 3 µg/mL
Copper, Cu	2,249 ± 5 µg/mL	Lead, Pb	998 ± 2 µg/mL	Manganese, Mn	497.9 ± 2.7 µg/mL
Strontium, Sr	998 ± 3 µg/mL				

Certified Density: 1.033 g/mL (measured at 22° C)

The following equations are used in the calculation of the certified value and the uncertainty. Reported uncertainties represent expanded uncertainties expressed at approximately the 95% confidence level using a coverage factor of k = 2.

$$\text{Certified Value } (\bar{x}) = \frac{\Sigma x_i}{n}$$

$$\text{Uncertainty } (\pm) = \frac{2[(\Sigma s_i)^2]^{1/2}}{(n)^{1/2}}$$

$(\bar{x})$ = mean
x_i = individual results
n = number of measurements
Σs_i = The summation of all significant estimated errors (Most common are the errors from instumental measurement, weighing, dilution to volume and the fixed error reported on the NIST SRM certificate of analysis)

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2.0 **DESCRIPTION OF CRM** Custom Solution

Catalog No.: STLNJ-CAL-1B

Lot Number: **D2-MEB327086**

Matrix: tr. HF, 3% HNO3(v/v)

2,000.00 µg/mL ea:
Ti,

500.00 µg/mL ea:
Mo,

200.00 µg/mL ea:
Sb, Sn

3.0 **CERTIFIED VALUES AND UNCERTAINTIES**

ELEMENT	CERTIFIED VALUE	ELEMENT	CERTIFIED VALUE	ELEMENT	CERTIFIED VALUE
Antimony, Sb	200.4 ± 0.5 µg/mL	Molybdenum, Mo	499.4 ± 1.8 µg/mL	Tin, Sn	200.4 ± 0.7 µg/mL
Titanium, Ti	1,993 ± 6 µg/mL				

Certified Density: 1.022 g/mL (measured at 22° C)

The following equations are used in the calculation of the certified value and the uncertainty. Reported uncertainties represent expanded uncertainties expressed at approximately the 95% confidence level using a coverage factor of k = 2.

$$\text{Certified Value } (\bar{x}) = \frac{\Sigma x_i}{n}$$

$$\text{Uncertainty } (\pm) = \frac{2[(\Sigma s_i)^2]^{1/2}}{(n)^{1/2}}$$

($\bar{x}$) = mean
x_i = individual results
n = number of measurements
Σs_i = The summation of all significant estimated errors
(Most common are the errors from instumental measurement, weighing, dilution to volume and the fixed error reported on the NIST SRM certificate of analysis)

4.0 **TRACEBILITY TO NIST AND VALUES OBTAINED BY INDEPENDENT METHODS**

- "Property of the result of a measurement or the value of a standard whereby it can be related to stated references, usually national or international standards, through an unbroken chain of comparisons all having stated uncertainties." (ISO VIM, 2nd ed., 1993, definition 6.10)
- This product is Traceable to NIST via an unbroken chain of comparisons. The uncertainties for each certified value are reported, taking into account the SRM uncertainty error and the measurement, weighing and volume dilution errors. In rare cases where no NIST SRMs are available, the term 'in-house std.' is specified.

Certificate of Analysis

Product Description:

Part Number:	**SM-606-122** Solution A
Lot Number:	**1218818**
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	12500.0 ± 62.5	3109a	050825	K	5000 ± 25	3141a	051220
Mg	12500.0 ± 62.5	3131a	050302	Na	12500.0 ± 62.5	3152a	010728

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 NBS 602 method.

High-Purity Standards is certified to ISO 9001:2008 and accredited to ISO/IEC 17025:2005 and ISO Guide 34:2009.

Certificate of Analysis

Product Description:

Part Number:	**SM-606-122** Solution B
Lot Number:	**1219217**
Matrix:	10% HNO_3
Purity:	99.99% - 99.999%

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	051031

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 NBS 602 method.

c. **Thermometer**
The standard thermometers are calibrated every year by the ISO 17025 accredited metrology service. The thermometers used in-house are verified against the standard thermometers yearly.

High-Purity Standards is certified to ISO 9001:2008 and accredited to ISO/IEC 17025:2005 and ISO Guide 34:2009.

Certificate of Analysis

Product Description:

1623262
ID: ME_Duo_CCV_D_00004
Int. CCV Soln D for Duo

Part Number:	**SM-606-122**
	Solution D
Lot Number:	**1218025**
Matrix:	2% HNO_3 + Tr HF
Purity:	99.98% - 99.999%

Certified Values:

Element	(µg/mL)	SRM ID	SRM Lot#	Element	(µg/mL)	SRM ID	SRM Lot#
Sb	100.0 ± 0.6	3102a	061229	Ag	125.00 ± 0.63	3151	992212
Mo	250.0 ± 1.5	3134	891307	Sn	100.0 ± 0.6	3161a	070330
Si	1000 ± 10	3150	071204	Ti	1000 ± 6	3162a	060808

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 NBS 602 method.

Lot No.: **1218025**
Rev. No.: 3.1.0
Page 1 of 2

High-Purity Standards is certified to ISO 9001:2008 and accredited to ISO/IEC 17025:2005 and ISO Guide 34:2009.

Certificate of Analysis

Product Description:

Part Number:	**SM-606-122** Solution C
Lot Number:	**1218026**
Matrix:	5% HNO_3
Purity:	99.995% - 99.9999%

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	3104a	070222	Mn	500 ± 5	3132	050429
Be	100 ± 1	3105a	090514	Ni	250.00 ± 1.25	3136	000612
B	100.0 ± 0.5	3107	070514	Se	250.0 ± 2.5	3149	100901
Cd	125.00 ± 0.63	3108	060531	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	011017	Zn	250.00 ± 1.25	3168a	080123

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.

High-Purity Standards is certified to ISO 9001:2008 and accredited to ISO/IEC 17025:2005 and ISO Guide 34:2009.

Revd 5/12/10 c.c.

INORGANIC VENTURES — CERTIFICATE OF ANALYSIS

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857935
ID: HE_ICSA_00027
Exp: Restrict Prod: CDC
ICP ICSA standard

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ACCREDITED ACCREDITED

2.0 **DESCRIPTION OF CRM**

	Stock Solution
Catalog No.:	CLPP-ICS-A
Lot Number:	**D2-MEB324151MCA**
Matrix:	2% HNO3(v/v)

5,000.00 µg/mL ea:
Al, Ca, Mg,

2,000.00 µg/mL ea:
Fe

3.0 **CERTIFIED VALUES AND UNCERTAINTIES**

ELEMENT	CERTIFIED VALUE	ELEMENT	CERTIFIED VALUE	ELEMENT	CERTIFIED VALUE
Aluminum, Al	5,000 ± 18 µg/mL	Calcium, Ca	5,000 ± 20 µg/mL	Iron, Fe	2,000 ± 6 µg/mL
Magnesium, Mg	5,000 ± 16 µg/mL				

Certified Density: 1.085 g/mL (measured at 20 ± 1° C)

The following equations are used in the calculation of the certified value and the uncertainty. Reported uncertainties represent expanded uncertainties expressed at approximately the 95% confidence level using a coverage factor of k = 2.

$$\text{Certified Value } (\bar{x}) = \frac{\Sigma x_i}{n}$$

$$\text{Uncertainty } (\pm) = \frac{2[(\Sigma s_i)^2]^{1/2}}{(n)^{1/2}}$$

$(\bar{x})$ = mean
x_i = individual results
n = number of measurements
Σs_i = The summation of all significant estimated errors (Most common are the errors from instrumental measurement, weighing, dilution to volume and the fixed error reported on the NIST SRM certificate of analysis)

4.0 **TRACEBILITY TO NIST AND VALUES OBTAINED BY INDEPENDENT METHODS**

- "Property of the result of a measurement or the value of a standard whereby it can be related to stated references, usually national or international standards, through an unbroken chain of comparisons all having stated uncertainties." (ISO VIM, 2nd ed., 1993, definition 6.10)
- This product is Traceable to NIST via an unbroken chain of comparisons. The uncertainties for each certified value are reported, taking into account the SRM uncertainty error and the measurement, weighing and volume dilution errors. In rare cases where no NIST SRMs are available, the term 'in-house std.' is specified.

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CERTIFICATE OF ANALYSIS

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Date Rec'd 6/22/2010 MP

2.0 **DESCRIPTION OF CRM** Stock Second Source Solution

Catalog No.: IV-7

Lot Number: **B2-MEB236098**

Matrix: 7% HNO3(v/v)

Second Source: Whenever possible, this solution was manufactured from a second set of concentrates in our manufacturing facility.

1,000.00 µg/mL ea:
K,

100.00 µg/mL ea:
Ag, Al, B, Ba, Na,

50.00 µg/mL ea:
Si

3.0 **CERTIFIED VALUES AND UNCERTAINTIES**

ELEMENT	CERTIFIED VALUE	ELEMENT	CERTIFIED VALUE	ELEMENT	CERTIFIED VALUE
Aluminum, Al	100.0 ± 0.4 µg/mL	Barium, Ba	99.8 ± 0.2 µg/mL	Boron, B	100.1 ± 0.3 µg/mL
Potassium, K	996 ± 3 µg/mL	Silicon, Si	50.67 ± 0.24 µg/mL	Silver, Ag	99.8 ± 0.1 µg/mL
Sodium, Na	99.9 ± 0.6 µg/mL				

Certified Density: 1.038 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.

$$\text{Certified Value } (\bar{x}) = \frac{\Sigma x_i}{n}$$

$$\text{Uncertainty } (\pm) = \frac{2\left(\Sigma s_i^2\right)^{1/2}}{(n)^{1/2}}$$

$(\bar{x})$ = mean
x_i = individual results
n = number of measurements
Σs_i = The summation of all significant estimated errors (Most common are the errors from instrumental measurement, weighing, dilution to volume, and the fixed error reported on the NIST SRM certificate of analysis.)

4.0 **TRACEABILITY TO NIST AND VALUES OBTAINED BY INDEPENDENT METHODS**

- "Property of the result of a measurement or the value of a standard whereby it can be related to stated references, usually national or international standards, through an unbroken chain of comparisons all having stated uncertainties." (ISO VIM, 2nd ed., 1993, definition 6.10)
- This product is Traceable to NIST via an unbroken chain of comparisons. The uncertainties for each certified value are reported, taking into account the SRM uncertainty error and the measurement, weighing and volume dilution errors. In rare cases where no NIST SRMs are available, the term "in-house std." is specified.

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Date Recd
6/22/2010
MR

2.0 **DESCRIPTION OF CRM** Stock Second Source Solution

Catalog No.: IV-19

Lot Number: **C2-MEB292064**

Matrix: tr. HF, 7% HNO3(v/v)

Second Source: Whenever possible, this solution was manufactured from a second set of concentrates in our manufacturing facility.

100.00 µg/mL ea:
As, Be, Ca, Cd, Co, Cr3, Cu, Fe, Mg, Mn, Mo, Ni, Pb, Sb, Se, Ti, Tl, V, Zn

3.0 **CERTIFIED VALUES AND UNCERTAINTIES**

ELEMENT	CERTIFIED VALUE	ELEMENT	CERTIFIED VALUE	ELEMENT	CERTIFIED VALUE
Antimony, Sb	100.0 ± 0.2 µg/mL	Arsenic, As	100.3 ± 0.4 µg/mL	Beryllium, Be	100.0 ± 0.2 µg/m
Cadmium, Cd	100.0 ± 0.2 µg/mL	Calcium, Ca	100.3 ± 0.3 µg/mL	Chromium+3, Cr3	100.3 ± 0.4 µg/m
Cobalt, Co	100.3 ± 0.2 µg/mL	Copper, Cu	99.9 ± 0.4 µg/mL	Iron, Fe	100.2 ± 0.2 µg/m
Lead, Pb	100.0 ± 0.4 µg/mL	Magnesium, Mg	100.1 ± 0.4 µg/mL	Manganese, Mn	100.0 ± 0.5 µg/m
Molybdenum, Mo	100.0 ± 0.3 µg/mL	Nickel, Ni	99.9 ± 0.2 µg/mL	Selenium, Se	100.0 ± 0.3 µg/m
Thallium, Tl	100.0 ± 0.2 µg/mL	Titanium, Ti	100.0 ± 0.3 µg/mL	Vanadium, V	100.0 ± 0.4 µg/m
Zinc, Zn	99.8 ± 0.3 µg/mL				

Certified Density: 1.036 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.

$$\text{Certified Value } (\bar{x}) = \frac{\Sigma x_i}{n}$$

$$\text{Uncertainty } (\pm) = \frac{2(\Sigma s_i)^2]^{1/2}}{(n)^{1/2}}$$

$(\bar{x})$ = mean
x_i = individual results
n = number of measurements
Σs_i = The summation of all significant estimated errors (Most common are the errors from instrumental measurement, weighing, dilution to volume, and the fixed error reported on the NIST SRM certificate of analysis.)

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CERTIFICATE OF ANALYSIS

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2.0 **DESCRIPTION OF CRM** **10000 µg/mL Sodium in 2% (v/v) HNO3**

Catalog Number:	CGNA10-1, CGNA10-2, and CGNA10-5
Lot Number:	**D2-NA03080**
Starting Material:	Na2CO3
Starting Material Purity (%):	99.997120
Starting Material Lot No:	C18157
Matrix:	2% (v/v) HNO3

Date Rev 4
2/30/10 MP

3.0 **CERTIFIED VALUES AND UNCERTAINTIES**

Certified Concentration: 10,039 ± 19 µg/mL

Certified Density: 1.034 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 $(\bar{x}) = \frac{\sum x_i}{n}$

Uncertainty $(\pm) = \frac{2\left[(\sum s_i)^2\right]^{\frac{1}{2}}}{(n)^{\frac{1}{2}}}$

($\bar{x}$) = mean
x_i = individual results
n = number of measurements
Σs_i = The summation of all significant estimated errors (Most common are the errors from instrumental measurement, weighing, dilution to volume and the fixed error reported on the NIST SRM certificate of analysis)

4.0 **TRACEABILITY TO NIST AND VALUES OBTAINED BY INDEPENDENT METHODS**

"Property of the result of a measurement or the value of a standard whereby it can be related to stated references, usually national or international standards, through an unbroken chain of comparisons all having stated uncertainties." (ISO VIM, 2nd ed., 1993, definition 6.10)

This product is Traceable to NIST via an unbroken chain of comparisons. The uncertainties for each certified value are reported, taking into account the SRM uncertainty error and the measurement, weighing and volume dilution errors. In rare cases where no NIST SRMs are available, the term "in-house std." is specified.

4.1 **Assay Method #1** **10,072 ± 41 µg/mL**
ICP Assay NIST SRM 3152a Lot Number: 010726

Assay Method #2 **10,039 ± 19 µg/mL**
Gravimetric NIST ISRM Lot Number: See Sec. 4.2

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2.0 **DESCRIPTION OF CRM**

	Custom Solution
Catalog No.:	TANJ-STD-3
Lot Number:	**D2-MEB325088**
Matrix:	5% HNO3(v/v)

500.00 µg/mL ea:
Ca, K, Mg, Na,
20.00 µg/mL ea:
Al, Ba,
15.00 µg/mL ea:
Fe,
5.00 µg/mL ea:
Co, V,
4.00 µg/mL ea:
Ni,
3.00 µg/mL ea:
Zn,
2.50 µg/mL ea:
Cu,
2.00 µg/mL ea:
Sr,
1.50 µg/mL ea:
Mn,
1.00 µg/mL ea:
Ag, Cr3, Tl,
0.50 µg/mL ea:
As, Pb, Se,
0.40 µg/mL ea:
Cd,
0.20 µg/mL ea:
Be

3.0 **CERTIFIED VALUES AND UNCERTAINTIES**

ELEMENT	CERTIFIED VALUE	ELEMENT	CERTIFIED VALUE	ELEMENT	CERTIFIED VALUE
Aluminum, Al	20.02 ± 0.04 µg/mL	Arsenic, As	0.4980 ± 0.0023 µg/mL	Barium, Ba	19.97 ± 0.04 µg/mL
Beryllium, Be	0.2000 ± 0.0005 µg/mL	Cadmium, Cd	0.4004 ± 0.0014 µg/mL	Calcium, Ca	499.6 ± 1.4 µg/mL
Chromium+3, Cr3	1.004 ± 0.002 µg/mL	Cobalt, Co	4.994 ± 0.011 µg/mL	Copper, Cu	2.495 ± 0.006 µg/mL

Rev'd 3/19/10 a.c.

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2.0 **DESCRIPTION OF CRM**

	Custom Solution
Catalog No.:	TANJ-STD-4
Lot Number:	**D2-MEB325087**
Matrix:	tr. HF, 5% HNO3(w/v)

5.00 µg/mL ea:
B, Sn,
2.00 µg/mL ea:
Mo, Ti,
1.00 µg/mL ea:
Sb

3.0 **CERTIFIED VALUES AND UNCERTAINTIES**

ELEMENT	CERTIFIED VALUE	ELEMENT	CERTIFIED VALUE	ELEMENT	CERTIFIED VALUE
Antimony, Sb	1.004 ± 0.007 µg/mL	Boron, B	5.018 ± 0.017 µg/mL	Molybdenum, Mo	1.999 ± 0.009 µg/mL
Tin, Sn	5.009 ± 0.013 µg/mL	Titanium, Ti	1.998 ± 0.009 µg/mL		

Certified Density: 1.024 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.

$$\text{Certified Value } (\bar{x}) = \frac{\Sigma x_i}{n}$$

$$\text{Uncertainty } (\pm) = \frac{2[(\Sigma s_i)^2]^{1/2}}{(n)^{1/2}}$$

$(\bar{x})$ = mean
x_i = individual results
n = number of measurements
Σs_i = The summation of all significant estimated errors (Most common are the errors from instrumental measurement, weighing, dilution to volume, and the fixed error reported on the NIST SRM certificate of analysis.)

4.0 **TRACEABILITY TO NIST AND VALUES OBTAINED BY INDEPENDENT METHODS**

"Property of the result of a measurement or the value of a standard whereby it can be related to stated references, usually national or international standards, through an unbroken chain of comparisons all having stated uncertainties." (ISO VIM, 2nd ed., 1993, definition 6.10)

This product is Traceable to NIST via an unbroken chain of comparisons. The uncertainties for each certified value are reported, taking into account the SRM uncertainty error and the measurement, weighing and volume dilution errors. In rare cases where no NIST SRMs are available, the term 'in-house std.' is specified.

Certificate of Analysis

Expiry: 14-Jun-16

Rec'd 1/8/15
C.G.

Part Number: 4400-100419DD01
Lot Number: 14L124
Shelf Life: 18 Months

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.

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ISO 9001:2008

Certificate of Analysis

Rec'd 1/8/15
a.c.

Expiry: 18-Jun-16

Part Number: 4400-100419DD01 **Solution B**
Lot Number: 14L126
Shelf Life: 18 Months

TestAmerica/Edison
Custom Standard
10% HNO3

Concentrations in µg/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 labcratory conditions. See attached MSDS for proper handling information.

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Certifying Officer

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ISO 9001:2008 17025

Certificate of Analysis

Expiry: 14-Jun-16

Rev'd 1/8/15 a.c.

Part Number: **4400-100419DD01** **Solution C**
Lot Number: **14L127**
Shelf Life: **18 Months**

5453214

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 1-800-876-7654 in the USA, +31 20 638 05 97 in Europe or visit our web-site at www.cpiinternational.com.

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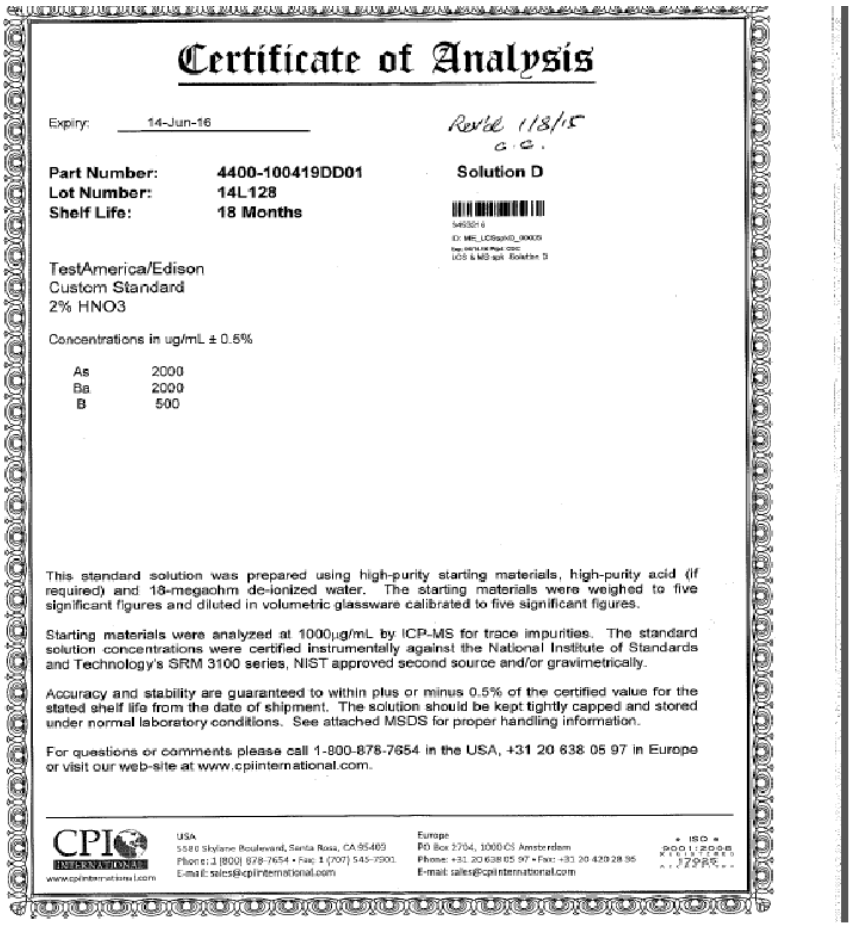

Certificate of Analysis

Expiry: 14-Jun-16

Revised 1/8/15 C.C.

Part Number: 4400-100419DD01
Lot Number: 14L128
Shelf Life: 18 Months

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.

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APPENDIX C: PROCEDURES CHECKLISTS

GENERAL PROCEDURES CHECKLIST

The following equipment will be requisitioned and inspected prior to the commencement of field activities.

Date: _____

Auditor: _____

	Y	N	N/A
1. PID with an 10.2 eV lamp (or equivalent) available:			
• Unit in working order			
• Unit calibrated according to manufacturer's specification			
2a. Water level probe available:			
• Unit is in working order			
• Unit has been decontaminated			
2b. pH/Conductivity/eH probe available:			
• Unit is in working order			
• Unit has been decontaminated			
2c. Temperature probe available:			
• Unit is in working order			
• Unit has been decontaminated			
2d. Turbidity meter available:			
• Unit is in working order			
• Unit has been decontaminated			
2e. Dust monitor available:			
• Unit is in working order			
2f. Disposable bailers and filters:			
• Units have been ordered and received			
• Spare parts are on hand			
3. Proper drilling/sampling tools available:			
• Equipment is in good shape			
• Equipment has been decontaminated			
4. All instruments successfully calibrated daily as required:			
5. Pry bar available for removing manway covers (if needed):			
6. Clean plastic sheeting available:			
7. Coolers available:			
8. Proper preservatives as listed in Section 4.0 of QAPP:			
9. Proper sample containers as listed in Section 4.0 of the QAPP Work Scope available:			
10. Field notebook and writing utensils available:			
11. Chain-of-Custody forms are available			

GENERAL PROCEDURES CHECKLIST

(continued)

	Y	N	N/A
12. Sample labels and custody seals are available			
13. Decontamination equipment/supplies are available:			
• Liquinox			
• Distilled water			
• Methanol			
• Nitric Acid			
• Cow trough & other buckets			
• Steam cleaner			
• Containers for waste water available			
14. Work gloves are available:			
15. Latex sampling gloves are available:			
16. Tools, spare fittings, fuses, batteries, etc.:			
17. Trip blanks and water for field blanks sent from the lab:			
18. Aluminum foil for head space analysis:			

N = No
Y = Yes
N/A = Not Applicable

SOIL SAMPLING PROCEDURES CHECKLIST

The following procedures will be followed during the collection of the soil samples.

Date: _____

Auditor: _____

	Y	N	N/A
1. Equipment decontaminated:			
• Decon area prepared			
• Cow trough & re-usable buckets steam cleaned			
• Clean plastic used			
• Equipment steam cleaned properly			
• Equipment reloaded or staged to prevent contamination			
2. PID properly calibrated :			
3. Presampling information logged in field log notebook (location, personnel, etc.):			
4. Sampling gloves worn prior to collecting the samples:			
5. Samples preserved and secured properly:			
6. Blanks and duplicates collected when required:			
7. Blanks with proper ID and clearly indicated in notebook:			
8. All field screening information logged for each boring location:			
9. All instruments successfully calibrated daily as required:			
10. "Hot Zone" sample determined for each boring location:			
11. Samples properly labeled and shipped to the lab:			
12. Disposable sampling equipment collected in a plastic bag for disposal:			
13. Decontamination procedures repeated when necessary:			
14. Holding times and shipment times for samples known:			
15. Samples, with chain-of-custody, shipped when necessary and proper cooler temperature confirmed immediately prior to shipment:			
16. Confirm that lab received the samples:			

Y = Yes
N = No
N/A = Not Applicable

WATER SAMPLING PROCEDURES CHECKLIST

The following procedures will be followed during the collection of the water samples.

Date: _____

Auditor: _____

	Y	N	N/A
1. Equipment decontaminated:			
• Decon area prepared			
• Cow trough steam cleaned			
• Clean plastic used			
• Equipment steam cleaned properly			
• Equipment reloaded or staged to prevent contamination			
2. PID properly calibrated			
3. Presampling information logged in field log notebook (location, personnel, etc.):			
4. Sampling gloves worn prior to collecting the samples:			
5. Samples preserved and secured properly: Note: If turbidity does not stabilize, collect additional sample if requested by the CPM . Field filter using field filters. Preserve and ship to lab. Sample ID should be XX-XF (for filtered).			
6. Blanks and duplicates collected when required:			
7. Blanks with proper ID and clearly indicated in notebook:			
8. All field screening information logged for each sampling location:			
9. All instruments successfully calibrated daily as required:			
10. Samples properly labeled and shipped to the lab:			
11. All disposable sampling equipment collected in a plastic bag for disposal:			
12. Decontamination procedures repeated when necessary:			
13. Holding times and shipment times for samples known:			
14. Samples, with chain-of-custody, shipped when necessary and proper cooler temperature confirmed immediately prior to shipment:			
15. Confirm that lab received the samples:			

Y = Yes
N = No
N/A = Not Applicable
F = Filtered

APPENDIX D: SAMPLING INSTRUCTIONS & FIELD OBSERVATION LOG

SAMPLING INSTRUCTIONS & FIELD OBSERVATION LOG

GROUNDWATER SAMPLING RECORD

SITE NAME	**Safety-Kleen Service Center**	**DATE**	
	60 Seabro Ave, N.Amityville, NY	**Weather**	
Sampler			

Well Name / ID	***GT-1***	***GT-2***	***GT-3***	***GT-4***	***DW-1***	***GT-5***	***GT-6***	***GT-7***	***VE-1R***	***VE-5***	**warehouse** ***VP-A***	***VP-B***
Lab Analysis - EPA 8260c VOCs	**Collect Samples as listed on the pre-printed Chain-of-Custody. Questions, contact Melissa Haas at Tel 203.944.1310.**											
Lab Analysis - EPA 8015d MSRO												
Natural Attenuation Parameters												
Split Samples	**Collect Samples as Directed in Separate Cooler provided with glassware.**											
Duplicate Sample:	**Collect Samples as listed on the pre-printed Chain-of-Custody. Questions, contact Melissa Haas.**											
Sample Equipment Rinse Blank												
MS/MSD												
ORC Socks Deployed	**Yes**	**No**	**Yes**	**No**	**No**	**Yes**	**Yes**	**No**	**Yes**	**No**	Yes	**Yes**
Socks Changed ("C") or Redeployed ("R")												
Collect Field Parameters	**Yes**	**Yes**	**Yes**	**Yes-Only**	**Yes**	**Yes**	**Yes**	**Yes**	**Yes**	**Yes**	Yes	**Yes**
Diameter of Well Casing	2 in	2 in	2 in	2 in	Manhole	2 in	2 in	2 in	4 in	1 in	2 in	2 in
Depth of Well (ft.)	25.54	27.40	27.48	26.18	10.50	24.85	28.2	28.1	24.1	24.80	25.49	23.0
Depth to Groundwater (ft.)												
Water Column Height (ft.)												
Volume Purged (gal)												
Purging Method	Bailer	Bailer	Bailer	Bailer	Bailer	Bailer	Bailer	Bailer	Bailer	Bailer	Bailer	Bailer
Collect additional sample for analysis of dissolved MSRO.	**Yes**						**Yes**		**Yes**			
Sampling Time												
Sample date												
GW Visual Observations												
color												
sheen (slight, moderate, heavy)												
odor (slight, moderate, heavy)												
carbon/particulates/settled matter (lo, med, high)												
GW Field Parameters												
Temperature (C)												
pH												
Conductivity in mS												
Dissolved Oxygen (mg/L)												
ORP (Eh (Mv))												
Turbidity (visual / NTU)												

Comments	**Containerize all fluids as directed by Terri Cowans at the facility. Tel: 631.443.4509 (cell). Coordinate with Terri in regards to moving all IDW back to the facility from wells GT-6 & GT-7. Under no circumstances are drums or debris to be left near wells GT-6 & 7. Both wells are located off-site. SK/consultants have permission from the property owner to access the wells.**	
	On-arrival at the facility, check-in at the main office, and notify Terri you are on-site. Follow all facility rules, and any direction with regard to well access, facility access,	
	Sample Collection Equipment: Collect samples with dedicated disposable bailers. DW-1 Soil Bottom Sample - Collect with Hand-Auger.	
	Complete field data in these rows.	Collect rinse blank if DW-1 is dry and a soil sample is collected.

APPENDIX E: TESTAMERICA LABORATORIES, INC. QUALITY ASSURANCE MANUALS

Limits for Project: 46008953 - 2016 Safety-Kleen Amityville

Analysis Group	Method Description	Method Code	Prep Method	Analyte Description	CAS Number	RL	MDL	LOD	Units	LCS - Low	LCS - High	LCS - RPD %	MS - Low	MS - High	MS - RPD %	Surrogate Low	Surrogate High
Solid - VOC/Mineral Spirits	Volatile Organic Compounds by GC/MS	8260C	5035A_M (medium level)	Chloromethane	74-87-3	50.0	11.0		ug/Kg	48	147	30	48	147	30		
				Bromomethane	74-83-9	50.0	9.00		ug/Kg	59	132	30	59	132	30		
				Vinyl chloride	75-01-4	50.0	10.0		ug/Kg	60	144	30	60	144	30		
				Chloroethane	75-00-3	50.0	18.5		ug/Kg	57	143	30	57	143	30		
				Methylene Chloride	75-09-2	50.0	10.5		ug/Kg	77	120	30	77	120	30		
				Acetone	67-64-1	250	53.5		ug/Kg	78	122	30	78	122	30		
				Carbon disulfide	75-15-0	50.0	11.0		ug/Kg	68	120	30	68	120	30		
				1,1-Dichloroethene	75-35-4	330	17.0		ug/Kg	77	120	30	77	120	30		
				1,1-Dichloroethane	75-34-3	270	12.0		ug/Kg	79	120	30	79	120	30		
				trans-1,2-Dichloroethene	156-60-5	190	9.00		ug/Kg	77	120	30	77	120	30		
				cis-1,2-Dichloroethene	156-59-2	250	13.0		ug/Kg	80	120	30	80	120	30		
				1,2-Dichloroethene, Total	540-59-0	100	9.00		ug/Kg	80	120	30	80	120	30		
				Chloroform	67-66-3	370	11.0		ug/Kg	80	120	30	80	120	30		
				1,2-Dichloroethane	107-06-2	50.0	12.5		ug/Kg	80	126	30	80	126	30		
				2-Butanone (MEK)	78-93-3	250	110		ug/Kg	54	128	30	54	128	30		
				1,1,1-Trichloroethane	71-55-6	680	14.0		ug/Kg	80	124	30	80	124	30		
				Carbon tetrachloride	56-23-5	760	16.5		ug/Kg	80	131	30	80	131	30		
				Bromodichloromethane	75-27-4	50.0	7.50		ug/Kg	80	120	30	80	120	30		
				1,2-Dichloropropane	78-87-5	50.0	9.00		ug/Kg	78	120	30	78	120	30		
				cis-1,3-Dichloropropene	10061-01-5	50.0	8.00		ug/Kg								
				Trichloroethene	79-01-6	470	11.0		ug/Kg	80	120	30	80	120	30		
				Dibromochloromethane	124-48-1	50.0	11.0		ug/Kg	79	120	30	79	120	30		
				1,1,2-Trichloroethane	79-00-5	50.0	4.00		ug/Kg	74	120	30	74	120	30		
				Benzene	71-43-2	60.0	9.50		ug/Kg	76	120	30	76	120	30		
				trans-1,3-Dichloropropene	10061-02-6	50.0	9.50		ug/Kg	69	120	30	69	120	30		
				2-Chloroethyl vinyl ether	110-75-8	50.0	8.50		ug/Kg								
				Bromoform	75-25-2	50.0	9.00		ug/Kg	77	124	30	77	124	30		
				4-Methyl-2-pentanone (MIBK)	108-10-1	250	31.5		ug/Kg	80	123	30	80	123	30		
				2-Hexanone	591-78-6	250	36.0		ug/Kg								
				Tetrachloroethene	127-18-4	1300	18.0		ug/Kg	77	125	30	77	125	30		
				1,1,2,2-Tetrachloroethane	79-34-5	50.0	9.50		ug/Kg	68	124	30	68	124	30		
				Toluene	108-88-3	700	12.5		ug/Kg	80	120	30	80	120	30		
				Chlorobenzene	108-90-7	1100	12.0		ug/Kg	80	120	30	80	120	30		
				Ethylbenzene	100-41-4	1000	15.0		ug/Kg	76	120	30	76	120	30		
				Styrene	100-42-5	50.0	8.50		ug/Kg	80	120	30	80	120	30		
				m&p-Xylene	179601-23-1	100	14.0		ug/Kg	78	120	30	78	120	30		
				o-Xylene	95-47-6	50.0	16.0		ug/Kg	80	120	30	80	120	30		
				Xylenes, Total	1330-20-7	260	14.0		ug/Kg	80	120	30	80	120	30		
				Acetonitrile	75-05-8	1000	65.0		ug/Kg								
				Vinyl acetate	108-05-4	100	24.0		ug/Kg								
				1,2-Dibromoethane	106-93-4	50.0	9.50		ug/Kg								
				1,3-Dichlorobenzene	541-73-1	2400	16.5		ug/Kg								
				1,4-Dichlorobenzene	106-46-7	1800	16.5		ug/Kg								
				1,2-Dichlorobenzene	95-50-1	1100	11.0		ug/Kg								
				Dichlorodifluoromethane	75-71-8	50.0	7.00		ug/Kg								
				1,1,1,2-Tetrachloroethane	630-20-6	50.0	11.0		ug/Kg	78	120	30	78	120	30		
				1,2,3-Trichloropropane	96-18-4	50.0	18.0		ug/Kg	72	123	30	72	123	30		
				1,2-Dibromo-3-Chloropropane	96-12-8	50.0	11.5		ug/Kg								
				Dibromomethane	74-95-3	50.0	6.50		ug/Kg	79	120	30	79	120	30		
				Methyl methacrylate	80-62-6	100	10.5		ug/Kg	76	132	30	76	132	30		
				Benzyl chloride	100-44-7	50.0	7.50		ug/Kg	45	123	30	45	123	30		
				Iodomethane	74-88-4	50.0	9.50		ug/Kg	78	124	30	78	124	30		
				trans-1,4-Dichloro-2-butene	110-57-6	50.0	9.00		ug/Kg	44	138	30	44	138	30		
				1,2-Dichloroethane-d4 (Surr)	17060-07-0				ug/Kg			30			30	69	143
				Toluene-d8 (Surr)	2037-26-5				ug/Kg			30			30	67	127
				4-Bromofluorobenzene	460-00-4				ug/Kg			30			30	61	137
				Dibromofluoromethane (Surr)	1868-53-7				ug/Kg								
Solid - VOC/Mineral Spirits	Volatile Organic Compounds by GC/MS	8260C	5035A_FW (low level)	Chloromethane	74-87-3	5.00	0.380		ug/Kg	66	128	30	66	128	30		
				Bromomethane	74-83-9	5.00	0.320		ug/Kg								
				Vinyl chloride	75-01-4	5.00	0.390		ug/Kg	70	134	30	70	134	30		
				Chloroethane	75-00-3	5.00	0.350		ug/Kg	50	139	30	50	139	30		
				Methylene Chloride	75-09-2	50.0	0.320		ug/Kg	79	128	30	79	128	30		
				Acetone	67-64-1	50.0	1.06		ug/Kg	75	120	30	75	120	30		
				Carbon disulfide	75-15-0	5.00	0.430		ug/Kg	74	130	30	74	130	30		
				1,1-Dichloroethene	75-35-4	270	0.410		ug/Kg	79	132	30	79	132	30		
				1,1-Dichloroethane	75-34-3	330	0.340		ug/Kg	80	124	30	80	124	30		
				trans-1,2-Dichloroethene	156-60-5	190	0.390		ug/Kg	80	129	30	80	129	30		
				cis-1,2-Dichloroethene	156-59-2	250	0.220		ug/Kg	80	123	30	80	123	30		
				1,2-Dichloroethene, Total	540-59-0	5.00	0.570		ug/Kg	80	126	30	80	126	30		
				Chloroform	67-66-3	370	0.210		ug/Kg	80	122	30	80	122	30		
				1,2-Dichloroethane	107-06-2	20.0	0.110		ug/Kg	68	120	30	68	120	30		
				2-Butanone (MEK)	78-93-3	120	0.770		ug/Kg	61	140	30	61	140	30		
				1,1,1-Trichloroethane	71-55-6	680	0.380		ug/Kg	80	125	30	80	125	30		
				Carbon tetrachloride	56-23-5	760	0.430		ug/Kg	77	138	30	77	138	30		
				Bromodichloromethane	75-27-4	5.00	0.380		ug/Kg	76	129	30	76	129	30		
				1,2-Dichloropropane	78-87-5	5.00	0.170		ug/Kg	77	124	30	77	124	30		
				cis-1,3-Dichloropropene	10061-01-5	5.00	0.150		ug/Kg	75	124	30	75	124	30		
				Trichloroethene	79-01-6	470	0.260		ug/Kg	79	122	30	79	122	30		
				Dibromochloromethane	124-48-1	5.00	0.150		ug/Kg	67	143	30	67	143	30		
				1,1,2-Trichloroethane	79-00-5	5.00	0.280		ug/Kg	76	124	30	76	124	30		
				Benzene	71-43-2	60.0	0.200		ug/Kg	75	127	30	75	127	30		
				trans-1,3-Dichloropropene	10061-02-6	5.00	0.100		ug/Kg	72	121	30	72	121	30		
				2-Chloroethyl vinyl ether	110-75-8	5.00	0.600		ug/Kg	76	124	30	76	124	30		
				Bromoform	75-25-2	5.00	0.130		ug/Kg	19	150	30	19	150	30		
				4-Methyl-2-pentanone (MIBK)	108-10-1	5.00	2.22		ug/Kg	80	120	30	80	120	30		
				2-Hexanone	591-78-6	10.0	0.940		ug/Kg	78	120	30	78	120	30		
				Tetrachloroethene	127-18-4	1300	0.280		ug/Kg	73	130	30	73	130	30		
				1,1,2,2-Tetrachloroethane	79-34-5	5.00	0.170		ug/Kg	72	131	30	72	131	30		
				Toluene	108-88-3	700	0.190		ug/Kg	75	122	30	75	122	30		
				Chlorobenzene	108-90-7	1100	0.140		ug/Kg	80	120	30	80	120	30		
				Ethylbenzene	100-41-4	1000	0.180		ug/Kg	79	124	30	79	124	30		
				Styrene	100-42-5	5.00	0.150		ug/Kg	78	123	30	78	123	30		
				m&p-Xylene	179601-23-1	5.00	0.110		ug/Kg	79	121	30	79	121	30		
				o-Xylene	95-47-6	5.00	0.160		ug/Kg	79	123	30	79	123	30		
				Xylenes, Total	1330-20-7	260	0.110		ug/Kg	79	122	30	79	122	30		
				Acetonitrile	75-05-8	50.0	4.07		ug/Kg	60	137	30	60	137	30		
				Vinyl acetate	108-05-4	20.0	0.600		ug/Kg	78	123	30	78	123	30		
				1,2-Dibromoethane	106-93-4	5.00	0.120		ug/Kg	80	122	30	80	122	30		
				1,3-Dichlorobenzene	541-73-1	2400	0.120		ug/Kg	79	124	30	79	124	30		
				1,4-Dichlorobenzene	106-46-7	1800	0.130		ug/Kg	79	121	30	79	121	30		
				1,2-Dichlorobenzene	95-50-1	1100	0.140		ug/Kg	80	121	30	80	121	30		

Limits for Project: 46008953 - 2016 Safety-Kleen Amityville

Analysis Group	Method Description	Method Code	Prep Method	Analyte Description	CAS Number	RL	MDL	LOD	Units	LCS - Low	LCS - High	LCS - RPD %	MS - Low	MS - High	MS - RPD %	Surrogate Low	Surrogate High
				Dichlorodifluoromethane	75-71-8	5.00	0.320		ug/Kg	72	127	30	72	127	30		
				1,1,1,2-Tetrachloroethane	630-20-6	5.00	0.410		ug/Kg	74	135	30	74	135	30		
				1,2,3-Trichloropropane	96-18-4	5.00	0.230		ug/Kg	77	120	30	77	120	30		
				1,2-Dibromo-3-Chloropropane	96-12-8	10.0	0.470		ug/Kg	65	129	30	65	129	30		
				Dibromomethane	74-95-3	5.00	0.140		ug/Kg	80	120	30	80	120	30		
				Methyl methacrylate	80-62-6	10.0	0.380		ug/Kg	74	120	30	74	120	30		
				Benzyl chloride	100-44-7	5.00	0.240		ug/Kg	63	135	30	63	135	30		
				Iodomethane	74-88-4	10.0	0.300		ug/Kg	79	128	30	79	128	30		
				trans-1,4-Dichloro-2-butene	110-57-6	10.0	0.210		ug/Kg	66	124	30	66	124	30		
				1,2-Dichloroethane-d4 (Surr)	17060-07-0				ug/Kg			30			30	78	135
				Toluene-d8 (Surr)	2037-26-5				ug/Kg			30			30	73	121
				4-Bromofluorobenzene	460-00-4				ug/Kg			30			30	67	126
				Dibromofluoromethane (Surr)	1868-53-7				ug/Kg								
Solid - VOC/Mineral Spirits	Hydrocarbon Product Identification (GC)	8015D_ID	3546	Mineral Spirits	64475-85-0	6700	2500		ug/Kg			30			30		
				o-Terphenyl	84-15-1				ug/Kg							25	132
Solid - VOC/Mineral Spirits	Percent Moisture	Moisture		Percent Moisture	STL00177	1.00			%								
				Percent Solids	STL00234	1.00			%								
Water - VOC/Mineral Spirits-total/dissolved	Volatile Organic Compounds by GC/MS	8260C	5030C	Acetone	67-64-1	50.0	1.07		ug/L	39	150	30	39	150	30		
				Acetonitrile	75-05-8	10.0	1.30		ug/L	37	150	30	37	150	30		
				Benzene	71-43-2	1.00	0.0900		ug/L	77	121	30	77	121	30		
				Benzyl chloride	100-44-7	10.0	0.150		ug/L	48	133	30	48	133	30		
				Bromodichloromethane	75-27-4	50.0	0.150		ug/L	76	120	30	76	120	30		
				Bromoform	75-25-2	5.00	0.180		ug/L	53	120	30	53	120	30		
				Bromomethane	74-83-9	5.00	0.180		ug/L	10	150	30	10	150	30		
				4-Methyl-2-pentanone (MIBK)	108-10-1	5.00	0.630		ug/L	78	124	30	78	124	30		
				Carbon disulfide	75-15-0	60.0	0.220		ug/L	69	133	30	69	133	30		
				Carbon tetrachloride	56-23-5	5.00	0.330		ug/L	70	132	30	70	132	30		
				Chlorobenzene	108-90-7	5.00	0.240		ug/L	80	120	30	80	120	30		
				Chloroethane	75-00-3	5.00	0.370		ug/L	52	150	30	52	150	30		
				2-Chloroethyl vinyl ether	110-75-8	20.0	0.170		ug/L	43	146	30	43	146	30		
				Chloroform	67-66-3	7.00	0.220		ug/L	80	120	30	80	120	30		
				Chloromethane	74-87-3	5.00	0.220		ug/L	56	131	30	56	131	30		
				cis-1,3-Dichloropropene	10061-01-5	1.00	0.160		ug/L	77	120	30	77	120	30		
				Dibromochloromethane	124-48-1	50.0	0.220		ug/L	73	120	30	73	120	30		
				Dibromomethane	74-95-3	5.00	0.130		ug/L	79	120	30	79	120	30		
				1,2-Dibromo-3-Chloropropane	96-12-8	1.00	0.230		ug/L	55	134	30	55	134	30		
				1,2-Dichlorobenzene	95-50-1	3.00	0.220		ug/L	80	120	30	80	120	30		
				1,3-Dichlorobenzene	541-73-1	3.00	0.330		ug/L	80	120	30	80	120	30		
				1,4-Dichlorobenzene	106-46-7	3.00	0.330		ug/L	80	120	30	80	120	30		
				Dichlorodifluoromethane	75-71-8	5.00	0.140		ug/L	50	131	30	50	131	30		
				1,1-Dichloroethane	75-34-3	5.00	0.240		ug/L	77	123	30	77	123	30		
				1,2-Dichloroethane	107-06-2	1.00	0.250		ug/L	76	121	30	76	121	30		
				1,1-Dichloroethene	75-35-4	5.00	0.340		ug/L	74	123	30	74	123	30		
				1,2-Dichloroethene, Total	540-59-0	2.00	0.180		ug/L	80	120	30	80	120	30		
				1,2-Dichloropropane	78-87-5	1.00	0.180		ug/L	77	123	30	77	123	30		
				Ethylbenzene	100-41-4	5.00	0.300		ug/L	80	120	30	80	120	30		
				2-Butanone (MEK)	78-93-3	50.0	2.20		ug/L	64	120	30	64	120	30		
				Methylene Chloride	75-09-2	5.00	0.210		ug/L	77	123	30	77	123	30		
				Methyl methacrylate	80-62-6	50.0	0.210		ug/L	71	124	30	71	124	30		
				m&p-Xylene	179601-23-1	10.0	0.280		ug/L	80	120	30	80	120	30		
				o-Xylene	95-47-6	5.00	0.320		ug/L	80	120	30	80	120	30		
				Styrene	100-42-5	5.00	0.170		ug/L	80	120	30	80	120	30		
				1,1,1,2-Tetrachloroethane	630-20-6	5.00	0.220		ug/L	77	120	30	77	120	30		
				1,1,2,2-Tetrachloroethane	79-34-5	5.00	0.190		ug/L	74	120	30	74	120	30		
				Tetrachloroethene	127-18-4	5.00	0.120		ug/L	78	122	30	78	122	30		
				trans-1,2-Dichloroethene	156-60-5	5.00	0.180		ug/L	79	120	30	79	120	30		
				trans-1,3-Dichloropropene	10061-02-6	1.00	0.190		ug/L	76	120	30	76	120	30		
				1,1,1-Trichloroethane	71-55-6	5.00	0.280		ug/L	75	125	30	75	125	30		
				1,1,2-Trichloroethane	79-00-5	1.00	0.0800		ug/L	78	120	30	78	120	30		
				Trichloroethene	79-01-6	5.00	0.220		ug/L	77	120	30	77	120	30		
				1,2,3-Trichloropropane	96-18-4	1.00	0.360		ug/L	76	120	30	76	120	30		
				Toluene	108-88-3	5.00	0.250		ug/L	80	120	30	80	120	30		
				Vinyl acetate	108-05-4	5.00	0.480		ug/L	10	150	30	10	150	30		
				Vinyl chloride	75-01-4	2.00	0.0600		ug/L	62	138	30	62	138	30		
				Xylenes, Total	1330-20-7	15.0	0.280		ug/L	80	120	30	80	120	30		
				2-Hexanone	591-78-6	50.0	0.720		ug/L	71	125	30	71	125	30		
				cis-1,2-Dichloroethene	156-59-2	5.00	0.260		ug/L	80	120	30	80	120	30		
				1,2-Dibromoethane	106-93-4	5.00	0.190		ug/L	80	120	30	80	120	30		
				Ethyl methacrylate	97-63-2	5.00	0.120		ug/L	70	121	30	70	121	30		
				Iodomethane	74-88-4	5.00	0.190		ug/L	10	147	30	10	147	30		
				trans-1,4-Dichloro-2-butene	110-57-6	5.00	0.180		ug/L	61	122	30	61	122	30		
				Methacrylonitrile	126-98-7	5.00	0.960		ug/L	77	130	30	77	130	30		
				1,2-Dichloroethane-d4 (Surr)	17060-07-0				ug/L			30			30	74	132
				4-Bromofluorobenzene	460-00-4				ug/L			30			30	77	124
				Toluene-d8 (Surr)	2037-26-5				ug/L			30			30	80	120
				Dibromofluoromethane (Surr)	1868-53-7				ug/L								
Water - VOC/Mineral Spirits-total/dissolved	Hydrocarbon Product Identification (GC)	8015D_ID	3510C	Mineral Spirits	64475-85-0	50.0	26.0		ug/L	70	130	30	70	130	30		
				o-Terphenyl	84-15-1				ug/L							34	158
Water - VOC/Mineral Spirits-total/dissolved	Hydrocarbon Product Identification (GC)	8015D_ID	3510C/FILTRATION	Mineral Spirits	64475-85-0	50.0	26.0		ug/L	70	130	30	70	130	30		
				o-Terphenyl	84-15-1				ug/L							34	158
Water - Natural Attenuation Tests	Alkalinity	2320B		Bicarbonate Alkalinity as CaCO3	STL00138	5.00	5.00		mg/L	90	110						
				Alkalinity	STL00171	5.00	5.00		mg/L	90	110						
Water - Natural Attenuation Tests	Anions, Ion Chromatography	300_ORGFM_28D		Sulfate	14808-79-8	0.600	0.127		mg/L	90	110	15	90	110	15		
Water - Natural Attenuation Tests	Ammonia	SM4500_NH3_H	SM4500NH3_B	Ammonia	7664-41-7	0.100	0.0720		mg/L	87	114		65	135	26		
Water - Natural Attenuation Tests	Dissolved Gases (GC)	RSK_175		Ethane	74-84-0	0.00500	0.00270		mg/L								
				Ethylene	74-85-1	0.00500	0.00220		mg/L								
				Methane	74-82-8	0.00500	0.00170		mg/L	80	120	33	46	142	30		
				Acetylene (Surr)	74-86-2		0.00250		mg/L							62	124
Water - Natural Attenuation Tests	Anions, Ion Chromatography	300_ORGFMS		Nitrate as N	14797-55-8	0.100	0.0300		mg/L	90	110	15	90	110	15		
				Nitrate Nitrite as N	STL00217	0.100	0.0300		mg/L	90	110	15	90	110	15		
				Nitrite as N	14797-65-0	0.120	0.0420		mg/L	90	110	15	90	110	15		

Limits for Project: 46008953 - 2016 Safety-Kleen Amityville

Analysis Group	Method Description	Method Code	Prep Method	Analyte Description	CAS Number	RL	MDL	LOD	Units	LCS - Low	LCS - High	LCS - RPD %	MS - Low	MS - High	MS - RPD %	Surrogate Low	Surrogate High
Water - Natural Attenuation Tests	Nitrogen, Nitrate-Nitrite	353.2		Nitrate as N	14797-55-8	0.100	0.0100	0.0100	mg/L	85	115		53	135	12		
		If needed		Nitrate Nitrite as N	STL00217	0.100	0.00910	0.00910	mg/L	85	115		77	114	10		
				Nitrite as N	14797-65-0	0.100	0.00300	0.00300	mg/L	85	115		81	111	10		
Water - Natural Attenuation Tests	Organic Carbon, Total (TOC)	SM5310B		TOC Result 2	STL00339	1.00			mg/L								
				TOC Result 1	STL00338	1.00			mg/L								
				Total Organic Carbon	7440-44-0	1.00	0.217		mg/L	85	115	10	85	115	10		
Water - Natural Attenuation Tests	Sulfide, Total	SM4500_S2_D		Sulfide as H2S	STL00456	0.106	0.0530	0.0530	mg/L	90	110	10	70	130	50		
Water - Natural Attenuation Tests	Carbon Dioxide and Forms of Alkalinity by Calcul	SM4500_CO2_D		Carbon Dioxide, Free	STL00334	5.00			mg/L								
Water - Natural Attenuation Tests	Phosphorus	4500_P_E	SM4500_P_B	Phosphate as PO4	STL00455	0.0900	0.0330		mg/L	85	115		75	123	10		
				Phosphorus as P	7723-14-0	0.0300	0.0110		mg/L								
Water - Natural Attenuation Tests	Metals (ICP)	200.7	200.7_P_TR/FILTRATION	Iron	7439-89-6	150	78.3		ug/L	85	115	20	70	130	20		
				Manganese	7439-96-5	15.0	4.89		ug/L	85	115	20	70	130	20		

Quality Assurance Manual

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Facility Distribution No. ELECTRONIC **Distributed To: FACILITY INTRANET**

Title Page:

Quality Assurance Manual
Approval Signatures

10/26/2015

Laboratory Director – Mark Acierno — Date

10/26/2015

Quality Assurance Manager - Carl Armbruster — Date

10/26/2015

Operations Manager – Donald Evans — Date

SECTION 2. TABLE OF CONTENTS

LIST OF TABLES

LIST OF FIGURES

LIST OF APPENDICES

REFERENCED CORPORATE SOPs AND POLICIES

SOP / Policy Reference	Title
CA-I-P-002	Electronic Reporting and Signature Policy
CA-L-P-002	Contract Compliance Policy
CA-L-S-002	Subcontracting Procedures
CA-Q-M-002	Corporate Quality Management Plan
CA-Q-S-001	Solvent and Acid Lot Testing and Approval
CA-Q-S-002	Acceptable Manual Integration Practices
CA-Q-S-006	Detection Limits
CA-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 of Potential Data Discrepancies and Determination for Data Recall
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

REFERENCED LABORATORY SOPs

SOP Reference	Title
ED-GEN-002	Document Control
ED-GEN-003	Control of Non-Conformances and Corrective Action
ED-GEN-022	Training
ED-GEN-024	Record Storage and Retention
ED-GEN-001	Data Management and Handling
ED-GEN-021	Data Review
ED-GEN-007	Subsampling
ED-SPM-001	Sample Receipt, Login, Identification, And Storage
ED-RP-001	Reports Production
ED-GEN-011	Calibration and Use of Pipettes
ED-FLD-008, -009	Groundwater Sampling and Flow Monitoring
ED-FLD-014	Wastewater Sampling
ED-FLD-001 thru -010	Field Analytical Parameters
ED-SPM-006	Acceptance and Handling of Regulated Domestic & Foreign Soils
ED-SPM-007	Disposal of Samples and Associated Laboratory Waste

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, and ISO/IEC Guide 17025:2005(E). In addition, the policies and procedures outlined in this manual are compliant with TestAmerica's Corporate Quality Management Plan (CQMP) and the various accreditation and certification programs listed in Appendix 3. The CQMP provides a summary of TestAmerica's quality and data integrity system. It contains requirements and general guidelines under which all TestAmerica facilities shall conduct their operations.

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

- EPA 600/4-88/039, *Methods for the Determination of Organic Compounds in Drinking Water,* EPA, Revised July 1991.
- EPA 600/R-95/131, *Methods for the Determination of Organic Compounds in Drinking Water,* Supplement III, EPA, August 1995.
- EPA 600/4-79-019, *Handbook for Analytical Quality Control in Water and Wastewater Laboratories,* EPA, March 1979.
- *Test Methods for Evaluating Solid Waste Physical/Chemical Methods (SW846), Third Edition, September 1986, Final Update I, July 1992, Final Update IIA, August 1993, Final Update II, September 1994; Final Update IIB, January 1995; Final Update III, December 1996; Final Update IV, January 2008.*
- 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.
- Toxic Substances Control Act (TSCA).

3.2 Terms and Definitions

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

constructive, documented problem solving, and provide a framework for continuous improvement within the organization.

Refer to Appendix 2 for the Glossary/Acronyms.

3.3 Scope / Fields of Testing

The laboratory analyzes a broad range of environmental and industrial samples every month. Sample matrices vary among drinking water, effluent water, groundwater, hazardous waste, sludge and soils. The Quality Assurance Program contains specific procedures and methods to test samples of differing matrices for chemical, physical and biological parameters. The Program also contains guidelines on maintaining documentation of analytical processes, reviewing results, servicing clients and tracking samples through the laboratory. The technical and service requirements of all analytical requests are thoroughly evaluated before commitments are made to accept the work. Measurements are made using published reference methods or methods developed and validated by the laboratory.

The methods covered by this manual include the most frequently requested methodologies needed to provide analytical services in the United States and its territories. The specific list of test methods used by the laboratory can be found in 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.

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., Chief Executive Officer (CEO), 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 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 the

VP of Operations (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.
- 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.

4.2.3 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.
- 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.

4.2.4 Quality Assurance (QA) Specialist

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

- Assist QA Manager in conducting QA training courses, including ethics training.
- Performs data audits.
- Assist in performing special audits as deemed necessary by data audits, client inquiries, etc.
- Assisting in, conducting and responding to external audits conducted by clients and regulatory agencies.
- Assisting in reviewing and/or writing of Quality Assurance Project Plans, and technical and QC specifications in contracts.
- Maintaining all necessary laboratory certifications.
- Arranging and managing PT samples.
- Reviewing laboratory SOPs. Writing SOPs as needed.
- Maintaining historical indices of all technical records including SOPs, QC records, laboratory data, etc.
- Ensuring maintenance of records archives.
- Assisting in and monitoring laboratory’s method compliance.
- Ensuring maintenance of DOCs for all analysts.
- Ensuring maintenance of training records for all employees.

- Assisting in identification of systematic problems within laboratories.
- Recommends resolutions for ongoing or recurring nonconformance.
- Providing statistical feedback to Departments on error rates, and assisting in identifying systematic improvements to minimize errors.
- Assists in tracking of customer complaints, providing statistical feedback to the laboratory, and assisting in identifying improvements.
- Overseeing and reviewing MDL studies.
- Ensuring control charts are generated; oversees and approves setting of control limits.
- Assists in monitoring new regulations and communicating them to the laboratory.

4.2.5 LAN Analyst

The LAN Analyst reports directly to the Regional Desktop Support Supervisor. Responsibilities include:

- Works with Corporate IT to solve information systems problems and to standardize laboratory IT equipment and processes.
- Monitors and supports office automation so that LAN is operational for internal and external communications.
- Troubleshoots problems throughout laboratory relating to computers, software, telephones and other electronic equipment.
- Responsible for new user setup on network, LIMS, telephone and voice mail.
- Installs or upgrades computers and other equipment.
- Maintains tape backups for multiple computer servers including LIMS.
- Maintains historical files of software, software operating procedures (manuals), software changes/modifications (Change Log) and software version numbers.
- Maintains log of repairs and service performed on LIMS hardware.
- Maintains awareness of any environmental conditions of the facility housing the LIMS that may compromise LIMS raw data and informs management.

4.2.6 Operations Manager

The Operations Manager manages and directs the analytical and reports production sections of the laboratory. He/She reports directly to the Laboratory Director. Specific responsibilities include:

- Maintains awareness of any environmental conditions of the facility housing the LIMS that may compromise LIMS raw data and informs management.
- Continuously evaluates production capacity and improves capacity utilization.
- Continuously evaluates turnaround time and addresses any problems that may hinder meeting the required and committed turnaround time from the various Departments.

- Develops and improves the training of all analysts in cooperation with the Laboratory Director and QA Manager and in compliance with regulatory requirements.
- Works with the Department (Technical) Managers to ensure that scheduled instrument maintenance is completed.
- Is responsible for efficient utilization of supplies.
- Constantly monitors and modifies the processing of samples through the Departments.
- Fully supports the quality system and, if called upon in the absence of the QA Manager, serves as his substitute in the interim.

4.2.7 Environmental, Health and Safety Manager

The Environmental, Health and Safety Manager reports directly to the Laboratory Director. The duties of this position consist of:

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

- Contacting the hazardous waste subcontractors for review of procedures and opportunities for minimization of waste.

4.2.8 EH&S/Facilities Coordinator

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

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

4.2.9 Technical Managers (Department Managers)

The Technical Managers (Department Managers) report directly to the Operations Manager. They are 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 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.
- Ensures that 100% of data review undergoes two documented levels of review. Likewise ensures that all non-conformance issues are properly documented.
- 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.
- Responsible for the timely and accurate completion of performance evaluation samples and MDLs, for the Department.
- Ensure all logbooks are maintained, current, and properly labeled or archived.
- Report all non-conformance conditions to the QA Manager, Operations Manager, and/or Laboratory Director.
- Ensure that preventive maintenance is performed on instrumentation as detailed in the QA Manual or SOPs. He is responsible for developing and implementing a system for preventive maintenance, troubleshooting, and repairing or arranging for repair of instruments.
- Maintain adequate and valid inventory of reagents, standards, spare parts, and other relevant resources required to perform daily analysis.
- Achieve optimum turnaround time on analyses and compliance with holding times.
- Provide written responses to external and internal audit issues and coordinates audit responses with the QA Manager.

4.2.10 Laboratory Analysts and Technicians

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

- Perform analyses by adhering to analytical and quality control protocols prescribed by current SOPs, this QA Manual, and project-specific plans honestly, accurately, timely, safely, and in the most cost-effective manner.
- Document standard and sample preparation, instrument calibration and maintenance, data calculations, sample matrix effects, and any observed non-conformance on worklists, benchsheets, lab notebooks and/or the Non-Conformance Database by means of Non-Conformance Memos (NCMs).
- Report all non-conformance situations, instrument problems, matrix problems and QC failures, which might affect the reliability of the data, to their Department (Technical) Manager, the Laboratory Director, and/or the QA Manager or member of QA staff.
- Perform 100% review of the data generated and document the review in the raw data and on the review checklist prior to entering and submitting for secondary level review.
- Suggest method improvements to the Department (Technical) Manager, the Laboratory Director, and the QA Manager. These improvements, if approved, will be incorporated within the constraints of the consensus reference methods.
- Work cohesively as a team in their Department to achieve the goals of accurate results, optimum turnaround time, cost effectiveness, cleanliness, complete documentation, and personal knowledge of environmental analysis.
- Adhere to all environmental, health and safety protocols and attend safety meetings as required.

- Attend and participate in all staff meetings.

4.2.11 Sample Control Manager

The Sample Control Manager reports to the Laboratory Director. The responsibilities are outlined below:

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

4.2.12 Client Services Manager

The Customer Service Manager reports to the Laboratory Director and serves as the primary interface between the laboratory and the Sales and Marketing staff. Responsibilities include:

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

4.2.13 Director of Project Management

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

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

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

4.2.14 Project Manager

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

- Ensure client specifications are met by communicating project and quality assurance requirements to the laboratory.
- Notify laboratory personnel of incoming projects and sample delivery schedules.
- Monitor the status of all projects in-house to ensure timely delivery of reports.
- Inform clients of project-related problems, resolving service issues and coordinating technical issues with the laboratory staff.
- Accountable for response to client inquiries concerning sample status.
- Responsible for assistance to clients regarding the resolution of problems concerning COC.
- Ensuring that client specifications, when known, are met by communicating project and quality assurance requirements to the laboratory.
- Notifying the supervisors of incoming projects and sample delivery schedules.
- Coordinate client requests for sample containers and other services.
- Schedule sample pick-ups from client offices or project sites and notifying the laboratory staff of incoming samples.
- Coordinate subcontract work.

- Respond to client inquiries concerning sample status.
- 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.

4.2.15 Project Management Assistant

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

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

4.2.16 Service Center Manager

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

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

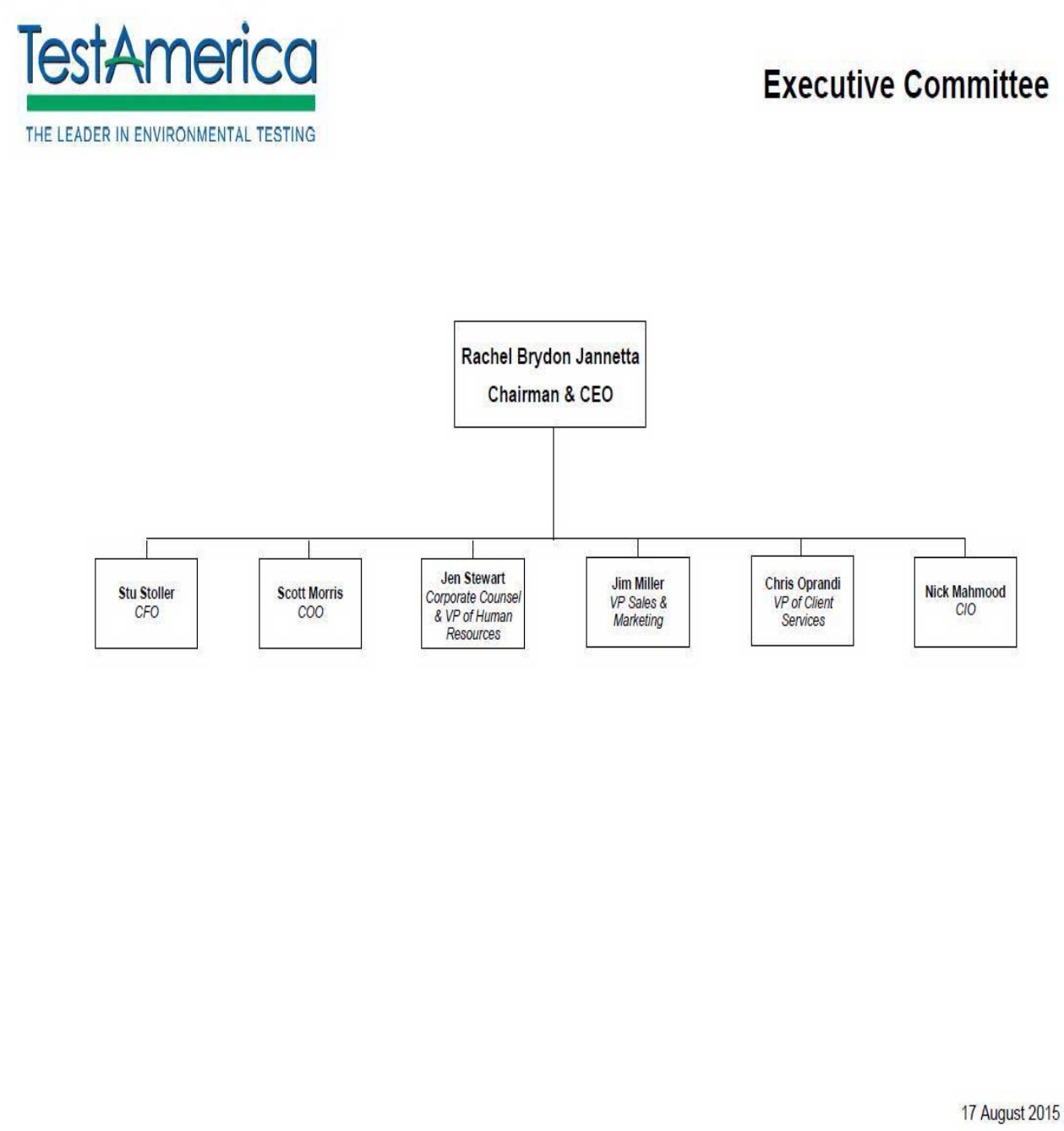
- Prepares proposals for new business opportunities.
- Orders supplies (bottles, coolers, etc.) for the service center

4.3 Deputies

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

Key Personnel	Deputy
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
Department (Technical) Managers	Donald Evans Laboratory Operations Manager
Mark Nemec Client Services Manager	Kenvwyn WIlliams Manager of Project Management
Kenwyn Williams Manager of Project Management	Kristyn Tempe Project Manager
Dan Helfrich EH&S Manager	Edward Roche EH&S Coordinator
Brian Bordieri Sample Control Manager	Donald Evans Laboratory Operations Manager
Tim Knollmeyer Kate Harrelson Service Center Managers	Bernard Sonnie Monica Verdi Field Services Supervisor

Figure 4-1. Corporate and Laboratory Organization Charts



TestAmerica
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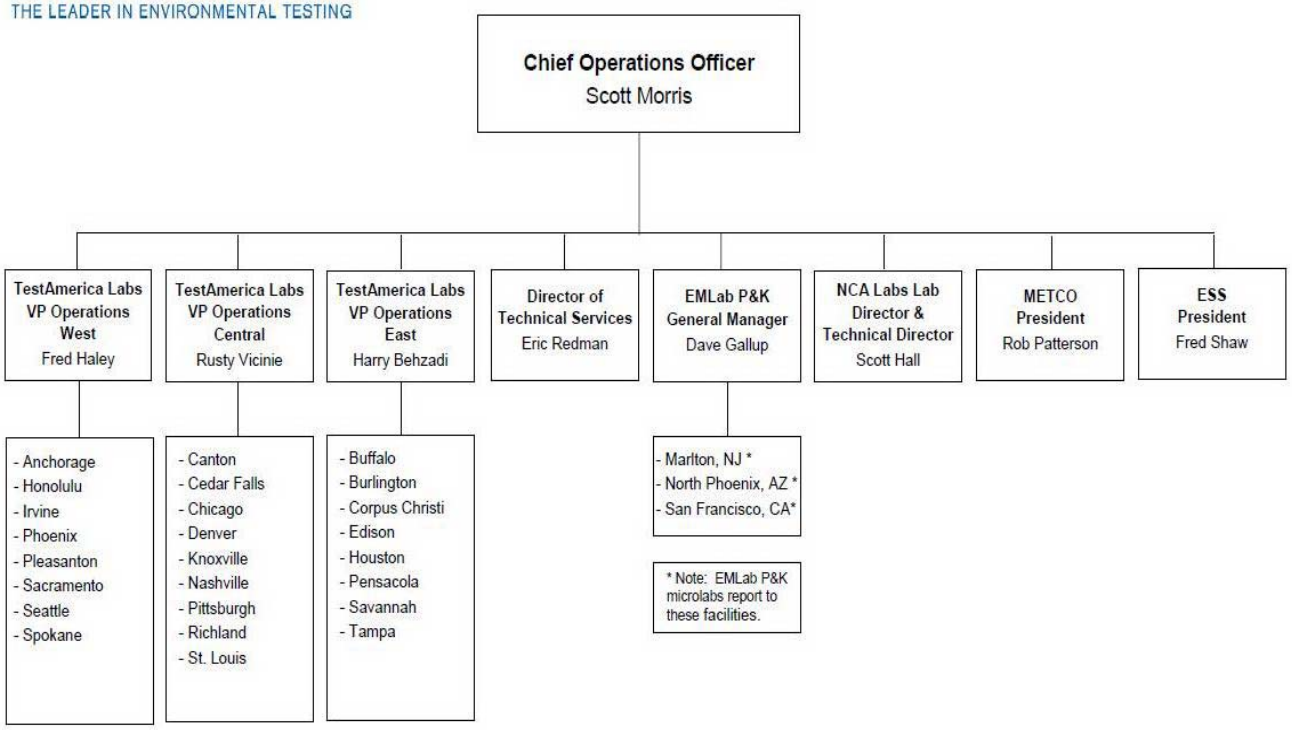
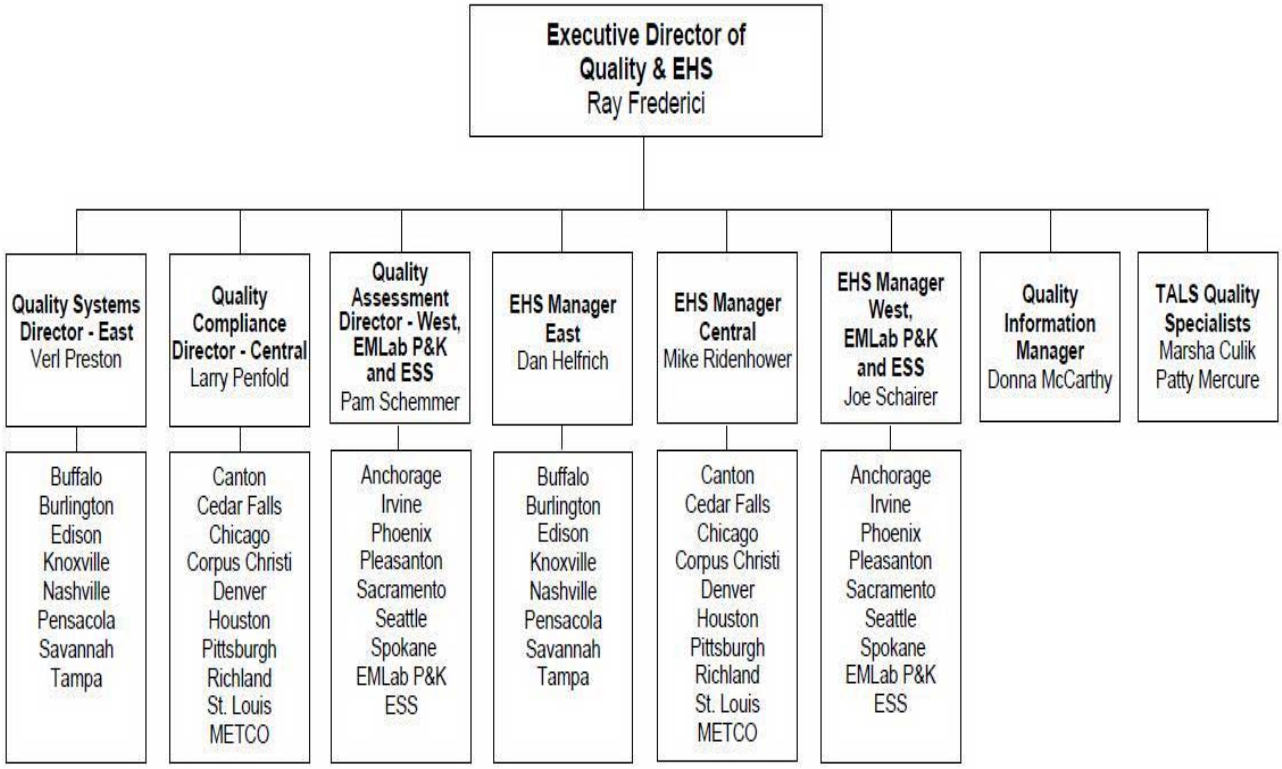
Operations



10 August 2015

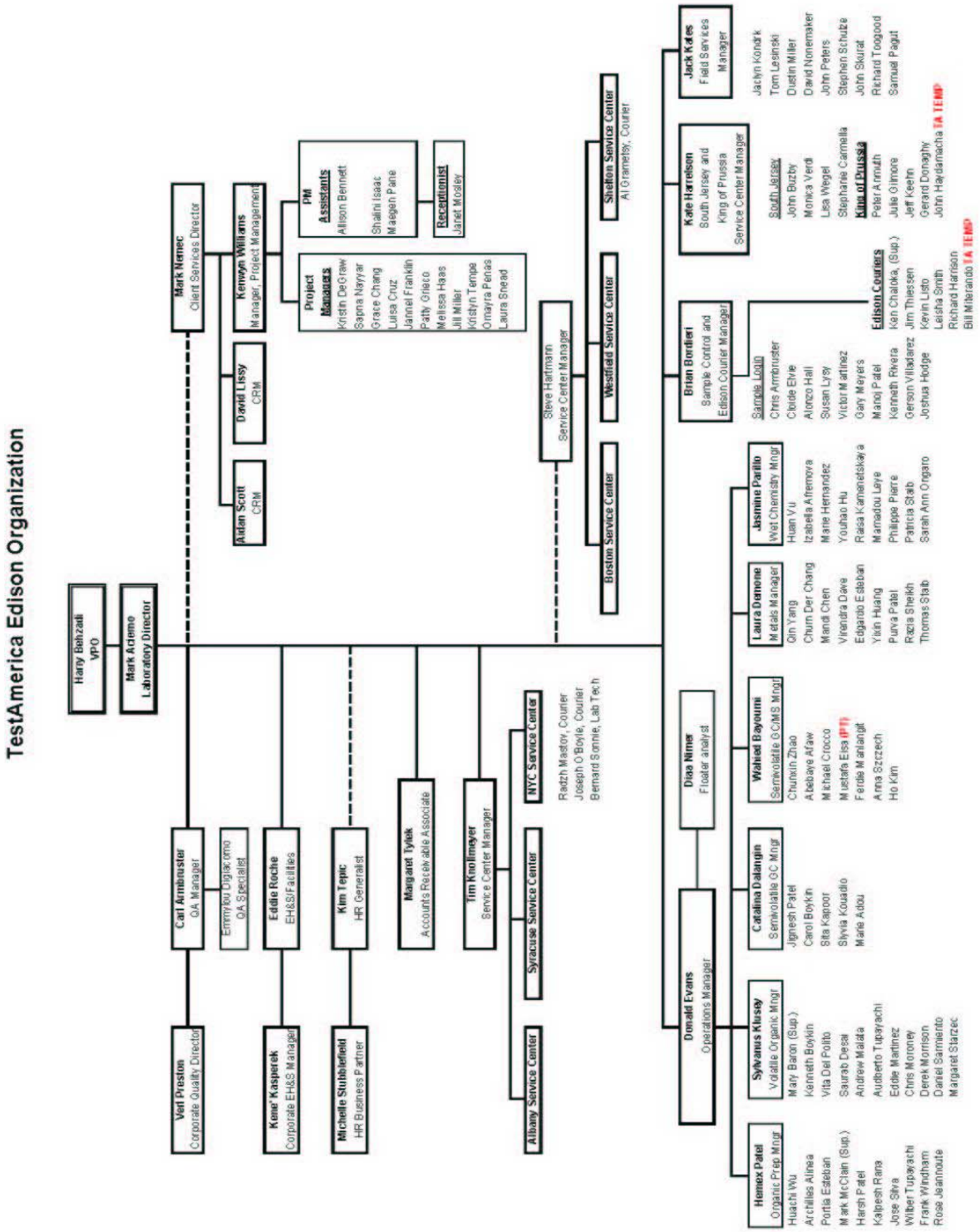
TestAmerica
THE LEADER IN ENVIRONMENTAL TESTING

Quality & EHS



Note: QA Managers and EH&S Coordinators have direct reporting relationship to their corporate manager and a strong dotted line reporting relationship to their Lab Director

6 July 2015


TestAmerica Edison Organization
Harry Behzadi
VP/O
Mark Arienne
Laboratory Director
Mark Nemec
Client Services Director
Kenwyn Williams
Manager, Project Management
PM Assistants
Allison Bennett
Shalini Isaac
Maegen Pane
Receptionist
Janet Mosley
Project Managers
Kristin DeGraw
Sapna Nayyar
Grace Chang
Luisa Cruz
Jannel Franklin
Patty Grieco
Melissa Haas
Jill Miller
Kristyn Tempe
Omayra Perias
Laura Snead
David Lissy
CRM
Aidan Scott
CRM
Steve Hartmann
Service Center Manager
Boston Service Center
Westfield Service Center
Shelton Service Center
Al Gramelsy, Courier
Carl Armbruster
QA Manager
Emmylou Digiacomo
QA Specialist
Eddie Roche
EH&S/Facilities
Kim Tepic
HR Generalist
Margaret Tylek
Accounts Receivable Associate
Tim Knollmeyer
Service Center Manager
NYC Service Center
Syracuse Service Center
Albany Service Center
Radzh Mastov, Courier
Joseph O'Boyle, Courier
Bernard Sonnie, Lab Tech
Veri Preston
Corporate Quality Director
Kena' Kasperek
Corporate EH&S Manager
Michelle Stubblefield
HR Business Partner
Donald Evans
Operations Manager
Diaa Nimer
Floater analyst
Brian Bordieri
Sample Control and Edison Courier Manager
Kate Harrelson
South Jersey and King of Prussia Service Center Manager
Jack Kates
Field Services Manager
Homex Patel
Organic Prep Mngr
Huachi Wu
Archilles Alinea
Portia Esteban
Mark McClain (Sup.)
Harish Patel
Kalpesh Rana
Jose Silva
Wilber Tupayachi
Frank Windham
Rose Jeannoute
Sylvanus Klesay
Volatile Organic Mngr
Mary Baron (Sup.)
Kenneth Boykin
Vita Del Polito
Saurab Desai
Andrew Malata
Audberto Tupayachi
Eddie Martinez
Chris Moroney
Derek Morrison
Daniel Sarmiento
Margaret Starzec
Catalina Dalangin
Semivolatile GC Mngr
Jignesh Patel
Carol Boykin
Sita Kapoor
Silvia Kouadio
Marie Abbu
Waheid Bayoumi
Semivolatile GC/MS Mngr
Chunxin Zhao
Abebaye Afaw
Michael Crocco
Mustafa Elsa (PT)
Feridie Marlangit
Anna Szczech
Ho Kim
Laura Damone
Metals Manager
Qin Yang
Churn Der Chang
Mandi Chen
Virendra Dave
Edgardo Esteban
Yikin Huang
Purva Patel
Razia Sheikh
Thomas Stalb
Jasmine Partlo
Wet Chemistry Mngr
Huan Vu
Izabella Artemova
Marie Hernandez
Youhao Hu
Raisa Kamenetskaya
Mamadou Leye
Philippe Pierre
Patricia Stalb
Sarah Ann Ongaro
Sample Login
Chris Armbruster
Cloide Elvir
Alonzo Hall
Susan Lysy
Victor Martinez
Gary Meyers
Manoj Patel
Kenneth Rivera
Gerson Villadarez
Kevin Listo
Joshua Hodge
Edison Couriers
Ken Chalotka, (Sup.)
Jim Thiessen
Laisha Smith
Richard Harrison
Bill Miserando (TA TEMP)
South Jersey
John Buzby
Monica Verdi
Lisa Wegel
Stephanie Carmella
King of Prussia
Peter Arnmuth
Julie Gilmore
Jeff Keehn
Gerard Donaghy
John Haydamacha (TA TEMP)
Jaclyn Kondrk
Tom Lesinski
Dustin Miller
David Nonemaker
John Peters
Stephen Schulze
John Skurat
Richard Toogood
Samuel Paput

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. CW-L-S-002).
- Effective external and internal monitoring system that includes procedures for internal audits (Section 15).
- Produce results, which are accurate and include QA/QC information that meets client pre-defined Data Quality Objectives (DQOs).
- Present services in a confidential, honest and forthright manner.

- Provide employees with guidelines and an understanding of the Ethical and Quality Standards of our Industry.
- Operate our facilities in a manner that protects the environment and the health and safety of employees and the public.
- Obey all pertinent federal, state and local laws and regulations and encourage other members of our industry to do the same.
- Educate clients as to the extent and kinds of services available.
- Assert competency only for work for which adequate personnel and equipment are available and for which adequate preparation has been made.
- Promote the status of environmental laboratories, their employees, and the value of services rendered by them.

5.3 Quality System Documentation

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

- Quality Assurance Manual – Each laboratory has a lab-specific quality assurance manual.
- Corporate SOPs and Policies – Corporate SOPs and Policies are developed for use by all relevant laboratories. They are incorporated into the laboratory's normal SOP distribution, training and tracking system. Corporate SOPs may be general or technical.
- Work Instructions – A subset of procedural steps, tasks or forms associated with an operation of a management system (e.g., checklists, preformatted bench sheets, forms).
- Laboratory SOPs – General and Technical
- Laboratory QA/QC Policy Memorandums

5.3.1 Order of Precedence

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

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

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

5.4 QA/QC Objectives for the Measurement of Data

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

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

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

Historically, laboratories have described their QC objectives in terms of precision, accuracy, representativeness, comparability, completeness, selectivity and sensitivity (PARCCSS).

5.4.1 Precision

The laboratory objective for precision is to meet the performance for precision demonstrated for the methods on similar samples and to meet data quality objectives of the EPA and/or other regulatory programs. Precision is defined as the degree of reproducibility of measurements under a given set of analytical conditions (exclusive of field sampling variability). Precision is documented on the basis of replicate analysis, usually duplicate or matrix spike (MS) duplicate samples.

5.4.2 Accuracy

The laboratory objective for accuracy is to meet the performance for accuracy demonstrated for the methods on similar samples and to meet data quality objectives of the EPA and/or other regulatory programs. Accuracy is defined as the degree of bias in a measurement system. Accuracy may be documented through the use of laboratory control samples (LCS) and/or MS. A statement of accuracy is expressed as an interval of acceptance recovery about the mean recovery.

5.4.3 Representativeness

The laboratory objective for representativeness is to provide data which is representative of the sampled medium. Representativeness is defined as the degree to which data represent a characteristic of a population or set of samples and is a measurement of both analytical and field sampling precision. The representativeness of the analytical data is a function of the procedures used in procuring and processing the samples. The representativeness can be documented by the relative percent difference between separately procured, but otherwise identical samples or sample aliquots.

The representativeness of the data from the sampling sites depends on both the sampling procedures and the analytical procedures. The laboratory may provide guidance to the client regarding proper sampling and handling methods in order to assure the integrity of the samples.

5.4.4 Comparability

The comparability objective is to provide analytical data for which the accuracy, precision, representativeness and reporting limit statistics are similar to these quality indicators generated by other laboratories for similar samples, and data generated by the laboratory over time.

The comparability objective is documented by inter-laboratory studies carried out by regulatory agencies or carried out for specific projects or contracts, by comparison of periodically generated statements of accuracy, precision and reporting limits with those of other laboratories.

5.4.5 Completeness

The completeness objective for data is 90% (or as specified by a particular project), expressed as the ratio of the valid data to the total data over the course of the project. Data will be considered valid if they are adequate for their intended use. Data usability will be defined in a QAPP, project scope or regulatory requirement. Data validation is the process for reviewing data to determine its usability and completeness. If the completeness objective is not met, actions will be taken internally and with the data user to improve performance. This may take the form of an audit to evaluate the methodology and procedures as possible sources for the difficulty or may result in a recommendation to use a different method.

5.4.6 Selectivity

Selectivity is defined as: The capability of a test method or instrument to respond to a target substance or constituent in the presence of non-target substances. Target analytes are separated from non-target constituents and subsequently identified/detected through one or more of the following, depending on the analytical method: extractions (separation), digestions (separation), interelement corrections (separation), use of matrix modifiers (separation), specific retention times (separation and identification), confirmations with different columns or detectors (separation and identification), specific wavelengths (identification), specific mass spectra (identification), specific electrodes (separation and identification), etc..

5.4.7 Sensitivity

Sensitivity refers to the amount of analyte necessary to produce a detector response that can be reliably detected (Method Detection Limit) or quantified (Reporting Limit).

5.5 Criteria for Quality Indicators

The laboratory maintains Quality Control Limits within the Method Limit Group tables in TALS (the laboratory's LIMS) that contains 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 certain regulatory 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. 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.

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 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. 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.

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

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

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

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

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

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

All contracts, QAPPs, Sampling and Analysis Plans (SAPs), contract amendments, and documented communications become part of the project record.

The same contract review process used for the initial review is repeated when there are amendments to the original contract by the client, and the participating personnel are informed of the changes.

7.2 Review Sequence and Key Personnel

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

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

For new, complex or large projects, the proposed contract is given to the Client Relations Manager (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.

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.

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.

SECTION 8. SUBCONTRACTING OF TESTS

8.1 Overview

For the purpose of this quality manual, the phrase subcontract laboratory refers to a laboratory external to the TestAmerica laboratories. The phrase "work sharing" refers to internal transfers

of samples between the TestAmerica laboratories. The term outsourcing refers to the act of subcontracting tests.

When contracting with our clients, the laboratory makes commitments regarding the services to be performed and the data quality for the results to be generated. When the need arises to outsource testing for our clients because project scope, changes in laboratory capabilities, capacity or unforeseen circumstances, we must be assured that the subcontractors or work sharing laboratories understand the requirements and will meet the same commitments we have made to the client. Refer to TestAmerica's Corporate SOP's on Subcontracting Procedures (CA-L-S-002).

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 (e.g, certain USACE projects) may require notification prior to placing such work**.**

8.2 Qualifying and Monitoring Subcontracators

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

- The first priority is to attempt to place the work in a qualified TestAmerica laboratory; Firms specified by the client for the task (Documentation that a subcontractor was designated by the client must be maintained with the project file. This documentation can be as simple as placing a copy of an e-mail from the client in the project folder);
- Firms listed as pre-qualified and currently under a subcontract with TestAmerica: A listing of all approved subcontracting laboratories is available on the TestAmerica intranet site. Supporting documentation is maintained by corporate offices and by the TestAmerica laboratory originally requesting approval of the subcontract lab. Verify necessary accreditation, where applicable, (e.g., on the subcontractors TNI, A2LA accreditation or State Certification).

- Firms identified in accordance with the company's Small Business Subcontracting program as small, women-owned, veteran-owned and/or minority-owned businesses;
- TNI or A2LA accredited laboratories.
- In addition, the firm must hold the appropriate certification to perform the work required.

All TestAmerica laboratories are pre-qualified for work sharing provided they hold the appropriate accreditations, can adhere to the project/program requirements, and the client approved sending samples to that laboratory. The client must provide acknowledgement that the samples can be sent to that facility (an e-mail is sufficient documentation or if acknowledgement is verbal, the date, time, and name of person providing acknowledgement must be documented). The originating laboratory is responsible for communicating all technical, quality, and deliverable requirements as well as other contract needs.

When the potential sub-contract laboratory has not been previously approved, Account Executives or PMs may nominate a laboratory as a subcontractor based on need. The decision to nominate a laboratory must be approved by the Laboratory Director. The Laboratory Director requests that the QA Manager begin the process of approving the subcontract laboratory as outlined in Corporate SOP No. CA-L-S-002, Subcontracting Procedures. The client must provide acknowledgement that the samples can be sent to that facility (an e-mail is sufficient documentation or if acknowledgement is verbal, the date, time, and name of person providing acknowledgement must be documented).

8.2.1 Once the appropriate accreditation and legal information is received by the laboratory, it is evaluated for acceptability (where applicable) and forwarded to the Corporate Quality Information Manager (QIM) for review. Once all documents are reviewed for completeness, the Corporate QIM will forward the documents to the Purchasing Manager for formal signature and contracting with the laboratory. The approved vendor will be added to the approved subcontractor list on the intranet site and the finance group is concurrently notified for JD Edwards.

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

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

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

intranet site and e-mailed to all Laboratory Directors, QA Managers and Sales Personnel.

8.3 Oversight and Reporting

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

Prior to sending samples to the subcontracted laboratory, the PM confirms their certification status to determine if it's current and scope-inclusive. The information is documented on a Subcontracted Sample Form (Figure 8-1) and the form is retained in the project folder. For TestAmerica laboratories, certifications can be viewed on the company's TotalAccess Database.

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

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

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

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

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

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

8.4 Contingency Planning

The Laboratory Director may waive the full qualification of a subcontractor process temporarily to meet emergency needs; however, this decision & justification must be documented in the project files, and the 'Purchase Order Terms And Conditions For Subcontracted Laboratory Services' must be sent with the samples and Chain-of-Custody. In the event this provision is utilized, the laboratory (e.g., PM) will be required to verify and document the applicable accreditations of the subcontractor. All other quality and accreditation requirements will still be applicable, but the subcontractor need not have signed a subcontract with TestAmerica at this time. The comprehensive approval process must then be initiated within 30 calendar days of subcontracting.

Figure 8-1.

Example - Subcontracted Sample Form

Date/Time: _____

Subcontracted Laboratory Information:

- Subcontractor's Name: _____
- Subcontractor Point of Contact: _____
- Subcontractor's Address: _____
- Subcontractor's Phone: _____
- Analyte/Method: _____
- Certified for State of Origin: _____
- TNI Certified: Yes_____No_____
- USDA Permit (__Domestic __ Foreign) Yes_____No_____
- A2LA (or ISO 17025) Certified: Yes_____No_____
- CLP-like Required: Yes_____No_____
  (Full doc required)
- Requested Sample Due Date: _____
  (Must be put on COC)

Project Manager: _____

Laboratory Sample # Range: _____
(Only of Subcontracted Samples)

Laboratory Project Number (Billing Control #): _____

All subcontracted samples are to be sent via bonded carrier and Priority Overnight. Please attach tracking number below and maintain these records in the project files.

PM Signature_____**Date**_____

SECTION 9. PURCHASING SERVICES AND SUPPLIES

9.1 Overview

Evaluation and selection of suppliers and vendors is performed, in part, on the basis of the quality of their products, their ability to meet the demand for their products on a continuous and short term basis, the overall quality of their services, their past history, and competitive pricing. This is achieved through evaluation of objective evidence of quality furnished by the supplier, which can include certificates of analysis, recommendations, and proof of historical compliance with similar programs for other clients. To ensure that quality critical consumables and equipment conform to specified requirements, which may affect quality, all purchases from specific vendors are approved by a member of the supervisory or management staff. Capital expenditures are made in accordance with TestAmerica's Corporate Controlled Purchases Procedure, SOP No. CW-F-S-007.

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

9.3.1 Purchasing

Chemical reagents, solvents, glassware, and general supplies are ordered as needed to maintain sufficient quantities on hand. Materials used in the analytical process must be of a known quality. The wide variety of materials and reagents available makes it advisable to specify recommendations for the name, brand, and grade of materials to be used in any determination. This information is contained in the method SOP. The 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 Facilities Coordinator to receive the shipment. It is the responsibility of the analyst who ordered the materials to document the date materials where received. Once the ordered reagents or materials are received, the analyst compares the information on the label or packaging to the original order to ensure that the purchase meets the quality level specified. Safety Data Sheets (SDSs) are available online through the Company's intranet website. Anyone may review these for relevant information on the safe handling and emergency precautions of on-site chemicals.

9.3.3 Specifications

Methods in use in the laboratory specify the grade of reagent that must be used in the procedure. If the quality of the reagent is not specified, analytical reagent grade will be used. It is the responsibility of the analyst to check the procedure carefully for the suitability of grade of reagent.

Chemicals must not be used past the manufacturer's expiration date and must not be used past the expiration time noted in a method SOP. If expiration dates are not provided, the laboratory may contact the manufacturer to determine an expiration date.

The laboratory assumes a five year expiration date on inorganic dry chemicals and solvents unless noted otherwise by the manufacturer or by the reference source method. Chemicals/solvents should not be used past the manufacturer's or SOPs expiration date unless 'verified' (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 megaohm-cm) at 25^{o}C. The specific conductivity is checked and recorded daily. If the water's specific conductivity is greater than the specified limit, the Facility Manager and appropriate Technical Managers must be notified immediately in order to notify all Departments, decide on cessation (based on intended use) of activities, and make arrangements for correction.

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

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

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

Upon receipt of a new or used piece of equipment, an 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.

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 Manager.

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

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

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

The Corporate Purchasing Group will work through the appropriate channels to gather the information required to clearly identify the problem and will contact the vendor to report the problem and to make any necessary arrangements for exchange, return authorization, credit, etc.
As deemed appropriate, the Vendor Performance Reports will be summarized and reviewed to determine corrective action necessary, or service improvements required by vendors

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

9.6.1 New Vendor Procedure

TestAmerica employees who wish to request the addition of a new vendor must complete a J.D. Edwards Vendor Add Request Form.

New vendors are evaluated based upon criteria appropriate to the products or services provided as well as their ability to provide those products and services at a competitive cost. Vendors are also evaluated to determine if there are ethical reasons or potential conflicts of interest with TestAmerica employees that would make it prohibitive to do business with them as well as their financial stability. The QA Department and/or the Technical Services Director are consulted with vendor and product selection that have an impact on quality.

SECTION 10. COMPLAINTS

10.1 Overview

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

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

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

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

The process of complaint resolution and documentation utilizes the procedures outlined in Section 12 (Corrective Actions) and is documented following the procedures in 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.

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

The general steps in the complaint handling process are:

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

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

10.3 Internal Complaints

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

10.4 Management Review

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

SECTION 11. CONTROL OF NON-CONFORMING WORK

11.1 Overview

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

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

Due to the frequently unique nature of environmental samples, sometimes departures from documented policies and procedures are needed. When an analyst encounters such a situation, the problem is presented to the Department (Technical) Manager for resolution. The manager may elect to discuss it with the Lab Director and/or QA Manager or have a representative contact the client to decide on a logical course of action. Once an approach is agreed upon, the analyst documents it using the laboratories corrective action system described in Section 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

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

Under certain circumstances, the Laboratory Director, the Lab Operations Manager, a Department (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, Laboratory Operations Manager, the QA Manager, and the Department (Technical) Managers The reporting of issues involving alleged violations of the company's Data Integrity or Manual Integration procedures must be conveyed to an Ethics and Compliance Officer (ECO), Executive Director of Quality & EHS and the laboratory's Quality Director within 24 hours of discovery.

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

The Laboratory Director, QA Manager, ECOs, Corporate Quality, Executive VP of Operations, VP of Operations and the Quality Directors have the authority and responsibility to halt work, withhold final reports, or suspend an analysis for due cause as well as authorize the resumption of work.

11.3 Evaluation of Significance and Actions Taken

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

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

11.4 Prevention of NonConforming Work

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

11.5 Method Suspension / Restriction (Stop Work Procedures)

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

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

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

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

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

Within 72 hours, the QA Manager will determine if compliance is now met and reports can be released, OR determine the plan of action to bring work into compliance, and release work. A team, with all principals involved (Laboratory Director, Laboratory Operations Manager, 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 Data Inquiry, Client Complaint and Corrective Action Report Form (CAR) (TestAmerica Edison Work Instruction No. EDS-WI-012) (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) – The CAR form is used to document the following types of corrective actions:

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

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

12.3.2 Selection and Implementation of Corrective Actions

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

12.3.3 Root Cause Analysis

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

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

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

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

12.3.4 Monitoring of the Corrective Actions

- The Department (Technical) Manager/Supervisor and QA Manager are responsible to ensure that the corrective action taken was effective.
- Ineffective actions are documented and re-evaluated until acceptable resolution is achieved. Department (Technical) Managers are accountable to the Laboratory Director to ensure final acceptable resolution is achieved and documented appropriately.
- Each CAR is entered into an Excel spreadsheet for tracking purposes and a monthly summary of all corrective actions is printed out for review to aid in ensuring that the corrective actions have taken effect.
- The QA Manager reviews monthly CARs for trends. Highlights are included in the QA monthly report (refer to Section 16). If a significant trend develops that adversely affects quality, an audit of the area is performed and corrective action implemented.

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

12.3.5 Follow-up Audits

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

(Also refer to Section 15.1.4, Special Audits.)

12.4 Technical Corrective Actions

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

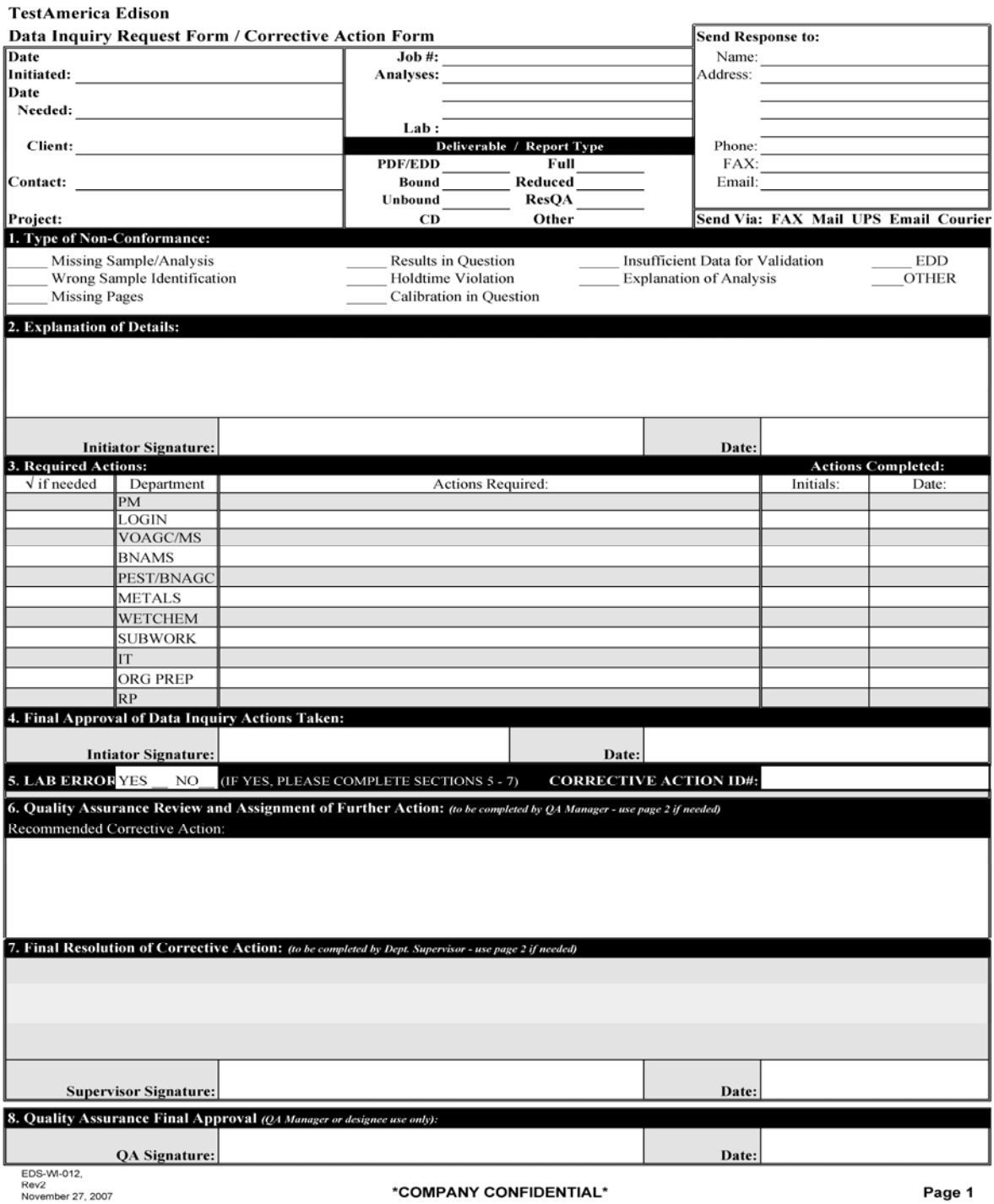
12.5 Basic Corrections

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

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

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

Figure 12-1.
Example - Corrective Action Report



TestAmerica Edison

Data Inquiry Request Form / Corrective Action Form

Date Initiated:	**Job #:**	**Send Response to:** Name:
Date Needed:	**Analyses:**	Address:
Client:	**Lab :**	
Contact:	**Deliverable / Report Type**	Phone:
Project:	**PDF/EDD** ____ **Full** ____	FAX:
	Bound ____ **Reduced** ____	Email:
	Unbound ____ **ResQA** ____	
	CD **Other**	**Send Via: FAX Mail UPS Email Courier**

1. Type of Non-Conformance:

____ Missing Sample/Analysis	____ Results in Question	____ Insufficient Data for Validation	____ EDD
____ Wrong Sample Identification	____ Holdtime Violation	____ Explanation of Analysis	____OTHER
____ Missing Pages	____ Calibration in Question		

2. Explanation of Details:

Initiator Signature: **Date:**

3. Required Actions: **Actions Completed:**

√ if needed	Department	Actions Required:	Initials:	Date:
	PM			
	LOGIN			
	VOAGC/MS			
	BNAMS			
	PEST/BNAGC			
	METALS			
	WETCHEM			
	SUBWORK			
	IT			
	ORG PREP			
	RP			

4. Final Approval of Data Inquiry Actions Taken:

Intiator Signature: **Date:**

5. LAB ERROR YES__ NO__ (IF YES, PLEASE COMPLETE SECTIONS 5 - 7) **CORRECTIVE ACTION ID#:**

6. Quality Assurance Review and Assignment of Further Action: *(to be completed by QA Manager - use page 2 if needed)*

Recommended Corrective Action:

7. Final Resolution of Corrective Action: *(to be completed by Dept. Supervisor - use page 2 if needed)*

Supervisor Signature: **Date:**

8. Quality Assurance Final Approval *(QA Manager or designee use only):*

QA Signature: **Date:**

EDS-WI-012,
Rev2
November 27, 2007

COMPANY CONFIDENTIAL

Page 1

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, Department Technical Manager)*	- 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, Department Technical Manager))*	- % 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 and/or Work Instructions	- If the acceptance criteria for duplicates or matrix spikes are not met because of matrix interferences, the acceptance of the analytical batch is determined by the validity of the LCS. - If the LCS is within acceptable limits the batch is acceptable. - The results of the duplicates, matrix spikes and the LCS are reported with the data set. - For matrix spike or duplicate results outside criteria the data for that sample shall be reported with qualifiers.

QC Activity *(Individual Responsible for Initiation/Assessment)*	Acceptance Criteria	Recommended Corrective Action
Laboratory Control Sample (LCS) *(Analyst, Data Reviewer)*	- % Recovery within limits specified in TALS and/or Work Instructions	- 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, Department Technical Manager)*	- Criteria supplied by PT Supplier.	- Any failures or warnings must be investigated for cause. Failures may result in the need to repeat a PT sample to show the problem is corrected.
Internal / External Audits *(QA Manager, Department Technical Manager)*	- Defined in Quality System documentation such as SOPs, QAM, etc..	- Non-conformances must be investigated through CAR system and necessary corrections must be made.

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, Department Technical Manager, QA Manager, Corporate QA, Corporate Management)	- SOP CW-L-S-002, Internal Investigation of Potential Data Discrepancies and Determination for Data Recall.	- Corrective action is determined by type of error. Follow the procedures in SOP CW-L-S-002 or the Corrective Action SOP (ED-GEN-003).
Client Complaints *(Project Managers, Lab Director Operations 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, Operations Manager, Department Technical Managers)	- QAM, SOPs.	- Corrective action is determined by the type of issue. For example, CARs for the month are reviewed and possible trends are investigated.
Health and Safety Violation (Safety Officer, Lab Director, Operations Manager, Department Technical Manager)	- Environmental Health and Safety (EHS) Manual.	- Non-conformance is investigated and corrected through CAR system.

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

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 during management system reviews, review of the monthly QA Metrics Report, evaluation of internal or external audits, results & evaluation of proficiency testing (PT) performance, review of control charts and QC results, data analysis & review processing operations, client complaints, staff observation, etc.

The monthly Management Systems Metrics Report shows performance indicators in all areas of the laboratory and quality system. These areas include revised reports, corrective actions, audit findings, internal auditing and data authenticity audits, client complaints, PT samples, holding time violations, SOPs, ethics training, etc. The metrics report is reviewed monthly be the laboratory management, Corporate QA and TestAmerica's Executive Committee. These metrics are used in evaluating the management and quality system performance on an ongoing basis and provide a tool for identifying areas for improvement.

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

The laboratory's corrective action process is integral to implementation of preventive actions. A critical piece of the corrective action process is the implementation of actions to prevent further occurrence of a non-compliance event. Historical review of corrective action and non-conformances provides a valuable mechanism for identifying preventive action opportunities.

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

- Identification of an opportunity for preventive action or process improvement.
- Process for the preventive action or improvement.
- Define the measurements of the effectiveness of the process once undertaken.
- Execution of the preventive action or improvement.

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

13.1.2 Any Preventive Actions/Process Improvement undertaken or attempted shall be taken into account during the annual Management Systems Review (Section 16). A highly detailed report is not required; however, a summary of successes and failures within the preventive action program is sufficient to provide management with a measurement for evaluation.

13.2 Management of Change

The Management of Change process is designed to manage significant events and changes that occur within the laboratory. Through these 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.

SECTION 14. CONTROL OF RECORDS

The laboratory maintains a records management system appropriate to its needs and that complies with applicable standards or regulations as required. The system produces unequivocal, accurate records that document all laboratory activities. The laboratory retains all

original observations, calculations and derived data, calibration records and a copy of the analytical report for a minimum of five years after it has been issued.

14.1 Overview

The laboratory has established procedures for identification, collection, indexing, access, filing, storage, maintenance and disposal of quality and technical records. A record index is listed in Table 14-1. Quality records are maintained by the QA Department in a database, which is backed up as part of the regular laboratory backup. Records are of two types; either electronic or hard copy paper formats depending on whether the record is computer or hand generated (some records may be in both formats). Technical records are maintained by Laboratory Operations under the direction of the Laboratory Operations Manager.

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	5 Years from document retirement date*
QA Records	- Internal & External Audits/Responses - Certifications - Corrective/Preventive Actions - Management Reviews - Method & Software Validation / Verification Data - 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)
Project Records	- Sample Receipt & COC Documentation - Contracts and Amendments - Correspondence - QAPP - SAP - Telephone Logbooks - Lab Reports	5 Years from analytical report issue*
Administrative Records	Finance and Accounting	10 years
	EH&S Manual, Permits,	7 years
	Disposal Records	Indefinitely
	Employee Handbook	Indefinitely

	Record Types [1]:	Retention Time:
	Personnel files, Employee Signature & Initials, Administrative Training Records (e.g., Ethics)	Refer to HR Manual
	Administrative Policies Technical Training Records	7 years

[1] Record Types encompass hardcopy and electronic records.
[2] Examples of Logbook types: Maintenance, Instrument Run, Preparation (standard and samples), Standard and Reagent Receipt, Archiving, Balance Calibration, Temperature (hardcopy or electronic records).
* Exceptions listed in Table 14-2.

14.1.1 All records are stored and retained in such a way that they are secure and readily retrievable at the laboratory facility or an offsite location that provides a suitable environment to prevent damage or deterioration and to prevent loss**.** All records shall be protected against fire, theft, loss, environmental deterioration, and vermin. In the case of electronic records, electronic or magnetic sources, storage media are protected from deterioration caused by magnetic fields and/or electronic deterioration.

Access to the data is limited to laboratory and company employees. Records archived off-site are stored in a secure location where a record is maintained of any entry into the storage facility. Whether on-site or off-site storage is used, logs are maintained in each storage box to note removal and return of records. Records are maintained for a minimum of five years unless otherwise specified by a client or regulatory requirement.

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

14.1.2 Programs with Longer Retention Requirements

Some regulatory programs have longer record retention requirements than the standard record retention time. These are detailed in Table 14-2 with their retention requirements. In these cases, the longer retention requirement is enacted. If special instructions exist such that client data cannot be destroyed prior to notification of the client, the container or box containing that data is marked as to who to contact for authorization prior to destroying the data.

Table 14-2. Example: Special Record Retention Requirements

Program	[1]Retention Requirement
Drinking Water – All States	5 years (project records) 10 years - Radiochemistry (project records)
Drinking Water Lead and Copper Rule	12 years (project records)
NY Potable Water NYCRR Part 55-2	10 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 chain of custody is stored in the laboratory's hard copy project file (in addition to the scanned copy included in the analytical report PDF). The chain of custody would indicate the name of the sampler. If any sampling notes are provided with a work order, they are kept in the project file as well. For additional details please refer to refer to TestAmerica Edison SOP No. ED-GEN-024 (Record Storage and Retention).

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

- The record keeping system facilitates the retrieval of all working files and archived records for inspection and verification purposes (e.g., set format for naming electronic files, set format for what is included with a given analytical data set. Reference TestAmerica Edison SOP No. ED-GEN-024 (Record Storage and Retention).

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

14.3 Laboratory Support Activities

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

- all original raw data, whether hard copy or electronic, for calibrations, samples and quality control measures, including analysts' work sheets and data output records (chromatograms, strip charts, and other instrument response readout records)**;**
- a written description or reference to the specific test method used which includes a description of the specific computational steps used to translate parametric observations into a reportable analytical value**;**
- copies of final reports**;**

- archived SOPs**;**
- correspondence relating to laboratory activities for a specific project**;**
- all corrective action reports, audits and audit responses**;**
- proficiency test results and raw data; and
- results of data review, verification, and crosschecking procedures

14.3.1 Sample Handling Records

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

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

14.4 Administrative Records

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

14.5 Records Management, Storage and Disposal

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

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

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

The laboratory has a record management system (a.k.a., document control) for control of laboratory notebooks, instrument logbooks, standards logbooks, and records for data reduction, validation, storage and reporting. Laboratory notebooks are issued on a per analysis basis, and are numbered sequentially. All data are recorded sequentially within a series of sequential notebooks. Bench sheets are filed sequentially. Standards are primarily maintained in the LIMS (this electronic record may be augmented by a logbook record. Records are considered archived when noted as such in the records management system (a.k.a., document control.)

14.5.1 Transfer of Ownership

In the event that the laboratory transfers ownership or goes out of business, the laboratory shall ensure that the records are maintained or transferred according to client's instructions. Upon ownership transfer, record retention requirements shall be addressed in the ownership transfer agreement and the responsibility for maintaining archives is clearly established. In addition, in cases of bankruptcy, appropriate regulatory and state legal requirements concerning laboratory records must be followed. In the event of the closure of the laboratory, all records will revert to the control of the corporate headquarters. Should the entire company cease to exist, as much notice as possible will be given to clients and the accrediting bodies who have worked with the laboratory during the previous 5 years of such action.

14.5.2 Records Disposal

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

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

If a third party records management company is hired to dispose of records, a "Certificate of Destruction" is required.

SECTION 15. AUDITS

15.1 Internal Audits

Internal audits are performed to verify that laboratory operations comply with the requirements of the lab's quality system and with the external quality programs under which the laboratory operates. Audits are planned and organized by the QA staff. Personnel conducting the audits should be independent of the area being evaluated. Auditors will have sufficient authority, access to work areas, and organizational freedom necessary to observe all activities affecting quality and to report the assessments to laboratory management and, when requested, to corporate management.

Audits are conducted and documented as described in the TestAmerica Corporate SOP on performing Internal Auditing, SOP No. CA-Q-S-003. The types and frequency of routine internal audits are described in Table 15-1. Special or ad hoc assessments may be conducted as needed under the direction of the QA staff.

Table 15-1. Types of Internal Audits and Frequency

Description	Performed by	Frequency
Quality Systems Audits	QA Department , QA approved designee, or Corporate QA	All areas of the laboratory annually
QA Technical Audits	Joint responsibility: a) QA Manager or designee b) Technical Manager or Designee (Refer to CA-Q-S-003)	Technical Audits Frequency: 50% of methods annually
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 are based on client projects, associated sample delivery groups, and the methods performed. Reported results are compared to raw data to verify the authenticity of results. The validity of calibrations and QC results are compared to data qualifiers, footnotes, and case narratives. Documentation is assessed by examining run logs and records of manual integrations. Manual calculations are checked. Where possible, electronic audit miner programs (e.g., MintMiner and Chrom AuditMiner) are used to identify unusual manipulations of the data deserving closer scrutiny. QA technical audits will include all methods within a two-year period.

15.1.3 SOP Method Compliance

Compliance of all SOPs with the source methods and compliance of the operational groups with the SOPs will be assessed by the Department Manager (i.e., 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 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.

15.3 Audit Findings

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

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

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

Clients must be notified promptly in writing, of any event such as the identification of defective measuring or test equipment that casts doubt on the validity of results given in any test report or amendment to a test report. The investigation must begin within 24-hours of discovery of the problem and all efforts are made to notify the client within two weeks after the completion of the investigation.

SECTION 16. MANAGEMENT REVIEWS

16.1 Quality Assurance Report

A comprehensive QA Report shall be prepared each month by the laboratory's QA Department and forwarded to the Laboratory Director, their Quality Director as well as the 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 VP of Operationss.

16.2 Annual Management Review

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

This management systems review (Corporate SOP No. CA-Q-S-004 & Work Instruction No. CA-Q-WI-003) uses information generated during the preceding year to assess the “big picture” by ensuring that routine actions taken and reviewed on a monthly basis are not components of larger systematic concerns. The monthly review should keep the quality systems current and effective, therefore, the annual review is a formal senior management process to review specific existing documentation. Significant issues from the following documentation are compiled or summarized by the QA Manager prior to the review meeting:

- Matters arising from the previous annual review.
- Prior Monthly QA Reports issues.
- Laboratory QA Metrics.
- Review of report reissue requests.
- Review of client feedback and complaints.
- Issues arising from any prior management or staff meetings.
- Minutes from prior senior lab management meetings. Issues that may be raised from these meetings include:
    - Adequacy of staff, equipment and facility resources.
    - Adequacy of policies and procedures.
    - Future plans for resources and testing capability and capacity.
- The annual internal double blind PT program sample performance (if performed),
- Compliance to the Ethics Policy and Data Integrity Plan. Including any evidence/incidents of inappropriate actions or vulnerabilities related to data Integrity.

A report is generated by the QA Manager and management. The report is distributed to the appropriate VP of Operations and the Quality Director. The report includes, but is not limited to:

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

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

16.3 Potential Integrity Related Managerial Reviews

Potential integrity issues (data or business related) must be handled and reviewed in a confidential manner until such time as a follow-up evaluation, full investigation, or other appropriate actions have been completed and issues clarified. TestAmerica's Corporate Data Investigation/Recall SOP shall be followed (SOP No. CW-L-S-002) . All investigations that result in finding of inappropriate activity are documented and include any disciplinary actions involved, corrective actions taken, and all appropriate notifications of clients.

TestAmerica's CEO, VP of Quality, Technical & Operations, VP of Operations and Quality Directors receive a monthly report from the Corporate Quality Director summarizing any current data integrity or data recall investigations. The VP of Operations are also made aware of progress on these issues for their specific labs.

SECTION 17. PERSONNEL

17.1 Overview

The laboratory's management believes that its highly qualified and professional staff is the single most important aspect in assuring a high level of data quality and service. The staff consists of professionals and support personnel as outlined in the organization chart in Figure 4-1.

All personnel must demonstrate competence in the areas where they have responsibility. Any staff that is undergoing training shall have appropriate supervision until they have demonstrated their ability to perform their job function on their own. Staff shall be qualified for their tasks based on appropriate education, training, experience and/or demonstrated skills as required.

The laboratory employs sufficient personnel with the necessary education, training, technical knowledge and experience for their assigned responsibilities.

All personnel are responsible for complying with all QA/QC requirements that pertain to the laboratory and their area of responsibility. Each staff member must have a combination of experience and education to adequately demonstrate a specific knowledge of their particular area of responsibility. Technical staff must also have a general knowledge of lab operations, test methods, QA/QC procedures and records management.

Laboratory management is responsible for formulating goals for lab staff with respect to education, training and skills and ensuring that the laboratory has a policy and procedures for identifying training needs and providing training of personnel. The training shall be relevant to the present and anticipated responsibilities of the lab staff.

The laboratory only uses personnel that are employed by or under contract to, the laboratory. Contracted personnel, when used, must meet competency standards of the laboratory and work in accordance to the laboratory's quality system.

17.2 Education and Experience Requirements for Technical Personnel

The laboratory makes every effort to hire analytical staffs that possess a college degree (AA, BA, BS) in an applied science with some chemistry in the curriculum. Exceptions can be made based upon the individual's experience and ability to learn. Selection of qualified candidates for laboratory employment begins with documentation of minimum education, training, and experience prerequisites needed to perform the prescribed task. Minimum education and training requirements for TestAmerica employees are outlined in job descriptions and are generally summarized for analytical staff in the table below.

The laboratory maintains job descriptions for all personnel who manage, perform or verify work affecting the quality of the environmental testing the laboratory performs. Job Descriptions are located on the TestAmerica intranet site's Human Resources web-page (Also see Section 4 for position descriptions/responsibilities).

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

As a general rule for analytical staff:

Specialty	Education	Experience
Extractions, Digestions, some electrode methods (pH, 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

Specialty	Education	Experience
Department Managers (i.e,Technical Managers) - **General**	Bachelors Degree in an applied science or engineering with 24 semester hours in chemistry An advanced (MS, PhD.) degree may substitute for one year of experience	And 2 years experience in environmental analysis of representative analytes for which they will oversee
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

When an analyst does not meet these requirements, they can perform a task under the direct supervision of a qualified analyst, peer reviewer or Department (i.e., 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).

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.

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 and temperature levels in the laboratory (when appropriate).

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

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

18.3 Work Areas

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

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

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

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

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

18.4 Floor Plan

A floor plan can be found in Appendix 1.

18.5 Building Security

Building keys 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.

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 SOPs), and where necessary, revised to ensure continuing suitability and compliance with applicable requirements.

19.3 Laboratory Methods Manual

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

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

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

19.4 Selection of Methods

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

19.4.1 Sources of Methods

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

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

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

- *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*
- *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.*
- *Annual Book of ASTM Standards, American Society for Testing & Materials (ASTM), Philadelphia, PA.*
- *National Status and Trends Program, National Oceanographic and Atmospheric Administration, Volume I-IV, 1985-1994.*
- *Manual for the Certification of Laboratories Analyzing Drinking Water (EPA 815-R-05-004, January 2005)*
- *Code of Federal Regulations (CFR) 40, Parts 136, 141, 172, 173, 178, 179 and 261*

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

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

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

19.4.2 Demonstration of Capability

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

A demonstration of capability (reference TestAmerica Edison Training SOP No. ED-GEN-022) is performed whenever there is a change in instrument type (e.g., new instrumentation), method or personnel (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).
- The laboratory's nominal or default reporting limit (RL) is equal to the quantitation limit (QL), must be at or above the lowest non-zero standard in the calibration curve and must be reliably determined. Project RLs are client specified reporting levels which may be higher than the QL. Results reported below the QL must be qualified as estimated values. Also see Section 19.6.1.3, Relationship of Limit of Detection (LOD) to Quantitation Limit (QL).
- The client request is documented and the lab informs the client of its procedure for working with unusual compounds. The final report must be footnoted: *Reporting Limit based on the low standard of the calibration curve.*

19.4.3 Initial Demonstration of Capability (IDOC) Procedures

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

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

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

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

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

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

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

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

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

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

Methods on line prior to the effective date of this Section shall be updated to the procedures outlined above as new analysts perform their demonstration of capability. A copy of the new record will replace that which was used for documentation in the past. At a minimum, the precision and accuracy of four mid-level laboratory control samples must have been compared to the laboratory's quality control acceptance limits.

19.5 Laboratory Developed Methods and Non-Standard Methods

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

19.6 Validation of Methods

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

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

19.6.1 Method Validation and Verification Activities for All New Methods

While method validation can take various courses, the following activities can be required as part of method validation. Method validation records are designated QC records and are archived accordingly.

19.6.1.1 Determination of Method Selectivity

Method selectivity is the demonstrated ability to discriminate the analyte(s) of interest from other compounds in the specific matrix or matrices from other analytes or interference. In some cases to achieve the required selectivity for an analyte, a confirmation analysis is required as part of the method.

19.6.1.2 Determination of Method Sensitivity

Sensitivity can be both estimated and demonstrated. Whether a study is required to estimate sensitivity depends on the level of method development required when applying a particular measurement system to a specific set of samples. Where estimations and/or demonstrations of sensitivity are required by regulation or client agreement, such as the procedure in 40 CFR Part 136 Appendix B, under the Clean Water Act, these shall be followed.

19.6.1.3 Relationship of Limit of Detection (LOD) to the Quantitation Limit (QL)

An important characteristic of expression of sensitivity is the difference in the LOD and the QL. The LOD is the minimum level at which the presence of an analyte can be reliably concluded. The QL is the minimum concentration of analyte that can be quantitatively determined with acceptable precision and bias. For most instrumental measurement systems, there is a region where semi-quantitative data is generated around the LOD (both above and below the estimated MDL or LOD) and below the QL. In this region, detection of an analyte may be confirmed but quantification of the analyte is unreliable within the accuracy and precision guidelines of the measurement system. When an analyte is detected below the QL, and the presence of the analyte is confirmed by meeting the qualitative identification criteria for the analyte, the analyte can be reliably reported, but the amount of the analyte can only be estimated. If data is to be reported in this region, it must be done so with a qualification that denotes the semi-quantitative nature of the result.

19.6.1.4 Determination of Interferences

A determination that the method is free from interferences in a blank matrix is performed.

19.6.1.5 Determination of Range

Where appropriate to the method, the quantitation range is determined by comparison of the response of an analyte in a curve to established or targeted criteria. Generally the upper

quantitation limit is defined by highest acceptable calibration concentration. The lower quantitation limit or QL cannot be lower than the lowest non-zero calibration level, and can be constrained by required levels of bias and precision.

19.6.1.6 Determination of Accuracy and Precision

Accuracy and precision studies are generally performed using replicate analyses, with a resulting percent recovery and measure of reproducibility (standard deviation, relative standard deviation) calculated and measured against a set of target criteria.

19.6.1.7 Documentation of Method

The method is formally documented in an SOP. If the method is a minor modification of a standard laboratory method that is already documented in an SOP, an SOP Attachment describing the specific differences in the new method is acceptable in place of a separate SOP.

19.6.1.8 Continued Demonstration of Method Performance

Continued demonstration of Method Performance is addressed in the SOP. Continued demonstration of method performance is generally accomplished by batch specific QC samples such as LCS, method blanks or PT samples.

19.7 Method Detection Limits (MDL) / Limits of Detection (LOD)

Method detection limits (MDL) are initially determined in accordance with 40 CFR Part 136, Appendix B or alternatively by other technically acceptable practices that have been accepted by regulators. MDL is also sometimes referred to as Limit of Detection (LOD). The MDL theoretically represents the concentration level for each analyte within a method at which the Analyst is 99% confident that the true value is not zero. The MDL is determined for each analyte initially during the method validation process and updated as required in the analytical methods, whenever there is a significant change in the procedure or equipment, or based on project specific requirements. Generally, the analyst prepares at least seven replicates of solution spiked at one to five times the estimated method detection limit (most often at the lowest standard in the calibration curve) into the applicable matrix with all the analytes of interest. Each of these aliquots is extracted (including any applicable clean-up procedures) and analyzed in the same manner as the samples. Where possible, the seven replicates should be analyzed over 2-4 days to provide a more realistic MDL. [To allow for some flexibility, this low level standard may be analyzed every batch or every week or some other frequency rather than doing the study all at once. In addition, a larger number of data points may be used if the appropriate t-value multiplier is used]

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

19.8 Instrument Detection Limits (IDL)

The IDL is sometimes used to assess the reasonableness of the MDLs or in some cases required by the analytical method or program requirements. IDLs are most used in metals analyses but may be useful in demonstration of instrument performance in other areas.

IDLs are calculated to determine an instrument's sensitivity independent of any preparation method. IDLs are calculated either using 7 replicate spike analyses, like MDL but without sample preparation, or by the analysis of 10 instrument blanks and calculating 3 x the absolute value of the standard deviation.

If IDL is > than the MDL, it may be used as the reported MDL.

19.9 Verification of Detection and Reporting Limits

Once an MDL is established, it must be verified, on each instrument, by analyzing a quality control sample (prepared as a sample) at no more than 3 times the calculated MDL for single analyte analyses (e.g. most wet chemistry methods, Atomic Absorption, etc.) and no more than 4 times the calculated MDL for multiple analyte methods (e.g. GC, GCMS, ICP, etc.). The analytes must be qualitatively identified. This verification does not apply to methods that are not readily spiked (e.g. pH, turbidity, etc.) or where the lab does not report to the MDL. 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.

19.12 Estimation of Uncertainty of Measurement

19.12.1 Uncertainty is "a parameter associated with the result of a measurement, that characterizes the dispersion of the values that could reasonably be attributed to the measurand" (as defined by the International Vocabulary of Basic and General Terms in Metrology, ISO Geneva, 1993, ISBN 92-67-10175-1). Knowledge of the uncertainty of a measurement provides additional confidence in a result's validity. Its value accounts for all the factors which could

possibly affect the result, such as adequacy of analyte definition, sampling, matrix effects and interferences, climatic conditions, variances in weights, volumes, and standards, analytical procedure, and random variation. Some national accreditation organizations require the use of an "expanded uncertainty": the range within which the value of the measurand is believed to lie within at least a 95% confidence level with the coverage factor k=2.

19.12.2 Uncertainty is not error. Error is a single value, the difference between the true result and the measured result. On environmental samples, the true result is never known. The measurement is the sum of the unknown true value and the unknown error. Unknown error is a combination of systematic error, or bias, and random error. Bias varies predictably, constantly, and independently from the number of measurements. Random error is unpredictable, assumed to be Gaussian in distribution, and reducible by increasing the number of measurements.

19.12.3 The minimum uncertainty associated with results generated by the laboratory can be determined by using the Laboratory Control Sample (LCS) accuracy range for a given analyte. The LCS limits are used to assess the performance of the measurement system since they take into consideration all of the laboratory variables associated with a given test over time (except for variability associated with the sampling and the variability due to matrix effects). The percent recovery of the LCS is compared either to the method-required LCS accuracy limits or to the statistical, historical, in-house LCS accuracy limits.

19.12.4 To calculate the uncertainty for the specific result reported, multiply the result by the decimal of the lower end of the LCS range percent value for the lower end of the uncertainty range, and multiply the result by the decimal of the upper end of the LCS range percent value for the upper end of the uncertainty range. These calculated values represent a 99% 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. Note: Client specific Contractual Terms & Conditions for reanalysis protocols may supersede the following items.

- Homogenous samples: If a reanalysis agrees with the original result to within the RPD limits for MS/MSD or Duplicate analyses, or within $\pm$ 1 reporting limit for samples $\leq$ 5x the reporting limit, the original analysis will be reported. At the client's request, both results may be reported on the same report but not on two separate reports.

- If the reanalysis does not agree (as defined above) with the original result, then the laboratory will investigate the discrepancy and reanalyze the sample a third time for confirmation if sufficient sample is available.

- Any potential charges related to reanalysis are discussed in the contract terms and conditions or discussed at the time of the request. The client will typically be charged for reanalysis unless it is determined that the lab was in error.

- Due to the potential for increased variability, reanalysis may not be applicable to Non-homogenous, Encore, and Sodium Bisulfate preserved samples. See the 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, 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.

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 Managers/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 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 Assurance 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 chemical relationships are evaluated, COC is followed, cover letters/ narratives are present, flags are appropriate, and project specific requirements are met.

19.14.4.7 Any project that requires a data package is subject to a tertiary data review for transcription errors and acceptable quality control requirements. The Project Manager then signs the final report. The accounting personnel also check the report for any clerical or invoicing errors. When complete, the report is sent out to the client.

19.14.5 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.

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

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. Manufacturers instructions for equipment use are readily accessible to all appropriate laboratory personnel.

20.2 Preventive Maintenance

The laboratory follows a well-defined maintenance program to ensure proper equipment operation and to prevent the failure of laboratory equipment or instrumentation during use. This program of preventive maintenance helps to avoid delays due to instrument failure.

Routine preventive maintenance procedures and frequency, such as cleaning and replacements, should be performed according to the procedures outlined in the manufacturer's manual. Qualified personnel must also perform maintenance when there is evidence of degradation of peak resolution, a shift in the calibration curve, loss of sensitivity, or failure to continually meet one of the quality control criteria.

Table 20-2 lists examples of scheduled routine maintenance. It is the responsibility of each Technical Manager to ensure that instrument maintenance logs are kept for all equipment in his/her Department. Preventative maintenance procedures may 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.

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 ± 0.1 pH units, and have a scale readability of at least 0.05 pH units. The meters automatically compensate for the temperature, and are calibrated with at least two working range buffer solutions before each use.

Conductivity meters are also calibrated before each use with a known standard to demonstrate the meters do not exceed an error of 1% or one umhos/cm.

Turbidity meters are also calibrated before each use. All of this information is documented in logs.

Consult pH and Conductivity, and Turbidity SOPs for further information.

20.3.3 Thermometers

All thermometers are calibrated on an annual basis with a NIST-traceable thermometer at temperatures bracketing the range of use. IR thermometers, digital probes and thermocouples are calibrated quarterly. IR Thermometers should be calibrated over the full range of use, including ambient, iced (4 degrees C) and frozen (0 to -5 degrees C), per the Drinking Water Manual.

The mercury 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 > 0ºC and ≤ 6 ºC.

Specific temperature settings/ranges for other refrigerators, ovens waterbaths, and incubators can be found in method specific SOPs.

All of this information is documented in Daily Temperature Logbooks and method-specific logbooks.

20.3.5 Autopipettors, Dilutors, and Syringes

Mechanical volumetric dispensing devices including burettes (except Class A Glassware) are given unique identification numbers and the delivery volumes are verified gravimetrically, at a minimum, on a quarterly basis.

For those dispensers that are not used for analytical measurements, a label is / can be applied to the device stating that it is not calibrated. Any device not regularly verified can not be used for any quantitative measurements. Refer to 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 manufacturers 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.

20.4.1 Calibration Standards

Calibration standards are prepared using the procedures indicated in the Reagents and Standards section of the determinative method SOP. If a reference method does not specify the number of calibration standards, a minimum of 3 calibration points (exception being ICP and ICP/MS methods) will be used.

Standards for instrument calibration are obtained from a variety of sources. All standards are traceable to national or international standards of measurement, or to national or international standard reference materials.

The lowest concentration calibration standard that is analyzed during an initial calibration must be at or below the stated reporting limit for the method based on the final volume of extract (or sample).

The other concentrations define the working range of the instrument/method or correspond to the expected range of concentrations found in actual samples that are also within the working range of the instrument/method. Results of samples not bracketed by initial instrument calibration standards (within calibration range to at least the same number of significant figures used to report the data) must be reported as having less certainty, e.g., defined qualifiers or flags (additional information may be included in the case narrative). The exception to these rules is ICP methods or other methods where the referenced method does not specify two or more standards.

All initial calibrations are verified with a standard obtained from a second source and traceable to a national standard, when available (or vendor certified different lot if a second source is not

available). For unique situations, such as air analysis where no other source or lot is available, a standard made by a different analyst at a different time or a different preparation would be considered a second source. This verification occurs immediately after the calibration curve has been analyzed, and before the analysis of any samples.

20.4.1.1 Calibration Verification

The calibration relationship established during the initial calibration must be verified initially and at least daily as specified in the laboratory method SOPs in accordance with the referenced analytical methods and in the 2009 TNI Standard. The process of calibration verification applies to both external standard and internal standard calibration techniques, as well as to linear and non-linear calibration models. Initial calibration verification is with a standard source secondary (second source standard) to the calibration standards, but continuing calibration verifications may use the same source standards as the calibration curve.

Note: The process of calibration verification referred to here is fundamentally different from the approach called "calibration" in some methods. As described in those methods, the calibration factors or response factors calculated during calibration are used to update the calibration factors or response factors used for sample quantitation. This approach, while employed in other EPA programs, amounts to a daily single-point calibration.

All target analytes and surrogates, including those reported as non-detects, must be included in periodic calibration verifications for purposes of retention time confirmation and to demonstrate that calibration verification criteria are being met, i.e., RPD, per 2009 TNI Std. EL-V1M4 Sec. 1.7.2.

All samples must be bracketed by periodic analyses of standards that meet the QC acceptance criteria (e.g., calibration and retention time). The frequency is found in the determinative methods or SOPs.

Note: If an internal standard calibration is being used (basically GCMS) then bracketing standards are not required, only daily verifications are needed. The results from these verification standards must meet the calibration verification criteria and the retention time criteria (if applicable).

Generally, the initial calibrations must be verified at the beginning of each 12-hour analytical shift during which samples are analyzed. (Some methods may specify more or less frequent verifications). The 12-hour analytical shift begins with the injection of the calibration verification standard (or the MS tuning standard in MS methods). The shift ends after the completion of the analysis of the last sample, QC, or standard that can be injected within 12 hours of the beginning of the shift.

A continuing instrument calibration verification (CCV) must be repeated at the beginning and, for methods that have quantitation by external calibration models, at the end of each analytical batch. Some methods have more frequent CCV requirements see specific SOPs. Most Inorganic methods require the CCV to be analyzed after ever 10 samples or injections, including matrix or batch QC samples.

Note: If an internal standard calibration is being used (basically GCMS) then bracketing standards are not required, only daily verifications are needed. The results from these verification standards must meet the calibration verification criteria and the retention time criteria (if applicable).

If the results of a CCV are outside the established acceptance criteria and analysis of a second consecutive (and immediate) CCV fails to produce results within acceptance criteria, corrective action shall be performed. Once corrective actions have been completed & documented, the laboratory shall demonstrate acceptable instrument / method performance by analyzing two consecutive CCVs, or a new initial instrument calibration shall be performed.

Sample analyses and reporting of data may not occur or continue until the analytical system is calibrated or calibration verified. However, data associated with an unacceptable calibration verification may be fully useable under the following special conditions:

a). when the acceptance criteria for the CCV are exceeded high (i.e., high bias) and the associated samples within the batch are non-detects, then those non-detects may be reported with a footnote or case narrative explaining the high bias. Otherwise the samples affected by the unacceptable CCV shall be re-analyzed after a new calibration curve has been established, evaluated and accepted; or

b). when the acceptance criteria for the CCV are exceeded low (i.e., low bias), those sample results may be reported if they exceed a maximum regulatory limit/decision level. Otherwise the samples affected by the unacceptable CCV shall be re-analyzed after a new calibration curve has been established, evaluated and accepted.

Samples reported by the 2 conditions identified above will be appropriately flagged.

20.4.1.2 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 acepted.

- When the acceptance criteria for the calibration verification are exceeded low, i.e., low bias, those sample results may be reported if they exceed a maximum regulatory limit/decision level. Otherwise, the samples affected by the unacceptable verification shall be reanalyzed after a new calibration curve has been established, evaluated and accepted. Alternatively, a reporting limit standard may be analyzed to demonstrate that the laboratory can still support non-detects at their reporting limit.

20.5 Tentatively Identified Compounds (TICs) – GC/MS Analysis

For samples containing components not associated with the calibration standards, a library search may be made for the purpose of tentative identification. The necessity to perform this type of identification will be determined by the purpose of the analyses being conducted. Data system library search routines should not use normalization routines that would misrepresent the library or unknown spectra when compared to each other.

Note: If the TIC compound is not part of the client target analyte list but is calibrated by the laboratory and is both qualitatively and/or quantitatively identifiable, it should not be reported as a TIC. If the compound is reported on the same form as true TICs, it should be qualified and/or narrated that the reported compound is qualitatively and quantitatively (if verification in control) reported compared to a known standard that is in control (where applicable).

For example, the RCRA permit or waste delisting requirements may require the reporting of non-target analytes. Only after visual comparison of sample spectra with the nearest library searches may the analyst assign a tentative identification.

20.6 GC/MS Tuning

Prior to any GCMS analytical sequence, including calibration, the instrument parameters for the tune and subsequent sample analyses within that sequence must be set.

Prior to tuning/auto-tuning the mass spec, the parameters may be adjusted within the specifications set by the manufacturer or the analytical method. These generally don't need any adjustment but it may be required based on the current instrument performance. If the tune verification does not pass it may be necessary to clean the source or perform additional maintenance. Any maintenance is documented in the maintenance log.

Table 20-1. Example: Instrumentation List

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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
ICP/MS (ICPMS2)	Agilent Technologies	7500CX	JP82802644	2010	New
Heat Exchanger	Agilent Technologies	3370	G57335	2006	Used
Autosampler	Agilent Technologies	ASX520	120536A520	2006	New
Autosampler	Thermo	SC-DX Fast	278-093100	2010	New
Mercury Analyzer	Leeman Labs	Hydra AA	HA-4012112-00101-1	2004	New
Mercury Analyzer	Leeman Labs	Hydra AA	HA-8016	2004	New
Mercury Analyzer	Leeman Labs	Hydra AA	HA-2008112-00064-1	2013	New
Hot Block (Hot Block 1)	Environmental Express Limited	SC-154	2772CE1378	2003	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 6)	Environmental Express Limited	SC-150	466CEC2183	2006	New
Hot Block (Hot Block 7)	Environmental Express Limited	SC-150	2772CDC1378	2006	New
Top Loading Balance (Balance #35)	Acculab	VIC-412	18255989	2005	New
Top Loading Balance (Balance #33)	Ohaus	AR5120	F0461200521139	2001	New
Semivoloatile GC/MS Department					
GC/MS System (BNAMS4)					
Gas Chromatograph	Hewlett Packard	5890 Series II	3108A34490	1986	New

Revised 09/15/2015

Page 1 of 18

TestAmerica
THE LEADER IN ENVIRONMENTAL TESTING

Instrument Type (Name)	Manufacturer	Model Number	Serial Number	Year Put into Service	Condition When Received
MS Detector	Hewlett Packard	5971A	3114A02077	1986	New
Autosampler Tower	Hewlett Packard	7673A	2546A02861	1986	New
Autosampler Tray	Hewlett Packard		2942A20598	1986	New
Controller	Hewlett Packard		2803A11211	1986	New
GC/MS System (BNAMS5)					
Gas Chromatograph	Agilent Technologies		CN10726100	2005	New
MS Detector	Agilent Technologies	5975C	US35120328	2005	New
Autosampler Tower	Agilent Technologies	7890A	CN72441261	2005	New
Autosampler Tray	Agilent Technologies		CN40427800	2005	New
Controller	Agilent Technologies		CN40427800	2005	New
GC/MS System (BNAMS6)					
Gas Chromatograph	Hewlett Packard		3336A54722	1990	New
MS Detector	Hewlett Packard	5971	3234A04274	1990	New
Autosampler Tower	Hewlett Packard	7673	2843A13155	1990	New
Autosampler Tray	Hewlett Packard		2933A11253	1990	New
Controller	Hewlett Packard		3018A21811	1990	New
GC/MS System (BNAMS9)					
Gas Chromatograph	Agilent Technologies		CN10349071	2004	New
MS Detector	Agilent Technologies	5973	US35120328	2004	New
Autosampler Tower	Agilent Technologies	7683	CN35134357	2004	New
Autosampler Tray	Agilent Technologies		CN40427800	2004	New
Controller	Agilent Technologies		CN40427800	2004	New
GC/MS System (BNAMS10)					
Gas Chromatograph	Agilent Technologies		CN10403063	2004	New
MS Detector	Agilent Technologies	5973	US35120373	2004	New

Revised 09/15/2015

TestAmerica
THE LEADER IN ENVIRONMENTAL TESTING

Instrument Type (Name)	Manufacturer	Model Number	Serial Number	Year Put into Service	Condition When Received
Autosampler Tower	Agilent Technologies	7683	CN40334758	2004	New
Autosampler Tray	Agilent Technologies		CN40327770	2004	New
Controller	Agilent Technologies		CN40327770	2004	New
GC/MS System (BNAMS11)					
Gas Chromatograph	Agilent Technologies		CN10727109	2007	New
MS Detector	Agilent Technologies	5975C	US71236621	2007	New
Autosampler Tower	Agilent Technologies	7890A	CN35134357	2007	New
Autosampler Tray	Agilent Technologies		CN72441255	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		CN61732705	2012	New
Autosampler Tray	Agilent Technologies		CN50932320	2012	New
Controller	Agilent Technologies				
GC/MS System (BNAMS13)					
Gas Chromatograph	Agilent Technologies	6890A	CN10529024	2012	New
MS Detector	Agilent Technologies	5975C	US52430481	2012	New
Autosampler Tower	Agilent Technologies		CN53427241	2012	New
Autosampler Tray	Agilent Technologies		CN1739524	2012	New
Controller	Agilent Technologies			2012	New
GC/MS System (BNAMS14)					
Gas Chromatograph	Agilent Technologies	6890A	CN10402079	2012	New
MS Detector	Agilent Technologies	5973	US35110172	2012	New
Autosampler Tower	Agilent Technologies		CN34433497	2012	New

Revised 09/15/2015

Page 3 of 18

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		CN40327583	2012	New
Controller	Agilent Technologies			2012	New
GC/MS System (BNAMS15)	Agilent Technologies				
Gas Chromatograph	Agilent Technologies	7890B	CN14393167	2015	New
MS Detector	Agilent Technologies	5977A	US1433L427	2015	New
Autosampler Tower	Agilent Technologies	G4513A	CN14410091	2015	New
Autosampler Tray	Agilent Technologies	7963	CN14360040	2015	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
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	Agilent Technologies	4551A	D60345B194	2006	New
Concentrator	Agilent Technologies	4660	D608466853	2006	New
Spiker	Agilent Technologies	SAM	E610475713	2006	New
GC/MS System (VOAMS2)					
Gas Chromatograph	Hewlett-Packard	7890A	CN10813013	2008	New
MS Detector	Hewlett-Packard	5975C	US80838709	2008	New
Autosampler	EST	Archon 51	15264	2008	New
Concentrator	EST	Encon Evolution	104041408	2008	New
GC/MS System (VOAMS3)					

TestAmerica
THE LEADER IN ENVIRONMENTAL TESTING

Instrument Type (Name)	Manufacturer	Model Number	Serial Number	Year Put into Service	Condition When Received
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	367060704	2004	New
Concentrator	EST	Encon	368060704	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	15266	2008	New
Concentrator	OI	2008	D809466076	2008	New
GC/MS System (VOAMS5)					
Gas Chromatograph	Hewlett-Packard	5890 II	3033A33368	1996	New
MS Detector	Hewlett-Packard	5971	3234A04198	1996	New
Autosampler	Archon	5100	11957-696A	1996	New
Concentrator	OI	4560	D310219	1996	New
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	5890 II	3126A36935	1998	New
MS Detector	Hewlett-Packard	5971	3118A02630	1998	New
Autosampler	EST Archon	5100A	12206	1998	New
Concentrator	OI	4560	I418460464	1998	New

Revised 09/15/2015

TestAmerica
THE LEADER IN ENVIRONMENTAL TESTING

Instrument Type (Name)	Manufacturer	Model Number	Serial Number	Year Put into Service	Condition When Received
GC/MS System (VOAMS9)					
Gas Chromatograph	Hewlett-Packard	5890 II	3203A40292	1998	New
MS Detector	Hewlett-Packard	5971	3118A03332	1998	New
Autosampler	EST Archon	5100A	12207	1998	New
Concentrator	OI	4560	C302089	1998	New
GC/MS System (VOAMS10)					
Gas Chromatograph	Hewlett-Packard	5890	2728414257	1997	New
MS Detector	Hewlett-Packard	5972	3307A00392	2000	New
Autosampler	Teledyne Tekmar	AquaTek	94312017	2008	New
Concentrator	Tekmar	3000	94087010	2008	New
GC/MS System (VOAMS11)					
Gas Chromatograph	Agilent Technologies	6890N	CN10324011	2003	New
MS Detector	Agilent Technologies	5973N	US30965664	2003	New
Autosampler	EST Archon	5100A	13970	2003	New
Concentrator	EST	Encon	279061703	2003	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/FID System (VOAGC1)					
Gas Chromatograph	Agilent Technologies	6890N	US10610006	2006	New
Autosampler	OI	4552	14608	2006	New

Revised 09/15/2015

Page 6 of 18

TestAmerica
THE LEADER IN ENVIRONMENTAL TESTING

Instrument Type (Name)	Manufacturer	Model Number	Serial Number	Year Put into Service	Condition When Received
Concentrator	OI	4660	D607466340	2006	New
Autosampler	OI	4551A	D60745B342	2006	New
Concentrator	OI	4660	D607466341	2006	New
Spiker	OI	SAM	E610475713	2006	New
GC/FID System (VOAGC2)					
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
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 (VOASCREEN1/2)					
Gas Chromatograph	Hewlett-Packard	5890 II	2950A29246	1989	New
Autosampler	Tekmar	7050	91025014	1989	New
Headspace Sampler	Tekmar	7000	91163066	1989	New
Autosampler	Tekmar	7050	91168012	1989	New
Headspace Sampler	Tekmar	7000	90255003	1989	New
GC/FID System (VOASCREEN3/4)					
Gas Chromatograph	Hewlett-Packard	5890	2908A21857	1998	New
Autosampler	Tekmar	7050	91346013	1998	New
Headspace Sampler	Tekmar	7000	91339015	1998	New
Autosampler	Tekmar	7050	90256011	1998	New
Headspace Sampler	Tekmar	7000	91025010	1998	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 #A1)	Denver Instruments	P214	1050008		

Revised 09/15/2015

TestAmerica
THE LEADER IN ENVIRONMENTAL TESTING

Instrument Type (Name)	Manufacturer	Model Number	Serial Number	Year Put into Service	Condition When Received
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
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
GC/FID System (BNAGC1)					
Gas Chromatograph	Agilent Technologies	6890N	US10248079		
Autosampler	Agilent Technologies	G2913A	CN43820804		
Autosampler	Agilent Technologies	G2914A	CN24428046		
Autosampler Tray	Agilent Technologies	G2914A	CN21420543		
GC/FID System (BNAGC2)					

TestAmerica
THE LEADER IN ENVIRONMENTAL TESTING

Instrument Type (Name)	Manufacturer	Model Number	Serial Number	Year Put into Service	Condition When Received
Gas Chromatograph	Agilent Technologies	6890N	US00005410		
Autosampler	Agilent Technologies	G2913A	CN24428026		
Autosampler	Agilent Technologies	G2914A			
Autosampler Tray	Agilent Technologies	G2914A	CN82949935		
GC/FID System (BNAGC3)					
Gas Chromatograph	Agilent Technologies	6890N	US10202132		
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	CN43820808	2006	New
Autosampler	Agilent Technologies	G2914A	CN43820804	2006	New
Autosampler Tray	Agilent Technologies	G2914A	CN43830663	2006	New
GC/FID System (BNAGC5)					
Gas Chromatograph	Hewlett-Packard	5890	2728A14513	1997	New
Autosampler	Hewlett-Packard	7673	2704A0854	1997	New
Autosampler Tray	Hewlett-Packard		2920A10887	1997	New
Controller	Hewlett-Packard		01866	1997	New
GC/FID System (BNAGC5)					
Gas Chromatograph	Hewlett-Packard	5890 II	3203A40054	1997	New
Autosampler	Hewlett-Packard	7673	3120A28315	1997	New
Autosampler	Hewlett-Packard		3202A27987	1997	New
Autosampler Tray	Hewlett-Packard		3228A29094	1997	New
Controller	Hewlett-Packard		3138A27180	1997	New

TestAmerica
THE LEADER IN ENVIRONMENTAL TESTING

Instrument Type (Name)	Manufacturer	Model Number	Serial Number	Year Put into Service	Condition When Received
GC/FID System (BNAGC6)					
Gas Chromatograph	Hewlett-Packard	5890	2443A03923	1999	New
Autosampler	Hewlett-Packard	7673A	2546A02013	1999	New
Autosampler Tray	Hewlett-Packard		2718A05293	1999	New
Controller	Hewlett-Packard		2929A15891	1999	New
GC/FID System (BNAGC8)					
Gas Chromatograph	Agilent Technologies	6890N	US10248079	2005	New
Autosampler	Agilent Technologies	G2913A	CN24428026	2005	New
Autosampler Tray	Agilent Technologies	G2914A	CN24322270	2005	New
GC/ECD System (PESTGC1)					
Gas Chromatograph	Hewlett-Packard	5890A	2612A07669	1992	New
Autosampler	Hewlett-Packard	G1513A	CN22321930	1992	New
Autosampler Tray	Hewlett-Packard	18596C	US72101578	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	3228A31372	1992	New
Autosampler Tray	Hewlett-Packard	18596B	3202A27453	1992	New
Controller	Hewlett-Packard	18594B	3049A23890	1992	New
GC/ECD System (PESTGC4)					
Gas Chromatograph	Hewlett-Packard	5890A Series II	336A54563	1997	New
Autosampler	Hewlett-Packard	18593B	3013A22344	1997	New
Autosampler Tray	Hewlett-Packard	18596B	3624A42191	1997	New
Controller	Hewlett-Packard	18594B	3227A29129	1997	New
GC/ECD System (PESTGC5)					

Revised 09/15/2015

Page 10 of 18

TestAmerica
THE LEADER IN ENVIRONMENTAL TESTING

Instrument Type (Name)	Manufacturer	Model Number	Serial Number	Year Put into Service	Condition When Received
Gas Chromatograph	Agilent Technologies	6890N	US10226033	2002	New
Autosampler	Agilent Technologies	G2613A	CN22025340	2002	New
Autosampler Tray	Agilent Technologies	G2614A	CN21420543	2002	New
GC/ECD System (PESTGC6)					
Gas Chromatograph	Hewlett-Packard	5890A	2950A26642	1998	New
Autosampler	Hewlett-Packard	G1513A	CN13420438	1998	New
Autosampler Tray	Hewlett-Packard	18596C	US20407961	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	C11144007141	1998	New
Autosampler Tray	Hewlett-Packard	18596A	C11154103504	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	CN15221154	2000	New
Autosampler Tray	Agilent Technologies	18596C	3050A23572	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	CN13420437	2001	New
Autosampler Tray	Agilent Technologies	18596C	US13807350	2001	New
Controller	Agilent Technologies	G1512A	CN00004150	2001	New
GC/ECD System (PESTGC11)					
Gas Chromatograph	Agilent Technologies	6890	US00008746	2003	New

Revised 09/15/2015

Page 11 of 18

TestAmerica
THE LEADER IN ENVIRONMENTAL TESTING

Instrument Type (Name)	Manufacturer	Model Number	Serial Number	Year Put into Service	Condition When Received
Autosampler	Agilent Technologies	G2513A	US64600228	2003	New
Autosampler Tray	Agilent Technologies	18596C	US22408138	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	HF Scientific	Micro 100	200604033	2006	New
Ion Selective Meter	Orion	720A	006825	1994	New
Ion Selective Meter	Orion	720A+	092904	2007	New
pH Meter	Orion	320	010005	2002	New
pH Meter	Orion	320	009986	2002	New
pH Meter	Orion	320	016995	2002	New
pH Meter	Orion	320	017414	2009	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	1409-MP	3403003		
Water Bath	Precision	50	9302-112	1995	New
Water Bath	Precision	50	9305-024	1995	New
FTIR	Perkin Elmer	1600	139038	1991	New
Printer	Epson	FX-870	61P107612	2003	New
Fixed Wavelength IR	Buck Scientific	404	1026	2004	New
COD Reactor	Hach	45600	980300017418	2007	New
COD Reactor	Hach	45600	900402268	2007	New
COD Reactor	Hach	DRB 200	1202323	2007	New
COD Reactor	Hach	DRB 200	1209887	2007	New
Flow Injection Autoanalyzer	Lachat	Quickem 8000	A83000	1997	New
Flow Injection Autoanalyzer	Lachat	8000 Series	8300-1658	2000	New

TestAmerica
THE LEADER IN ENVIRONMENTAL TESTING

Instrument Type (Name)	Manufacturer	Model Number	Serial Number	Year Put into Service	Condition When Received
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
Incubatore	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
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
BOD Aerator	Thomas Scientific	DOA-P104D-AA	1187	1998	New
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

TestAmerica
THE LEADER IN ENVIRONMENTAL TESTING

Instrument Type (Name)	Manufacturer	Model Number	Serial Number	Year Put into Service	Condition When Received
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
Organic Prep Department					
Analytical Balance (Balance #28)	A&D	HR200C	12315879		
Analytical Balance (Balance #30)	A&D	HR200C	12315880		
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-Evao #2	Organomation	112	1694	1990	New
N-Evao #3	Organomation	112	59284	2014	New
N-Evao #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

Revised 09/15/2015

TestAmerica
THE LEADER IN ENVIRONMENTAL TESTING

Instrument Type (Name)	Manufacturer	Model Number	Serial Number	Year Put into Service	Condition When Received
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
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/Temp Meter	Thermo Orion	250A+	15035	2000	New
Conductivity Meter	HACH	Sension 5	21000005660	2002	New
DO Meter	HACH	Sension 6	0200001321	2002	New
DO Meter	HACH	Sension 6	001200002352	2000	New
Turbidity Meter	La Motte	2020	0119-0997	1998	New
Turbidity Meter	La Motte	2020	3897-5102	2002	New
Turbidity Meter	La Motte	2020	3649-3802	2002	New
pH/ORP Meter	Cole Palmer	05669-20	643409		New
pH/ORP Meter	HACH	Sension 1	31100003358	2005	New
Conductivity/Salinity/TDS Meter	HACH	Sension 5	30500006215		New
pH/ORP Meter	HACH	Sension 1	050400020239	2005	New
pH/ORP Meter	HACH	Sension 1	050400022762	2005	New
Conductivity/Salinity/TDS Meter	YSI	Sension 5	050200013668	2005	New
Conductivity/Salinity/TDS Meter	La Motte	22	93L12159		New
Turbidity Meter	La Motte	2020E	ME 10036	2005	New
Turbidity Meter	HACH	2020E	ME 10117	2005	New
Conductivity/Salinity/TDS Meter	HACH	Sension 5	050506C50148	2005	New
DO Meter	HACH	Sension 6	050500C60212	2005	New
DO Meter	HACH	Sension 6	050500C60066	2005	New
pH/ORP Meter	HACH	Sension 1	060600C10445	2005	New
pH/ORP Meter	HACH	Sension 1	4030004162	2005	New
DO Meter	HACH		040800001267	2006	New
Conductivity Meter	HACH		05010002708	2006	New

TestAmerica
THE LEADER IN ENVIRONMENTAL TESTING

Instrument Type (Name)	Manufacturer	Model Number	Serial Number	Year Put into Service	Condition When Received
DO Meter	HACH		040700001191	2006	New
pH/mv Meter	HACH		040200003831	2006	New
Conductivity Meter	HACH		050100002707	2006	New
DO Meter	HACH		030500007618	2006	New
pH/mv Meter	HACH		041200004666	2006	New
Turbidity Meter	La Motte		4969-1604	2006	New
Turbidity Meter	La Motte		4943-1604	2006	New
Turbidity Meter	La Motte		1909-2900	2006	New
pH/mv Meter	HACH		041200002902	2006	New
pH/mv Meter E-019	HACH	Sension 1	41200002933	2006	New
Conductivity Meter E-027	HACH	Sension 5	050500C50193	2006	New
pH Meter E-028	HACH	Sension 1	040800010007	2006	New
pH/mv Meter M-039	HACH	Sension 1	0804C410063	2006	New
pH/mv Meter M-034	HACH	Sension 1	06070C710134	2006	New
Conductivity Meter M-028	HACH	Sension 5	050500C50288	2005	New
pH/mv Meter M-036	HACH	Sension 1	07080C710259	2007	New
pH/mv Meter M-030	HACH	Sension 1	050600C10468	2005	New
pH/mv Meter M-037	HACH	Sension 1	08020C110145	2008	New
DO Meter	HACH	Sension 6	07120C260018	2008	New
pH Meter E-031	Thermo Orion	230	018168		New
pH/ORP Meter E-029	HACH	Sension 1	07070C610178	2008	New
DO Meter E-032	YSI	55/25 F	01F0708AA		New
pH Meter E-033	Thermo Orion	230A	017788		New
pH Meter E-034	Thermo Orion	230A	017630		New
Chlorine Meter CL-007	HACH	Pocket Colorimeter II	040200011290	2006	New
Chlorine Meter CL-002	HACH	Pocket Colorimeter	020100174404	2006	New
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
Colorimeter M-040	HACH	48450-60	041050031426		New
Water Level Meter	Solonist		37993	2005	New
Water Level Meter	Solonist		37995	2005	New
Water Level Meter	Solonist		42807	2006	New
Water Level Meter	Fischer				New
PID Meter	RAE Systems	PGM7600	110-010953	2005	New
PID Meter	RAE Systems	Mini RAE 2000	110-010984	2005	New
PID Meter	RAE Systems	Mini-RAE 2000	110-01094	2005	New
PID Meter	RAE Systems	Plus Classic	103958	2005	New
PID Meter	PE Photovac	2020	DQGD302		New

TestAmerica
THE LEADER IN ENVIRONMENTAL TESTING

Instrument Type (Name)	Manufacturer	Model Number	Serial Number	Year Put into Service	Condition When Received
Composite Sampler	ISCO	603704001-3700	205C01376	2005	New
Composite Sampler	ISCO	6037040012-3700	205C01380	2005	New
Composite Sampler	ISCO	3700	204G00984		New
Composite Sampler	ISCO	2700	05248-001		New
Composite Sampler	ISCO	2700			New
Composite Sampler	ISCO	2700			New
COMPOSITE SAMPLER	ISCO	2700			New
DO Meter M-032	HACH	Sension 6	05070C360249	2006	New
Bladder Pump	QED	MP-SPK-4P	11191	2005	New
Bladder Pump	QED	MP-SPK-4P	11192	2005	New
Bladder Pump	QED	MP-SPK-4P	11512	2005	New
Bladder Pump	QED	MP-SPK-4P	10948	2005	New
Bladder Pump	QED	MP-SPK-4P	10949	2005	New
Bladder Pump	QED	MP-SPK-4P		2005	New
Bladder Pump	QED	MP-SPK-4P			New
Bladder Pump	QED	MP-SPK-4P			New
Peristaltic Pump	Solonist	410	002562		New
Peristaltic Pump	Solonist	410	002071		New
Peristaltic Pump	Solonist	410	001979		New
Peristaltic Pump	Solonist	4110	002642		New
Peristaltic Pump	ISCO	Accuwell 150 portable pump			New
Peristaltic Pump	ISCO	Accuwell 150 portable pump			New
Peristaltic Pump	ISCO	Accuwell 150 portable pump			New
Peristaltic Pump	ISCO	Accuwell 150 portable pump			New
Peristaltic Pump	ISCO	Accuwell 150 portable pump			New
Peristaltic Pump	ISCO	Accuwell 150 portable pump			New
Centrifugal Pump	Teel	2P110B	3021		New
Centrifugal Pump	Teel	2P110B	0036		New
Centrifugal Pump	Teel	2P110B	0034		New
Centrifugal Pump	Teel	2P110B	1962		New
Centrifugal Pump	Teel	2P110B			New
Compressor	Coleman/Honda	CT5090412	D021812339	2005	New
Compressor	Honda/Campbell Hausfeld		VT697203AJ		New
Multi-Probe Meter	YSI	55 MPS	06F1362AC	2006	New
GPS	Ashtech	110454-01	10564		New
Oil/Water Interface Probe	Testwell				New
Oil/Water Interface Probe	Testwell				New
Oil/Water Interface Probe	Solonist	122	122-008699-1	2007	New
Oil/Water Interface Probe	Solonist		122 007364-1	2006	New

Revised 09/15/2015

Page 17 of 18

TestAmerica
THE LEADER IN ENVIRONMENTAL TESTING

Instrument Type (Name)	Manufacturer	Model Number	Serial Number	Year Put into Service	Condition When Received
Submersible Pump	Grundfos	MP-1/1A106003	05141-8349	2005	New
Submersible Pump	Grundfos	MP-1/1A106003	05141-8361	2005	New
Submersible Pump	Grundfos	A1A106003P1	0621-0014	2006	New
Submersible Pump	Grundfos		06029591		New
Submersible Pump	Grundfos		98490294		New
Submersible Pump	Grundfos				New
Submersible Pump	Grundfos				New
Submersible Pump	Grundfos				New
Submersible Pump	ProActive	SS Monsoon	1371	2006	New
Pump Control Box	Grundfos	91126028	H0412210120	2005	New
Pump Control Box	Grundfos	91126028	H0412210120	2005	New
Pump Control Box	Grundfos		P1940304254	2005	New
Pump Control Box	Grundfos		203831	2005	New
Pump Control Box	Grundfos		H0303130012	2005	New
Pump Control Box	Grundfos		9517	2005	New
Pump Control Box	Grundfos			2005	New
Pump Control Box	ProActive	Low flow with power booster		2006	New
Trash Pump	North Star	10633	E06	2007	New
Generator	Honda	EZGP-1145763	EB-3000C	2005	New
Generator	Honda	EZGP-1151238	EB-3000C	2005	New
Generator	Honda	EB-3000C	EZGL 1002930	2005	New
Generator	Honda				New
Generator	Honda				New
Control Pack	QED	MP-15	MP15-1300	2005	New
Control Pack	QED	MP-15	MP15-1297	2005	New
Control Pack	QED	MP-15	MP15-1298	2005	New
Control Pack	QED	MP-15	MP15-1299	2005	New
Control Pack	QED	MP-15		2005	New
Control Pack	QED	MP-15		2005	New
Control Pack	QED	MP-15		2005	New
Control Pack	QED	MP-15		2005	New
Control Pack	QED	MP-15		2005	New
Control Pack	QED	MP-15		2005	New
Bladder Pump	QED	MP-SPK-4P	10993	2005	New
Bladder Pump	QED	MP-SPK-4P	10997	2005	New
Bladder Pump	QED	MP-SPK-4P	10995	2005	New
Bladder Pump	QED	MP-SPK-4P	10996	2005	New

Table 21-2. 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
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	

Table 21-2. Example: Schedule of Routine Maintenance

Instrument	Procedure	Frequency
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	0.01M 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
Drying Ovens	Temperature monitoring	Daily
	Temperature adjustments	As required
Refrigerators/ Freezers	Temperature monitoring	Daily
	Temperature adjustment	As required
	Defrosting/cleaning	As required
Vacuum Pumps/ Air Compressor	Drained	Weekly
	Belts checked	Monthly
	Lubricated	Semi-annually
pH/Specific Ion Meter	Calibration/check slope	Daily
	Clean electrode	As required

Table 21-2. Example: Schedule of Routine Maintenance

Instrument	Procedure	Frequency
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

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 of 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

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 in the applicable analytical Departments. Records must be kept of the date of receipt and date of expiration of standards, reagents and reference materials. In addition, records of preparation of laboratory standards, reagents, and reference materials must be retained, stored appropriately, and be readily available for use and inspection. For detailed information on documentation and labeling, please refer to method specific SOPs.

Commercial materials purchased for preparation of calibration solutions, spike solutions, etc.., are usually accompanied with an assay certificate or the purity is noted on the label. If the assay purity is 96% or better, the weight provided by the vendor may be used without correction. If the assay purity is less than 96% a correction will be made to concentrations applied to solutions prepared from the stock commercial material. Blended gas standard cylinders use a nominal concentration if the certified value is within +/-15%, otherwise the certified values is used for the canister concentration.

21.4.1 All standards, reagents, and reference materials must be labeled in an unambiguous manner. Standards are logged into the laboratory's LIMS system, and are assigned a unique identification number. The following information is typically recorded in the electronic database 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 (either electronically or hard-copy) for standard and reference material preparation. These records show the traceability to purchased stocks or neat compounds. These records also include method of preparation, date of preparation, expiration date and preparer's name or initials. Preparation procedures are provided in the Method SOPs.

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 (***Specify from LIMS or logbook***)
- Special Health/Safety warnings if applicable

Records must also be maintained of the date of receipt for commercially purchased items or date of preparation for laboratory prepared items. Special Health/Safety warnings must also be available to the analyst. This information is maintained 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)
- 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 the following sampling and field 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)

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 provided by the supplier 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.

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
- 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.

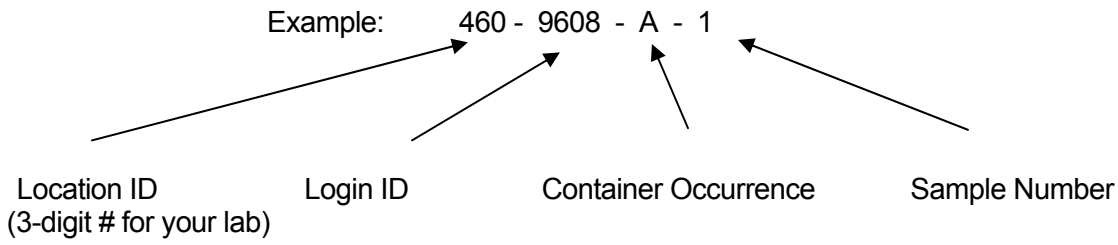
23.2.1 Laboratory Receipt

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 via the Sample Receipt application within TALS (the laboratory LIMS) and brought to the immediate attention of the appropriate Project Manager who will, in turn, contact the client. The COC, shipping documents, documentation of any non-conformance, irregularity, or compromised sample receipt, record of client contact, and resulting instructions become part of the project record.

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);
- 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 an 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).

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 is 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 Soil).

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. 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.

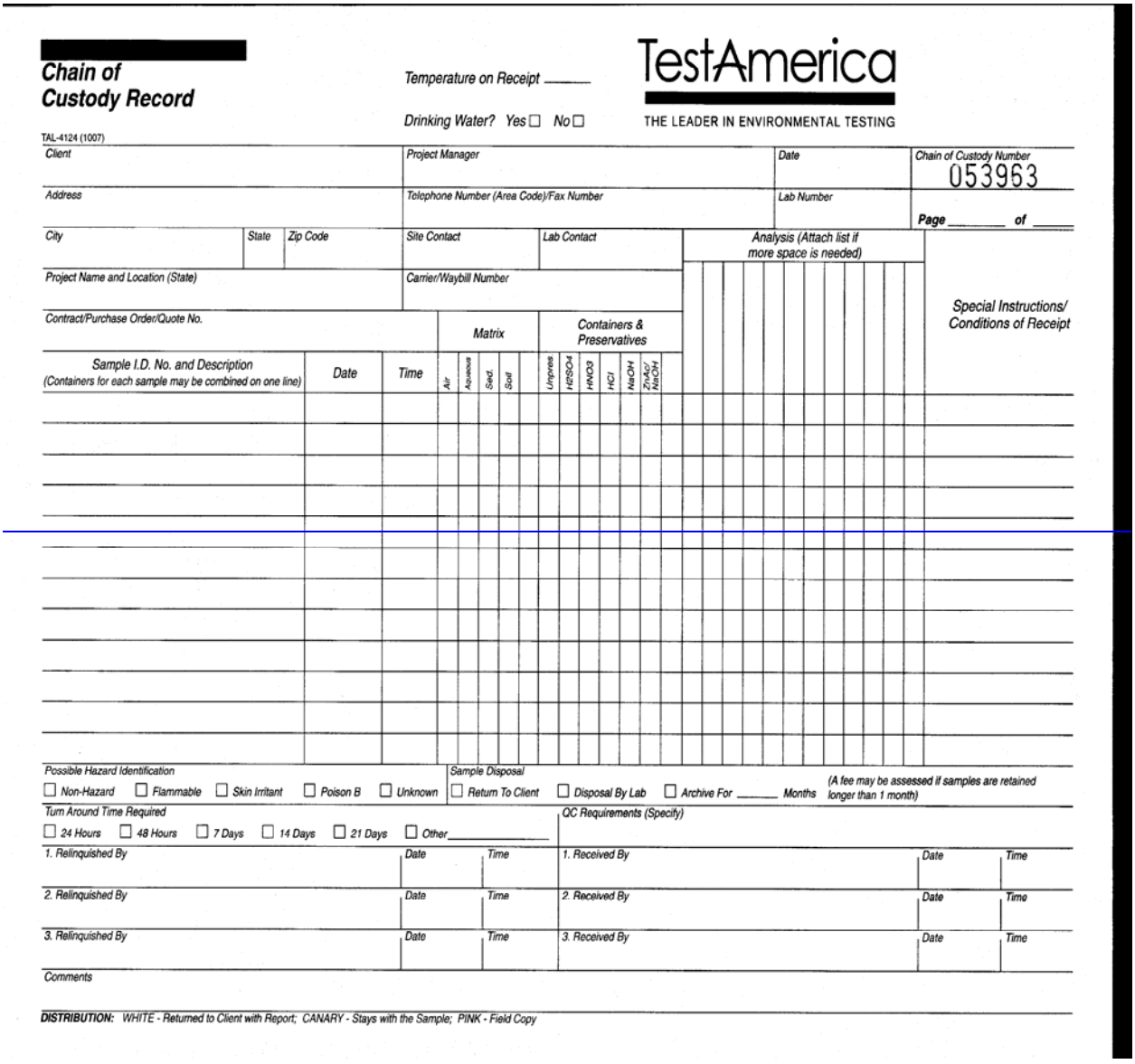
Note: If a client does not request trip blank analysis on the COC or other paperwork, the laboratory will not analyze the trip blanks that were supplied. However, in the interest of good client service, the laboratory will advise the client at the time of sample receipt that it was noted that they did 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 may be disposed of in accordance with the laboratory's waste disposal procedures, TestAmerica Edison SOP No. ED-SPM-007 (Disposal of Samples and Associated Laboratory Waste). All procedures in the laboratory Environmental, Health and Safety Manual are followed during disposal. Samples are normally maintained in the laboratory no longer than 2 months from receipt unless otherwise requested. Unused portions of samples found or suspected to be hazardous according to state or federal guidelines may be returned to the client upon completion of the analytical work.

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).

Figure 23-1. Chain of Custody (COC)



Chain of Custody Record

Temperature on Receipt _____

Drinking Water? Yes ☐ No ☐

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TAL-4124 (1007)

Client	Project Manager	Date	Chain of Custody Number 053963
Address	Telephone Number (Area Code)/Fax Number	Lab Number	Page _____ of _____
City / State / Zip Code	Site Contact	Lab Contact	Analysis (Attach list if more space is needed)
Project Name and Location (State)	Carrier/Waybill Number		Special Instructions/ Conditions of Receipt
Contract/Purchase Order/Quote No.	Matrix	Containers & Preservatives	

Sample I.D. No. and Description (Containers for each sample may be combined on one line)	Date	Time	Air	Aqueous	Sed.	Soil	Unpres	H_2SO_4	HNO_3	HCl	NaOH	ZnAc/ NaOH

Possible Hazard Identification
☐ Non-Hazard ☐ Flammable ☐ Skin Irritant ☐ Poison B ☐ Unknown

Sample Disposal
☐ Return To Client ☐ Disposal By Lab ☐ Archive For _____ Months
(A fee may be assessed if samples are retained longer than 1 month)

Turn Around Time Required
☐ 24 Hours ☐ 48 Hours ☐ 7 Days ☐ 14 Days ☐ 21 Days ☐ Other_____

QC Requirements (Specify)

1. Relinquished By	Date	Time	1. Received By	Date	Time
2. Relinquished By	Date	Time	2. Received By	Date	Time
3. Relinquished By	Date	Time	3. Received By	Date	Time

Comments

DISTRIBUTION: WHITE - Returned to Client with Report; CANARY - Stays with the Sample; PINK - Field Copy

Figure 23-2. Example: Sample Acceptance Policy

All incoming work will be evaluated against the criteria listed below. Where applicable, data from any samples that do not meet the criteria listed below will be noted on the laboratory report defining the nature and substance of the variation. In addition the client will be notified either by telephone, fax or e-mail ASAP after the receipt of the samples.

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.

4) Samples must be preserved according to the requirements of the requested analytical method.

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.

- 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.

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.

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

field samples. Where there is no preparation taken for an analysis (such as in aqueous volatiles), or when all samples and standards undergo the same preparation and analysis process (such as Phosphorus), a calibration verification standard is reported as the LCS. In some instances where there is no practical clean solid matrix available, aqueous LCS's may be processed for solid matrices; final results may be calculated as mg/kg or ug/kg, assuming 100% solids and a weight equivalent to the aliquot used for the corresponding field samples, to facilitate comparison with the field samples.

Certified pre-made reference material purchased from a NIST/A2LA accredited vendor may also be used for the LCS when the material represents the sample matrix or the analyte is not easily spiked (e.g. solid matrix LCS for metals, TDS, etc.).

The specific frequency of use for LCS during the analytical sequence is defined in the specific standard operating procedure for each analysis. It is generally 1 for each batch of samples; not to exceed 20 environmental samples.

If the mandated or requested test method, or project requirements, do not specify the spiking components, the laboratory shall spike all reportable components to be reported in the Laboratory Control Sample (and Matrix Spike) where applicable (e.g. no spike of pH). However, in cases where the components interfere with accurate assessment (such as simultaneously spiking chlordane, toxaphene and PCBs in Method 608), the test method has an extremely long list of components or components are incompatible, at a minimum, a representative number of the listed components (see below) shall be used to control the test method. The selected components of each spiking mix shall represent all chemistries, elution patterns and masses, permit specified analytes and other client requested components. However, the laboratory shall ensure that all reported components are used in the spike mixture within a two-year time period.

- For methods that have 1-10 target analytes, spike all components.
- For methods that include 11-20 target analytes, spike at least 10 or 80%, whichever is greater.
- For methods with more than 20 target analytes, spike at least 16 components.
- Exception: Due to analyte incompatibility in pesticides, Toxaphene and Chlordane are only spiked at client request based on specific project needs.
- Exception: Due to analyte incompatibility between the various PCB aroclors, aroclors 1016 and 1260 are used for spiking as they cover the range of all of the aroclors. Specific aroclors may be used by request on a project specific basis.

24.5 Sample Matrix Controls

Table 24-3. Sample Matrix Control

Control Type	Details	
Matrix Spikes (MS)	Use	used to assess the effect sample matrix of the spiked sample has on the precision and accuracy of the results generated by the method used;
	Typical Frequency [1]	At a minimum, with each matrix-specific batch of samples processed, an MS is carried through the complete analytical procedure. Unless specified by the client, samples used for spiking are randomly selected and rotated between different client projects.If the mandated or requested test method does not specify the spiking components, the laboratory shall spike all reportable components to be reported in the Laboratory Control Sample and Matrix Spike. Refer to the method SOP for complete details
	Description	essentially a sample fortified with a known amount of the test analyte(s).
Surrogate	Use	Measures method performance to sample matrix (organics only).
	Typical Frequency [1]	Are added to all samples, standards, and blanks, for all organic chromatography methods except when the matrix precludes its use or when a surrogate is not available. The recovery of the surrogates is compared to the acceptance limits for the specific method. Poor surrogate recovery may indicate a problem with sample composition and shall be reported, with data qualifiers, to the client whose sample produced poor recovery.
	Description	Are similar to matrix spikes except the analytes are compounds with properties that mimic the analyte of interest and are unlikely to be found in environment samples.
Duplicates[2]	Use	For a measure of analytical precision, with each matrix-specific batch of samples processed, a matrix duplicate (MD or DUP) sample, matrix spike duplicate (MSD), or LCS duplicate (LCSD) is carried through the complete analytical procedure.
	Typical Frequency [1]	Duplicate samples are usually analyzed with methods that do not require matrix spike analysis.
	Description	Performed by analyzing two aliquots of the same field sample independently or an additional LCS.
Internal Standards	Use	Are spiked into all environmental and quality control samples (including the initial calibration standards) to monitor the qualitative aspect of organic and some inorganic analytical measurements.
	Typical Frequency [1]	All organic and ICP methods as required by the analytical method.
	Description	Used to correct for matrix effects and to help troubleshoot variability in analytical response and are assessed after data acquisition. Possible sources of poor internal standard response are sample matrix, poor analytical technique or instrument performance.

[1] See the specific analytical SOP for type and frequency of sample matrix control samples.

[2] LCSD's are normally not performed except when regulatory agencies or client specifications require them. The recoveries for the spiked duplicate samples must meet the same laboratory established recovery limits as the accuracy QC samples. If an LCSD is analyzed both the LCS and LCSD must meet the same recovery criteria and be included in the final report. The precision measurement is reported as "Relative Percent Difference" (RPD). Poor precision between duplicates (except LCS/LCSD) may indicate non-homogeneous matrix or sampling.

24.6 Acceptance Criteria (Control Limits)

As mandated by the test method and regulation, each individual analyte in the LCS, MS, or Surrogate Spike is evaluated against the control limits published in the test method. Where there are no established acceptance criteria, the laboratory calculates in-house control limits with the use of control charts or, in some cases, utilizes client project specific control limits. When this occurs, the regulatory or project limits will supersede the laboratory's in-house limits.

Note: For methods, analytes and matrices with very limited data (e.g., unusual matrices not analyzed often), interim limits are established using available data or by analogy to similar methods or matrices.

Once control limits have been established, they are verified, reviewed, and updated if necessary on an annual basis unless the method requires more frequent updating. Control limits are established per method (as opposed to per instrument) regardless of the number of instruments utilized.

Laboratory generated % Recovery acceptance (control) limits are generally established by taking ± 3 Standard Deviations (99% confidence level) from the average recovery of a minimum of 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.

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.

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

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

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. Signatories are appointed by the Lab Director.

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.

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 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.

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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. All faxed reports are followed by hardcopy. Procedures used to ensure client confidentiality are outlined in Section 25.6.

25.3.1 Electronic Data Deliverables (EDDs)

EDDs are routinely offered as part of TestAmerica's services in addition to the test report as described in Section 25.2. When NELAP accreditation is required and both a test report and EDD are provided to the client, the official version of the test report will be the combined information of the report and the EDD. TestAmerica 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 necessitates a limited scope of interpretation, and this work is performed by the QA Department. This is the only form of "interpretation" of data that is routinely performed by the laboratory.

When opinions or interpretations are included in the report, the laboratory provides an explanation as to the basis upon which the opinions and interpretations have been made. Opinions and interpretations are clearly noted as such and where applicable, a comment should be added suggesting that the client verify the opinion or interpretation with their regulator.

25.5 Environmental Testing Obtained From Subcontractors

If the laboratory is not able to provide the client the requested analysis, the samples would be subcontracted following the procedures outlined in the Corporate SOP on Subcontracting (SOP No. CA-L-S-002).

Data reported from analyses performed by a subcontractor laboratory are clearly identified as such on the analytical report provided to the client. Results from a subcontract laboratory outside of TestAmerica are reported to the client on the subcontract laboratory's original report stationary and the report includes any accompanying documentation.

25.6 Client Confidentiality

In situations involving the transmission of environmental test results by telephone, facsimile or other electronic means, client confidentiality must be maintained.

TestAmerica will not intentionally divulge to any person (other than the Client or any other person designated by the Client in writing) any information regarding the services provided by TestAmerica or any information disclosed to TestAmerica by the Client. Furthermore, information known to be potentially endangering to national security or an entity's proprietary rights will not be released.

Note: This shall not apply to the extent that the information is required to be disclosed by TestAmerica under the compulsion of legal process. TestAmerica will, to the extent feasible, provide reasonable notice to the client before disclosing the information.

Note: Authorized representatives of an accrediting authority are permitted to make copies of any analyses or records relevant to the accreditation process, and copies may be removed from the laboratory for purposes of assessment.

25.6.1 Report deliverable formats are discussed with each new client. If a client requests that reports be faxed or e-mailed, the reports are faxed with a cover sheet or e-mailed with the following note that includes a confidentiality statement similar to the following:

This material is intended only for the use of the individual(s) or entity to whom it is addressed, and may contain information that is privileged and confidential. If you are not the intended recipient, or the employee or agent responsible for delivering this material to the intended recipient, you are hereby notified that any dissemination, distribution or copying of this communication is strictly prohibited. If you have received this communication in error, please notify us immediately by telephone at the 1-800-765-0980 (or for e-mails: please notify us immediately by e-mail or by phone (1-800-765-0980) and delete this material from any computer).

25.7 Format of Reports

The format of reports is designed to accommodate each type of environmental test carried out and to minimize the possibility of misunderstanding or misuse.

25.8 Amendments to Test Reports

Corrections, additions, or deletions to reports are only made when justification arises through supplemental documentation. Justification is documented using the laboratory's corrective action system (refer to Section 12).

The revised report is retained on the Archive data server, as is the original report. The revised report is stored in the Archive data server under the sample number followed by "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.

25.9 Policies on Client Requests for Amendments

25.9.1 Policy on Data Omissions or Reporting Limit Increases

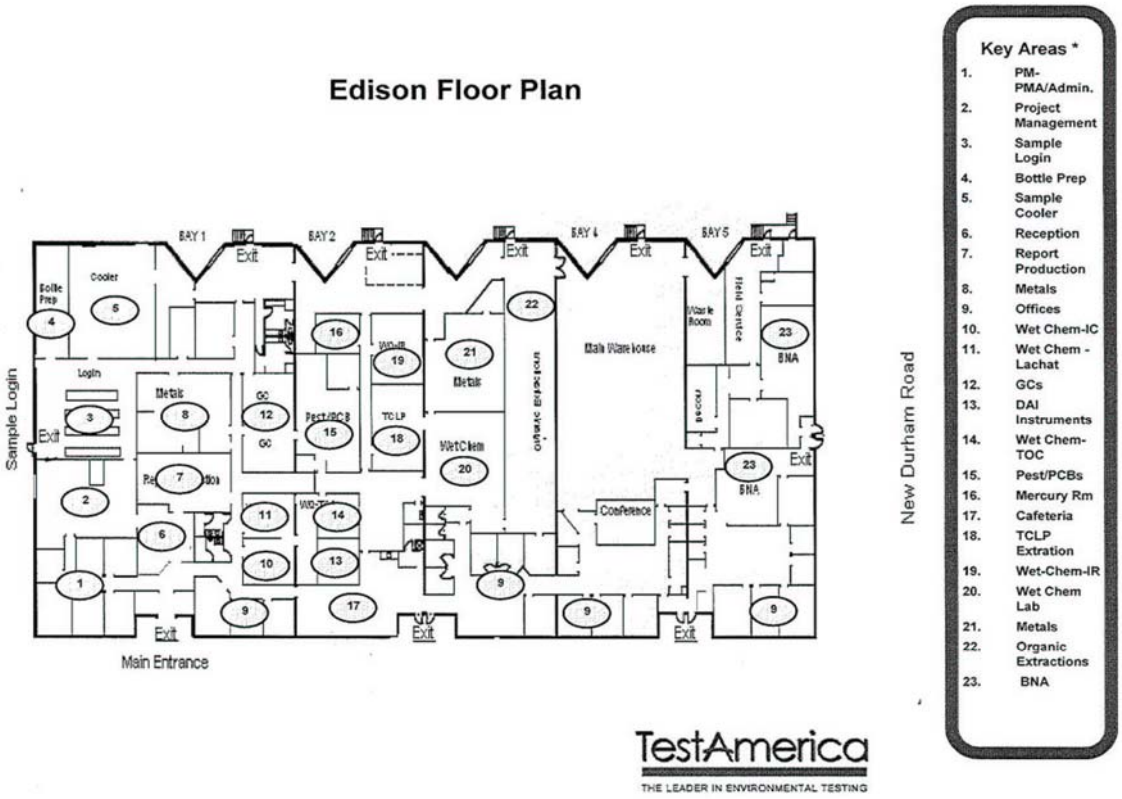
Fundamentally, our policy is simply to not omit previously reported results (including data qualifiers) or to not raise reporting limits and report sample results as ND. This policy has few exceptions. Exceptions are:

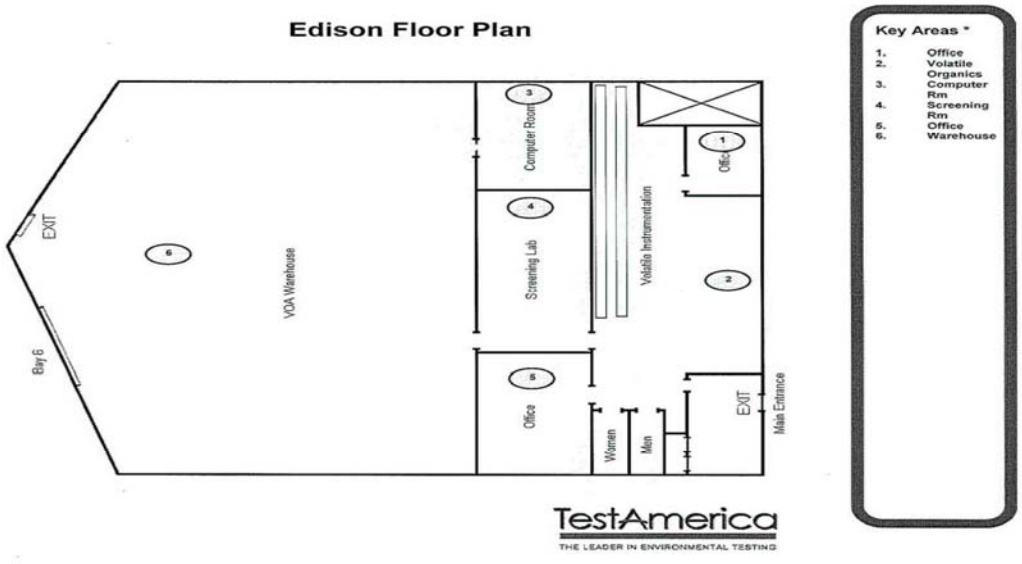
- Laboratory error.
- Sample identification is indeterminate (confusion between COC and sample labels).
- An incorrect analysis (not analyte) was requested (e.g., COC lists 8315 but client wanted 8310). A written request for the change is required.
- Incorrect limits reported based on regulatory requirements.
- The requested change has absolutely *no possible* impact on the interpretation of the analytical results and there is *no possibility* of the change being interpreted as misrepresentation by anyone inside or outside of our company.

25.9.2 Multiple Reports

TestAmerica does not issue multiple reports for the same work order where there is different information on each report (this does not refer to copies of the same report) unless required to meet regulatory needs and approved by QA.

Appendix 1. Laboratory Floor Plan





Appendix 2. Glossary/Acronyms (EL-V1M2 Sec. 3.1)

Glossary:

Acceptance Criteria: Specified limits placed on characteristics of an item, process, or service defined in requirement documents. (ASQC)

Accreditation: The process by which an agency or organization evaluates and recognizes a laboratory as meeting certain predetermined qualifications or standards, thereby accrediting the laboratory.

Accuracy: The degree of agreement between an observed value and an accepted reference value. Accuracy includes a combination of random error (precision) and systematic error (bias) components which are due to sampling and analytical operations; a data quality indicator. (QAMS)

Analyst: The designated individual who performs the "hands-on" analytical methods and associated techniques and who is the one responsible for applying required laboratory practices and other pertinent quality controls to meet the required level of quality.

Analytical Uncertainty: A subset of Measurement Uncertainty that includes all laboratory activities performed as part of the analysis. (TNI)

Anomaly: A condition or event, other than a deficiency, that may affect the quality of the data, whether in the laboratory's control or not.

Assessment: The evaluation process used to measure or establish the performance, effectiveness, and conformance of an organization and/or its systems to defined criteria (to the standards and requirements of laboratory accreditation). (TNI)

Audit: A systematic and independent examination of facilities, equipment, personnel, training, procedures, record-keeping, data validation, data management, and reporting aspects of a system to determine whether QA/QC and technical activities are being conducted as planned and whether these activities will effectively achieve quality objectives. (TNI)

Batch: Environmental samples that are prepared and/or analyzed together with the same process and personnel, using the same lot(s) of reagents. A **preparation batch** is composed of one (1) to twenty (20) environmental samples of the same quality systems matrix, meeting the above mentioned criteria and with a maximum time between the start of processing of the first and last sample in the batch to be twenty-four (24) hours. An **analytical batch** is composed of prepared environmental samples (extracts, digestates or concentrates) which are analyzed together as a group. An analytical batch can include prepared samples originating from various quality system matrices and can exceed twenty (20) samples. (TNI)

Bias: The systematic or persistent distortion of a measurement process, which causes errors in one direction (i.e., the expected sample measurement is different from the sample's true value). (TNI)

Blank: A sample that has not been exposed to the analyzed sample stream in order to monitor contamination during sampling, transport, storage or analysis. The blank is subjected to the usual analytical and measurement process to establish a zero baseline or background value and is sometimes used to adjust or correct routine analytical results. (ASQC)

Calibration: A set of operations that establish, under specified conditions, the relationship between values of quantities indicated by a measuring instrument or measuring system, or values represented by a material measure or a reference material, and the corresponding values realized by standards. (TNI)

1) In calibration of support equipment the values realized by standards are established through the use of reference standards that are traceable to the International System of Units (SI).

2) In calibration according to methods, the values realized by standards are typically established through the use of Reference Materials that are either purchased by the laboratory with a certificate of analysis or purity, or prepared by the laboratory using support equipment that has been calibrated or verified to meet specifications.

Calibration Curve: The mathematical relationship between the known values, such as concentrations, of a series of calibration standards and their instrument response. (TNI)

Calibration Standard: A substance or reference material used to calibrate an instrument (QAMS)

Certified Reference Material (CRM): A reference material accompanied by a certificate, having a value, measurement uncertainty, and stated metrological traceability chain to a national metrology institute. (TNI).

Chain of Custody (COC) Form: Record that documents the possession of the samples from the time of collection to receipt in the laboratory. This record generally includes: the number and types of containers; the mode of collection; the collector; time of collection; preservation; and requested analyses. (TNI)

Compromised Samples: Those samples which are improperly sampled, insufficiently documented (chain of custody and other sample records and/or labels), improperly preserved, collected in improper containers, or exceeding holding times when delivered to a laboratory. Under normal conditions, compromised samples are not analyzed. If emergency situation require analysis, the results must be appropriately qualified.

Confidential Business Information (CBI): Information that an organization designates as having the potential of providing a competitor with inappropriate insight into its management, operation or products. TNI and its representatives agree to safeguarding identified CBI and to maintain all information identified as such in full confidentiality.

Confirmation: Verification of the identity of a component through the use of an approach with a different scientific principle from the original method. These may include, but are not limited to Second Column Confirmation; Alternate wavelength; Derivatization; Mass spectral interpretation; Alternative detectors or Additional Cleanup procedures. (TNI)

Conformance: An affirmative indication or judgment that a product or service has met the requirements of the relevant specifications, contract, or regulation; also the state of meeting the requirements. (ANSI/ASQC E4-1994)

Correction: Actions necessary to correct or repair analysis specific non-conformances. The acceptance criteria for method specific QC and protocols as well as the associated corrective actions. The analyst will most frequently be the one to identify the need for this action as a result of calibration checks and QC sample analysis**.** No significant action is taken to change behavior, process or procedure.

Corrective Action: The action taken to eliminate the causes of an existing nonconformity, defect or other undesirable situation in order to prevent recurrence. (ISO 8402)

Data Audit: A qualitative and quantitative evaluation of the documentation and procedures associated with environmental measurements to verify that the resulting data re of acceptable quality (i.e., that they meet specified acceptance criteria).

Data Reduction: The process of transforming the number of data items by arithmetic or statistical calculations, standard curves, and concentration factors, and collation into a more useable form. (TNI)

Deficiency: An unauthorized deviation from acceptable procedures or practices, or a defect in an item. (ASQC), whether in the laboratory's control or not.

Demonstration of Capability: A procedure to establish the ability of the analyst to generate analytical results of acceptable accuracy and precision. (TNI)

Document Control: The act of ensuring that documents (and revisions thereto) are proposed, reviewed for accuracy, approved for release by authorized personnel, distributed properly, and controlled to ensure use of the correct version at the location where the prescribed activity if performed. (ASQC)

Duplicate Analyses: The analyses or measurements of the variable of interest performed identically on two subsamples of the same sample. The results from duplicate analyses are used to evaluate analytical or measurement precision but not the precision of sampling, preservation or storage internal to the laboratory. (EPA-QAD)

Equipment Blank: Sample of analyte-free media which has been used to rinse common sampling equipment to check effectiveness of decontamination procedures.

External Standard Calibration: Calibrations for methods that do not utilize internal standards to compensate for changes in instrument conditions.

Field Blank: Blank prepared in the field by filing a clean container with pure de-ionized water and appropriate preservative, if any, for the specific sampling activity being undertaken (EPA OSWER)

Field of Accreditation: Those matrix, technology/method, and analyte combinations for which the accreditation body offers accreditation.

Holding Times: The maximum times that samples may be held prior to analyses and still be considered valid or not compromised. (40 CFR Part 136)

Internal Standard: A known amount of standard added to a test portion of a sample as a reference for evaluating and controlling the precision and bias of the applied analytical test method. (TNI)

Internal Standard Calibration: Calibrations for methods that utilize internal standards to compensate for changes in instrument conditions.

Instrument Blank: A clean sample (e.g., distilled water) processed through the instrumental steps of the measurement process; used to determine instrument contamination. (EPA-QAD)

Instrument Detection Limit (IDL): The minimum amount of a substance that can be measured with a specified degree of confidence that the amount is greater than zero using a specific instrument. The IDL is associated with the instrumental portion of a specific method only, and sample preparation steps are not considered in its derivation. The IDL is a statistical estimation at a specified confidence interval of the concentration at which the relative uncertainty is ± 100%. The IDL represents a range where qualitative detection occurs on a specific instrument. Quantitative results are not produced in this range.

Laboratory Control Sample (however named, such as laboratory fortified blank, spiked blank, or QC check sample): A sample matrix, free from the analytes of interest, spiked with verified known amounts of analytes or a material containing known and verified amounts of analytes, taken through all preparation and analysis steps of the procedure unless otherwise noted in a reference method. It is generally used to establish intra-laboratory or analyst specific precision and bias or to assess the performance of all or a portion of the measurement system.

An LCS shall be prepared at a minimum of 1 per batch of 20 or less samples per matrix type per sample extraction or preparation method except for analytes for which spiking solutions are not available such as total suspended solids, total dissolved solids, total volatile solids, total solids, pH, color, odor, temperature, dissolved oxygen or turbidity. The results of these samples shall be used to determine batch acceptance.

Least Squares Regression (1st Order Curve): The least squares regression is a mathematical calculation of a straight line over two axes. The y axis represents the instrument response (or Response ratio) of a standard or sample and the x axis represents the concentration. The regression calculation will generate a correlation coefficient (r) that is a measure of the "goodness of fit" of the regression line to the data. A value of 1.00 indicates a perfect fit. In order to be used for quantitative purposes, r must be greater than or equal to 0.99 for organics and 0.995 for inorganics.

Limit(s) of Detection (LOD) [a.k.a., Method Detection Limit (MDL)]: A laboratory's estimate of the minimum amount of an analyte in a given matrix that an analytical process can reliably detect in their facility. (TNI)

LOD Verification [a.k.a., MDL Verification]: A processed QC sample in the matrix of interest, spiked with the analyte at no more than 3X the LOD for single analyte tests and 4X the LOD for multiple analyte tests and processed through the entire analytical procedure.

Limit(s) of Quantitation (LOQ) [a.k.a., Reporting Limit]: The minimum levels, concentrations, or quantities of a target variable (e.g., target analyte) that can be reported with a specified degree of confidence. (TNI)

Quality System (QS) Matrix: The component or substrate that contains the analyte of interest. For purposes of batch and QC requirement determinations, the following matrix distinctions shall be used:

Aqueous: Any aqueous sample excluded from the definition of Drinking Water or Saline/Estuarine —Includes surface water, groundwater, effluents, and TCLP or other extracts.

Drinking Water: Any aqueous sample that has been designated as a potable or potential potable water source.

Saline/Estuarine: Any aqueous sample from an ocean or estuary, or other salt water source such as the Great Salt Lake.

Non-Aqueous Liquid: Any organic liquid with <15% settleable solids.

Biological Tissue: Any sample of a biological origin such as fish tissue, shellfish, or plant material. Such samples shall be grouped according to origin.

Solids: Includes soils, sediments, sludges, and other matrices with >15% settleable solids.

Chemical Waste: A product or by-product of an industrial process that results in a matrix not previously defined.

Air & Emissions: Whole gas or vapor samples including those contained in flexible or rigid wall containers and the extracted concentrated analytes of interest from a gas or vapor that are collected with a sorbant tube, impinger solution, filter, or other device. (TNI)

Matrix Spike (spiked sample or fortified sample): A sample prepared, taken through all sample preparation and analytical steps of the procedure unless otherwise noted in a referenced method, by adding a known amount of target analyte to a specified amount of sample for which an independent test result of target analyte concentration is available. Matrix spikes are used, for example, to determine the effect of the matrix on a method's recovery efficiency.

Matrix Spike Duplicate (spiked sample or fortified sample duplicate): A replicate matrix spike prepared and analyzed to obtain a measure of the precision of the recovery for each analyte.

Method Blank: A sample of a matrix similar to the batch of associated samples (when available) that is free from the analytes of interest and is processed simultaneously with and under the same conditions as samples through all steps of the analytical procedures, and in which no target analytes or interferences are present at concentrations that impact the analytical results for sample analyses.

Method Detection Limit: The minimum concentration of a substance (an analyte) that can be measured and reported with 99% confidence that the analyte concentration is greater than zero and is determined from analysis of a sample in a given matrix containing the analyte. (40 CFR Part 136, Appendix B)

Negative Control: Measures taken to ensure that a test, its components, or the environment do not cause undesired effects, or produce incorrect test results.

Non-conformance: An indication, judgment, or state of not having met the requirements of the relevant specifications, contract, or regulation.

Observation: A record of phenomena that (1) may assist in evaluation of the sample data; (2) may be of importance to the project manager and/or the client, and yet not at the time of the observation have any known effect on quality.

Performance Audit: The routine comparison of independently obtained qualitative and quantitative measurement system data with routinely obtained data in order to evaluate the proficiency of an analyst or laboratory.

Positive Control: Measures taken to ensure that a test and/or its components are working properly and producing correct or expected results from positive test subjects.

Precision: The degree to which a set of observations or measurements of the same property, obtained under similar conditions, conform to themselves; a data quality indicator. Precision is usually expressed as standard deviation, variance or range, in either absolute or relative terms. (TNI)

Preservation: Any conditions under which a sample must be kept in order to maintain chemical and/or biological integrity prior to analysis. (TNI)

Proficiency Testing: A means of evaluating a laboratory's performance under controlled conditions relative to a given set of criteria through analysis of unknown samples provided by an external source. (TNI)

Proficiency Testing Program: The aggregate of providing rigorously controlled and standardized environmental samples to a laboratory for analysis, reporting of results, statistical evaluation of the results and the collective demographics and results summary of all participating laboratories. (TNI)

Proficiency Test Sample (PT): A sample, the composition of which is unknown to the laboratory and is provided to test whether the laboratory can produce analytical results within specified acceptance criteria. (TNI)

Quality Assurance: An integrated system of management activities involving planning, implementation, assessment, reporting and quality improvement to ensure that a process, item, or service is of the type of quality needed and expected by the client. (TNI)

Quality Assurance [Project] Plan (QAPP): A formal document describing the detailed quality control procedures by which the quality requirements defined for the data and decisions pertaining to a specific project are to be achieved. (EAP-QAD)

Quality Control: The overall system of technical activities that measures the attributes and performance of a process, item, or service against defined standards to verify that they meet the stated requirements established by the customer; operational techniques and activities that are used to fulfill requirements for quality; also the system of activities and checks used to ensure that measurement systems are maintained within prescribed limits, providing protection against "out of control" conditions and ensuring that the results are of acceptable quality. (TNI)

Quality Control Sample: A sample used to assess the performance of all or a portion of the measurement system. One of any number of samples, such as Certified Reference Materials, a quality system matrix fortified by spiking, or actual samples fortified by spiking, intended to demonstrate that a measurement system or activity is in control. (TNI)

Quality Manual: A document stating the management policies, objectives, principles, organizational structure and authority, responsibilities, accountability, and implementation of an agency, organization, or laboratory, to ensure the quality of its product and the utility of its product to its users. (TNI)

Quality System: A structured and documented management system describing the policies, objectives, principles, organizational authority, responsibilities, accountability, and implementation plan of an organization for ensuring quality in its work processes, products (items), and services. The quality system provides the framework for planning, implementing, and assessing work performed by the organization and for carrying out required QA and QC activities. (TNI)

Raw Data: The documentation generated during sampling and analysis. This documentation includes, but is not limited to, field notes, electronic data, magnetic tapes, untabulated sample results, QC sample results, print outs of chromatograms, instrument outputs, and handwritten records. (TNI)

Record Retention: The systematic collection, indexing and storing of documented information under secure conditions.

Reference Material: Material or substance one or more properties of which are sufficiently homogeneous and well established to be used for the calibration of an apparatus, the assessment of a measurement method, or for assigning values to materials. (TNI)

Reference Standard: Standard used for the calibration of working measurement standards in a given organization or a given location. (TNI)

Sampling: Activity related to obtaining a representative sample of the object of conformity assessment, according to a procedure.

Second Order Polynomial Curve (Quadratic): The 2nd order curves are a mathematical calculation of a slightly curved line over two axis. The y axis represents the instrument response (or Response ratio) of a standard or sample and the x axis represents the concentration. The 2nd order regression will generate a

coefficient of determination (COD or r^2) that is a measure of the "goodness of fit" of the quadratic curvature the data. A value of 1.00 indicates a perfect fit. In order to be used for quantitative purposes, r^2 must be greater than or equal to 0.99.

Selectivity: The ability to analyze, distinguish, and determine a specific analyte or parameter from another component that may be a potential interferent or that may behave similarly to the target analyte or parameter within the measurement system. (TNI)

Sensitivity: The capability of a method or instrument to discriminate between measurement responses representing different levels (e.g., concentrations) of a variable of interest. (TNI)

Spike: A known mass of target analyte added to a blank, sample or sub-sample; used to determine recovery efficiency or for other quality control purposes.

Standard: The document describing the elements of laboratory accreditation that has been developed and established within the consensus principles of standard setting and meets the approval requirements of standard adoption organizations procedures and policies. (TNI)

Standard Operating Procedures (SOPs): A written document which details the method for an operation, analysis, or action, with thoroughly prescribed techniques and steps. SOPs are officially approved as the methods for performing certain routine or repetitive tasks. (TNI)

Storage Blank: A blank matrix stored with field samples of a similar matrix (volatiles only) that measures storage contribution to any source of contamination.

Surrogate: A substance with properties that mimic the analyte of interest. It is unlikely to be found in environment samples and is added to them for quality control purposes.

Surrogate compounds must be added to all samples, standards, and blanks, for all organic chromatography methods except when the matrix precludes its use or when a surrogate is not available. Poor surrogate recovery may indicate a problem with sample composition and shall be reported to the client whose sample produced poor recovery. (QAMS)

Systems Audit (also Technical Systems Audit): A thorough, systematic, qualitative on-site assessment of the facilities, equipment, personnel, training, procedures, record keeping, data validation, data management, and reporting aspects of a total measurement system. (EPA-QAD)

Technical Manager: A member of the staff of an environmental laboratory who exercises actual day-to-day supervision of laboratory operations for the appropriate fields of accreditation and reporting of results

Technology: A specific arrangement of analytical instruments, detection systems, and/or preparation techniques.

Traceability: The ability to trace the history, application, or location of an entity by means of recorded identifications. In a calibration sense, traceability relates measuring equipment to national or international standards, primary standards, basic physical constants or properties, or reference materials. In a data collection sense, it relates calculations and data generated throughout the project back to the requirements for the quality of the project. (TNI)

Trip Blank: A blank matrix placed in a sealed container at the laboratory that is shipped, held unopened in the field, and returned to the laboratory in the shipping container with the field samples.

Uncertainty: A parameter associated with the result of a measurement that characterizes the dispersion of the value that could reasonably be attributed to the measured value.

Acronyms:

CAR – Corrective Action Report
CCV – Continuing Calibration Verification
CF – Calibration Factor
CFR – Code of Federal Regulations
COC – Chain of Custody
DOC – Demonstration of Capability
DQO – Data Quality Objectives
DUP - Duplicate
EHS – Environment, Health and Safety
EPA – Environmental Protection Agency
GC - Gas Chromatography
GC/MS - Gas Chromatography/Mass Spectrometry
HPLC - High Performance Liquid Chromatography
ICP - Inductively Coupled Plasma Atomic Emission Spectroscopy
ICP/MS – ICP/Mass Spectrometry
ICV – Initial Calibration Verification
IDL – Instrument Detection Limit
IH – Industrial Hygiene
IS – Internal Standard
LCS – Laboratory Control Sample
LCSD – Laboratory Control Sample Duplicate
LIMS – Laboratory Information Management System
LOD – Limit of Detection
LOQ – Limit of Quantitation
MDL – Method Detection Limit
MDLCK – MDL Check Standard
MDLV – MDL Verification Check Standard
MRL – Method Reporting Limit Check Standard
MS – Matrix Spike
MSD – Matrix Spike Duplicate
NELAP - National Environmental Laboratory Accreditation Program
PT – Performance Testing
TNI – The NELAC Institute
QAM – Quality Assurance Manual
QA/QC – Quality Assurance / Quality Control
QAPP – Quality Assurance Project Plan
RF – Response Factor
RPD – Relative Percent Difference
RSD – Relative Standard Deviation
SD – Standard Deviation
SDS – Safety Data Sheet
SOP – Standard Operating Procedure
TAT – Turn-Around-Time
VOA – Volatiles
VOC – Volatile Organic Compound

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 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	04/04/2017
TestAmerica Edison	NELAP	New Jersey	12028	06/30/2015 *
TestAmerica Edison	NELAP	New York	11452	03/31/2016
TestAmerica Edison	NELAP	Pennsylvania	68-00522	02/28/2016
TestAmerica Edison	State Program	Connecticut	PH-0200	09/30/2016
TestAmerica Edison	State Program	DE Haz. Subst. Cleanup Act (HSCA)	N/A	12/31/2015
TestAmerica Edison	State Program	Rhode Island	LAO00132	12/30/2015

Page 1 of 1 10/23/15

* Certification Valid - Laboratory is Pending Renewal with the Program Authority

For more information, or to contact a local TestAmerica representative nearest you, please visit our website at www.testamericainc.com

The certificates and accredited parameter lists are available, for each State/Program at www.testamericainc.com under Analytical Services Search – Certifications.

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Quality Assurance Manual

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Effective Date of Revision 24: August 1, 2014

Facility Distribution No. 0001 **Distributed To: QA Server, 02, 03, 04, 05, 06, 07, 10, 12**

Title Page:

Quality Assurance Manual
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SECTION 2. TABLE OF CONTENTS

LIST OF TABLES

LIST OF FIGURES

LIST OF APPENDICES

REFERENCED CORPORATE SOPs AND POLICIES

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

REFERENCED LABORATORY SOPs

SOP Reference	Title
NV02-1	Sample Receiving
NV02-174	Project Management
NV03-212	Thermometer Calibration / Temperature Monitoring and Documentation
NV03-213	Balance Calibration
NV08-139	Data Package Procedure
NV08-152	Document Control
NV08-161	Management of Change System
NV08-194	Archiving of Technical and Quality Department Paper Records
NV08-199	Training Procedures for Environmental Technical Staff
NV08-202	Procedure for the Determination of Method Detection Limits
NV08-203	Procedure for Method Start-Up or Modification
NV08-214	Reagent and Standard Purchase, Preparation, Control Documentation
NV08-220	Organization of Quality Department Documents
NV08-229	Sample Homogenization, Subsampling, and Compositing
NV08-232	Purchasing
NV09-12	Lab Data Archiving System
NV10-83	Waste Disposal
NV10-162	Handling of Soils Regulated by USDA

SECTION 3. INTRODUCTION, SCOPE AND APPLICABILITY

3.1 Introduction and Compliance References

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

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

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

- EPA 600/4-88/039, *Methods for the Determination of Organic Compounds in Drinking Water,* EPA, Revised July 1991*.*
- EPA 600/R-95/131, *Methods for the Determination of Organic Compounds in Drinking Water*, Supplement III, EPA, August 1995.
- EPA 600/4-79-019, *Handbook for Analytical Quality Control in Water and Wastewater Laboratories*, EPA, March 1979.
- EPA SW-846, *Test Methods for Evaluating Solid Waste Physical/Chemical Methods (SW846), Third Edition*, September 1986, Final Update 1, July 1992; Final Update IIA, August 1993; Final Update II, September 1994; Final Update IIB, January 1995; Final Update III, December 1996; Final Update IIIB, June 2005, and Final Update IV, January 2008.
- Federal Register, 40 CFR Parts 136, 141, 172, 173, 178, 179 and 261.
- *Manual for the Certification of Laboratories Analyzing Drinking Water* (EPA-815-R-05-004, January 2005)
- *Statement of Work for Inorganics & Organics Analysis, Multi-Media, Multi-Concentration, SOM and ISM*, current revisions, USEPA Contract Laboratory Program
- APHA, *Standard Methods for the Examination of Water and Wastewater*, 18th Edition, 19th, 20th, 21st and on-line Editions.
- Toxic Substances Control Act (TSCA).

3.2 Terms and Definitions

The Quality Assurance Program is a company-wide system designed to ensure that data produced by TestAmerica Nashville 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

TestAmerica Nashville analyzes a broad range of environmental and industrial samples. Sample matrices vary among drinking water, effluent water, groundwater, hazardous waste, sludge, and soils. The Quality Assurance Program contains specific procedures and methods to test samples of differing matrices for chemical and physical parameters. The QA Program also contains guidelines on maintaining documentation of analytical processes, reviewing results, servicing clients, and tracking samples through the laboratory. The technical and service requirements of all analytical requests are thoroughly evaluated before commitments are made to accept the work. Measurements are made using published reference methods or methods developed and validated by the laboratory.

The methods covered by this manual include the most frequently requested methodologies needed to provide analytical services in the United States and its territories. The specific list of test methods used by the laboratory can be found on TestAmerica's TotalAccess website. 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 TestAmerica Nashville meet these criteria, as appropriate. In some instances, quality assurance project plans (QAPPs), project specific data quality objectives (DQOs), or local regulations may require criteria other than those contained in this manual. In these cases, the laboratory will abide by the requested criteria following review and acceptance of the requirements by the Laboratory Director, the Technical Director, and the Quality Assurance Manager. In some cases, QAPPs and DQOs may specify less stringent requirements. The Laboratory Director, the Technical Director, and the QA Manager must determine if it is in the laboratory's best interest to follow the less stringent requirements.

3.4 Management of the Manual

3.4.1 Review Process

The template on which this manual is based is reviewed annually by Corporate Quality Management Personnel to assure that it remains in compliance with Section 3.1. This manual itself is reviewed biannually by senior laboratory management to assure that it reflects current practices and meets the requirements of TestAmerica Nashville'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 reviews the changes in the normal course of business and incorporate changes into revised sections of the document. All updates will be reviewed by senior laboratory management staff. The laboratory updates and approves such changes according to the SOPs *Document Control* / NV08-152 and/or *Management of Change* / NV08-161.

SECTION 4. MANAGEMENT REQUIREMENTS

4.1 Overview

TestAmerica Nashville is a local operating unit of TestAmerica Laboratories, Inc. The organizational structure, responsibilities, and authorities of the corporate staff of TestAmerica Laboratories, Inc are presented in the CQMP. The laboratory has day-to-day independent operational authority overseen by corporate officers (e.g., Chief Executive Officer (CEO), Executive Vice President (VP) Operations, Corporate Quality, etc.). The laboratory operational and support staff work under direction of the Laboratory Director. The organizational structure for both Corporate and TestAmerica Nashville 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. Due to the variable nature of terminology among the various States, the use of the words “Director” and “Manager” should be considered equivalent. When necessary, more extensive job descriptions may be maintained by laboratory management.

4.2.1 Additional Requirements for Laboratories

The responsibility for quality resides with every employee of TestAmerica Nashville. 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 Nashville laboratory.

4.2.2 Laboratory Director

TestAmerica Nashville’s Laboratory Director supervises all laboratory personnel and provides leadership and direction as needed. The Laboratory Director is also responsible for ensuring compliance and integration of facility operations with corporate and regulatory policies and procedures.

Specific responsibilities include, but are not limited to:

- Manages the laboratory to provide a positive operating margin and meet annual budgetary goals.
- Builds and maintains positive customer relationships and sales growth through direct interaction with customers and TestAmerica sales personnel.
- Establishes and maintains an active and positive safety culture and ensures that the employee Health and Safety procedures are implemented and followed to maintain facility operations that are compliant with appropriate policies and regulations.
- Reviews and approves Quality Assurance SOPs for the facility and ensures their implementation such that the facility is operated in a compliant manner which allows the production of defensible data.

- Prioritizes the activities of the operations groups to ensure key goals are achieved and customer service needs are addressed.
- Communicates facility–specific goals and objectives to employees. Communicates and implements company iniatives designed to foster teamwork and communication.
- Evaluates and maintains appropriate staffing levels to support operational needs and budgetary requirements.
- Supports Human Resources function to ensure that all laboratory policies and programs are applied consistently throughout the laboratory.
- Provides periodic reports to General Manager to ensure that goals and objectives are being achieved and to recognize opportunities for development.
- Works with Project Managers and Manager of Project Managers to ensure forecasting is accurate, the lab is providing quality data to clients and meeting client expectations.
- Provides supervisory resposnsibilities to direct reports to achieve strong staff performance. Mentors key employees.
- Actively participates in TestAmerica's best practice process to spread best technical practices and develop TestAmerica Standard Operating Procedures. Leads the implementation and follow-up of the best practices and SOPs in the laboratory.
- Pursues and maintains appropriate laboratory certification and contract approvals. Supports ISO 17025 requirements.

4.2.3 Quality Assurance (QA) Manager or Designee

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

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

- Serves as the focal point for QA/QC in the laboratory
- Maintains and updating the QAM.
- Monitors and evaluating laboratory certifications; schedules proficiency testing samples.
- Monitors and communicates regulatory changes that may affect the laboratory to management.
- Trains and advises the laboratory staff on quality assurance/quality control procedures that are pertinent to their daily activities.
- Arranges for or conducts internal audits on quality systems and the technical operation.
- Acts as a focal point for ethics and data integrity issues
- Maintains records of all ethics-related training, including the type and proof of attendance.

- Maintains training records, including DOCs, for all analysts.
- Reviews and approves SOPs. Writes SOPs as needed.
- Maintains, improves, and evaluates the corrective action database and the corrective and preventive action systems.
- Notifies laboratory management of deficiencies in the quality system and ensures corrective action is taken. Procedures that do not meet the standards set forth in the QAM or laboratory SOPs shall be investigated following procedures outlined in Section 12 and if deemed necessary, may be temporarily suspended during the investigation.
- Objectively monitors standards of performance in quality control and quality assurance without outside (e.g., managerial) influence.
- Coordinates document control of SOPs, MDLs, control limits, miscellaneous forms, and information.
- Authorized to stop work when procedures do not conform to the requirements of the QAM or laboratory SOPs
- Reviews a percentage of all final data reports for internal consistency, including 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.
- Establishes a reporting schedule and prepares various quality reports for the Laboratory Director, clients, and/or Corporate QA.
- Develops suggestions and recommendations to improve quality systems.
- Researches of current state and federal requirements and guidelines.
- Tracks customer complaints and assists in identifying improvements.
- Oversees and approves MDL studies and verifications.
- Captains the QA team to enable communication and to distribute duties and responsibilities.
- Ensures communication and monitors standards of performance to ensure that systems are in place to produce the level of quality defined in this document.

4.2.4 Technical Director or Designee

The Technical Director Serves as a technical resource for TestAmerica's personnel and clients in their field of expertise. Researches, develops and implements new analytical methods and recommends process improvements to existing analyses. Manages technical projects and conducts evaluations of technologies. Writes and reviews laboratory SOPs and trains employees on methods and procedures. Maintains and troubleshoots laboratory instruments.. Specific responsibilities include, but are not limited to:

- Solves technical problems in the laboratory including troubleshooting instruments and develops or modifies methods as needed to meet customer requirements. Consults with clients regulators, and others regarding technical aspects of analyses.
- Suggests and implements process improvements to maximize productivity, save costs and decrease turnaround time.

- Develops and amends departmental SOPs.
- Participates in TestAmerica's best practice process to spread best technical practices and develop TestAmerica Standard Operating Procedures. Leads the implementation and follow-up of the best practices and SOPs in the laboratory.
- Evaluates and adapts new technologies and methodologies.
- Accountable for ensuring all analysts and analyses are compliant with ISO 17025. Assists analysts with maintainance and repairs of analytical equipment to reduce down time. Performs non-routine analysis as required to meet the needs of current long-term clients or as a means to capture new clients in support of business development efforts. Consults with Project Managers and sales staff regarding analytical techniques, programs, and capabilities. Mentors associates in the laboratory.
- Manages technical projects. Prepares reports and recommendations for senior laboratory management.
- Contributes technical information and evaluation for deciding major new equipment purchases and capital expenditures.
- Reviews Quality Assurance Protocols from clients for technical anomalies or guidance that vary from routine laboratory procedures. Makes recommendations on alternate methodology or guidance for the laboratory on how to meet the requested protocols. Meets with staff from laboratory director to analysts to ensure that guidance is understood and followed throughout the projects. Provides training and development programs to applicable laboratory staff as new hires and, subsequently, on a scheduled basis. Training may include instruction on calculations and instrumentation management to include troubleshooting and preventive maintenance.
- Investigates issues raised by clients, QA, sales and other departments to find root cause and implement corrective action and proper response.
- Coordinates audit responses with Operations Managers, Department Supervisors, and QA Manager.

4.2.5 Operations Manager(s)

The Operations Manager manages and directs the analytical production sections of the laboratory. For Ohio VAP, this position is known as a Department Operations Manager. He/She manages the day-to-day operational activities of the laboratory, directly supervises exempt group leaders, and coordinates the efforts of the analytical operating groups. More specifically, he/she:

- Responsible for reporting laboratory groups compliance with health, safety and quality assurance programs.
- Coordinates the work of the laboratory groups and assists in setting work priorities in an effort to minimize costs, scheduling conflicts or delays.
- Reviews and modifies group schedules based on the current and projected workloads and staffing in each department.
- Prepares production reports and ensures status updates for each department are maintained daily.

- Assists the Technical Director with review and development of standard operating procedures and prepares recommendations as the need arises for new or revised analytical methods.
- Works with the Manager of Project Managers to schedule and direct work to achieve established sales and profit goals of the laboratory.
- Responsible for maintaining adequate staffing to meet sales and workload goals. Adjusts personnel to meet peak load demands and must support decisions in economic terms.
- Meets with group leaders daily to resolve schedule conflicts and delivery issues.
- Works with department managers and supervisors with their staffing and production efforts to ensure that the laboratory is optimizing all manpower and equipment for increased throughput as necessary.
- Develops and improves the training of all analysts in cooperation with the Technical Director and QA Manager and in compliance with regulatory requirements.

4.2.6 Department Managers

Department Managers report to the Operations Manager. Department Managers are responsible for the overall operations of a specific laboratory area. These responsibilities include but are not limited to meeting client satisfaction goals, managing the human resources with in the department and ensuring health, safety and quality assurance plan compliance is followed. Serves as a technical resource to department employees, as well as Project Managers, sales personnel, and clients. Make recommendations to laboratory management in regards to process improvements. Each one is responsible to:

- Coordinates work projects with project managers to appropriately schedule laboratory workload to meet client requirements.
- Prioritize samples for analysis to ensure OTD and TAT requirements are met.
- Supervise supervisors to ensure test procedures are in compliance with method SOP's and QA requirements and maximize productivity.
- Schedules employees in regard to workload and backlog to improve efficiency.
- Reviews data produced in assigned department and authorizes its release.
- Communicates department issues and provides status reports to Laboratory Director and Project Managers.
- Partners with laboratory management to evaluate new work opportunities and plan implementation.
- With regard to analysts, oversees management of the selection, training (as documented in Section 18.3), development of performance objectives and standards of performance, appraisal (measurement of objectives), scheduling, counseling, discipline, and motivation of analysts and documents these activities in accordance with systems developed by the QA and Personnel Departments. They evaluate staffing sufficiency and overtime needs. Training consists of familiarization with SOPs, QC, Safety, and computer systems.

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

4.2.7 Laboratory Analysts

Laboratory analysts are responsible for performing analyses and/or preps of samples using standard analytical techniques in compliance with TestAmerica's Quality Assurance Program and SOP's. Responsibilities of the analysts are listed below:

- Perform analyses by adhering to analytical and quality control protocols prescribed by current SOPs, this QA Manual, and project-specific plans honestly, accurately, timely, safely, and in the most cost-effective manner.
- Responsible for meeting TestAmerica Health and Safety guidelines as appropriate for their job functions.
- Performs routine lab tasks such as preparation of samples using standard techniques, chemical analyses using standard techniques, calculating data, recording and reporting unusual test occurences to the supervisor, routine maintenance of instruments and conforming to all lab QA/QC policies.
- Completion of initial demonstration of capability and maintenance of ongoing demonstration of capability.
- Repsonsible for general housekeeping and maintenance in his/her work area.

- Perform 100% review of the data generated prior to entering and submitting for secondary level review.
- Suggest method improvements to their Department Manager, the Technical Director, and the QA Manager. These improvements, if approved, are incorporated. Ideas for the optimum performance of their assigned area, for example, through the proper cleaning and maintenance of the assigned instruments and equipment, are encouraged.
- Work cohesively as a team in their department to achieve the goals of accurate results, optimum turnaround time, cost effectiveness, cleanliness, complete documentation, and personal knowledge of environmental analysis.

4.2.8 Environmental Health & Safety (EH&S) Manager

The EH&S Manager reports to the Laboratory Director and ensures that systems are maintained for the safe operation of the laboratory. For Ohio VAP, this is position is the Health & Safety Manager/Coordinator. The EH&S Manager is responsible to:

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

4.2.9 Manager of Project Management

The Manager of Project Management reports to the Client Services Director and serves as the interface between the laboratory’s analytical departments and the laboratory’s clients. For Ohio VAP, this position is the Project Management Operations Manager. 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 PM team.
- Technical liaison for the PM team.
- Human resource management of the PM team.
- Responsible to ensure that clients receive the proper sampling supplies.
- Coordinating status meetings with Department Managers.
- 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 department managers of incoming projects and sample delivery schedules.
- Accountable to clients for communicating sample progress/status.
- Responsible for discussing with client any project-related problems, resolving service issues, and coordinating technical details with the laboratory staff.
- Responsible for staff familiarization with specific quotes, sample log-in review, and final report completeness.
- Monitor the status of all data package projects in-house to ensure timely and accurate delivery of reports.
- Inform clients of data package-related problems and resolve service issues.
- Coordinate requests for sample containers and other services (data packages).

4.2.10 Project Managers

The Project Management team consists of Project Managers who report to the Manager of Project Management. The Project Managers are responsible to:

- Monitor analytical and QA project requirements for a specified project
- Ensure that client specifications, when known, are met by communicating project and quality assurance requirements to the laboratory.
- Notify laboratory personnel of incoming projects and sample delivery schedules.
- Be accountable to clients for communicating sample progress/status.
- Ensure that clients receive the proper sampling supplies.
- Coordinate subcontract work.
- Inform clients of project related problems, resolving service issues, and coordinating technical issues with the laboratory staff.
- Provide assistance to clients regarding resolution of problems concerning COC.
- Monitor the status of all projects in-house to ensure timely delivery of reports.
- Perform review of projects for completeness and compliance to client needs prior to release to client.
- Generate final laboratory reports and has signature authority (where approved).

4.2.11 Sample Receipt/Control Manager and Shipping Department Manager

The Sample Receipt/Control Manager and Shipping Department Manager reports to the Laboratory Director. He/She:

- Coordinates sample log-in to LIMS.
- Manages sample labeling.
- Manages the appropriate storage of samples.
- Oversees the preparation of bottle kits.
- Coordinates the proper storage, packing, and disposal of laboratory wastes according to Department of Transportation (DOT) and Resource Conservation and Recovery Act (RCRA) regulations.
- Manages the shipment of subcontracted samples to designated laboratories.
- Schedules waste pick-ups and prepare paperwork according to DOT, RCRA regulations and Treatment, Storage, Disposal Facility (TSDF) requirements.
- Maintains waste disposal records (manifests, land ban forms, and certificates of destruction, POTW reports/documentation) in a stand-alone and legally defensible fashion.
- Coordinates hazardous waste minimization program to meet regulatory requirements.
- Conducts weekly inspections of satellite accumulation areas and all hazardous waste storage areas. Submits reports to area leaders, hazardous waste representatives, and Environmental Health and Safety (EH&S) Coordinator. Ensures corrective actions noted are completed.
- Labpacks expired chemicals for disposal according to regulatory requirements.
- Tracks volume of waste generated for reporting to corporate and EPA.
- Prepares and tracks implementation of Waste Minimization Plan.
- Maintains the inventory control system for all departments.
- Receives and distributes incoming supplies to appropriate departments.
- Maintains bottle and cooler inventory.
- Performs daily facility maintenance activities.
- Performs routine preventative maintenance throughout facility.
- Performs preventative maintenance on company vehicles.
- Contacts outside contractors as necessary to repair/maintain items outside the realm of reasonable maintenance.
- Oversees storage facilities, files, and outside storage.
- Monitors vendor service and responds appropriately to any problems.
- Completes all controlled purchases.

4.3 Deputies

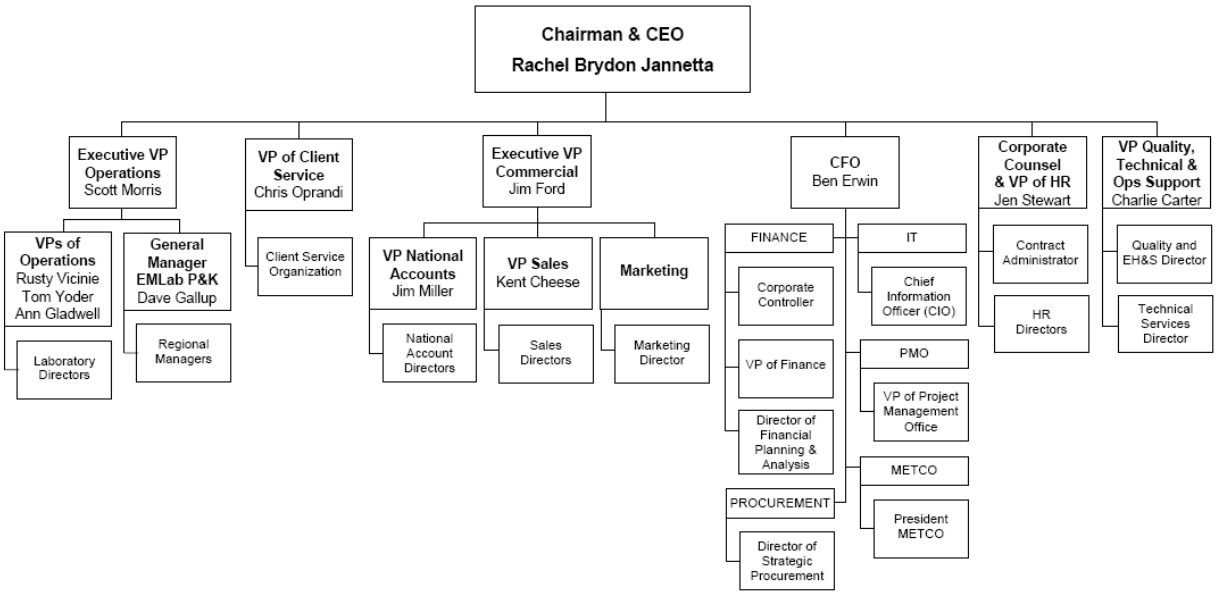
The following table defines who assumes the responsibilities of key personnel in their absence:

Table 4-1. Deputies

Key Personnel	Primary Deputy	Support Deputy
Laboratory Director	Technical Director	QA Manager Operations Manager
QA Manager	Laboratory Director	Technical Director
Technical Director	Laboratory Director	QA Manager Operations Managers
EHS Manager	Purchasing Manager	Laboratory Director

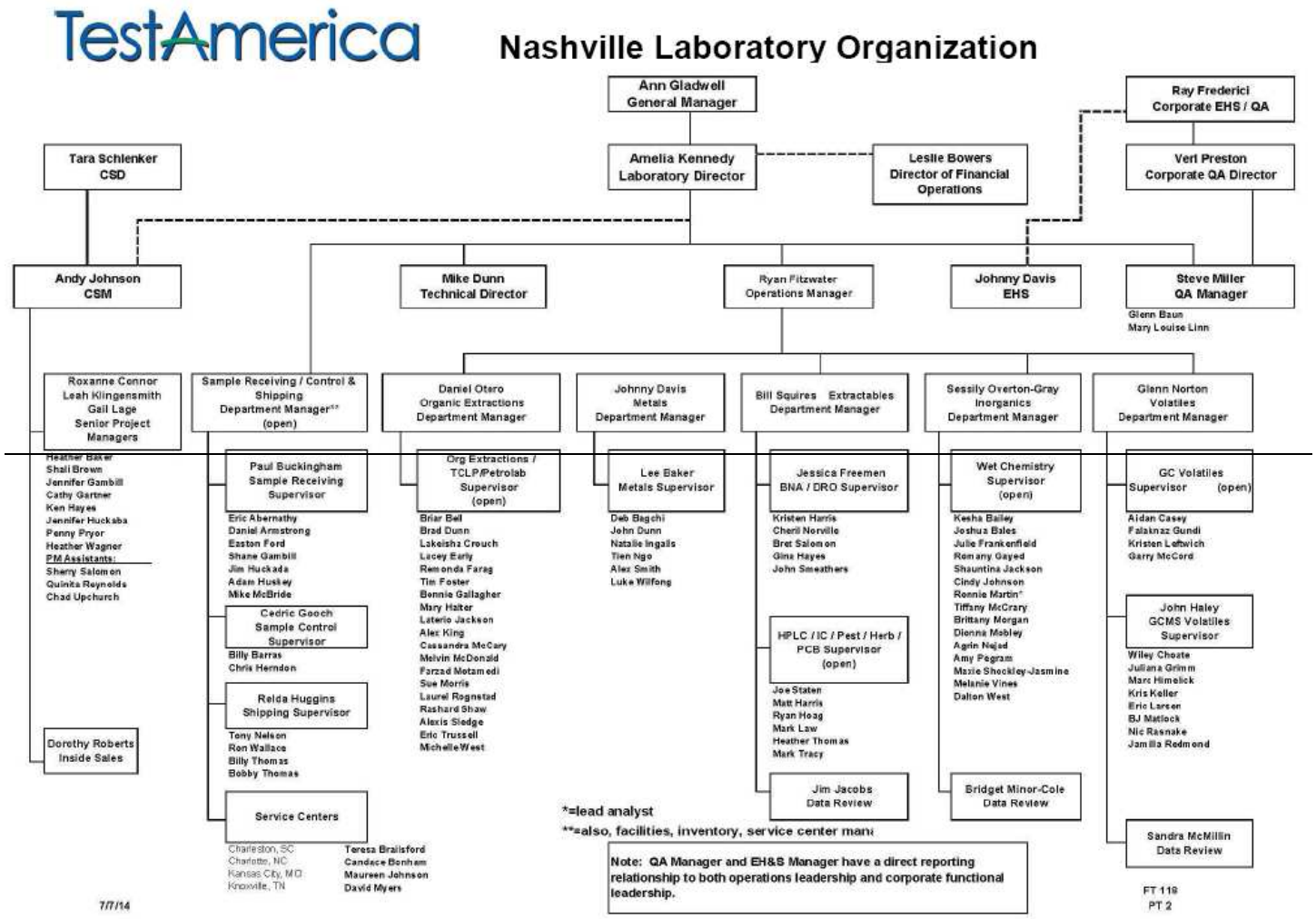
Figure 4-1. Corporate and Laboratory Organization Charts

Corporate Organization Chart



Note: Subject to change.

Laboratory Organization Chart



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 continually improve the effectiveness of the management system.

Every staff member at TestAmerica Nashville 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 CW-L-S-002).
- Procedures and guidance for recalling data if necessary (Corporate SOP CW-L-S-002).
- An effective external and internal monitoring system that includes procedures for internal audits (Section 16).
- Production of results which are accurate and include QA/QC information that meets a client's pre-defined Data Quality Objectives (DQOs).
- Presentation of services in a confidential, honest, and forthright manner.
- Provision of employees with guidelines and an understanding of the ethical and quality standards of our industry.

- Operation our facilities in a manner that protects the environment and the health and safety of employees and the public.
- Adherence to all pertinent federal, state, and local laws and regulations and encourage other members of our industry to do the same.
- Education clients as to the extent and kinds of services available.
- Assertion of competency only for work for which adequate personnel and equipment are available and for which adequate preparation has been made.
- Promotion of 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 laboratory-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 Memoranda

5.3.1 Order of Precedence

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

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

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

5.4 QA/QC Objectives for the Measurement of Data

Quality Assurance (QA) and Quality Control (QC) are activities undertaken to achieve the goal of producing data that accurately characterize the sites or materials that have been sampled. Quality Assurance is generally understood to be more comprehensive than Quality Control.

Quality Assurance can be defined as the integrated system of activities that ensures that a product or service meets defined standards.

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

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

Historically, laboratories have described their QC objectives in terms of precision, accuracy, representativeness, comparability, completeness, selectivity, and sensitivity (PARCCSS).

5.4.1 Precision

The laboratory objective for precision is to meet the performance for precision demonstrated for the methods on similar samples and to meet data quality objectives of the EPA and/or other regulatory programs. Precision is defined as the degree of reproducibility of measurements under a given set of analytical conditions (exclusive of field sampling variability). Precision is documented on the basis of replicate analysis, usually duplicate or matrix spike (MS) duplicate samples.

5.4.2 Accuracy

The laboratory objective for accuracy is to meet the performance for accuracy demonstrated for the methods on similar samples and to meet data quality objectives of the EPA and/or other regulatory programs. Accuracy is defined as the degree of bias in a measurement system. Accuracy may be documented through the use of laboratory control samples (LCS) and/or MS. A statement of accuracy is expressed as an interval of acceptance recovery about the mean recovery.

5.4.3 Representativeness

The laboratory objective for representativeness is to provide data which is representative of the sampled medium. Representativeness is defined as the degree to which data represent a characteristic of a population or set of samples and is a measurement of both analytical and field sampling precision. The representativeness of the analytical data is a function of the procedures used in procuring and processing the samples. The representativeness can be documented by the relative percent difference between separately procured, but otherwise identical samples or sample aliquots.

The representativeness of the data from the sampling sites depends on both the sampling procedures and the analytical procedures. The laboratory may provide guidance to the client regarding proper sampling and handling methods in order to assure the integrity of the samples.

5.4.4 Comparability

The comparability objective is to provide analytical data for which the accuracy, precision, representativeness, and reporting limit statistics are similar to these quality indicators generated by other laboratories for similar samples and data generated by the laboratory over time.

The comparability objective is documented by inter-laboratory studies carried out by regulatory agencies or carried out for specific projects or contracts by comparison of periodically generated statements of accuracy, precision, and reporting limits with those of other laboratories.

5.4.5 Completeness

The completeness objective for data is 90% (or as specified by a particular project) expressed as the ratio of the valid data to the total data over the course of the project. Data will be considered valid if they are adequate for their intended use. Data usability will be defined in a QAPP, project scope, or regulatory requirement. Data validation is the process for reviewing data to determine its usability and completeness. If the completeness objective is not met, actions must be taken internally and with the data user to improve performance. This may take the form of an audit to evaluate the methodology and procedures as possible sources for the difficulty or may result in a recommendation to use a different method.

5.4.6 Selectivity

Selectivity is defined as: The capability of a test method or instrument to respond to a target substance or constituent in the presence of non-target substances. Target analytes are separated from non-target constituents and subsequently identified/detected through one or more of the following, depending on the analytical method: extractions (separation), digestions (separation), interelement corrections (separation), use of matrix modifiers (separation), specific retention times (separation and identification), confirmations with different columns or detectors (separation and identification), specific wavelengths (identification), specific mass spectra (identification), specific electrodes (separation and identification), etc.

5.4.7 Sensitivity

Sensitivity refers to the amount of analyte necessary to produce a detector response that can be reliably detected (Method Detection Limit) or quantified (Reporting Limit).

5.5 Criteria for Quality Indicators

Unless otherwise noted, limits within LIMS are laboratory generated. Some acceptability limits are derived from U.S. EPA methods when they are required. Where U.S. EPA method limits are not required, TestAmerica Nashville has developed limits from evaluation of data from similar matrices. Criteria for development of control limits are contained in Section 24.

5.6 Statistical Quality Control

Statistically-derived precision and accuracy limits are required by selected methods (such as SW-846) and programs. TestAmerica Nashville routinely utilizes statistically-derived limits to evaluate method performance and determine when corrective action is appropriate. The analysts use the current limits in the laboratory (dated and approved by the Technical Director and QA Manager) and entered into the Laboratory Information Management System (LIMS). The Quality Assurance department maintains an archive of all limits used within the laboratory (see SOP *Archiving of Technical and Quality Department Paper Records* / NV08-194). If a method defines the QC limits, the method limits are used.

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

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 re-analyzed or re-extracted/re-analyzed or if a comment is to be added to the report explaining the reason for the QC outlier.

5.6.1 QC Charts

As the QC limits are calculated, QC charts are generated showing warning and control limits for the purpose of evaluating trends. The QA Manager evaluates these to determine if adjustments need to be made or for corrective actions to methods. All findings are documented and kept on file.

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 company intranet site. Printed copies are considered uncontrolled unless the laboratory physically distributes them as controlled documents. A detailed description of the procedures for issuing, authorizing, controlling, distributing, and archiving corporate documents is found in Corporate SOP CW-Q-S-001, *Corporate Document Control and Archiving*. The laboratory's internal document control procedure is defined in the following laboratory SOPs: *Document Control* / NV08-152, *Archiving of Technical and Quality Department Paper Records* / NV08-194, and *Organization of Quality Department Documents* / NV08-220.

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

The laboratory maintains control of records for raw analytical data and supporting records such as audit reports and responses, logbooks, standard logs, training files, MDL studies, Proficiency Testing (PT) studies, certifications and related correspondence, and corrective action reports. Raw analytical data may consist 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 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 department is responsible for the maintenance of the system.

Controlled documents are authorized by the QA Department and other management. In order to develop a new document, a manager submits a paper or electronic draft to the QA Department for suggestions and approval before use. Upon approval, QA personnel add the identifying version information to the document and retain that document as the official document on file. That document is then provided as needed 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 are reviewed at a minimum of every two years except for drinking water SOPs which are reviewed every year and revised as appropriate. Changes to documents occur when a procedural change warrants a revision of the document.

6.3 Procedures for Document Control Policy

For changes to the QA Manual, refer to SOP Document Control / NV08-152. Uncontrolled copies must not be used within the laboratory. Previous revisions and back-up data are stored by the QA department. Electronic copies are stored on the QA server.

For changes to SOPs, refer to SOP No. CW-Q-S-002, *Writing a Standard Operating Procedure (SOP)* and SOP *Document Control* / NV08-152. The QA Department has a complete file of all current and previous versions, showing changes, of each SOP. Additionally, there are controlled notebooks of current SOPs in the lab. These are updated by the QA department. There is a table of contents. Electronic versions of current, previous, and in-transition SOPs are maintained on a QA hard drive that is backed-up weekly. Current, unchangeable versions are stored on the QA server/SOPs.

Changes to facilities, the QA Manual, certifications, personnel, safety/health, and capabilities are documented in the Management of Change log as prescribed in Section 14.

Forms, worksheets, work instructions and information are organized on the QA server. There is a table of contents. The procedure for the care of these documents is in SOP *Document Control* / NV08-152.

Reference books, regulations, and other external protocols are listed, with location, in the QA office. This list is updated as needed.

Logbooks and preparation worksheets are initialized and stored in an archiving system described in SOP *Archiving of Technical and Quality Department Paper Records* / NV08-194 for easy tracking and retrieval.

Certification correspondence, audit reports and responses, control charts, MDLs, training files, subcontractor credentials, and PT studies are stored in the QA office in appropriate files and/or on the QA server. These documents are not uniquely identified.

6.4 Obsolete Documents

The laboratory has specific procedures (as described above) to ensure that all invalid or obsolete documents are removed, or otherwise prevented from unintended use. In general, obsolete documents are collected from employees according to distribution lists and are marked obsolete on the cover or destroyed. At least one copy of the obsolete document is archived as described in SOP *Document Control* / NV08-152.

SECTION 7. SERVICE TO THE CLIENT

7.1 Overview

TestAmerica Nashville 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.

A thorough review of technical and QC requirements contained in contracts is performed to ensure project success. The appropriateness of requested methods, and the laboratory's capability to perform them must be established. Projects, proposals, and contracts are reviewed for adequately defined requirements and TestAmerica Nashville's capability to meet

those requirements. Alternate test methods that are capable of meeting the clients' requirements may be proposed by the laboratory. A review of the laboratory'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 (method detection and reporting limits), 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 determines 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 and intends to subcontract such services (whether to another TestAmerica facility or to an outside firm), then the subcontracted services must 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 laboratory to complete the work satisfactorily. Any discrepancy between the client's requirements and TestAmerica's ability to meet those requirements must be resolved in writing before acceptance of the contract. It is necessary that the contract be acceptable to both the laboratory and the client. Amendments initiated by the client and/or TestAmerica are documented in writing.

All contracts, QAPPs, Sampling and Analysis Plans (SAPs), contract amendments, and documented communications become part of the project record.

The same contract review process used for the initial review is repeated when there are amendments to the original contract by the client, and the participating personnel are informed of the changes.

7.2 Review Sequence and Key Personnel

Appropriate personnel must 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 laboratory has the capacity to meet the clients turn around needs. Project Managers contact the appropriate laboratory management or corporate personnel if any of the details are unknown, differ from what standard payment policy and insurance coverage includes, or if the task appears to fall outside the Project Manager's job responsibilities.

For new, complex, or large projects, the proposed contract is given to the Director of National Accounts, who decides which laboratory 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 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 the scope of the contract, to evaluate all of the requirements shown above (not necessarily in the order below):

- Contract Administrator
- VP of Operations
- Manager of Project Management
- Laboratory Project Manager
- Laboratory and/or Corporate Technical 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 fulfills the review requirements.

The Contracts Department maintains copies of all 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. Information is stored in the LIMS system or on the server.

The contract will be maintained on the contracts drive for Nashville:
\\corp-fs-04.tai.com\Contracts_Management\Nashville Contract Docs

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 correspondence records (i.e., e-mail) and a phone log of conversations with the client. These records are archived by the laboratory facility for a minimum of five years.

7.3.1 Project-Specific Quality Planning

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

PMs are the primary client contact and they ensure resources are available to meet project requirements. Although PMs 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/supervisors at these meetings. The laboratory staff is then introduced to the modified requirements via the individual department managers/supervisors. After the modification is implemented into the laboratory process, documentation of the modification is made in the case narrative of the data report(s).

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

7.4 Special Services

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

Note: ISO/IEC 17025 states that a laboratory "shall afford clients or their representatives cooperation to clarify the client's request". This topic is discussed in Section 7.

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

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

7.5 Client Communication

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

The Technical Director or QA Manager is available to discuss any technical questions or quality 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. Client comments are also collected from the TotalAccess public website.

SECTION 8. SUBCONTRACTING OF TESTS

8.1 Overview

For the purpose of this quality manual, the phrase subcontracting laboratory refers to a laboratory external to the TestAmerica laboratory network. The phrase "work sharing" refers to internal transfers of samples between 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 the SOP on Subcontracting Procedures (CA-L-S-002).

When outsourcing analytical services, the PM or other laboratory staff must assure, to the extent necessary, that the subcontracting 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 subcontracting facility. Additionally, work requiring accreditation is placed with an appropriately accredited laboratory. The laboratory performing the subcontracted work is identified in the final report, as is non-TNI accredited work where required.

Project Managers (PMs), Manager of Project management, or Account Executives (AEs) for the Export Laboratory (i.e., the TestAmerica laboratory transferring samples to a nother laboratory) are responsible for obtaining client approval prior to subcontracting any samples. The laboratory will advise the client of a subcontracting arrangement in writing and when possible approval from the client is retained in the project folder. Standard TestAmerica Terms & Conditions include the flexibility to subcontract samples within the TestAmerica laboratory network. 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 regulatory agencies (e.g., USDA) or contracts may require notification prior to placing such work.

8.2 Qualifying and Monitoring Subcontractors

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

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

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

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 the 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.

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

8.2.1 Once the appropriate accreditation and legal information is received by the laboratory, it is evaluated for acceptability (where applicable) by laboratory management and forwarded to the Corporate Quality Information Manager (QIM) for review. Once all documents are reviewed for completeness, the Corporate QIM will forward the documents to the Purchasing Manager for formal signature and contracting with the laboratory. The approved vendor will be added to the approved subcontractor list on the intranet site and the finance group is concurrently notified for JD Edwards.

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

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

- Complaints shall be investigated. Documentation of the complaint, investigation, and corrective action is 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 subcontracting laboratories.
- Subcontractors in good standing are retained on the intranet listing. The QA Manager will notify all TestAmerica laboratories, Corporate Quality, and Corporate Contracts if any laboratory requires removal from the intranet site. This notification is posted on the intranet site and e-mailed to all Laboratory Directors, QA Managers, and Sales Personnel.

8.3 Oversight and Reporting

The PM must request that the selected subcontractor be presented with a subcontract if one is not already executed between the laboratory and the subcontractor. The subcontract must include terms which reflect the requirements of our clients, either in the subcontract itself or through the mechanism of work orders relating to individual projects. A standard subcontract

and the Laboratory Subcontractor Vendor Package (posted on the intranet) can be used to accomplish this, and Corporate Counsel can tailor the document or assist with negotiations, if needed. The PM (or EDs, AEs, or CSM, etc.) responsible for the project must advise and obtain client consent to the subcontract as appropriate and provide the scope of work to ensure that the proper requirements are made a part of the subcontract and are made known to the subcontractor.

Prior to sending samples to the subcontracting laboratory, the PM confirms their certification status to determine if it is current and scope-inclusive. The information is documented on a Subcontracted Sample Form (Figure 8-1) and the form is retained in the project folder. For TestAmerica laboratories, certifications can be viewed on the company's Total Access Database.

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

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

Through communication with the subcontracting 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 subcontracting laboratories are not altered and are included in their original form in the final project report. The added report clearly identifies the data as being produced by a subcontracting facility. If subcontracting laboratory data is incorporated into the laboratory's EDD (i.e., imported), the report must explicitly indicate which laboratory 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 laboratory are identified on the final report. The report must explicitly indicate which laboratory produced the data for which methods and samples. The final report must include a copy of the completed COC for all work sharing reports.

8.4 Contingency Planning

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

with TestAmerica at this time. The comprehensive approval process must then be initiated within 30 calendar days of subcontracting.

Figure 8-1.

Subcontracted Sample Form

Date/Time: _____

Subcontracted Laboratory Information:

- Subcontractor's Name: _____
- Subcontractor Point of Contact: _____
- Subcontractor's Address: _____
- Subcontractor's Phone: _____
- Analyte/Method: _____
- Certified for State of Origin: _____
- NELAC Accredited: Yes_____No_____
- A2LA (or ISO 17025) Accredited: Yes_____No_____
- AIHA Accredited: Yes_____No_____
- CLP-like Required: (Full doc required) Yes_____No_____
- Requested Sample Due Date: (Must be put on COC) _____
- Client POC Approval on-file to Subcontract Samples to Sub-Laboratory: Yes_____No_____

Project Manager: _____

Laboratory Sample # Range: _____
(Only of Subcontracted Samples)

Laboratory Project Number (Billing Control #): _____

All subcontracted samples are to be sent via bonded carrier and Priority Overnight. Please attach tracking number below and maintain these records in the project files.

PM Signature_____**Date**_____

SECTION 9. PURCHASING SERVICES AND SUPPLIES

9.1 Overview

Capital expenditures are normally made in accordance with TestAmerica's Corporate SOP *Capital Expenditure, Controlled Purchase Requests, and Fixed Asset Capitalization*, CW-F-S-007. TestAmerica Nashville may approve some expenditures according the guidance given in SOP *Purchasing* / NV08-232

Evaluation and selection of suppliers and vendors are 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.

Contracts are signed in accordance with TestAmerica's *Company-Wide Authorization Matrix Policy*, CW-F-P-002. Requests 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 by the manufacturer or according to laboratory procedure. Pyrex (or equivalent) glass is used where possible.

9.3 Reagents, Standards, and Supplies

Purchasing guidelines for equipment and reagents must meet the requirements of the specific method and testing procedures for which they are being purchased. Standards should be purchased through ISO Guide 34 compliant vendors. Solvents and acids are pre-tested in accordance with Corporate SOP on *Solvent & Acid Lot Testing & Approval*, SOP No. CA-Q-S-001.

9.3.1 Purchasing

Chemical reagents, solvents, glassware, and general supplies are ordered as needed to maintain sufficient quantities on hand. Materials used in the analytical process must be of a known quality. The wide variety of materials and reagents available makes it advisable to specify recommendations for the name, brand, and grade of materials to be used in any determination. This information is contained in the method SOP. The analyst may check the item out of the on-site consignment system that contains items approved for laboratory use.

Any substitution of items requires the approval of the Technical Director, QA Manager, or Laboratory Director, using a Supply Item Substitution Approval Form as described in SOP *Reagent and Standard Purchase, Preparation, Control Documentation* / NV08-214. Completed Supply Item Substitution forms are filed with the Purchasing Manager. The department managers and the Purchasing Manager place the orders. See SOP *Purchasing* / NV08-232 for further details.

9.3.2 Receiving

It is the responsibility of the Purchasing Manager or Operations Manager to acknowledge receipt the shipment into J.D. Edwards. 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. 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, reagent grade will be used. It is the responsibility of the analyst to check the procedure carefully for the suitability of grade of reagent.

Chemicals must not be used past the manufacturer's expiration date and must not be used past the expiration time noted in a method SOP or the SOP Reagent and Standard Purchase, Preparation, Control Documentation / NV08-214. 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 must not be used past the manufacturer's or SOP's expiration date unless 'verified' (refer to the 2nd bulleted item 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 by the department manager.

Wherever possible, standards must be traceable to national or international standards of measurement or to national or international reference materials (i.e., 9001, IT024, and ISO 34 as applicable). Records to that effect must be available to the user.

Compressed gases in use are checked for pressure and secure positioning daily. The minimum total pressure must be 500 psig or the tank must be replaced. To prevent a tank from going to dryness or introducing potential impurities, close observation of the tank gauge must take place as pressure decreases toward 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 be free of target analytes at the concentration of the MDL.

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 must be documented.

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

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

Records of manufacturer's certification and traceability statements are maintained in files or binders in each laboratory section or electronically. 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 a previous one for the same purpose and that no difference is noted, unless approved and so documented by the Technical Director, Quality Assurance Manager, or Corporate.

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

Upon receipt of a new or used piece of equipment, an identification name is assigned and added to the equipment list that is maintained by the QA Department, and IT must be notified so that it can be linked to the server 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 (see

Section 19). For software, its operation must be deemed reliable and evidence of instrument verification must be retained at the instrument and in the QA Department. Software certificates supplied by the vendors are filed with the IT department. The manufacturer's operation manual is retained at the bench.

9.5 Services

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

9.6 Suppliers

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

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

Any issues of vendor performance are to be reported immediately by the laboratory staff to the Corporate Purchasing Group by completing a *Vendor Performance Report* (CW-F-WI-009).

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

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

The laboratory has access to a master 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 to be used for technical purchases must also undergo vendor evaluation as described in laboratory SOP *Purchasing* / NV08-232.

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 Corporate QA Department and/or the Corporate Technical Services Director are consulted with vendor and product selection that have an impact on quality.

SECTION 10. COMPLAINTS

10.1 Overview

TestAmerica Nashville 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 stands behind its data, service obligations, and products.

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

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

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

The process of complaint resolution and documentation utilizes the procedures outlined in Section 12 (Corrective Actions) and is documented in a non-conformance database or in a Corrective Action Report (CAR).

10.2 External Complaints

An employee that receives a complaint initiates the complaint resolution process and the documentation of the complaint.

Complaints fall into two categories: correctable and non-correctable. An example of a correctable complaint would be one where a report re-issue would resolve the complaint. An example of a

non-correctable complaint would be one where a client complains that their data was repeatedly late. Non-correctable complaints are 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 Information Technology (IT) may initiate a complaint by contacting the laboratory or through the corrective action system described in Section 12.

10.4 Management Review

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

SECTION 11. CONTROL OF NON-CONFORMING WORK

11.1 Overview

When data discrepancies are discovered or deviations and departures from laboratory standard procedures, policies, and/or client requests have occurred, corrective action is taken immediately. First, the laboratory evaluates the significance of the nonconforming work and generates a non-conformance memo (NCM) in LIMS, and notifies laboratory management (using the “Nashville-Mgr411” emailing group). Then, an 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 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 NCM and/or corrective action system (refer to Section 12).

Due to the frequently unique nature of environmental samples, sometimes departures from documented policies and procedures are needed. When an analyst encounters such a situation, the problem is presented to the supervisor for resolution. The supervisor may elect to discuss it

with the Technical Director or have a representative contact the client to decide on a logical course of action. Once an approach is agreed upon, the analyst documents it using the laboratories NCM system described in Section 13. This information can then be supplied to the client in the form of a footnote or a case narrative with the report. [**Note:** For Ohio VAP, the laboratory must not deviate from approved SOPs or the QA Manual.]

Project Management may encounter situations where a client may request that a special procedure be applied to a sample that is not standard laboratory practice. Based on a technical evaluation, the laboratory 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 laboratory does not normally report. The laboratory would not have validated the method for this compound following the procedures in Section 19. The client may request that the compound be reported based only on the calibration. Such a request would need to be approved by the Technical 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.

On a monthly basis, the laboratory management team reviews the non-conformances to determine if any trends are present. If trends are found, such as repeated occurrences, further corrective action is taken to eliminate the reoccurrences as outline in Section 12.

11.2 Responsibilities and Authorities

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

Under certain circumstances the Laboratory Director, the Technical Director, a Department Manager, or the QA Manager may authorize departures from documented procedures or policies. The departures may be a result of procedural changes due to the nature of the sample; a one-time procedure for a client; QC failures with insufficient sample to reanalyze, etc. In most cases, the client must 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 need to be documented in logbooks and/or data review checklists and in LIMS, 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 Director. The reporting of issues involving alleged violations of the company's data integrity or manual integration procedures must be conveyed to an Ethics and Compliance Officer (ECO), the Director of Quality & Client Advocacy, and Corporate 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 Department Manager or Operations Manager must notify the QA Manager, Technical Director, and Laboratory Director when the reporting of inaccurate data has been confirmed.

The Laboratory Director, QA Manager, Ethics and Compliance Officers, Corporate Quality, Executive VP of Operations, VP of Operations, and the Corporate 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

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

11.4 Prevention of Non-Conforming Work

If it is determined that the nonconforming work could recur, corrective actions must be made following the laboratory's corrective action system.

On a monthly basis, the Senior Management team evaluates non-conformances to determine if any nonconforming work has been repeated multiple times. If so, the laboratory's corrective action process is 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 must be respected, the problem and the required corrective and preventive action must be stated in writing and presented to the Laboratory Director.

The Laboratory Director must arrange for the appropriate personnel to meet with the QA Manager as needed. This meeting must be held to confirm that there is a problem, that suspension/restriction of the method is required and must be concluded with a discussion of the steps necessary to bring the method/target or test fully back on line. In some cases a formal group meeting 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 also initiates a corrective action report as described in Section 12 if one has not already been started. A copy of any meeting notes and agreed upon steps should be faxed or e-

mailed by the laboratory to the appropriate General Manager and member of Corporate QA. This fax/e-mail acts as notification of the incident.

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

Within 72 hours, the QA Manager determines if compliance is now met and reports can be released, OR determines the plan of action to bring work into compliance, and releases work. A team, with all principals involved (Laboratory Director, Technical Director, QA Manager, Department Manager) can devise a start-up plan to cover all steps from client notification through compliance and release of reports. Project Management and the Director of Client Services and Sales and Marketing are 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. Approval is given by final signature on the completed corrective action report.

SECTION 12. CORRECTIVE ACTION

12.1 Overview

A major component of TestAmerica's Quality Assurance (QA) Program is the problem investigation and feedback mechanism designed to keep the laboratory staff informed on quality related issues and to provide insight to problem resolution. When nonconforming work or departures from policies and procedures in the quality system or technical operations are identified, the corrective action procedure provides a systematic approach to assess the issues, restore the laboratory's system integrity, and prevent reoccurrence. Corrective actions are documented using Non-Conformance Memos (NCM) and Corrective Action Reports (CAR) (refer to Figure 12-1).

12.2 General

Problems within the quality system or within analytical operations may be discovered in a variety of ways, such as QC sample failures, internal or external audits, proficiency testing (PT) performance, client complaints, staff observation, etc.

The purpose of a corrective action system is to:

- Identify non-conforming events and assign responsibility for investigation.
- Resolve non-conforming events and assign responsibility for any required corrective action.
- Identify systematic problems before they become serious.
- Identify and track client complaints and provide resolution (see more on client complaints in Section 11).

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 versus manufacturer's packing slips

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

- Questionable trends that are found in the review of NCMs
- Issues found while reviewing NCMs that warrant further investigation
- Internal and external audit findings
- Failed or unacceptable PT results
- Corrective actions that cross multiple departments in the laboratory
- Systematic reporting / calculation errors
- Client complaints
- Data recall investigations
- Identified poor process or method performance trends
- Excessive revised reports
- Health and safety violations

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

12.3 Closed Loop Corrective Action Process

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

12.3.1 Cause Analysis

- Upon discovery of a non-conformance event, the event must be defined and documented. An NCM or CAR must be initiated, someone is assigned to investigate the issue and the event is investigated for cause. Table 12-1 provides some general guidelines on determining responsibility for assessment.
- The cause analysis step is the key to the process since a long-term corrective action cannot be determined until the cause is determined

- If the root cause is not obvious, the Department Manager, Technical Director, 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 must 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 must 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 must document and implement the changes. The NCM or CAR is used for this documentation.

12.3.3 Root Cause Analysis

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

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 Director, Department Manager/Supervisor, and QA Manager are responsible to ensure that the corrective action taken was effective.
- Ineffective actions are documented and re-evaluated until acceptable resolution is achieved. Department Managers are accountable to the Laboratory Director to ensure final acceptable resolution is achieved and documented appropriately.
- Each NCM and CAR is entered into a database for tracking purposes, and a monthly summary of all corrective actions is printed out for review to aid in ensuring that the corrective actions have taken effect.

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

12.3.5 Follow-up Audits

- Follow-up audits may be initiated by the QA Manager and are 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 accomplished through the use of an NCM or CAR.

Table 12-1 includes examples of general technical corrective actions. For specific criteria and corrective actions refer to the analytical methods or method-specific 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 must be treated if associated QC measurements are unacceptable. Specific procedures are included in method SOPs, work instructions, and QAM Sections 19 and 20). All corrective actions are reviewed monthly, at a minimum, by the QA Manager and highlights are included in the QA monthly report.

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

12.5 Basic Corrections

When mistakes occur in records, each mistake must be crossed-out, and not erased, deleted, made illegible, or otherwise obliterated (e.g., no white-out), and the correct value entered alongside the original value. All such corrections must be initialed (or signed) and dated by the person making the correction (see Figure 12-2). 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 original initial/date 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) must also be documented.

Figure 12-1. Corrective Action Report

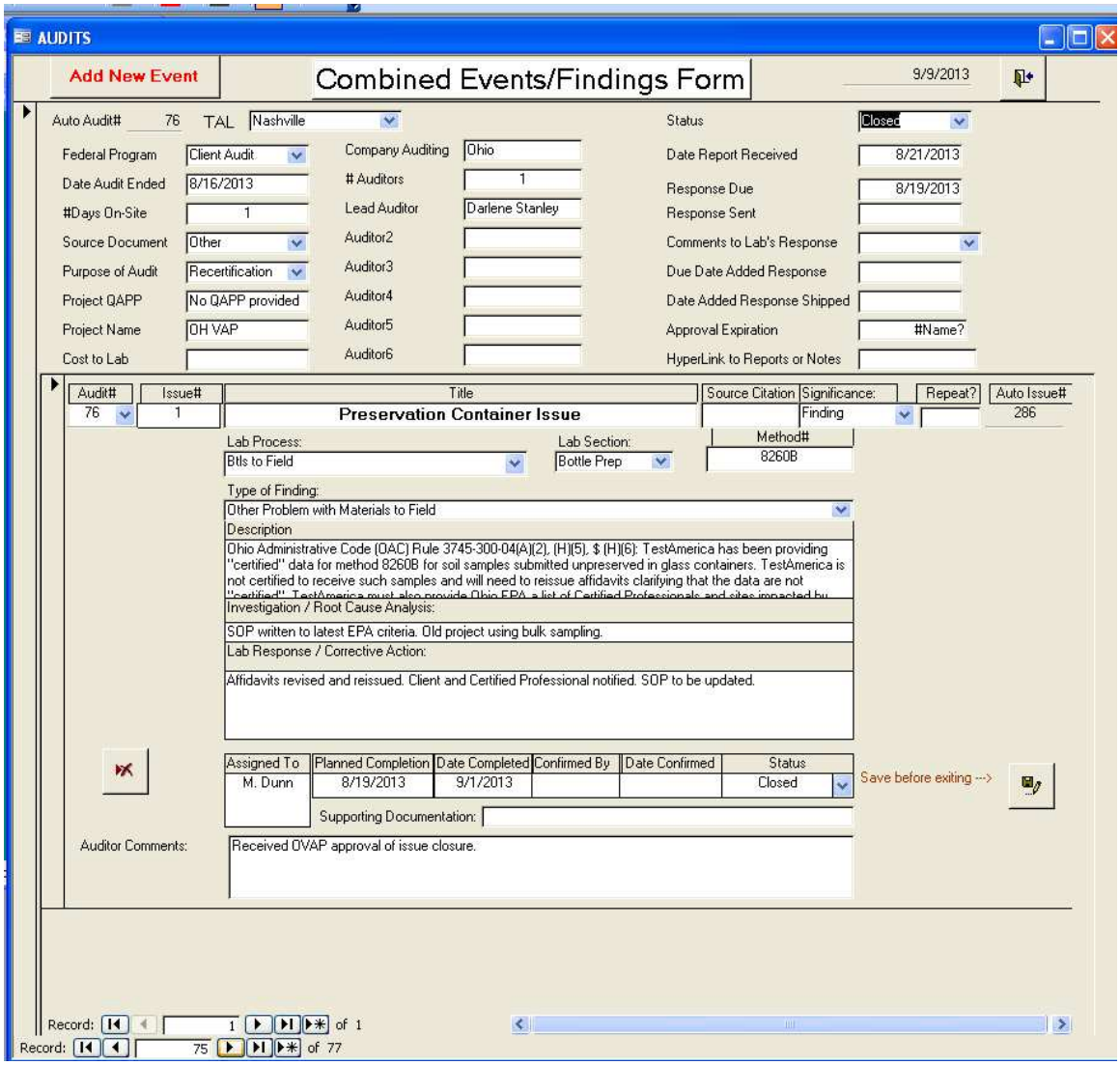


Figure 12-2. Example of Acceptable Error Correction

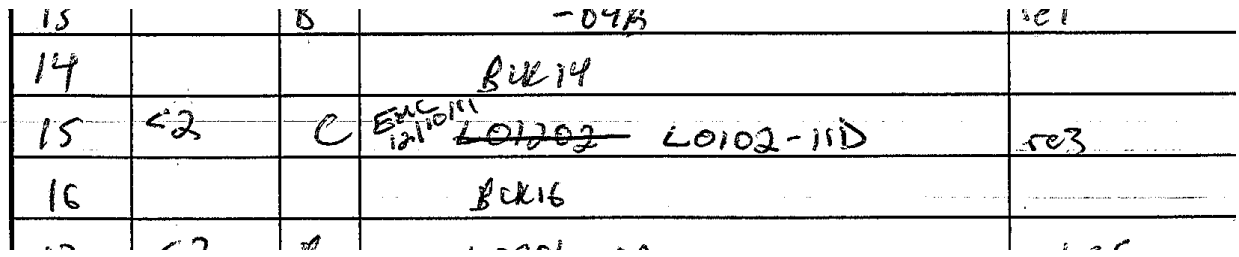


Table 12-1. General Corrective Action Procedures

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, Department Manager)*	- Correlation coefficient r ≥ 0.995 (r2 ≥ 0.990) 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, Department Manager)*	- % Recovery within control limits.	- Remake once 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 once. - If still unacceptable, then recalibrate and rerun affected samples.
Matrix Spike / Matrix Spike Duplicate (MS/MSD) *(Analyst, Data Reviewer)*	- % Recovery within limits documented in the LIMS	- If the acceptance criteria for duplicates or matrix spikes are not met because of matrix interferences, the acceptance of the analytical batch is determined by the validity of the LCS. - If both MS and MSD are similarly outside acceptable limits and the LCS is within acceptable limits, the batch is acceptable - If one analysis of the MS/MSD pair is within acceptable limits and the other is outside acceptable limits, repeat the analysis exhibiting unacceptable results. - 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. Note: It is acceptable to not report MS/MSD results for projects when a sample from that project was not spiked.

QC Activity *(Individual Responsible for Initiation/Assessment)*	Acceptance Criteria	Recommended Corrective Action
Laboratory Control Sample (LCS) *(Analyst, Data Reviewer)*	- % Recovery within limits specified in LIMS	- 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: This may not be used for the Ohio VAP. Samples with detects (i.e., high or low exceedance) must be reanalyzed as well as samples with non-detects and the low exceedance assuming sufficient sample remains. - 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 as specified in LIMS.	- Individual samples must be repeated. Place comment in LIMS. - Surrogate results outside criteria shall be reported with qualifiers - When surrogates fail in a method blank or LCS, re-analyze an aliquot of the blank or LCS. If the same anomaly occurs upon re-analysis, the batch (i.e. blank, LCS and associated samples) must be re-prepared and re-analyzed. The exceptions are as follows: (a) insufficient sample for re-preparation, or (b) the surrogates are biased high and the samples are non-detect.

QC Activity *(Individual Responsible for Initiation/Assessment)*	Acceptance Criteria	Recommended Corrective Action
- 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 a sample (not applicable for samples analyzed under the Ohio VAP – if sufficient sample remains, reprocess the batch.)
Proficiency Testing (PT) Samples *(QA Manager, Department Manager)*	- Criteria supplied by PT Supplier.	- Any failures or warnings must be investigated for cause. Failures may result in the need to repeat a PT sample to show the problem is corrected.
Internal / External Audits *(QA Manager, Department Manager, Operations Manager, Technical Director, Laboratory Director)*	- Defined in Quality System documentation such as SOPs, QAM, etc.	- Non-conformances must be investigated through CAR system and necessary corrections must be made.
Reporting / Calculation Errors (Depends on issue – possible individuals include: Analysts, Data Reviewers, Project Managers, Department Manager, QA Manager, Corporate QA, Corporate Management)	- SOP CW-L-S-002, Internal Investigation of Potential Data Discrepancies and Determination for Data Recall.	- Corrective action is determined by type of error. Follow the procedures in SOP CW-L-S-002.
Client Complaints *(Project Managers, Laboratory Director, Sales and Marketing)*	-	- Corrective action is determined by the type of complaint. For example, a complaint regarding an incorrect address on a report must 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).

QC Activity *(Individual Responsible for Initiation/Assessment)*	Acceptance Criteria	Recommended Corrective Action
QA Monthly Report (Refer to Section 16 for an example) (QA *Manager*, Laboratory Director, Department Managers)	- QAM, SOPs.	- Corrective action is determined by the type of issue. For example, CARs for the month are reviewed and possible trends are investigated.
Health and Safety Violation (Health & Safety Officer, Laboratory Director, Department Manager)	- Environmental Health and Safety (EHS) Manual.	- Non-conformance is investigated and corrected through CAR system.

Note:
1. Except as noted below for certain compounds, the method blank must be below the detection limit. Concentrations up to five times the reporting limit are allowed for the ubiquitous laboratory and reagent contaminants: methylene chloride, toluene, acetone, 2-butanone and phthalates **provided** they appear in similar levels in the reagent blank and samples. This allowance presumes that the detection limit is significantly below any regulatory limit to which the data are to be compared and that blank subtraction is not performed. For benzene and ethylene dibromide (EDB) and other analytes for which regulatory limits are extremely close to the detection limit, the method blank must be below the method detection limit. For Ohio VAP, the method blank must be below the reporting limit for all analytes that are a contaminant of concern for the project site.

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 TestAmerica Nashville's commitment to its Quality Program. It is beneficial to identify and address negative trends before they develop into complaints, problems, and corrective actions. Additionally, customer service and client satisfaction can be improved through continuous improvements to laboratory systems.

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

The monthly Management Systems Metrics Report shows performance indicators in all areas of the laboratory and quality system. These areas include revised reports, corrective actions, audit findings, internal auditing and data authenticity audits, client complaints, PT samples, holding time violations, SOPs, ethics training, etc. These metrics are used in evaluating the management and quality system performance on an ongoing basis and provide a tool for identifying areas for improvement.

The laboratory's corrective action process is integral to implementation of preventive actions. A critical piece of the corrective action process is the implementation of actions to prevent further occurrence of a non-compliance event. Historical review of corrective action provides a valuable mechanism for identifying preventive action opportunities.

13.1.1 The following elements are part of a preventive action system:

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

13.1.2 Any preventive actions performed or attempted must be evaluated during the annual Management Systems Review (Section 16). A highly detailed report is not required; however, a summary of success and failure within the preventive action program is sufficient to provide management with a measurement for evaluation.

13.2 Management of Change

The Management of Change process is designed to manage significant events and changes that occur within the laboratory. Through these procedures, the potential risks inherent with a new event or change are identified and evaluated. The risks are minimized or eliminated through pre-planning and the development of preventive measures. The types of changes covered under this system include: facility changes, major accreditation changes, addition or deletion to the laboratory's capabilities or instrumentation, key personnel changes, laboratory information management system (LIMS) changes, etc. This process is discussed in further detail in SOP *Management of Change* / NV08-161.

SECTION 14. CONTROL OF RECORDS

TestAmerica Nashville 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 they has been issued. See SOP *Archiving of Technical and Quality Department Paper Records* / NV08-194 for the archiving procedures.

14.1 Overview

The laboratory has established procedures for identification, collection, indexing, access, filing, storage, maintenance, and disposal of quality and technical records. A record index is listed in Table 14-1. Quality records are maintained by the Quality Assurance (QA) department in a database, which is backed-up as part of the regular network back-up. 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). Where identical electronic and hard copy documents both exist, the electronic version is the primary record. Technical records are maintained by the QA Department.

Table 14-1. Record Index1

	Record Types [1]:	Retention Time:
Technical Records	- Raw Data - Logbooks[2] - Standards - Certificates - Analytical Records - MDLs/IDLs/DOCs - Laboratory Reports	5 Years from analytical report issue*

	Record Types [1]:	Retention Time:
Official Documents	- Quality Assurance Manual (QAM) - Work Instructions - Policies - SOPs - Policy Memorandums - Manuals	5 Years from document retirement date*
QA Records	- Internal & External Audits/Responses - Certifications - Corrective/Preventive Actions - Management Reviews - Method & Software Validation / Verification Data - 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)
Project Records	- Sample Receipt & COC Documentation - Contracts and Amendments - Correspondence - QAPP - SAP - Telephone Logbooks - Laboratory Reports	5 Years from analytical report issue*
Administrative Records	Finance and Accounting	10 years
	EH&S Manual, Permits	7 years
	Disposal Records	Indefinitely
	Employee Handbook	Indefinitely
	Personnel files, Employee Signature & Initials, Administrative Training Records (e.g., Ethics)	7 Years (HR Personnel Files must be maintained indefinitely)
	Administrative Policies Technical Training Records	7 years

[1] Record types encompass hardcopy and electronic records.
[2] Examples of Logbook Types: Maintenance, Instrument Run, Preparation (standard and samples), Standard and Reagent Receipt, Archiving, Balance Calibration, Temperature (hardcopy or electronic records).
* Exceptions listed in Table 14-2.

14.1.1 All records are stored and retained in such a way that they are secure and readily retrievable at the laboratory facility or an offsite location that provides a suitable environment to prevent damage, deterioration, and loss. All records are protected against fire, theft, loss, environmental deterioration, and vermin. In the case of electronic records, storage media are protected from deterioration caused by magnetic fields and/or electronic deterioration.

Access to the data is limited to laboratory and company employees and shall be documented with an access log**.** Records archived off-site are stored in a secure location where a record is maintained of any entry into the storage facility. Whether on-site or off-site storage is used, logs are maintained in each storage box to note removal and return of records. Retention of records are maintained on-site at the laboratory for at least 1 year after their generation and moved offsite for the remainder of the required storage time. Records are maintained for a minimum of five years unless otherwise specified by a client or regulatory requirement or as specified in Table 14-2.

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

14.1.2 Programs with Longer Retention Requirements

Some regulatory programs have longer record retention requirements than the standard record retention time. These are detailed in Table 14-2 with their retention requirements. In these cases, the longer retention requirement is enacted. If special instructions exist such that client data cannot be destroyed prior to notification of the client, the container or box containing that data is marked as to whom to contact for authorization prior to destroying the data.

Table 14-2. Special Record Retention Requirements

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

[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, see Section 19.14.1 for more information.

14.1.4 The record keeping system allows for historical reconstruction of all laboratory activities that produced the analytical data as well as rapid recovery of historical data (i.e., records stored off-site are accessible within 72 hours of the request). The history of the sample from initial laboratory receipt must be readily understood through the documentation (including inter-laboratory transfers of samples and/or extracts).

- The records include the identities of personnel involved in sampling, sample receipt, preparation, and analysis. All analytical work contains the initials (at a minimum) of the personnel involved. The laboratory's copy of the chain of custody is stored with the project folder and an electronic copy is stored on the server. The chain of custody indicates 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 LIMS maintains an audit trail of data verification steps.
- The record keeping system facilitates the retrieval of all working files and archived records for inspection and verification purposes (e.g., a defined format for naming electronic files, a defined format for items included with a particular analytical data set). At a minimum, a data folder includes: benchsheet, prep sheets (where applicable), sample data, and associated QC data. Analytical data is stored sequentially by date analyzed and by department. Run logs are maintained for each instrument or method. A copy of each day's run long or instrument sequence is stored with the data to aid in reconstructing an analytical sequence. Where an analysis is performed without an instrument, bound logbooks or bench sheets are used to record and file data. Standard and reagent information is recorded in logbooks or entered into the LIMS for each method as required. Each folder containing a batch of data is labeled, at a minimum, with the date analyzed and the benchsheet or sequence number.
- Changes to hardcopy records must follow the procedures outlined in Sections 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 by including phrases 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. Scanned documents are considered the original document and hard copy documents may then be destroyed. Prior to the destruction of any of the hard copy information that was scanned, the scanning process must 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.
- 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, 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 or in Table 14-2. The records for each analysis must contain sufficient information to enable the analysis to be repeated under conditions as close as possible to the original conditions. The records must include the identities of laboratory personnel responsible for the sample collection, performance of each analysis, and analytical review.

14.2.2 Observations, analytical data, and calculations are recorded at the time they are made and are identifiable to the specific task.

14.2.3 Changes to hardcopy records must follow the procedures outlined in Sections 12 and 19. Changes to electronic records in LIMS or instrument data are recorded in audit trails.

The essential information to be associated with analysis, such as strip charts, tabular printouts, computer data files, analytical notebooks, and run logs, include:

- Laboratory sample ID code
- Date of analysis. (Time of analysis is also required if the holding time is seventy-two (72) hours or less, or when time critical steps are included in the analysis (e.g., drying times, incubations, etc.). Instrumental analyses have the date and time of analysis recorded as part of their general operations. Where a time-critical step exists in an analysis, location for such a time is included as part of the documentation in a specific logbook or on a benchsheet.)
- Instrumentation identifier and instrument operating conditions/parameters (Operating conditions/parameters are recorded in instrument maintenance logs, where available)
- Analysis type;
- All manual calculations and manual integrations
- Analyst's or operator's initials/signature
- Sample preparation information including cleanup, separation protocols, incubation periods, ID codes, volumes, weights, instrument printouts, meter readings, calculations, reagents, etc.
- 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, back-ups, and records of any changes to automated data entries
- Method performance criteria including expected quality control requirements (these are indicated both in the LIMS and on specific analytical report formats)

14.2.4 For the Ohio VAP, TestAmerica Nashville will attest, via affadavit, that data is VAP certified only when the analyses are performed within its current certification and both of the following conditions must be true.

- Ohio certification held at the time of analysis must be valid for the analytes, parameter groups, and methods utilized
- Analyses were performed in a manner consistent with SOPs and Quality Assurance Program Plan (QAPP) approved by the Ohio EPA

Affidavits must accompany either every report or series of reports for an ongoing project.

14.2.5 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 indicate 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
- 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)
- 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. See Table 14-1.

14.5 Records Management, Storage, and Disposal

All records (including those pertaining to test equipment), certificates, and reports are safely

stored and 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 (including personal computers) have hard copy, write-protected back-up copies, or an electronic audit trail controlling access. Personnel must access and use P: and L: drives and the OASIS intranet site.

TestAmerica Nashville has a record management system (a.k.a document control) for control of laboratory notebooks, instrument logbooks, and standards logbooks as well as records for data reduction, validation, storage, and reporting. Laboratory notebooks are issued on a per instrument basis and are numbered sequentially within a given analysis. No instrument has more than one active notebook at a time, so all data are recorded sequentially within a series of notebooks. Bench sheets are filed sequentially in folders. Standards preparation information is maintained in the LIMS; therefore, logbooks are not used to record standards preparation data.

14.5.1 Transfer of Ownership

In the event that the laboratory transfers ownership or goes out of business, TestAmerica Nashville must ensure that the records are maintained or transferred according to client's instructions. Upon ownership transfer, record retention requirements must be addressed in the ownership transfer agreement where the responsibility for maintaining archives will be clearly established. In case of bankruptcy, appropriate regulatory and state legal requirements concerning laboratory records will be followed. In the event of the closure of the laboratory, all records will revert to the control of the corporate headquarters. If the entire company ceases to exist, as much notice as possible must be given to clients and the accrediting bodies who have worked with the laboratory during the previous 5 years.

14.5.2 Records Disposal

Records may be removed from the archive and destroyed after 5 years unless otherwise specified by a client or regulatory requirement (as noted in Tables 14-1 and 14-2). 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.

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 company is hired to dispose of records, a "Certificate of Destruction" will be required.

SECTION 15. AUDITS

15.1 Internal Audits

Internal audits are performed to verify that laboratory operations comply with the requirements of the laboratory'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 have the sufficient authority, access to work areas, and organizational freedom necessary to observe all activities affecting quality, to report the assessments to laboratory management, and when requested to corporate management.

Audits are conducted and documented as described in the TestAmerica Corporate SOP on performing Internal Auditing, SOP No. CW-Q-S-003. The types and frequency of routine internal audits are described in Table 15-1. Special or ad hoc assessments may be conducted as needed under the direction of the QA staff.

Table 15-1. Types of Internal Audits and Frequency

Description	Performed by	Frequency
Quality Systems Audits	QA Department, QA – approved designee, or Corporate QA	All areas of the laboratory annually
QA Technical Audits	Joint responsibility: a) QA Manager or designee b) Technical Manager or Designee (Refer to CW-Q-S-003)	Technical Audits Frequency: 50% of methods annually
SOP Method Compliance	Joint responsibility: c) QA Manager or designee d) Technical Manager or Designee (Refer to CW-Q-S-003)	SOP Compliance Review Frequency: Every 2 years (Annualy for Drinking Water methods)
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, NELAP quality systems, ISO 17025, A2LA, client, and state requirements, and the effectiveness of the internal controls of the

analytical process including but not limited to data review, quality controls, preventive action, and corrective action. The completeness of earlier corrective actions is assessed for effectiveness and sustainability. The audit is divided into sections for each operating or support area of the lab, and each section is comprehensive for a given area. The area audits may be performed on a rotating schedule throughout the year to ensure adequate coverage of all areas. This schedule may change as situations in the laboratory warrant.

15.1.2 QA Technical Audits

QA technical audits are based on client projects, associated sample delivery groups, and the methods performed. Reported results are compared to raw data to verify the authenticity of results. The validity of calibrations and QC results are compared to data qualifiers, footnotes, and case narratives. Documentation is assessed by examining run logs and records of manual integrations. Manual calculations are checked. Where possible, electronic audit miner programs (e.g., MintMiner, Chrom Auditminer) is used to identify unusual manipulations of the data deserving closer scrutiny. QA technical audits must include all methods within a two-year period.

15.1.3 SOP Method Compliance

Compliance of all SOPs with the source methods and compliance of the operational groups with the SOPs must be assessed by the Technical Director and QA department at least every two years. The work of each newly hired analyst is assessed within 3 months of working independently, (e.g., completion of method IDOC). As analysts add methods to their capabilities, reviews of the analyst work products must be performed within 3 months of completing the documented training (i.e., new IDOCs).

15.1.4 Special Audits

Special audits are conducted on an as needed basis, generally as a follow up to specific issues such as client complaints, corrective actions, PT results, data audits, system audits, validation comments, regulatory audits, or suspected ethical improprieties. Special audits are focused on a specific issue and their report formats, distribution lists, 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, nonpotable water, and soil.

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

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

15.2 External Audits

External audits are performed when certifying agencies or clients conduct on-site inspections or submit performance testing samples for analysis. It is TestAmerica's policy to cooperate fully with regulatory authorities and clients. The laboratory makes every effort to provide the auditors with access to personnel, documentation, and assistance. Laboratory Department Managers and Technical Director 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 laboratory's 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. Upon entry into the TestAmerica Nashville laboratory, auditors sign in using a document that includes a confidentiality clause. 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 must not be obscured from the information. Additional information regarding CBI can be found in within the 2009 TNI standards.

15.3 AUDIT FINDINGS

Audit findings are documented using the corrective action process and database. The laboratory's corrective action responses for both types of audits (internal and external) are completed within a predetermined timeframe. When the corrective action has not yet been completed within that predefined response timeframe, a completion date must set and agreed to by operations management and the QA Manager.

Developing and implementing corrective actions to findings is the responsibility of the Department Manager where the finding originated. Findings that are not corrected by specified due dates are reported monthly to management in the QA monthly report. When requested, a copy of the audit report and the laboratory's corrective action plan are 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 must take timely corrective action and must notify clients in writing if the investigations show that the laboratory results have been affected.

Once corrective action is implemented, a follow-up audit is scheduled to ensure that the problem has been corrected.

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 must be 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 is prepared each month by the laboratory's QA Department and forwarded to the Laboratory Director, Technical Managers, their Quality Director, as well as the VP of Operations. All aspects of the QA system are reviewed to evaluate the suitability of policies and procedures. During the course of the year, the Laboratory Director, VP of Operations, or Corporate QA may request that additional information be added to the report.

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

16.2 Annual Management Review

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

This management systems review (Corporate SOP CW-Q-S-004 & Work Instruction No. CW-Q-WI-003) uses information generated during the preceding year to assess the "big picture" by ensuring that routine actions taken and reviewed on a monthly basis are not components of larger systematic concerns. The monthly review keeps 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 laboratory senior management meetings - Issues that may be raised from these meetings include:
    - Adequacy of staff, equipment and facility resources
    - Adequacy of policies and procedures
    - Future plans for resources and testing capability and capacity
- Compliance to the Ethics Policy and Data Integrity Plan, including any evidence/incidents of inappropriate actions or vulnerabilities related to data integrity.

A report is generated by the QA Manager and management. The report is distributed to the appropriate General Manager and Corporate Quality Director. The report includes, but is not limited to:

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

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

16.3 Potential Integrity-Related Managerial Reviews

Potential integrity issues (data or business related) must be handled and reviewed in a confidential manner until such time as a follow-up evaluation, full investigation, or other appropriate actions have been completed and issues clarified. TestAmerica's Corporate Data Investigation/ Recall SOP must be followed (SOP No. CW-L-S-002). All investigations that result in finding of inappropriate activity are documented and include any disciplinary actions involved, corrective actions taken, and all appropriate notifications of clients.

TestAmerica's CEO, Executive VP of Operations, VP of Client and Technical Services, VP of Operations, and Quality Directors receive a monthly report compiled from the Corporate Quality and EHS Director summarizing any current data integrity or data recall investigations. The VPs of Operations are also made aware of progress on these issues for their specific labs.

SECTION 17. PERSONNEL

17.1 Overview

The laboratory's management believes that its highly qualified and professional staff is the single most important aspect in assuring a high level of data quality and service. The staff consists of professionals and support personnel as outlined in the organization chart in Figure 4-1 (subject to change).

All personnel must demonstrate competence in the areas where they have responsibility. Any staff member that is undergoing training must 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 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 laboratory operations, test methods, QA/QC procedures, and records management.

Laboratory management is responsible for formulating goals for laboratory staff with respect to education, training, and skills as well as ensuring that the laboratory has policies and procedures for identifying training needs and providing training of personnel. The training must be relevant to the present and anticipated responsibilities of the laboratory staff.

The laboratory only uses personnel that are employed by or under contract to the laboratory. Contracted personnel, when used, must meet competency standards of the laboratory and work in accordance to the laboratory's quality system.

17.2 Education and Experience Requirements for Technical Personnel

The laboratory makes every effort to hire analytical staff that possesses a college degree (AA, BA, BS) in an applied science with some chemistry in the curriculum. Exceptions can be made based upon the individual's experience and ability to learn. Experience and specialized training are occasionally accepted in lieu of a college degree (basic laboratory skills such as using a balance, etc. are also considered). For any analysis, thorough training and working with another experienced staff member until proficiency has been demonstrated is required.

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).

Table 17-1. Education/Experience Requirements

Specialty	Education	Experience
Extractions, Digestions, some electrode methods (pH, DO, Redox, etc.), or Titrimetric and Gravimetric Analyses	H.S. Diploma	On the job training (OJT)
CVAA, Single component, or short list Chromatography (e.g., Fuels, BTEX-GC, IC	A college degree in an applied science or 2 years of college and at least 1 year of college chemistry	Or 2 years prior analytical experience is required
ICP, ICPMS, Long List or complex chromatography (e.g., Pesticides, PCB, Herbicides, HPLC, etc.), GCMS	A college degree in an applied science or 2 years of college applied sciences	Or 5 years of prior analytical experience
Spectra Interpretation	A college degree in an applied science or 2 years of college applied sciences	And 2 years relevant experience Or 5 years of prior analytical experience
Operations Managers/Department Managers /Supervisors	Bachelor's Degree in an applied science or engineering An advanced (MS, PhD.) degree may substitute for one year of experience	And two years experience in environmental analysis of representative analytes for which they will oversee
Laboratory Director	Bachelor's Degree in science or a minor in chemistry	Or 10 years experience in the environmental laboratory industry
Quality Assurance Manager	Bachelor's degree in a basic or applied science and 1 year nonacademic analytical chemistry; Training in statistics or quality control procedures.	Or 4 years nonacademic analytical chemistry experience.
Technical Director	Bachelor's degree in basic or applied science or engineering and at least 24 college semester credit hours in chemistry	At least two years experience in environmental analysis; masters or doctoral degree may substitute for one year experience.

Analysts-in-training perform tasks under the direct supervision of a qualified analyst or Department Manager/Supervisor. The person supervising an analyst-in-training is accountable for the quality of the analytical data. The trainer must review and approve the trainee's data and any 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:

Table 17-2. Training Requirements

Required Training	Time Frame*	Employee Type
Environmental Health & Safety	Prior to laboratory 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 19.4.2.

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 is maintained in their training file.
- Documentation of proficiency (refer to Section 19).
- An Ethics Agreement signed by each staff member (renewed each year) and evidence of annual ethics training.
- A Confidentiality Agreement signed by each staff member signed at the time of employment.
- Human Resources maintains documentation and attestation forms on employment status and records; benefit programs; timekeeping/payroll; and employee conduct (e.g., ethics). This information is maintained in the employee's secured personnel file.

Evidence of successful training could include such items as:

- Adequate documentation of training within operational areas, including one-on-one technical training for individual technologies, and particularly for people cross-trained.
- Analyst's knowledge to refer to QA Manual for quality issues.
- Analyst following SOPs, i.e., practice matches SOPs.
- Analysts regularly communicate to supervisors and QA if SOPs need revision, rather than waiting for auditors to find problems.
- Analyst successfully analyzes a PT sample.

Further details of the laboratory's training program are described in the SOP *Training Procedures for Environmental Technical Staff* / NV08-199.

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 an 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 are not tolerated. Employees who violate this policy are 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 including confidential/anonymous 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 or criminal prosecution
- Importance of proper written narration / data qualification by the analyst and project manager with respect to those cases where the data may still be usable but are, in one sense or another, partially deficient

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

SECTION 18. ACCOMMODATIONS AND ENVIRONMENTAL CONDITIONS

18.1 Overview

TestAmerica Nashville is an approximately 50,000 ft^2, secure, laboratory facility with controlled access, designed to accommodate an efficient workflow and to provide a safe and comfortable work environment for employees. All visitors must sign in and are escorted by laboratory personnel. Access is controlled by various measures.

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

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

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

18.2 Environment

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

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

The laboratory provides for the effective monitoring, control, and recording of environmental conditions that may affect the results of environmental tests as required by the relevant specifications, methods, and procedures. Such environmental conditions include humidity, voltage, and temperature in the laboratory.

When any of the method- or regulatory-required environmental conditions change such that they may adversely affect test results, analytical testing is 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. For example, volatile organic chemical handling areas, including sample preparation and waste disposal, are separated from volatile organic chemical analysis areas.

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

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

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

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

18.4 Floor Plan

A floor plan can be found in Appendix 1.

18.5 Building Security

Building keys are distributed to employees as necessary. All doors have automatic closures which lock when closed, except the two main entrances which are locked at 5:30 p.m. each business day.

Visitors to the laboratory sign in and out in a visitors' logbook. A visitor is defined as any person who visits the laboratory who is not an employee of TestAmerica Nashville. The visitors' logbook is used to ensure that everyone gets out of the building safely in case of emergency. The visitor is provided with a "Visitor" identification card to wear while in the laboratory. The Environmental Health and Safety Manual contains requirements for visitors and vendors including the 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 visitors' logbook. Visitors sign a document that includes a requirement to treat all observations in a confidential manner.

SECTION 19. TEST METHODS AND METHOD VALIDATION

19.1 Overview

TestAmerica Nashville uses methods that are appropriate to meet our clients' requirements and that are within the scope of the laboratory's capabilities. These include sampling, handling, transport, storage and preparation of samples, and, where appropriate, an estimation of the measurement of uncertainty as well as statistical techniques for analysis of environmental data.

Instructions are available in the laboratory for the operation of equipment as well as for the handling and preparation of samples. All instructions, Standard Operating Procedures (SOPs), reference methods, and manuals relevant to the working of the laboratory are readily available to all staff. Deviations from published methods are documented (with justification) in the laboratory's approved SOPs. SOPs are submitted to clients for review at their request. Significant deviations from published methods require client approval and regulatory approval where applicable.

19.2 Standard Operating Procedures (SOPS)

TestAmerica Nashville maintains SOPs that accurately reflect all phases of the work performed by laboratory personnel such as assessing data integrity, corrective actions, and 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 TestAmerica Nashville. Modifications or clarifications to published methods are clearly noted in the SOPs. All SOPs used by laboratory personnel are controlled copies :

- All SOPs contain a revision number, effective date, and appropriate approval signatures. Controlled copies are available to all staff.
- Procedures for SOP preparation, review, revision and control are incorporated by reference to SOPs: CW-Q-S-002 (*Writing a Standard Operating Procedure (SOP)*) and SOP *Document Control* / NV08-152.
- SOPs are reviewed at a minimum of every two years (annually for Drinking Water SOPs), and where necessary, revised to ensure continuing suitability and compliance with applicable requirements.

19.3 Laboratory Methods Manual

For each test method, the laboratory has 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 demonstrates 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, they (and other pertinent information) are summarized by the Project Manager. These mechanisms ensure that the proper analytical methods are applied when the samples arrive for log-in. For non-routine analytical services (e.g., special matrices, non-routine compound lists, etc.), the method of choice is selected based on client needs and available technology. The methods selected must be capable of measuring the specific parameter of interest, in the concentration range of interest, and with the required precision and accuracy.

19.4.1 Sources of Methods

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

When clients do not specify the method to be used or specific methods are not required, the methods used must 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:

- HASL-300 28th Edition, Environmental Measurements Laboratory (EML), 1997.
- *Compendium of Methods for the Determination of Toxic Organic Compounds in Ambient Air*, US EPA, January 1996.
- *Guidelines Establishing Test Procedures for the Analysis of Pollutants Under the Clean Water Act*, and Appendix A-C; 40 CFR Part 136, USEPA Office of Water. Revised as of July 1, 1995, Appendix A to Part 136 - Methods for Organic Chemical Analysis of Municipal and Industrial Wastewater (EPA 600 Series)
- *Methods for Chemical Analysis of Water and Wastes*, EPA 600 (4-79-020), 1983.

- *Methods for the Determination of Inorganic Substances in Environmental Samples*, EPA-600/R-93/100, August 1993.
- *Methods for the Determination of Metals in Environmental Samples*, EPA/600/4-91/010, June 1991. Supplement I: EPA-600/R-94/111, May 1994.
- *Methods for the Determination of Organic Compounds in Drinking Water*, EPA-600/4-88-039, December 1988, Revised, July 1991, Supplement I, EPA-600-4-90-020, July 1990, Supplement II, EPA-600/R-92-129, August 1992. Supplement III EPA/600/R-95/131 - August 1995 (EPA 500 Series) (EPA 500 Series methods)
- *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*, various editions /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 IIIB, June 2005; and Final Update IV, January 2008, including New Methods on-line.
- *Annual Book of ASTM Standards*, American Society for Testing & Materials (ASTM), Philadelphia, PA.
- *Manual for the Certification of Laboratories Analyzing Drinking Water* (EPA 815-R-05-004, January 2005)
- Code of Federal Regulations (CFR) 40, Parts 136, 141, 172, 173, 178, 179 and 261.

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 the latest versions of each approved method as regulations allow or require. See controlled document QAF-132 (Method Capabilities) for a complete listing of methods currently used at TestAmerica Nashville.

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 determines the method utilized.

The laboratory must 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 must be documented.

19.4.2 Demonstration of Capability

Before the laboratory may institute a new method and begin reporting results, it must be confirmed that the method can be performed properly. 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 (see SOP *Training Procedure for Environmental Technical Staff* / NV08-199) is performed whenever there is a change in instrument type, matrix, method, or personnel (e.g., analyst has not performed the test within the last 12 months). A Demonstration of Capability is also performed when an instrument (new or used) is moved into a department and brought online for the first time in that analytical department.

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

The initial demonstration of capability must be thoroughly documented and approved by the Technical Director and QA Manager prior to independently analyzing client samples. All associated documentation must be retained in accordance with the laboratory's archiving procedures.

The laboratory must have an approved SOP, demonstrate satisfactory performance, and conduct a method detection limit study (when applicable). There may be other requirements as stated within the published method or regulations (i.e., retention time window study) (see SOP *Procedure for Method Start- Up or Modification* / NV08-203).

Note: In some instances, a situation may arise in which 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 must be calibrated for the unusual analyte using the method criteria and ICV/CCV criteria for the method (unless an ICV/CCV is not required by the method or criteria are per project specifications).
- The laboratory's nominal or default reporting limit (RL) must be at or above the lowest non-zero standard in the calibration curve and must be reliably determined. Project RLs (determined during Work Request Review, see Section 7) are client specified reporting limits which may be higher than the laboratory's RL. Results reported below the RL must be qualified as estimated values (i.e., "J" flagged). Also see Section 19.6.1.3, *Relationship of Method Detection Limit (MDL) to Reporting Limit (RL)*.
- The client request must be documented and the laboratory must inform 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.

- Refer to Section 11 (Control of Non-Conforming Work).

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 (i.e., a second source).

19.4.3.2 The analyte(s) are diluted in a volume of clean matrix sufficient to prepare four aliquots at the concentration specified by a method or the laboratory SOP or if unspecified to a concentration 1-4 times the laboratory RL.

19.4.3.3 At least four aliquots are prepared (including any applicable clean-up procedures) and analyzed according to the test method (either concurrently or over a period of days). Note: Results of successive LCS analyses can be used to fulfill the DOC requirement.

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 assesses 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, confirms 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.

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

Methods on line prior to the effective date of this Section must be updated to the procedures outlined above as new analysts perform their demonstration of capability. A copy of the new record replaces 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 is 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 (see SOP *Procedure for Method Start-Up or Modification* / NV08-203).

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. Estimations and/or demonstrations of sensitivity required by regulation or client agreement (such as the procedure in 40 CFR Part 136 Appendix B, under the Clean Water Act) must be followed.

19.6.1.3 Relationship of Method Detection Limit (MDL) to the Reporting Limit (RL)

An important characteristic of expression of sensitivity is the difference in the MDL and the RL. The MDL is the minimum level at which the presence of an analyte can be reliably detected. The RL 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 MDL (both above and below the estimated MDL) and below the RL. In this region, detection of an analyte may be confirmed but quantification of the analyte is unreliable within the accuracy and precision guidelines of the measurement system. When an analyte is detected below the RL, 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.

Note: For the purposes of Ohio VAP, the terms MDL and reporting limit (RL) are used exclusively by the laboratory.

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 RL 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 or replicate spikes, 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 LCSs, method blanks, or PT samples.

19.7 Method Detection Limits (MDL)

Method detection limits (MDL) are initially determined in accordance with 40 CFR Part 136, Appendix B or alternatively by other technically acceptable practices that have been accepted by regulators. MDL is also sometimes referred to as Limit of Detection (LOD). The MDL theoretically represents the concentration level for each analyte within a method at which the analyst is 99% confident that the true value is not zero. The MDL is determined for each analyte initially during the method validation process and updated as required in the analytical methods, whenever there is a significant change in the procedure or equipment, or based on project specific requirements. 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 or ½ the lowest calibration curve standard) 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 are analyzed over 2-4 days to provide a more realistic MDL value.

In some instances, evaluation of blanks over time may be used to determine MDL values and the values are then verified on each instrument. Alternatively, the least sensitive instrument

representing a specific technology may be used to determine MDL values and the values are then verified on similar instrumentation.

Note: For the purposes of Ohio VAP, the term MDL is used exclusively by the laboratory.

Refer to the Corporate SOP No. CA-Q-S-006 or the SOP *Procedure for the Determination of Method Detection Limits* / NV08-202 for details on the laboratory's MDL process.

19.8 Instrument Detection Limits (IDL)

The IDL is sometimes used to assess the reasonableness of the MDLs or in some cases required by the analytical method or program requirements. IDLs are most used in metals analyses but may be useful in demonstration of instrument performance in other areas.

IDLs are calculated to determine an instrument's sensitivity independent of any preparation method. IDLs are calculated by the analysis of multiple instrument blanks and calculating, nominally, 3 x the absolute value of the standard deviation.

If the IDL is > than the MDL, then the MDL is adjusted to a value equal to or greater than the IDL.

19.9 Verification of Detection and Reporting Limits

Once an MDL is established, it must be verified, on each instrument representing the technology, by analyzing a quality control sample (prepared as a sample) at no more than 1-2 times (nominal) the calculated MDL. 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 laboratory does not report to the MDL. If the MDL is not verified, then the laboratory must 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 (Quarterly for Texas, Monthly for Minnesota).

When the laboratory establishes a quantitation limit, it must be initially verified by the analysis of a low level standard or QC sample at 1-2 times the reporting limit and annually thereafter. The annual requirement is waved for methods that have an annually verified MDL. The laboratory must comply with any regulatory requirements.

19.10 Retention Time Windows

For every chromatography analysis, or as specified in the reference method, each analyte has a specific time of elution from injection on 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 phase 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 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, and specific electrode response characteristics.

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. The true result of an environmental sample 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 analytical (measurement) 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 statistically-calculated, historical, in-house LCS accuracy limits. Overall uncertainty uses matrix spike results to include sample uncertainty.

19.12.4 To calculate the measurement uncertainty for the specific result, multiply the result by the decimal value 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 value of the upper end of the LCS range percent value for the upper end of the uncertainty range. These calculated values represent a 99%-certain range for the reported result. As an example, suppose that the result reported is 1.0 mg/L, and the LCS percent recovery range is 50 to 150%. The uncertainty range would be 0.5 to 1.5 mg/L, which could also be written as 1.0 +/- 0.5 mg/L.

19.12.5 In the case where a well recognized test method specifies limits to the values of major sources of uncertainty of measurement (e.g., 524.2, 525, etc) and specifies the form of presentation of calculated results, no further discussion of uncertainty is required.

19.12.6 There are a series of required steps for estimation of measurement uncertainty:

- Inventory all components of uncertainty in the test (e.g., sampling, subsampling, calibration, etc)

- Determine the significance of each component, eliminating any component that is insignificant
- Identifying all available data that can be used in the uncertainty estimate and identifying the component that it applies to (e.g., duplicate data, spike recovery data, etc.)
- Identify any gaps in data
- Use the available data and logically derived estimates where gaps exist to calculate the expanded uncertainty

These criteria are met when using EPA methods, Standard Methods, ASTM, etc. Specific criteria are represented by the use of LCS measurement, sample plus MS/MSD measurement, or duplicates and participation in PT programs. Control charting is used to trend and update uncertainty as percent acceptance.

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.**

Note: Refer to the applicable SOP for approved corrective actions when analyzing samples for the Ohio VAP.

- Homogenous samples: If a reanalysis agrees with the original result to within the RPD limits for MS/MSD or Duplicate analyses, or within $\pm$ 1 reporting limit for samples $\leq$ 5x the reporting limit, the original analysis will be reported. At the client's request, both results may be reported on the same report but not on two separate reports.
- If the reanalysis does not agree (as defined above) with the original result, then the laboratory will investigate the discrepancy and reanalyze the sample a third time for confirmation if sufficient sample is available. The Department Manager/Supervisor should notify the QA Manager, the Technical Director, and the Laboratory Director when an error is suspected (i.e., sample switch, ICAL issue, etc.)
- Any potential charges related to reanalysis are discussed in the contract terms and conditions or discussed at the time of the request. The client will typically be charged for reanalysis unless it is determined that the lab was in error.
- Due to the potential for increased variability, reanalysis may not be applicable to non-homogenous, Encore, and Sodium Bisulfate preserved samples. See the Department Supervisor or Laboratory Director if unsure.

19.14 Control of Data

The laboratory has policies and procedures in place to ensure the authenticity, integrity, and accuracy of the analytical data generated by the laboratory.

19.14.1 Computer and Electronic Data Related Requirements

The three basic objectives of our computer security procedures and policies are shown below. More detail is outlined in SOP *Lab Data Archiving System* / NV09-171.

19.14.1.1 Maintain the Database Integrity: Assurance that data is reliable and accurate through data verification (review) procedures, password-protected access, anti-virus protection, data change requirements, and 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.
- **Note**: "Commercial off-the-shelf software (e.g. word processing, database, and statistical programs) in general use within the designed application range may be considered to be sufficiently validated." From TNI Standard EL-V1M2-ISO-2009. However, laboratory specific configurations or modifications are validated prior to use.
- Instrument hardware and software adjustments are safeguarded through maintenance logs, audit trails, and controlled access.

19.14.1.2 Ensure Information Availability: Protection against loss of information or service is ensured through scheduled back-ups, stable file server network architecture, secure storage of media, line filter, uninterruptible power supply (UPS) use, 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 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.

Note: The Ohio VAP requires that all calculations be performed through the use of spreadsheets or other computerized software (such as LIMS).

For manual data entry, e.g., Wet Chemistry, the data is reduced by the analyst and then verified by the Department Manager or alternate analyst prior to updating the data in LIMS. The spreadsheets, or any other type of applicable documents, are signed by both the analyst and alternate reviewer to confirm the accuracy of the manual entry(s). Applicable documents are scanned and linked to the batch in LIMS. The scanned documents are considered the originals.

All manual integrations of peaks must be documented and reviewed, and the raw data must be flagged in accordance with Section 20.13.5 and the TestAmerica Corporate SOP CA-Q-S-002, *Acceptable Manual Integration Practices*.

Analytical results are reduced to appropriate concentration units specified by the analytical method, taking into account factors such as dilution, sample weight or volume, etc. Blank correction will be applied only when required by the method or per manufacturer's indication; otherwise, it is not 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/batch 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. The units "mg/L" and "mg/kg" are the same as "parts per million (ppm)". The units "µg/L" and "µg/kg" are the same as "parts per billion (ppb)." For values greater than 10,000 mg/L, results can be reported in percent, i.e., 10,000 mg/L = 1%. LIMS includes the capability to select several unit options.

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 into LIMS with at least three significant figures. In general, results are reported to three 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, the raw results and dilution factors are entered directly into LIMS by the analyst, and the software calculates the final result for the analytical report. LIMS has a defined significant figure criterion for each analyte.

19.14.2.5 The laboratory strives to import data directly from instruments or calculation spreadsheets to ensure that the reported data are free from transcription and calculation errors. For those analyses with an instrumental output compatible with the LIMS, the raw results and dilution factors are transferred into LIMS electronically after reviewing the quantitation report and removing unrequested or poor spectrally-matched compounds. The analyst prints a copy of what has been entered to check for errors. This printout and the instrument's printout of calibrations, concentrations, retention times, chromatograms, and mass spectra, if applicable, are retained with the data file. The data file is stored in a monthly folder on the instrument computer. Periodically, this stored file is transferred to the server.

19.14.3 Logbook / Worksheet Use Guidelines

Logbooks and worksheets are filled out "real time" and have enough information entered to trace the events of the applicable analysis/task (e.g., calibrations, standards, analyst, sample ID, date, time on short holding time tests, temperatures when applicable, calculations are traceable, etc.)

- Corrections are made following the procedures outlined in Section 12.
- Logbooks are controlled by the QA department. A record is maintained of all logbooks in the lab.

- Unused portions of pages must be "Z"'d out, signed, and dated.
- Worksheets are created with the approval of the Technical Director or QA Manager at the facility. The QA Department controls all worksheets following the procedures in Section 6.

19.14.4 Review / Verification Procedures

Review procedures are outlined in several SOPs (e.g., *Sample Receiving* / NV02-01, *Project Management* / NV02-174, *Data Package Procedure* / NV08-139) to ensure that reported data are free from calculation and transcription errors, that QC parameters have been reviewed and evaluated before data is reported. The laboratory also has an SOP discussing Manual Integrations to ensure the authenticity of the data (CA-Q-S-002). The general review concepts are discussed below, more specific information can be found in the SOPs.

19.14.4.1 The data review process at TestAmerica Nashville starts at the Sample Control level. Sample Control personnel review chain-of-custody forms and input the sample information and required analyses into a LIMS. The Sample Control Supervisor reviews the chain-of-custody forms against the entered information. The Project Managers perform final review of the chain-of-custody forms and entered information.

19.14.4.2 The next level of data review occurs with the analysts. As results are generated, analysts review their work to ensure that the results generated meet QC requirements, relevant EPA methodologies, and any project-specific criteria. The analysts transfer the data into the LIMS and add data qualifiers if applicable (see Appendix 4 for list of common data qualifiers). To ensure data compliance, a Department Manager (or other designee) performs a second level of review. Second level review is accomplished by checking reported results against raw data and evaluating the results for accuracy. During the second level review, blank runs, QA/QC check results, initial and continuing calibration results, laboratory control samples, sample data, qualifiers, and spike information are evaluated. Where calibration is not required on a daily basis, secondary review of the initial calibration results may be conducted at the time of calibration. Data review checklists are used to document both analyst and second level reviews. Issues that require further review include the following occurrences:

- 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 have unusually high results
- Results exceed a known regulatory limit
- Raw data indicates contamination or poor technique
- Inconsistent peak integration
- Transcription errors
- Results are outside of the calibration range

19.14.4.3 Unacceptable analytical results may require reanalysis of the samples. Any issues are brought to the attention of the Laboratory Director, Project Manager, Quality Assurance Manager,

Technical Director, Manager of Project Managers, or Department Manager for further investigation. Corrective action is initiated as necessary.

19.14.4.4 The results are then entered or directly transferred into the LIMS and a hard copy (or .pdf) is printed for the client. Scanned information is considered the original documentation.

19.14.4.5 As a final review prior to the release of the report, the Project Manager reviews the results for appropriateness and completeness. This review and approval ensures that client requirements have been met and that the final report has been properly completed. The process includes, but is not limited to, verifying that chemical relationships are evaluated, COC is followed, cover letters/ narratives are present, flags are appropriate, and project specific requirements are met.

19.14.4.6 Any project that requires a data package is subject to a tertiary data review for transcription errors and acceptable quality control requirements. The Project Manager then signs the final report. Accounting personnel also check the report for any clerical or invoicing errors. When complete, the report is sent out to the client.

19.14.4.7 A visual summary of the flow of samples and information through the laboratory, as well as data review and validation, is presented in Figure 19-2.

19.14.5 Manual Integrations

Computerized data systems provide the analyst with the ability to re-integrate raw instrument data in order to optimize the interpretation of the data. Though manual integration of data is an invaluable tool for resolving variations in instrument performance and some sample matrix problems, when used improperly, this technique would make unacceptable data appear to meet quality control acceptance limits. Improper re-integrations lead to legally indefensible data, a poor reputation, or possible laboratory decertification. Because guidelines for re-integration of data are not provided in the methods and most methods were written prior to widespread implementation of computerized data systems, the laboratory trains all analytical staff on proper manual integration techniques using SOP CA-Q-S-002 as the guidelines**.**

19.14.5.1 The analyst must adjust baseline or the area of a peak in some situations. Examples include 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 must not increase or decrease peak areas for the sole purpose of achieving acceptable QC recoveries that would have otherwise been unacceptable. The intentional recording or reporting of incorrect information (or the intentional omission of correct information) is against company principals and policy and is grounds for immediate termination.

19.14.5.3 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. Scales should not be over-expanded (e.g., >100 times).

Figure 19-1. Demonstration of Capability Documentation

2013 DEMONSTRATION OF CAPABILITY

☐ *Initial*
☐ *Ongoing*

Analyte: ***Matrix:***
Method:
Analyst:
Date(s) of Analysis:

ANALYTE	*TRUE CONC. mg/L*	ANALYTICAL RUNS (mg/L) *1*	*2*	*3*	*4*	*AVG. % REC*	*RSD*
						#DIV/0!	*#DIV/0!*
Acceptable Range						90-110	< 20

RSD=Percent relative standard deviation =100s/X percent.

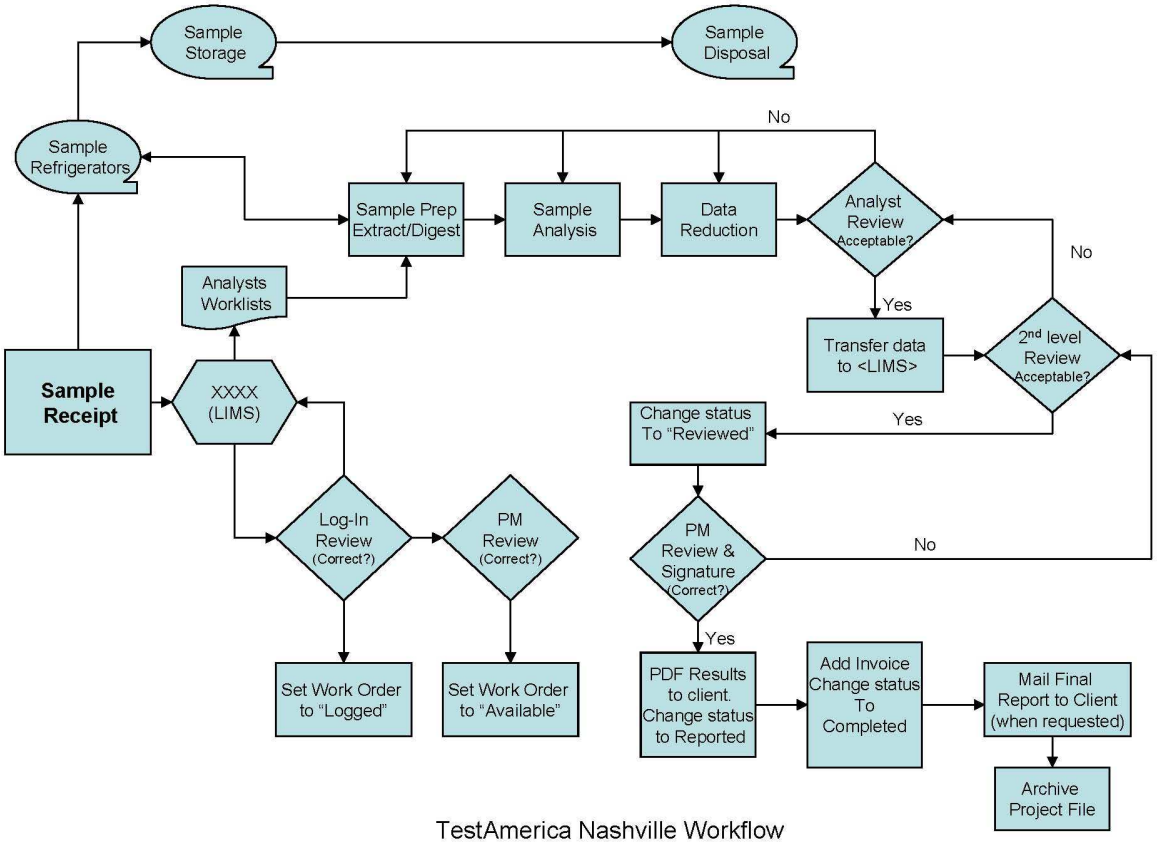
Certification Statement:

We, the undersigned, certify that:

1. The analyst identified above, using the cited test method, which is in use at this facility for the analyses of samples under the National Environmental Laboratory Accreditation Program, has met the Demonstration of Capability.
2. The test method was performed by the analyst 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 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 by authorized inspectors.

Analyst	*Signature*	*Date*
Technical Director	*Signature*	*Date*

Figure 19-2. Work Flow



SECTION 20. EQUIPMENT and CALIBRATIONS

20.1 Overview

TestAmerica purchases technically advanced analytical instrumentation for sample analyses. Instrumentation is purchased on the basis of accuracy, dependability, efficiency and sensitivity. The laboratory is furnished with all items of 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 is calibrated and checked to establish that it meets its intended specification. The calibration routines for analytical instruments establish the range of quantitation. Calibration procedures are specified in laboratory SOPs. A list of laboratory equipment and instrumentation is presented in Table 20-1.

Equipment is only operated by authorized and trained personnel. Manufacturers' instructions for equipment use are readily accessible to all appropriate laboratory personnel.

20.2 Preventive Maintenance

TestAmerica Nashville 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 lubrication, cleaning, and replacements, should be performed according to the procedures outlined in the manufacturer's manual. Qualified personnel must also perform maintenance when there is evidence of degradation of peak resolution, a shift in the calibration curve, loss of sensitivity, or failure to continually meet one of the quality control criteria.

Table 20-2 lists examples of schedules for routine maintenance. It is the responsibility of each Department Manager to ensure that instrument maintenance logs, containing records of both preventive and corrective maintenance, are kept for all equipment in his/her department. Preventative maintenance procedures may also be outlined in analytical SOPs or instrument manuals.

Instrument maintenance logs are controlled and are used to document instrument problems, instrument repair, and maintenance activities. Maintenance logs must be kept for all major pieces of equipment. Instrument maintenance logs are also 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 (i.e., 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.).

- 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. The service representative may complete relevant sections of the maintenance 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 must 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 to be adequately operational by calibration, verification, and/or other test to demonstrate acceptable performance. The laboratory must examine the effect of this defect on previous analyses.

In the event of equipment malfunction that cannot be resolved, service must be obtained from the instrument vendor, manufacturer, or a qualified service technician, if such a service can be tendered. If on-site service is unavailable, arrangements are made to have the instrument shipped back to the manufacturer for repair. Back-up instruments, which have been approved for the analysis, 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 must 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 laboratory operations.

20.3 Support Equipment

This section applies to all devices that may not be the actual test instrument, but are necessary to support laboratory operations. These include but are not limited to: balances, ovens, refrigerators, freezers, incubators, water baths, temperature measuring devices, 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 that is A2LA accredited. 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 at least annually by a qualified service representative (i.e., A2LA accredited), 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 per the requirements of SOP *Balance Calibration* / NV03-213.

20.3.2 pH, Conductivity, and Turbidity Meters

The pH meters used in the laboratory are accurate to at least + 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 that bracket the expected pH 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% umhos/cm.

Turbidity meters are also calibrated before each use. All of this information is documented in logs.

Consult pH and Conductivity, and Turbidity SOPs for further information.

20.3.3 Thermometers

All thermometers are calibrated on an annual basis with a NIST-traceable thermometer. IR thermometers, digital probes and thermocouples are calibrated quarterly.

Mercury/digital NIST thermometers are recalibrated annually (unless thermometer has been exposed to temperature extremes or apparent separation of internal liquid) by an approved outside service that is A2LA accredited and the provided certificate of traceability, including uncertainty, is kept on file. The NIST thermometer(s) have increments of 1 degree (0.5 degree or less increments are required for drinking water microbiological laboratories), and have ranges applicable to method and certification requirements. The NIST traceable thermometer is used for no other purpose than to calibrate other thermometers.

All of this information is documented in logbooks. Monitoring method-specific temperatures, including incubators, heating blocks, water baths, and ovens, is documented in method-specific logbooks. More information on this subject can be found in the SOP *Thermometer Calibration / Temperature Monitoring and Documentation* / NV03-212.

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 continuously.

Ovens, waterbaths and incubators are monitored on days of use.

All of this equipment has a unique identification number, and is assigned a unique temperature device for monitoring.

Sample storage refrigerator temperatures are nominally > 0ºC and < 6 ºC.

Specific temperature settings/ranges for other refrigerators, ovens waterbaths, and incubators can be found in method specific SOPs.

All of this information is documented in Daily Temperature Logbooks and method-specific logbooks.

20.3.5 Autopipettors, Dilutors, and Syringes

Mechanical volumetric dispensing devices including burettes (except Class A Glassware and Glass microliter syringes) are given unique identification numbers and the delivery volumes are verified gravimetrically, at a minimum, on a quarterly basis.

For those dispensers that are not used for analytical measurements, a label is / can be applied to the device stating that it is not calibrated. Any device not regularly verified cannot be used for any quantitative measurements.

Micro-syringes are purchased from Hamilton Company. Each syringe is traceable to NIST. The laboratory keeps on file an "Accuracy and Precision Statement of Conformance" from Hamilton attesting established accuracy.

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 (average RF or CF, regression 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 reanalysis is not possible, any data associated with an unacceptable initial calibration must be reported with appropriate data qualifiers (refer to Section 12).

Note: Instruments are calibrated initially and as needed after that and at least annually.

20.4.1 Calibration Standards

Calibration standards are prepared using the procedures indicated in the Reagents and Standards section of the determinative method SOP. If a reference method does not specify the number of calibration standards, a minimum of 3 calibration points will be used.

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. They must be obtained from an ISO Guide 34 verified vendor.

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. 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., flagged with defined qualifiers or additional information may be included in the case narrative). The exception to these rules is ICP methods or other methods where the referenced method does not specify two or more standards.

All initial calibrations are verified with a standard obtained from a second source and traceable to a national standard, when available (or vendor certified different lot if a second source is not available). For unique situations, such as air analysis where no other source or lot is available, a standard made by a different analyst at a different time or a different preparation would be considered a second source. This verification occurs immediately after the calibration curve has been analyzed, and before the analysis of any samples.

20.4.1.1 Calibration Verification

The calibration relationship established during the initial calibration must be verified at periodic intervals 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. Continuing calibration verifications use the same source standards as the calibration curve.

All target analytes and surrogates, including those reported as non-detects, must be included in periodic calibration verifications for purposes of retention time confirmation and to demonstrate that calibration verification criteria are being met.

All samples must be bracketed by periodic analyses of standards that meet the QC acceptance criteria (e.g., calibration and retention time). The frequency is found in the determinative methods or SOPs.

Note: If an internal standard calibration is being used (i.e., GCMS) then bracketing standards are not required, only initial 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 or standard that can be injected within 12 hours of the beginning of the shift.

A continuing instrument calibration verification (CCV) standard must be repeated at the beginning and at the end of each analytical batch for non-GC/MS methods. Some methods have more frequent CCV requirements see specific SOPs. Most inorganic methods require the CCV to be analyzed after every 10 samples.

Note: If an internal standard calibration is being used (i.e., GCMS) then bracketing standards are not required, only initial daily verifications are needed. The results from these verification standards must meet the calibration verification criteria and the retention time criteria (if applicable).

If the results of a CCV are outside the established acceptance criteria and analysis of a second consecutive (and immediate) CCV fails to produce results within acceptance criteria, corrective action shall be performed. Once corrective actions have been completed & documented, the laboratory shall demonstrate acceptable instrument / method performance by analyzing **two** consecutive CCVs, or a new initial instrument calibration shall be performed.

Sample analyses and reporting of data may not occur or continue until the analytical system is calibrated or calibration verified. However, data associated with an unacceptable calibration verification may be fully useable under the following special conditions: and reported based upon discussion and approval of the client:

a). When the acceptance criteria for the CCV are exceeded high (i.e., high bias) and the associated samples within the batch are non-detects, then those non-detects may be reported with a footnote or case narrative explaining the high bias in the CCV. 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.

Note: Refer to the applicable SOP for approved corrective actions when analyzing samples for the Ohio VAP.

20.4.1.2 Verification of Linear and Non-Linear Calibrations

Calibration verification involves the calculation of the percent drift or the percent difference of the instrument response between the initial calibration and each subsequent analysis of the verification standard. Use the equations shown in method SOPs to calculate % Drift or % Difference. Verification standards are evaluated based on the % Difference from the average CF or RF of the initial calibration or based on % Drift or % Recovery if a linear or quadratic curve is used.

Regardless of whether a linear or non-linear calibration model is used, if initial verification criterion is not met, then no sample analyses may take place until the calibration has been verified or a new initial calibration is performed that meets the specifications listed in the method SOPs. If the calibration cannot be verified after the analysis of a single verification standard, then adjust the

instrument operating conditions and/or perform instrument maintenance, and analyze another aliquot of the verification standard. If the calibration cannot be verified with the second standard, then a new initial calibration is performed.

- When the acceptance criteria for the calibration verification are exceeded high, i.e., high bias, and there are associated samples that are non-detects, then those non-detects may be reported. Otherwise, the samples affected by the unacceptable calibration verification shall be reanalyzed after a new calibration curve has been established, evaluated and accepted.
- When the acceptance criteria for the calibration verification are exceeded low, i.e., low bias, those sample results may be reported if they exceed a maximum regulatory limit/decision level. Otherwise, the samples affected by the unacceptable verification shall be reanalyzed after a new calibration curve has been established, evaluated and accepted. Alternatively, a reporting limit standard may be analyzed to demonstrate that the laboratory can still support non-detects at their reporting limit.

Note: Refer to the applicable SOP for approved corrective actions when analyzing sample for the Ohio VAP.

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 is determined by the purpose of the analyses being conducted. Data system library search routines do 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 must not be reported as a TIC. If the compound is reported on the same form as true TICs, it must be qualified and/or narrated that the reported compound is qualitatively and quantitatively (if verification in control) reported compared to a known standard that is in control (where applicable).

Note: TestAmerica Nashville cannot provide 'certified data' for TICs under the Ohio VAP.

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 likely library spectra may the analyst assign a tentative identification.

20.6 GC/MS Tuning

Prior to beginning any GCMS analytical sequence, including calibration, the instrument parameters for the tune and subsequent sample analyses within that sequence must be set.

Prior to tuning/auto-tuning the mass spectrometer, operating parameters may be adjusted within the specifications set by the manufacturer or the analytical method. These generally do not need any adjustment, but alteration 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 must be documented in the maintenance log.

Table 20-1. Laboratory Equipment and Instrumentation

Table 20-1: Equipment/ Instrument	Manufacturer	Model Number	Serial Number	Year Put into Service	Condition When Received
ABSORPTION UNIT	MITSUBISHI	Sigma 10	7520591	1996	USED
ACCELERATED SOLVENT EXTRACTOR	DIONEX	ASE200	02060437	2002	NEW
ACCELERATED SOLVENT EXTRACTOR	DIONEX	ASE200	02060440	2002	NEW
AUTO BOAT	MITSUBISHI	ABC	75A10673	1996	USED
AUTOSAMPLER	CENTAC TECHNOLOGIES	ASX-510	120347ASX	2005	NEW
AUTOSAMPLER	AMERICAN PRECISION	3i9A	9033	1992	[1]
AUTOSAMPLER	AMERICAN PRECISION	32E-6108AY	9205	1992	[1]
AUTOSAMPLER	DIONEX	ASI-100	4290509	2005	NEW
AUTOSAMPLER	DIONEX	ASI-100	4440407	2005	NEW
AUTOSAMPLER	EST	CENTURION	CENT123020504	[1]	[1]
AUTOSAMPLER	EST	CENTURION	CENT153112904	[1]	[1]
AUTOSAMPLER	EST	CENTURION	CENT124030104	2004	[1]
AUTOSAMPLER	EST	CENTURION	CENT126030104	[1]	[1]
AUTOSAMPLER	EST	CENTURION	CENT197042106	[1]	[1]
AUTOSAMPLER	EST	CENTURION	CENT196041706	[1]	[1]
AUTOSAMPLER	EST	CENTURION	CENT171060705	[1]	[1]
AUTOSAMPLER	EST	CENTURION	CENT172060705	[1]	[1]
AUTOSAMPLER	EST	CENTURION	CENT176102105	[1]	[1]
AUTOSAMPLER	EST	CENTURION	CENT174060705	[1]	[1]
AUTOSAMPLER	EST	CENTURION	CENT198050106	[1]	[1]
AUTOSAMPLER	EST	ENCON	215081902	[1]	[1]
AUTOSAMPLER	EST	ENCON	219082202	[1]	[1]
AUTOSAMPLER	HEWLETT PACKARD	718593B	3114A25542	[1]	[1]
AUTOSAMPLER	HEWLETT PACKARD	HP7673	3247A30576	2005	[1]
AUTOSAMPLER	HEWLETT PACKARD	HP7673	3514A42318	[1]	[1]
AUTOSAMPLER	HEWLETT PACKARD	HP7673A	2843A1152	[1]	[1]
AUTOSAMPLER	HEWLETT PACKARD	HP7673	3248A33147	[1]	[1]
AUTOSAMPLER	HEWLETT PACKARD	HP7673	2546A01716	[1]	[1]
AUTOSAMPLER	HEWLETT PACKARD	HP7673	3514A42318	2007	USED
AUTOSAMPLER	SHIMADZU	SIL-10A	60341F	1997	NEW
AUTOSAMPLER	SHIMADZU	SIL-10A	C20363506886	1999	NEW
AUTOSAMPLER	SHIMADZU	SIL-10A	C20363506585	1999	NEW
AUTOSAMPLER	SHIMADZU	AOC20i	C11143602454	[1]	[1]
AUTOSAMPLER	SUPERIOR ELECTRIC	MO61-LF-517	A218524	1992 OR 93	[1]

Table 20-1: Equipment/ Instrument	Manufacturer	Model Number	Serial Number	Year Put into Service	Condition When Received
AUTOSAMPLER	SUPERIOR ELECTRIC	MO61-LF-517	033473653	2003	[1]
AUTOSAMPLER	SUPERIOR ELECTRIC	MO61-LF-517	040939389	2004	[1]
AUTOSAMPLER	THERMO JARRELL ASH	AS300	12677003	1994	NEW
AUTOSAMPLER	THERMO JARRELL ASH	AS300	0492	1997	NEW
AUTOSAMPLER	THERMO JARRELL ASH	AS300	67247	1998	USED
AUTOSAMPLER	OI ANALYTICAL	4552	LR92489	1998	NEW
AUTOSAMPLER	VARIAN	ARCHON	11798-895	[1]	[1]
AUTOSAMPLER	VARIAN	ARCHON	12314	1998	NEW
AUTOSAMPLER	VARIAN	ARCHON	13735	2002	NEW
AUTOSAMPLER	VARIAN	ARCHON	13790	2002	NEW
AUTOSAMPLER	VARIAN	ARCHON	13800	2002	NEW
AUTOSAMPLER	VARIAN	ARCHON	12201	1998	NEW
AUTOSAMPLER	VARIAN	ARCHON	12194	1998	NEW
AUTOSAMPLER	VARIAN	ARCHON	12307	1998	NEW
AUTOSAMPLER	VARIAN	ARCHON	12761	1998	NEW
AUTOSAMPLER	VARIAN	ARCHON	12974	1999	NEW
AUTOSAMPLER	VARIAN	ARCHON	13161	[1]	[1]
AUTOSAMPLER	VARIAN	ARCHON	13812	[1]	[1]
AUTOSAMPLER	VARIAN	ARCHON	13765	[1]	[1]
AUTOSAMPLER	VARIAN	ARCHON	12945	[1]	[1]
AUTOSAMPLER	VARIAN	ARCHON	13722	[1]	[1]
AUTOSAMPLER	VARIAN	ARCHON	12920	[1]	[1]
AUTOSAMPLER	VARIAN	ARCHON	13600	[1]	[1]
AUTOSAMPLER	VARIAN	ARCHON	13641	[1]	[1]
AUTOSAMPLER	VARIAN	ARCHON	13087	1997	NEW
AUTOSAMPLER	VARIAN	ARCHON	12958	1996	USED
AUTOSAMPLER	VARIAN	ARCHON	13748	1995	NEW
AUTOSAMPLER	VARIAN	ARCHON	13291	1994	NEW
AUTOSAMPLER	VARIAN	ARCHON	13077	[1]	[1]
AUTOSAMPLER	VARIAN	ARCHON	13723	2006	USED
AUTOSAMPLER	VARIAN	ARCHON	13165	1994	NEW
AUTOSAMPLER TOWER	HEWLETT PACKARD	18596C	3530A43392	[1]	[1]
AUTOSAMPLER TOWER	HEWLETT PACKARD	18596C	3120A26333	[1]	[1]
AUTOSAMPLER TOWER	HEWLETT PACKARD	18596C	3120A27415	[1]	[1]
AUTOSAMPLER TOWER	HEWLETT PACKARD	18593A	2843A1162	[1]	[1]
AUTOSAMPLER TOWER	HEWLETT PACKARD	18593B	3114A25542	[1]	[1]
AUTOSAMPLER TOWER	HEWLETT PACKARD	18593B	3447A40940	[1]	[1]
AUTOSAMPLER TOWER	AGILENT	7683	US03815233	[1]	[1]

Table 20-1: Equipment/ Instrument	Manufacturer	Model Number	Serial Number	Year Put into Service	Condition When Received
AUTOSAMPLER TOWER	AGILENT	7683	US91807060	[1]	[1]
AUTOSAMPLER TOWER	AGILENT	7683	US94209739	[1]	[1]
AUTOSAMPLER TOWER	AGILENT	7683	US00411367	[1]	[1]
AUTOSAMPLER TOWER	AGILENT	7683	CN30729442	2010	USED
AUTOSAMPLER TOWER	AGILENT	1200	DE64775625	2010	NEW
AUTOSAMPLER TOWER	HEWLETT PACKARD	7673	3351A37407	2010	AUTOSAMPLER
AUTOSAMPLER TRAY	HEWLETT PACKARD	18593B	3106A21456	[1]	[1]
AUTOSAMPLER TRAY	HEWLETT PACKARD	18593B	3415A35076	[1]	[1]
AUTOSAMPLER TRAY	HEWLETT PACKARD	18593B	3339A33311	[1]	[1]
AUTOSAMPLER TRAY	HEWLETT PACKARD		3348A31236	[1]	[1]
AUTOSAMPLER TRAY	HEWLETT PACKARD		(unreadable)	[1]	[1]
AUTOSAMPLER TRAY	HEWLETT PACKARD		3131A25799	[1]	[1]
AUTOSAMPLER TRAY	HEWLETT PACKARD		3304A31194	[1]	[1]
AUTOSAMPLER TRAY	HEWLETT PACKARD	6890	3514A42318	2010	USED
AUTOSAMPLER TRAY	HEWLETT PACKARD	6890	3415A35076	2010	USED
AUTOSAMPLER TRAY	AGILENT	7683	US82601220	[1]	[1]
AUTOSAMPLER TRAY	AGILENT	7683	US81100420	[1]	[1]
AUTOSAMPLER TRAY	AGILENT	7683	US24314927	[1]	[1]
AUTOSAMPLER TRAY	AGILENT	7683	US00407128	[1]	[1]
AUTOSAMPLER TRAY	AGILENT	7683	CN91653019	2010	USED
AUTOSAMPLER TRAY	AGILENT	1200	DE64775625	2010	NEW
AUTOSAMPLER TRAY	EST	CENTURION WS	CENTS157052610	2010 USED	
AUTOSAMPLER WITH HEADSPACE	MARKELOV	HS9000	HS121053007	2007	NEW
AUTOSAMPLER WITH HEADSPACE	MARKELOV	HS9000	HS120053007	2007	NEW
BALANCE	METTLER	AE240	N28161	1993	NEW
BALANCE	METTLER	PB602-S	1118381704	[1]	[1]

Table 20-1: Equipment/ Instrument	Manufacturer	Model Number	Serial Number	Year Put into Service	Condition When Received
BALANCE	METTLER	PB602	1117421936	[1]	[1]
BALANCE	METTLER	PB602-S	1126143319	2005	NEW
BALANCE	METTLER	PG802	1115352767	[1]	[1]
BALANCE	METTLER	PB602-S	1120430697	[1]	NEW
BALANCE	METTLER	AE200	F15224	[1]	[1]
BALANCE	METTLER	PM600T	H19877	[1]	[1]
BALANCE	METTLER	PB302	1116383230	[1]	[1]
BALANCE	METTLER	AE200	L94571	[1]	[1]
BALANCE	METTLER	PG203-S	119331062	[1]	NEW
BALANCE	METTLER	AE200	J63202	[1]	NEW
BALANCE	METTLER	PL601-S	1202140224	[1]	NEW
BALANCE	METTLER	AG204	1122173241	2003	NEW
BLOCK DIGESTOR	LACHAT	BE – 46	100400000973	2010	NEW
BOD ANALYZER	MANTEC	PC-BOD	PC-1805-00	2013	USED
BOD INCUBATOR	FISHER	307	WB05105311	1990	NEW
BOD INCUBATOR	FISHER	FU199ERWI	FGL52777	1989	USED
BOD INCUBATOR	KENMORE	253.65802508	BA73420257	2008	NEW
BOD INCUBATOR	EQUATHERM	HA757376	TA14SLB	1992	USED
CALORIMETER	PARR	1341	5427	1986	NEW
CENTRIFUGE	BECKMAN	ALLEGRA 6	ALS08D16	2009	NEW
CENTRIFUGE	CLAY ADAMS	DYNAC III 420104	3820031	[1]	NEW
CENTRIFUGE	CLAY ADAMS	DYNAC III 420104	4150009	[1]	NEW
CHILLER	NESLAB	CFT-33	293223	1993	NEW
COD REACTOR	HACH	DR 3900	1415997	2011	NEW
COD REACTOR	HACH	DR 6000	1509235	2013	NEW
CONDUCTIVITY METER	WTW	330i	0148004	2002	NEW
DISCRETE ANALYZER	KONELAB	AQUA 20	E0719526	2004	NEW
DISCRETE ANALYZER	KONELAB	AQUA 20	S2419226	2009	USED
DISCRETE ANALYZER	KONELAB	AQUA 20	E07 19524	2011	USED
DISSOLVED OXYGEN METER	YSI	5100	00B0314	2000	NEW
DISSOLVED OXYGEN METER	YSI	5100	00D0552	2000	NEW
DISSOLVED OXYGEN METER	YSI	5100	08C 101838	2013	NEW
FLASHPOINT TESTER, AUTO	PETROLAB	PMA-4	741	1997	NEW
FLASHPOINT TESTER, AUTO	PETROLAB	PMA-4	741	1997	NEW
FLASHPOINT, OPEN CUP	FISHER	--	201N0005	2002	NEW
FLASHPOINT, PENSKY-MARTENS	FISHER	13-497-5	1787	1986	NEW
FLOW INJECTION ANALYZER	LACHAT	8000	A83000234	1995	NEW

Table 20-1: Equipment/ Instrument	Manufacturer	Model Number	Serial Number	Year Put into Service	Condition When Received
FLOW INJECTION ANALYZER	LACHAT	8500 FIA	050600000167	2008	USED
FLOW INJECTION ANALYZER	LACHAT	8500	090400001098	2009	NEW
FLOW INJECTION ANALYZER	LACHAT	8500 Series 2	1208400000144 5	2012	New
FLUOROMETER	SEQUOIA - TURNER	450	B002262TV	1992	NEW
FURNACE	THERMOLYN	30400	10599810039263	1984	NEW
GC w/ECD/ECD	PERKIN ELMER	AUTOSYS	610N9122009	2000	NEW
GC w/ECD/ECD	HEWLETT PACKARD	6890	US00011427	2000	NEW
GC w/ECD/ECD	HEWLETT PACKARD	5890	3336A61865	2006	USED
GC w/ECD/ECD	HEWLETT PACKARD	5890	3336A51644	2007	USED
GC w/ECD/ECD	HEWLETT PACKARD	7890A	CN10734082	2009	NEW
GC w/FID	HEWLETT PACKARD	5890	3336A52049	1998	NEW
GC w/FID	HEWLETT PACKARD	5890	2950A27678	[1]	[1]
GC w/FID	HEWLETT PACKARD	5890	3108A34413	1999	USED
GC w/FID	HEWLETT PACKARD	5890	3235A45233	2006	USED
GC w/FID	HEWLETT PACKARD	5890	2950A26161	2006	USED
GC w/FID	HEWLETT PACKARD	5890/7673	3235A45233	2006	USED
GC w/FID	HEWLETT PACKARD	5890	3336A51997	2006	USED
GC w/FID	HEWLETT PACKARD	5890	3235A46759	2009	USED
GC w/FID	HEWLETT PACKARD	6890/7683	US00029922	2010	USED
GC w/FID	HEWLETT PACKARD	6890	US00010165	2010	USED
GC w/FID	HEWLETT PACKARD	5890	3336A61864	2010	USED
GC w/FID	HEWLETT PACKARD	6890	CN10727030	2010	NEW
GC w/FID	SHIMADZU	17A	C11123580493	1998	NEW
GC w/FID	SHIMADZU	17A	C11123781625	1999	NEW
GC w/FID	SHIMADZU	17A	C11123781731	1999	NEW
GC w/NPD/FPD	PERKIN ELMER	CLARUS 500	650N4061602	2004	NEW
GC w/PID/FID	HEWLETT PACKARD	5890	3027A29703	2001	USED
GC w/PID/FID	HEWLETT PACKARD	5890	3126A36232	1998	USED

Table 20-1: Equipment/ Instrument	Manufacturer	Model Number	Serial Number	Year Put into Service	Condition When Received
GC w/PID/FID	HEWLETT PACKARD	5890	2908A91269	1990	USED
GC w/PID/FID	HEWLETT PACKARD	5890	3133A37656	1996	USED
GC w/PID/FID	HEWLETT PACKARD	5890	3203A42091	1996	USED
GC w/PID/FID	HEWLETT PACKARD	5890	3336A58503	1998	USED
GC w/PID/FID	HEWLETT PACKARD	5890	2938A24989	1998	USED
GC w/PID/FID	HEWLETT PACKARD	5890	3336A50264	1998	USED
GC w/PID/FID	HEWLETT PACKARD	5890	2938A25493	1998	USED
GC w/PID/FID	HEWLETT PACKARD	6890	US00041322	2002	NEW
GC w/PID/FID	HEWLETT PACKARD	6890	US00041324	2002	NEW
GC w/PID/FID	HEWLETT PACKARD	6890	US00027429	2002	NEW
GC w/PID/FID	HEWLETT PACKARD	5890	3336A60618	2003	USED
GC w/PID/FID	HEWLETT PACKARD	5890	DE00003878	2003	NEW
GC w/PID/FID	HEWLETT PACKARD	6890N	US10350005	2004	NEW
GC w/PID/FID	HEWLETT PACKARD	6890N	US10350002	2004	NEW
GC w/PID/FID	HEWLETT PACKARD	5890	2950A27370	2005	USED
GC w/PID/FID	HEWLETT PACKARD	5890	3223A43282	2005	USED
GC w/PID/FID	HEWLETT PACKARD	5890	3019A28662	2005	USED
GC w/PID/FID	HEWLETT PACKARD	5890	3203A41711	2006	USED
GC w/PID/FID	HEWLETT PACKARD	5890	3203A41407	2007	USED
GC/MS	HEWLETT PACKARD	5890II/5972	3307A00396	1997	USED
GC/MS	HEWLETT PACKARD	5890II/5972	3329A00693	1994	NEW
GC/MS	HEWLETT PACKARD	5890II5972	3501A02393	1995	NEW
GC/MS	HEWLETT PACKARD	6890/5973	US82311321	1999	USED
GC/MS	HEWLETT PACKARD	6890/5973	US80210937	1999	USED
GC/MS	HEWLETT PACKARD	6890/5973	US94222434	2000	NEW
GC/MS	HEWLETT PACKARD	6890/5973N	US01140208s	2000	NEW
GC/MS	HEWLETT PACKARD	G1530A/ G1099A	US91421544	1999	NEW

Table 20-1: Equipment/ Instrument	Manufacturer	Model Number	Serial Number	Year Put into Service	Condition When Received
GC/MS	HEWLETT PACKARD	6890/5973	US81221486	2000	NEW
GC/MS	HEWLETT PACKARD	6890/5973	US91921735	2000	NEW
GC/MS	HEWLETT PACKARD	6890/5973	US94212231	2000	NEW
GC/MS	HEWLETT PACKARD	6890/5973	US94211203	2000	NEW
GC/MS	HEWLETT PACKARD	6890/5973	US94260109	2001	USED
GC/MS	HEWLETT PACKARD	6890/5973N	US03340475	2001	NEW
GC/MS	HEWLETT PACKARD	6890/5973N	US03340479	2002	NEW
GC/MS	HEWLETT PACKARD	6890/5973N	US21844021	2002	NEW
GC/MS	HEWLETT PACKARD	6890/5973N	US21844025	2002	NEW
GC/MS	HEWLETT PACKARD	5890/5972	3336A57928	2004	USED
GC/MS	HEWLETT PACKARD	5890/5972	3235A44162	2005	USED
GC/MS	HEWLETT PACKARD	5890/5972	2423A01966	2005	USED
GC/MS	HEWLETT PACKARD	5890/5973	US00027481	2005	USED
GC/MS	HEWLETT PACKARD	6890/5973	4510250045	2005	USED
GC/MS	HEWLETT PACKARD	6890/5973	US00037997	2006	USED
GC/MS	HEWLETT PACKARD	6890/5973	US00037995	2006	USED
GC/MS	HEWLETT PACKARD	6890/5973	US00027177	2006	USED
GC/MS	HEWLETT PACKARD	6890/5973	US00033436	2006	USED
GC/MS	HEWLETT PACKARD	6890/5973	US00008026	2006	USED
GC/MS	HEWLETT PACKARD	6890/5973	US00022412	2008	USED
GC/MS	HEWLETT PACKARD	6890/5973	US10462174	2009	NEW
GC/MS	HEWLETT PACKARD	6890/5973	US10204013	2010	USED
GC/MS	HEWLETT PACKARD	6890/5973	US00006290	2013	USED
GC/MS w/ENTEC CONCENTRATOR	HEWLETT PACKARD	6890/5973N	US10450528	2001	NEW
HEADSPACE ANALYZER	TEKMAR	7000	91242001	2001	USED
HOTPLATES	THERMODYNE	CIMAREC 3		1991 -	NEW
HPLC	DIONEX	TCC-100MSV	1280301	2005	NEW
HPLC	AGILENT	1200	DE60555493	2010	NEW
HPLC	AGILENT	1200	DE62964850	2012	NEW

Table 20-1: Equipment/ Instrument	Manufacturer	Model Number	Serial Number	Year Put into Service	Condition When Received
ICP – TRACE	THERMO-FISHER	6500	20100905	2010	NEW
ICP – TRACE	THERMO SCIENTIFIC	ICAP 6000 SERIES	20084103	2009	NEW
ICP – TRACE	THERMO SCIENTIFIC	ICAP 6000 SERIES	20122509	2012	NEW
ICP/MS	HEWLETT PACKARD	7500CE	JP51201460	2005	NEW
ICP/MS	HEWLETT PACKARD	7500CX	JP51202170	2013	NEW
ION CHROMATO-GRAPH	METROHM	861	04168	2010	NEW
ION CHROMATO-GRAPH	METROHM	861	07200	2010	NEW
ION CHROMATO-GRAPH	METROHM	861	03135	2010	NEW
MERCURY ANALYZER	LEEMAN	Hydra II	0031	2010	NEW
MERCURY ANALYZER	LEEMAN	HYDRA II	0002	2010	NEW
MICROWAVE	CEM	MARS XPRESS 907501	MD2979	2009	NEW
MICROWAVE	CEM	MARS XPRESS 907501	MD8469	2006	NEW
MIDI-DISTILLATION	ANDREWS GLASS		MCVA1290374	1994	NEW
MIDI-DISTILLATION	WESTCO	EASY-DIST	EA-1006	1996	NEW
OVEN	BLUE M	SW11TA1	SW3034	1989	NEW
OVEN	BLUE M	SW17TA1	SW7252	1993	NEW
OVEN	BLUE M	SWIITA-1	SW3990	[1]	[1]
OVEN	FISHER	ISOTEMP 650G	803N0038	[1]	[1]
OVEN	FISHER	ISOTEMP 516G	903N0083	[1]	[1]
OVEN	FISHER	500	51100409	[1]	[1]
OVEN	NAPCO	620	(ILLEGIBLE)	[1]	[1]
OVEN	FISHER	ISOTEMP 650G	202N0036	2002	NEW
OVEN	FISHER	6926	608516-30	2010	NEW
OVEN	FISHER	6926	275812-45	2011	NEW
OVEN	FISHER	6926	275828-62	2011	New
OXYGEN PROBE	ORION	97-08-00		1991	NEW
pH METER	ORION	330	32663	1999	NEW
pH METER	ORION	330	32663	1999	NEW
pH/mv METER	ACCUMET	AB15	AB92331859	2009	NEW
pH/mv METER	ACCUMET	AB15	642122	2011	NEW
pH/mv METER	ACCUMET	AB15	AB92340881	2013	NEW
pH/mv METER	ACCUMET	AB15	AB92333068	2013	NEW
pH/mv METER	ACCUMET	AB150	AB92340683	2013	NEW
pH/mv METER	ACCUMET	AB15	AB92337680	[1]	[1]
PURGE/TRAP	EST	ENCON	217082202 E	1997	NEW
PURGE/TRAP	EST	ENCON	126112900 E	1997	NEW

Table 20-1: Equipment/ Instrument	Manufacturer	Model Number	Serial Number	Year Put into Service	Condition When Received
PURGE/TRAP	EST	ENCON	216081902 E	1994	NEW
PURGE/TRAP	EST	ENCON	218082202 E	1994	NEW
PURGE/TRAP	EST	ENCON	215081902E/P	1	1
PURGE/TRAP	EST	ENCON	219082202E/P	1	1
PURGE/TRAP	EST	ENCON	237082022	2006	USED
PURGE/TRAP	EST	ENCON	126112900	2006	USED
PURGE/TRAP	HEWLETT PACKARD	G1900-60500	3651A10630	1995	NEW
PURGE/TRAP	HEWLETT PACKARD	3000	3636A10579	1	1
PURGE/TRAP	TEKMAR	3000	93251007	1993	NEW
PURGE/TRAP	TEKMAR	3000	95093006	1995	NEW
PURGE/TRAP	TEKMAR	3000	99053016	2003	1
PURGE/TRAP	TEKMAR	3000	94057008	1	1
PURGE/TRAP	TEKMAR	3000	98236009	1	1
PURGE/TRAP	TEKMAR	3000	98139001	1	1
PURGE/TRAP	TEKMAR	3000	98098001	1	1
PURGE/TRAP	TEKMAR	3000	98364008	1	1
PURGE/TRAP	TEKMAR	3000	93316011	1	1
PURGE/TRAP	TEKMAR	3000	94252002	1	1
PURGE/TRAP	TEKMAR	3000	98111004	1	1
PURGE/TRAP	TEKMAR	3000	93334005	1	1
PURGE/TRAP	TEKMAR	3000	95144001	1	1
PURGE/TRAP	TEKMAR	3000	95166007	1	1
PURGE/TRAP	TEKMAR	3000	94266005	1	1
PURGE/TRAP	TEKMAR	3000	99196020	1	1
PURGE/TRAP	TEKMAR	3000	97009013	1	1
PURGE/TRAP	TEKMAR	3000	98092002	1	1
PURGE/TRAP	TEKMAR	3000	995207010	1	1
PURGE/TRAP	TEKMAR	3000	98008002	1	1
PURGE/TRAP	TEKMAR	3000	3449A10215	1	1
PURGE/TRAP	TEKMAR	3000	95153007	1	1
PURGE/TRAP	TEKMAR	3000	98229017	1	1
PURGE/TRAP	TEKMAR	3000	94005035	1	1
PURGE/TRAP	TEKMAR	3000	94005015	1	1
PURGE/TRAP	TEKMAR	3000	96325013	1	1
PURGE/TRAP	TEKMAR	3000	93170002	1	1
PURGE/TRAP	TEKMAR	3000	99076004	1	1
PURGE/TRAP	TEKMAR	LCS-3000	94067010	2002	NEW
PURGE/TRAP	TEKMAR	3100	02115005	1	1
PURGE/TRAP	TEKMAR	3100	00104011	1	1
PURGE/TRAP	TEKMAR	3100	00104012	1996	USED
PURGE/TRAP	TEKMAR	3100	99305025	1	1
PURGE/TRAP	TEKMAR	3100	US01354002	2004	1
PURGE/TRAP	TEKMAR	3100	US02249003	2004	1
PURGE/TRAP	TEKMAR	3100	US02155008	1	1
PURGE/TRAP	TEKMAR	3100	US02326003	1	1
PURGE/TRAP	TEKMAR	3100	95129003	1	1
PURGE/TRAP	TEKMAR	3100	94104002	1	1

Table 20-1: Equipment/ Instrument	Manufacturer	Model Number	Serial Number	Year Put into Service	Condition When Received
PURGE/TRAP	TEKMAR	3100	97273002	[1]	[1]
RECIPROCAL WATER BATH	NEW BRUNSWICK SCIENTIFIC	R 76	400364888	2003	NEW
ROTATOR	ASSOC. DESIGN	3740-48BRE		1994	NEW
SOLID PHASE EXTRACTOR	HORIZON TECHNOLOGY	SPE-3000XL	01-1200	2004	NEW
SONICATOR	SONICS & MATERIALS	VCX600	28785F	1999	NEW
SONICATOR	SONICS & MATERIALS	VC750	31469F	2000	NEW
SONICATOR	SONICS & MATERIALS	VC750	37577G	2000	NEW
SONICATOR	SONICS & MATERIALS	VC750	59927AC	2003	NEW
SONICATOR	SONICS & MATERIALS	VC750	59933AC	2010	NEW
SONICATOR	MISONIX	XL2020	G4428	2012	USED
SOXHLET EXTRACTOR, AUTOMATED	GERHARDT	MULTISTAT SX/PC	NA	2007	NEW
SOXHLET EXTRACTOR, AUTOMATED	GERHARDT	MULTISTAT SX/PC	NA	2007	NEW
SOXHLET EXTRACTOR, AUTOMATED	GERHARDT	MULTISTAT SX/PC	NA	2007	NEW
SOXHLET EXTRACTOR, AUTOMATED	GERHARDT	MULTISTAT SX/PC	NA	2007	NEW
THERMOMETER (NIST)	ERTCO	ASTM 62C	4240	2004	NEW
THERMOMETER (NIST)	ERTCO	ASTM 62C	4254	2004	NEW
THERMOMETER (NIST)	ERTCO	1007	3161	2006	NEW
THERMOMETER (NIST)	ERTCO	1007	3437	2009	NEW
TITRATOR, AUTO	MAN-TECH	PCM-1104-00	MS-OC7-752	2008	NEW
TITRATOR, AUTO	MAN-TECH	PC-1000.688	MS-1A1-603	2011	NEW
TITRATOR, AUTO	MAN-TECH	PCM-1104-00	MS-OG2-312	2012	USED
TOC	SHIMADZU	TOC-VCSN	H51204635241	2009	NEW
TOC	O.I. ANALYTICAL	1030W	F923730891 AUTOSAMPLER: 612788821	2009	NEW
TOC W/SOLIDS OPTION	SHIMADZU	TOC-VCSN	H51204635241	2009	NEW
TOX	MITSUBISHI	SIGMA 10	75R00110	1998	USED
TOX	MITSUBISHI	TOX-100	A7M41934	2009	USED
TOX	MITSUBISHI	TOX-10Σ	75R0328A	2013	USED
TOX	MITSUBISHI	TOX-100	A7M21109	2013	USED
TOX	MITSUBISHI	AOX-200	E7B20262	2012	USED
TURBIDIMETER	HF	DRT – 100B	602164	2000	NEW

Table 20-1: Equipment/ Instrument	Manufacturer	Model Number	Serial Number	Year Put into Service	Condition When Received
TURBIDIMETER	HF	MICRO 100	201008129	2010	NEW
UV/VIS	SHIMADZU	1601	A10753782132	2001	NEW
UV/VIS	SHIMADZU	UV-1800	A11454830564	2010	NEW
UV/VIS	THERMO	SPECTRONIC 200	8A2R232004	2013	NEW
ZHE PRESSURE	MILLIPORE	T316	22361028	1993	NEW
ZHE ROTATOR (4)	MILLIPORE	Z3	455TR4042	1990	NEW

[1]Although equipment is operational and calibration maintained, this information is not available.

Table 20-2. Preventive Maintenance Procedures for Laboratory Equipment

Table 20-2: Instrument/Equipment Type	Activity	Frequency
Gas Chromatograph	Change septum	Monthly
	Check gases	Daily
	Replace or clip column	Yearly
	Check autosampler seals	Each day of use
	Replace liners	Monthly
	Clean or replace PID lamp	Quarterly
Liquid Chromatography	Check seals for leakage	Each day of use
	Replace seals/valves	Yearly
	Replace column	Yearly
GC/MS	Change Merlin (SVOC only)	Quarterly
	Bake trap (VOC only)	Each day of use
	Clean source	Quarterly
	Change vacuum pump oil	Biannually
	Replace liner (SVOC only)	Each day of use
	Replace column	Yearly
ICP	Torch inspection	Each day of use
	Inspect filters	Each day of use
	Change filters	Yearly
	Inspect pump tubing	Each day of use
	Change pump tubing	Quarterly
	Inspect contact rings	Each day of use
	Clean windows	Each day of use
	Align lamp	Each day of use
	Clean Cores	Quarterly
Mercury Analyzer	Inspect tubes and reagents	Each day of use
Microwave	Check power output	Weekly
	Check rotation	Each day of use
pH Meter	Inspect electrode	Each day of use
TCLP Extractors And Zero Headspace Extractors	Verify rotation	Each day of use
	Check for leakage	Each day of use
UV/VIS Spectrometer	Clean sample compartment	Monthly
	Auto-check calibration	Daily at start-up
	Wavelength calibration	Biannually
Total Organic Carbon Analyzer	Check gas flow	Each day of use
	Check fluid level (IC reservoirs)	Each day of use
	Replace “O” rings	Biannually
	Check needle	Each day of use
	Replace scrubbers (halogen and CO_2)	Yearly
	Replace catalyst	Yearly
	Check acids/oxidizer	Each day of use

Table 20-2: Instrument/Equipment Type	Activity	Frequency
Total Organic Halogen Analyzer	Clean cell	Each day of use
	Check electrode levels	Each day of use
Weighing balances	Clean pan	Each day of use
	Check calibration	Each day of use
Temperature devices: refrigerators, incubators, evaporators, flash point tester, COD reactor, water circulator, drying ovens	Monitor temperature	Daily or when used (refrigerators 2 times per day)
Ultrasonic Disruptors	Clean, Tune by depressing tuning button while adjusting % output knob until lowest reading is obtained. For dual head, disconnect one horn at a time.	Each day of use
Discrete Analyzer	Perform “start-up”	Monthly
	Clear daily files	Each day of use
	Check water and waste container; empty if needed	Weekly
	Check cuvette bin	Weekly
	Check syringe plunger tip; replace syringe	Biannually
	Save database to CD	Monthly
	Delete messages	Monthly
	Replace lamp	Biannually

Table 20-3. Periodic Calibration

Table 20-3: Instrument	Type of Calibration/ Number of Standards	Frequency	Acceptance Limits	Corrective Action
Analytical Balance	Accuracy determined using A2LA-accredited NIST weights. Minimum of 2 standards bracketing the weight of interest.	Calibrated semi-annually. Daily calibration verification prior to use.	± 0.2%	Clean, check level, insure lack of drafts, and that unit is warmed up, recheck. If fails, call service.
Top Loading Balance	Accuracy determined using A2LA-accredited NIST weights. Minimum of 2 standards bracketing the weight of interest.	Calibrated semi-annually. Daily calibration verification prior to use.	± 0.5%	Clean. Replace.
A2LA-accredited NIST Weights	Accuracy determined by accredited weights and measurement laboratory.	1 year	As per certificate.	Replace.
NIST-Traceable Thermo-meter	Accuracy determined by A2LA-accredited weights and measurement laboratory.	1 year	As per certificate.	Replace.
Thermo-meter (liquid)	Against NIST-traceable thermometer	Yearly at appropriate temperature range for intended use	± 2.0°C	Replace
Minimum-Maximum Thermo-meters	Against NIST-traceable thermometer	Yearly	± 2.0°C	Replace
InfraRed Temperature Guns	Against NIST-traceable thermometer	Quarterly at appropriate temperature range for intended use.	± 1.5°C	Repair/replace
Dial-type Therm-ometers	Against NIST-traceable thermometer	Yearly at appropriate temperature range for intended use.	± 2.0°C	Replace
Digital Therm-ometers	Against NIST-traceable thermometer	Quarterly	±2.0°C	Replace

Table 20-3: Instrument	Type of Calibration/ Number of Standards	Frequency	Acceptance Limits	Corrective Action
Refrigerator	Temperature checked using NIST-traceable thermometer.	Daily. If out of range, check again after two readings (four hours).	2.7 ± 1.7°C	Adjust. Repair. While waiting for repair, seal door, attach "Out of Service" sign, move items to functional unit. Notify supervisor.
Freezer	Temperature checked using NIST-traceable thermometer	Daily. If out of range, check again after two readings (four hours).	(-10)-(-20)°C	Adjust. Repair. While waiting for repair, seal door, attach "Out of Service" sign, move items to functional unit. Notify supervisor.
Oven	Temperature checked using NIST-traceable thermometer.	When in use.	103 ± 1°C (drying) 180 ± 2°C (TDS)	Adjust. Replace.
Incubator	Temperature checked using NIST-traceable thermometer.	When in use.	BOD: 20 ± 1.0°C	Adjust. Replace.
Water Bath	Temperature checked using NIST-traceable thermometer.	When in use.	± 2°C	Adjust. Replace.
Volumetric Dispensing Devices (Eppendorf ® pipette, automatic dilutor or dispensing devices)	One delivery by weight. Using DI water or Methanol, dispense into tared vessel. Record weight with device ID number. Note: For dispensers not used for analytical measurements, a label is applied to the device stating that it is not calibrated.	Quarterly	± 2% Calculate accuracy by using controlled spreadsheets.	Adjust. Replace.
Glass Microliter Syringes	Syringes greater than 25 ul require calibration	Semi-annually	± 2%	Replace.
Conductivity Meter	Cell impedance calibrated with three KCl standards.	Each use.	r ≥ 0.99 +/- check std methods	Recalibrate.
Deionized Water	Check in-line conductivity meter on system with conductivity meter in Inorganics Department.	Weekly	<10 µmhos/cm^2	Record on log. Report discrepancies to Technical Manager.

Table 20-3: Instrument	Type of Calibration/ Number of Standards	Frequency	Acceptance Limits	Corrective Action
Barometer	Barometric pressure checked against 17025 standards	Yearly	±5 mBars	Replace.

SECTION 21. MEASUREMENT TRACEABILITY

21.1 Overview

Traceability of measurements shall be assured using a system of documentation, calibration, and analysis of reference standards. Items of 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 are subject to ongoing certifications of accuracy. At a minimum, these must include procedures for checking specifications of ancillary equipment: balances, thermometers, temperature, deionized (DI) and reverse osmosis (RO) water systems, automatic pipettes, and other volumetric measuring devices. With the exception of Class A glassware and glass microliter syringes that have a certificate of accuracy, quarterly accuracy checks are performed for all mechanical volumetric devices. Microsyringes are verified at least semiannually or disposed of after 6 months. 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 other deformity (e.g., bent needle). If the Class A glassware or syringe is suspect, the accuracy of the item must be assessed prior to use or it must be replaced.

21.2 NIST-Traceable Weights and Thermometers

Reference standards of measurement are used for calibration only and for no other purpose unless it can be shown that their performance as reference standards would not be invalidated.

For NIST-traceable weights and thermometers, the laboratory requires that all calibrations be conducted by a calibration laboratory accredited by A2LA, NVLAP (National Voluntary Laboratory Accreditation Program), APLAC (Asia-Pacific Laboratory Accreditation Cooperation), or EA (European Cooperation for Accreditation). A calibration certificate and scope of accreditation are kept on file at the laboratory.

The calibration laboratory's policy for achieving measurement traceability is defined and includes the following elements of uncertainty:

- The uncertainty calculations of the calibration laboratory are supported by uncertainty budgets and are represented by expanded uncertainties typically using a coverage factor of k=2 to approximate the 95% confidence level. This explanation must accompany the measurement result and the associated uncertainty value.
- The tolerance uncertainty ratio (TUR) must be calculated using the expanded uncertainty of the measurement, not the collective uncertainty of the measurement standards. A statement to this effect must accompany the TUR along with the coverage factor and confidence level.

The calibration report or certificate submitted to TestAmerica Nashville must contain, in a well designed format, a traceability statement, the conditions under which the calibrations were made in the context of any potential influence, a compliance statement with an identified metrological specification and the pertinent clauses, a clearly identified record of the quantities and functional test results before and after re-calibration, and no recommendation on the calibration interval. Opinions and interpretations of results are presented along with the basis upon which they were made and identified as such. The report may be submitted by facsimile

or other electronic means as long as the requirements of the International Standard are achieved. If significant amendments are made to a calibration certificate, a supplemental certificate for the serial-number-specified piece of equipment is so identified. When a new certificate is offered, it uniquely identifies and references the one it replaces. All calibration reports are filed in the QA Office or scanned to a network folder.

The calibration laboratory supports in-house calibration systems: documented procedures for in-house calibrations, evidence by a report, certificate, or sticker, for an appropriate amount of time; training records of calibration personnel; certificates from accreditation services demonstrating traceability to national or international standards of measurement; procedures for evaluating measurement uncertainty; timely and documented recalibration of reference standards. When subcontracting to a calibration laboratory, TestAmerica Nashville does not use a firm who subcontracts the work.

An external certified service engineer services laboratory balances on an annual basis. This service is documented on each balance with a signed and dated certification sticker. Balance calibrations are checked each day of use. All mercury thermometers are calibrated annually against a traceable reference thermometer. Temperature readings of ovens, refrigerators, and incubators are checked on each day of use.

21.3 Reference Standards / Materials

Reference standards/materials, where commercially available, are traceable to certified reference materials. Commercially prepared reference standards, to the extent available, are purchased from vendors that are accredited to ISO Guide 34 and ISO/IEC Guide 17025. All reference standards from commercial vendors shall be accompanied with a certificate that includes at least the following information:

- Manufacturer
- Analytes or parameters calibrated
- Identification or lot number
- Calibration method
- Concentration with associated uncertainties
- Purity

If a standard cannot be purchased from a vendor that supplies a Certificate of Analysis, the purity of the standard is documented by analysis. The receipt of all reference standards must be documented. Reference standards are labeled with a description of the standard, a unique Standard Identification Number and expiration date. All documentation received with the reference standard is retained as a QC record and references the Standard Identification Number.

All reference, primary, and working standards/materials, whether commercially purchased or laboratory prepared, must be checked regularly to ensure that the variability of the standard or material from the ‘true’ value does not exceed method requirements. The accuracy of calibration standards is checked by comparison with a standard from a second source. In cases where a second standard manufacturer is not available, a vendor certified different lot is acceptable for use as a second source. For unique situations, such as air analysis where no

other source or lot is available, a standard made by a different analyst would be considered a second source. The appropriate Quality Control (QC) criteria for specific standards are defined in laboratory SOPs. In most cases, the analysis of an Initial Calibration Verification (ICV) or LCS (where there is no sample preparation) is used as the second source confirmation. These checks are generally performed as an integral part of the analysis method (e.g. calibration checks, laboratory control samples).

All standards and materials must be stored and handled according to method or manufacturer's requirements in order to prevent contamination or deterioration. Refer to the Corporate Environmental Health & Safety Manual or laboratory SOPs. For safety requirements, please refer to method SOPs and the laboratory Environmental Health and Safety Manual.

Standards and reference materials shall not be used after their expiration dates unless their reliability is verified by the laboratory and their use is approved by the Quality Assurance Manager. The laboratory must have documented contingency procedures for re-verifying expired standards.

21.4 Documentation and Labeling of Standards, Reagents, and Reference Materials

At a minimum, reagents must be the purity cited in the applicable test method. The date of reagent receipt and the expiration date are documented. The lots for most of the common solvents and acids are tested for acceptability prior to company wide purchase. Refer to SOP No. CA-Q-S-001, *Solvent and Acid Lot Testing and Approval*.

All manufacturer- or vendor-supplied Certificates of Analysis or Purity must be retained, stored appropriately, and readily available for use and inspection. These records are maintained in the analytical departments of the laboratory or are scanned electronically to folder on the network. Records must be kept of the date of receipt and date of expiration of standards, reagents, and reference materials. In addition, records of preparation of laboratory standards, reagents, and reference materials must be retained, stored appropriately, and be readily available for use and inspection. For detailed information on documentation and labeling, please refer to method specific SOPs and SOP *Reagent and Standard Purchase, Preparation, Control Documentation* / NV08-214.

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 must 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%, oetherwise the certified value is used for the canister concentration.

21.4.1 All standards, reagents, and reference materials must be labeled in an unambiguous manner. Standards are logged into the laboratory's LIMS system and are assigned a unique identification number. The following information is typically recorded in the electronic database 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. See EH&S manual for further information.

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.

21.4.2.1 In addition, the following information may be helpful:

- Date opened (for multi-use containers, if applicable)
- Description of standard (if different from manufacturer's label or if standard was prepared in the laboratory)
- Recommended storage conditions
- Concentration (if applicable)
- Initials of analyst preparing standard or opening container

All containers of prepared reagents must include an expiration date and an ID number to trace back to preparation.

Procedures for preparation of reagents can be found in the Method SOPs.

To maintain traceability, standard ID numbers must be noted on all 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 outlined in laboratory Method SOPs

3) With requirements in SOP *Reagent and Standard Purchase, Preparation, Control Documentation* / NV08-214

SECTION 22. SAMPLING

22.1 Overview

TestAmerica Nashville does not provide sample collection services. The laboratory's responsibility in the sample collection process lies in supplying the sampler with the necessary coolers, sample containers, preservatives, sample labels, custody seals, COC forms, and packing materials required to properly preserve, pack, and ship samples to the laboratory

22.2 Sampling Containers

The laboratory offers clean sampling containers for use by clients. These containers are obtained from reputable container manufacturers and meet EPA specifications as required. Certificates of cleanliness provided by the supplier 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 most cases, containers may be purchased pre-preserved from the container supplier. Whether prepared by the laboratory or bought pre-preserved, the grades of the preservatives are at a minimum:

- Hydrochloric Acid – Reagent ACS (Certified VOA Free) or equivalent
- Methanol – Purge and Trap grade
- Nitric Acid – Instra-Analyzed or equivalent
- Sodium Bisulfate – ACS Grade or equivalent
- Sodium Hydroxide – Instra-Analyzed or equivalent
- Sulfuric Acid – Instra-Analyzed or equivalent
- Sodium Thiosulfate – ACS Grade or equivalent

22.3 Definition of Holding Time

The date and time of sampling documented on the chain-of-custody (COC) form establishes the day and time zero. As a general rule, when the maximum allowable holding time is expressed in "days" (e.g., 14 days, 28 days), the holding time is based on calendar days measured. Holding times expressed in "hours" (e.g., 6 hours, 24 hours, etc.) are measured from date and time zero. The first day of holding time ends twenty-four hours after sampling. Holding times for analysis include any necessary reanalysis. However, there are some programs that determine all holding times based on the date and specific time of sampling or to the hour, versus "days" approach.

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. LIMS will designate a time of field test.

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 placed inside the container, and the homogeneity of the sample need consideration when subsampling for sample preparation. It is the laboratory's responsibility to take a representative subsample or aliquot of the sample provided for analysis.

Each sample is handled by analysts as if it is potentially dangerous. At a minimum, safety glasses, gloves, and laboratory coats must be worn when preparing aliquots for analysis.

Refer to SOP Sample Homogenization, Subsampling, and Compositing / NV08-229 for specific details on taking sample aliquots and subsampling.

SECTION 23. HANDLING OF SAMPLES

Sample management procedures at TestAmerica Nashville 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 24-1). This form includes information such as:

- Client name, address, phone number
- Project name and/or number
- The sample identification
- Date, time and location of sampling
- Sample collectors name
- The matrix description
- The container description
- The total number of each type of container
- Preservatives used
- Analysis requested
- Requested turnaround time (TAT)
- Any special instructions
- Purchase order number or billing information (e.g., quote number) if available
- The date and time that each person relinquished or received the sample(s), including their signed name.

When the sampling personnel deliver the samples directly to TestAmerica personnel, the samples are stored in a cooler with ice, as applicable, and remain solely in the possession of the client's field technician until the samples are delivered to the laboratory. The sample collector must assure that each container is in his/her physical possession or in his/her view at all times, or stored in such a place and manner to preclude tampering. The field technician relinquishes the samples in writing on the COC form to the sample control personnel at the laboratory or to a TestAmerica courier. When sampling personnel deliver the samples through a common carrier (Fed-Ex, UPS), the COC relinquished date/time is completed by the field personnel and samples are released to the carrier. Samples are only considered to be received by laboratory when personnel at the laboratory have physical contact with the samples.

Note: Independent couriers are not required to sign the COC form. The COC is usually kept in the sealed sample cooler. The receipt from the courier is stored in Sample Control by date; it lists all receipts each date.

23.1.2 Legal / Evidentiary Chain-of-Custody

If samples are identified for legal/evidentiary purposes on the COC or by the client, Sample Control completes the custody seal, retains the shipping record with the COC. Sample movement within the laboratory as well as disposal is tracked using an internal COC included in the LIMS.

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 is assigned a unique sample identification number that is cross-referenced to the client identification number such that traceability of test samples is unambiguous and documented. Each sample container is affixed with a durable sample identification label. Sample acceptance, receipt, tracking and storage procedures are summarized in the following sections and in full detail in SOP *Sample Receiving* / NV02-01.

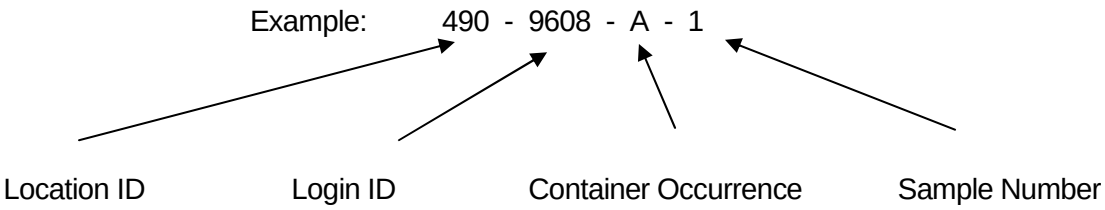
23.2.1 Laboratory Receipt

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 Cooler Receipt Form and brought to the immediate attention of the Project Manager and client. The COC, shipping documents, documentation of any non-conformance, irregularity, or comprised sample receipt, record of client contact, and resulting instructions become part of the project record and are usually scanned into LIMS.

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 Nashville Laboratory (Location 490). 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: 490 - 9608 - A - 1 - A ⟵ Secondary Container Occurrence

Example: 490-9608-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 23-3) that clearly outlines the circumstances under which samples are accepted or rejected. These include:

- A COC must be filled out completely
- Samples must be properly labeled
- Samples must be submitted in proper containers with adequate volume for the analysis and necessary QC
- Samples must be preserved according to the requirements of the requested analytical method
- Adequate sample holding times must remain for preparation and analysis
- All shipping containers including samples for water/solid volatile organic analyses must hold a trip blank
- Sample containers must be received in good condition
- The Project Manager is notified if any sample fails to meet the criteria listed in this sample acceptance policy.

Data from samples which do not meet these criteria are flagged and the nature of the variation from policy is defined. A copy of the sample acceptance policy is provided with all laboratory-supplied container shipments.

23.3.1 Inspection of samples includes a check for:

- Complete documentation to include sample identification, location (i.e., state), date and time of collection, collector's name, preservation type, sample type and any additional comments concerning the samples.
- Complete sample labels to include unique identification in indelible ink.
- Use of appropriate sample containers
- Adherence to holding times as specified in the test method and/or summarized in Section 24.

- Adequate sample volume for required analyses.
- Damage or signs of contamination to sample container. Volatile vials are also inspected for headspace.

23.3.2 Check and record the temperature of the samples that require thermal preservation.

- Samples are deemed acceptable if upon arrival they are not frozen (excluding VOAs) and are less than or equal to 6.0°C. Samples that are hand-delivered immediately after collection may not be at the required temperatures; however, if there is evidence that the chilling process has begun, such as the arrival on ice, the samples are considered acceptable. Condition upon receipt at the laboratory must be documented on the COC.
- If the samples were shipped in ice and solid ice is still present and in direct contact with samples, report the samples as "received on ice." Direct contact means samples must be surrounded by ice cubes or crushed ice. Ice present in a plastic bottle or other container does not constitute direct contact. Samples shipped with only “blue ice” may not be reported as “received on ice.”

23.3.3 Verify sample preservation as specified in the test method. Check for correct pH as specified in the test method. The results are documented on the Cooler Receipt Form and in LIMS. In the case of volatiles, pH is recorded after analysis on the run log and benchsheet. Chlorine is checked on samples requiring extractable organics, BOD, TOX, cyanide, fluoride, ammonia, TKN, and CBOD, and nitrate. Chlorine presence or absence is recorded for these analyses.

23.3.4 After inspecting the samples, the sample control receiving personnel sign and date the COC form, make any necessary notes of the samples' conditions on the Cooler Receipt Form, and store them in appropriate refrigerators or storage locations.

23.3.5 If samples are received without a COC, TestAmerica provides a generic COC form to be completed by the client when the samples are brought to the laboratory. The client is always provided with a copy of the completed COC form for their records.

23.3.6 If analyses with short holding times are requested, the dates and times are inspected to ensure that holding times have not already expired.

23.3.7 For samples received after normal working hours, the receipt time, date, and temperature are recorded at a minimum. The person delivering the samples signs the COC if they are the client or records their initials if they are a courier (e.g., FedEx or UPS). For samples not removed from the coolers, the coolers containing the samples are placed in a walk-in refrigerator. If samples are removed from the coolers, then the person receiving the samples must record the receipt date and time, the presence or absence of ice and custody seals, the temperature of samples, presence and type of packing material, and their initials. These samples are also placed in appropriate refrigerators.

23.3.8 Any deviations from these checks that question the suitability of the sample for analysis, or incomplete documentation as to the tests required will be resolved by consultation with the client. If the sample acceptance policy criteria are not met, the laboratory shall either:

- Retain all correspondence and/or records of communications with the client regarding the disposition of rejected samples, or

- Fully document any decision to proceed with sample analysis that does not meet sample acceptance criteria.

> **Note**: North Carolina requires that they be notified when samples are processed that do not meet sample acceptance criteria.

Once sample acceptance is verified, the samples are logged into the LIMS according SOP *Sample Receiving* / NV02-1

23.3.9 The COC, shipping documents, documentation of any non-conformance, irregularity, or compromised sample receipt, record of client contact, and resulting instructions become part of the project record.

23.4 Sample Storage

In order to avoid deterioration, contamination, or damage to a sample during storage and handling from the time of receipt until all analyses are complete, samples are stored in refrigerators, freezers, or protected locations suitable for the sample matrix. In addition, samples to be analyzed for volatile organic parameters are stored in separate refrigerators designated for volatile organic parameters only. Samples are never stored with reagents, standards, or materials that may create contamination.

To ensure the integrity of the samples during storage, refrigerator blanks are maintained in the volatile sample refrigerators and analyzed, at minimum, 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. After all analyses are complete, samples are placed into sample storage for at least 60 days. This area is at room temperature. At the end of approximately 60 days, samples are disposed of in accordance with SOP *Waste Disposal* / NV10-83.

Access to the laboratory is controlled such that sample storage devices do not require locking mechanisms. 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 designated, isolated areas designated for hazardous waste only. Any sample that is known to be hazardous at the time of receipt or after completion of analysis is placed in one of two refrigerators designated for highly contaminated samples. Foreign and U.S. soils requiring separate storage (as specified in SOP *Handling of Soils Regulated by USDA* / NV10-162) due to potential contamination with foreign organisms are tagged with an orange sticker and placed in specified locations. Foreign soils are heat-treated prior to disposal.

23.6 Sample Shipping

In the event that the laboratory needs to ship samples, the samples are placed in a cooler with enough ice to ensure the samples remain just above freezing and at or below 6.0°C during transit. The samples are carefully surrounded by packing material to avoid breakage (yet maintain appropriate temperature). For sample shipments which include water/solid volatile organic analyses, a trip blank is enclosed when required by method specifications, state requirements, or regulatory programs (see Note). The chain-of-custody form is signed by the sample control technician and attached to the shipping paperwork. Samples are generally shipped via overnight courier 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 are retained for a minimum of 30 days after the project report is sent; however, provisions may be made for earlier disposal of samples once the holding time is exceeded. Some samples are required to be held for longer periods based on regulatory or client requirements (e.g., 60 days after project report is sent). The laboratory must follow the longer sample retention requirements where required by regulation or client agreement.

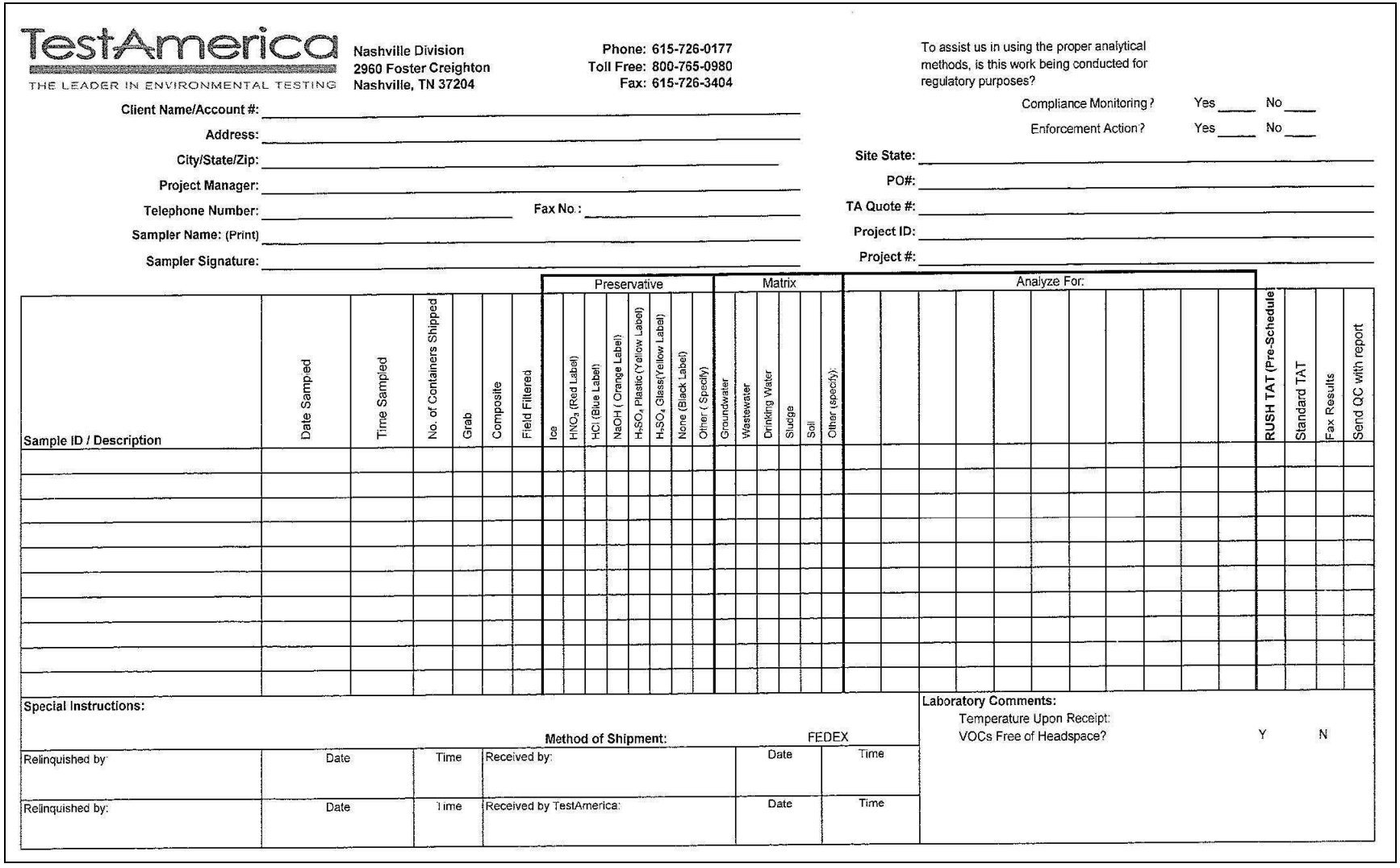
Several possibilities for sample disposal exist: the sample may be consumed completely during analysis, the sample may be returned to the customer or location of sampling for disposal, or the sample may be disposed of in accordance with the laboratory's waste disposal procedures (SOP *Waste Disposal* / NV10-83). 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 legal action, 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), and names of individuals who conducted the arrangements and physically completed the task. The laboratory removes or defaces sample labels prior to disposal, unless this is accomplished through the disposal method (e.g., samples are incinerated). Waste disposal is tracked in the LIMS.

Waste management practices are conducted in accordance with all applicable rules and regulations. Excess reagents, samples, and method process wastes must be stored, managed, and disposed of in accordance with all federal and state laws and regulations. Waste

description rules and land disposal restrictions are followed. Waste disposal procedures are incorporated by reference to this document and SOP *Waste Disposal* / NV10-83.

Figure 23-1. Chain of Custody (COC)



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To assist us in using the proper analytical methods, is this work being conducted for regulatory purposes?

Compliance Monitoring? Yes ____ No ____

Enforcement Action? Yes ____ No ____

Client Name/Account #: _____

Address: _____

City/State/Zip: _____

Project Manager: _____

Telephone Number: _____ Fax No.: _____

Sampler Name: (Print) _____

Sampler Signature: _____

Site State: _____

PO#: _____

TA Quote #: _____

Project ID: _____

Project #: _____

Sample ID / Description	Date Sampled	Time Sampled	No. of Containers Shipped	Grab	Composite	Field Filtered	Preservative: Ice	Preservative: HNO_3 (Red Label)	Preservative: HCl (Blue Label)	Preservative: NaOH (Orange Label)	Preservative: H_2SO_4 Plastic (Yellow Label)	Preservative: H_2SO_4 Glass(Yellow Label)	Preservative: None (Black Label)	Preservative: Other (Specify)	Matrix: Groundwater	Matrix: Wastewater	Matrix: Drinking Water	Matrix: Sludge	Matrix: Soil	Matrix: Other (specify):	Analyze For:										RUSH TAT (Pre-Schedule)	Standard TAT	Fax Results	Send QC with report

Special Instructions:

Method of Shipment: FEDEX

Laboratory Comments:
Temperature Upon Receipt:
VOCs Free of Headspace? Y N

Relinquished by:	Date	Time	Received by:	Date	Time
Relinquished by:	Date	Time	Received by TestAmerica:	Date	Time

Figure 23-2. **Custody Seal**

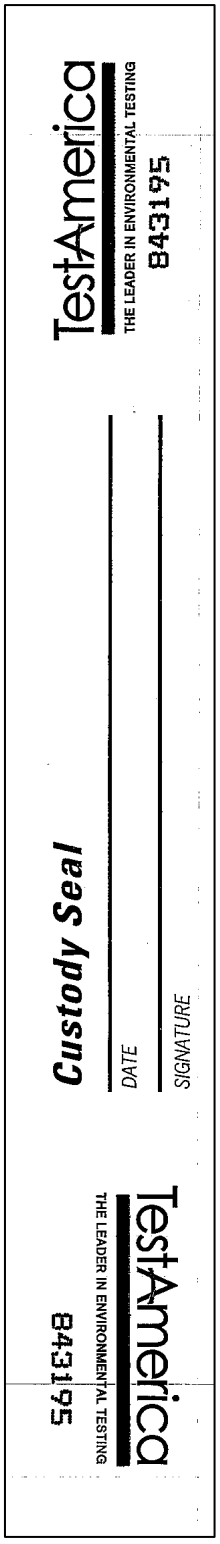

Figure 23-3. Sample Acceptance Policy

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 TesAmerica 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. See LIMS.

4) Samples must be preserved according to the requirements of the requested analytical method (See LIMS)**.**

Most analytical methods require chilling samples to 4^{o} C (other than water samples for metals analysis). For these methods, the criteria are met if the samples are chilled to below 6^{o} C and above freezing (0^{o}C). For methods with other temperature criteria (e.g. some bacteriological methods require $\leq$ 10 ^{o}C), the samples must arrive within $\pm$ 2^{o} C of the required temperature or within the method specified range. **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.

- 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 (working days) remaining on the holding time for us to ensure analysis.

   - Analyses that are designated as "field" analyses (Odor, pH, Dissolved Oxygen, Disinfectant Residual; a.k.a. Residual Chlorine, and Redox Potential) should be analyzed ASAP by the field sampler prior to delivering to the lab (within 15 minutes). However, if the analyses are to be performed in the laboratory, TestAmerica will make every effort to analyze the samples within 24 hours from receipt of the samples in the testing laboratory. Samples for "field" analyses received after 4:00 pm on Friday or on the weekend will be analyzed no later than the next business day after receipt (Monday unless a holiday). Samples will remain refrigerated and sealed until the time of analysis. The actual times of all "field" sample analyses are noted on the "Short Hold Time Detail Report" in the final report. Samples analyzed in the laboratory will be qualified on the final report with an 'H' to indicate holding time exceedance.

7) All samples submitted for Volatile Organic analyses must have a Trip Blank submitted at the same time. TestAmerica will supply a blank with the bottle order.

8) The project manager will be notified if any sample is received in damaged condition. TestAmerica will request that a sample be resubmitted for analysis.

9) Recommendations for packing samples for shipment.

   - Pack samples in Ice rather than "Blue" ice packs.

   - Soil samples should be placed in plastic zip-lock bags. The containers often have dirt around the top and do not seal very well and are prone to intrusion from the water from melted ice.

   - Water samples would be best if wrapped with bubble-wrap or paper (newspaper, or paper towels work) and then placed in plastic zip-lock bags.

   - Fill extra cooler space with bubble wrap.

Figure 23-4. Cooler Receipt Form

Cooler Received/Opened On_____

1. Tracking #_____(last 4 digits, FedEx)

Courier: _____ IR Gun ID_____

2. Temperature of rep. sample or temp blank when opened:_____*Degrees Celsius*

3. If Item #2 temperature is 0°C or less, was the representative sample or temp blank frozen? YES NO…NA

4. Were custody seals on outside of cooler? YES…NO…NA

If yes, how many and where:_____

5. Were the seals intact, signed, and dated correctly? YES…NO…NA

6. Were custody papers inside cooler? YES…NO…NA

I certify that I opened the cooler and answered questions 1-6 (intial) _____

7. Were custody seals on containers: YES…NO…NA

Were these signed and dated correctly? YES…NO…NA

8. Packing mat'l used? Bubblewrap Plastic bag Peanuts Vermiculite Foam Insert Paper Other None

9. Cooling process: Ice Ice-pack Ice (direct contact) Dry ice Other None

10. Did all containers arrive in good condition (unbroken)? YES…NO…NA

11. Were all container labels complete (#, date, signed, pres., etc)? YES…NO…NA

12. Did all container labels and tags agree with custody papers? YES…NO…NA

13a. Were VOA vials received? YES…NO…NA

b. Was there any observable headspace present in any VOA vial? YES…NO…NA

14. Was there a Trip Blank in this cooler? YES…NO…NA If multiple coolers, sequence #_____

I certify that I unloaded the cooler and answered questions 7-14 (intial) _____

15a. On pres'd bottles, did pH test strips suggest preservation reached the correct pH level? YES..NO..NA

b. Did the bottle labels indicate that the correct preservatives were used YES…NO…NA

16. Was residual chlorine present? YES…NO…NA

I certify that I checked for chlorine and pH as per SOP and answered questions 15-16 (intial) _____

17. Were custody papers properly filled out (ink, signed, etc)? YES…NO…NA

18. Did you sign the custody papers in the appropriate place? YES…NO…NA

19. Were correct containers used for the analysis requested? YES…NO…NA

20. Was sufficient amount of sample sent in each container? YES…NO…NA

I certify that I entered this project into LIMS and answered questions 17-20 (intial)_____

I certify that I attached a label with the unique LIMS number to each container (intial)_____

21. Were there Non-Conformance issues at login? YES…NO Documented in LIMS? YES…NO…#_____

SECTION 24. ASSURING THE QUALITY OF TEST RESULTS

24.1 Overview

In order to assure the validity of the data produced at TestAmerica Nashville, laboratory personnel continuously evaluate 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), duplicate matrix spikes (MSD), duplicates (DUP), surrogates, internal standards (IS)). These quality control checks are performed as required by the applicable methods or regulations to assess precision and accuracy. In addition to the routine process quality control samples, Proficiency Testing (PT) samples (at 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, acid digestion, distillation, 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. 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, Teflon chips, 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. For Ohio VAP, Correct the problem then re-prepare and re-analyze the method blank and all samples processed with the contaminated blank.
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 solvents or reagent water that may be processed during an analytical sequence in order to assess contamination in the analytical system. In general, instrument blanks are used to differentiate between contamination caused by the analytical system and that caused by the sample handling or sample prep process. Instrument blanks may also be inserted throughout the analytical sequence to minimize the effect of carryover from samples with high analyte content.

Table 24-1. 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 reagent water that has been purged to remove any volatile compounds. Appropriate preservatives are also added to the container. The trip blank is sent with the bottle order and is intended to reflect the environment that the containers are subjected to throughout shipping and handling and help identify possible sources if contamination is found. The field sampler returns the trip blank in the cooler with the field samples.
Field Blanks [1]	Are sometimes used for specific projects by the field samplers. A field blank prepared in the field by filling a clean container with pure reagent water and appropriate preservative, if any, for the specific sampling activity being undertaken. (EPA OSWER)
Equipment Blanks [1]	Are also sometimes created in the field for specific projects. An equipment blank is a sample of analyte-free media which has been used to rinse common sampling equipment to check effectiveness of decontamination procedures. (TNI)
Holding Blanks	Also referred to as refrigerator or freezer blanks, are used to monitor the sample storage units for volatile organic compounds during the storage of VOA samples in the laboratory

[1] When known, these field QC samples should not be selected for matrix QC as it does not provide information on the behavior of the target compounds in the field samples. Usually, the client sample ID will provide information to identify the field blanks with labels such as "FB", "EB", or "TB."

Evaluation criteria and corrective action for these controls are defined in the specific standard operating procedure for each analysis.

24.4 Positive Controls

Control samples (e.g., QC indicators) are analyzed with each batch of samples to evaluate data based upon (1) Method Performance (Laboratory Control Sample (LCS) or Blank Spike (BS)), which entails both the preparation and measurement steps; and (2) Matrix Effects (Matrix Spike (MS) (matrix spikes are not applicable to air) or Sample Duplicate (MSD, DUP), which evaluate 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, Teflon chips, 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 and is subjected to all preparatory and analytical steps along with the field samples. Where there is no preparation performed for an analysis (such as in aqueous volatiles) or when all samples and standards undergo the same preparation and analysis process (such as for total 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 representing the sample matrix or the analyte is not easily spiked (e.g., solid matrix LCS for metals, TDS, etc.).

The LCS may be made from different source material than that used to generate the calibration standard.

As stated in the opening of this section, the LCS goes through all of the steps of the process (including as necessary: filtration, clean-ups, etc.).

The specific frequency for inclusion of a 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 spikes all components to be reported in the LCS (and matrix spike) where applicable (e.g. no spike of pH). However, in certain 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 subset of the components to be reported (see below) are used to spike the LCS (and matrix spike). The selected components of each spiking mix must represent all chemistries, elution patterns and masses, permit-specified analytes, and other client-requested components. However, the laboratory must also ensure that all reported components are used in the spike mixture within a two-year time period.

- 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. Aroclors 1242, 1248, and 1254 are rotated as the spike throughout a year's time. Specific Aroclors may be used by request on a project specific basis.

24.5 Sample Matrix Controls

Table 24-2. Sample Matrix Control

Control Type	Details	
Matrix Spikes (MS/MSD)	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 environmental 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 most organic and some inorganic analytical measurements.
	Typical Frequency [1]	All organic and ICP methods as required by the analytical method (except Method 8015).
	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/MSD, 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 or regulatory mandated control limits. When this occurs, the regulatory or project limits supersede the laboratory's in-house limits.

Note: For methods, analytes, and matrices with very limited data (e.g., unusual matrices not analyzed often), interim limits are established using available data or by analogy to similar methods or matrices.

Once control limits have been established, they are reviewed and updated, if necessary, on an annual basis. A more frequent evaluation of a particular method's control limits will be performed whenever a significant change is made to the laboratory's method conditions. Control limits are established per method (as opposed to per instrument) regardless of the number of instruments utilized.

Laboratory generated % Recovery acceptance (control) limits are generally established by taking $\pm$ 3 Standard Deviations (at a 99% confidence level) from the average recovery of a minimum of 20-30 data points (more points are preferred).

- Regardless of the calculated values, the limit is no tighter than the calibration verification (ICV/CCV) (unless the analytical method specifies a tighter limit).
- With exception for certain poorly performing analytes (i.e., "bad actors"), 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 is 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 is 150%.
- The maximum acceptable RPD limit is 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 the laboratory's 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.

Client- or contract-required control limits are evaluated against the laboratory's statistically-derived control limits to determine if the data quality objectives (DQOs) can be achieved. If laboratory control limits are not consistent with DQOs, then alternatives must be considered (such as method improvements or use of an alternate analytical method).

24.6.2 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 are 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. A NCM (see Section 12) is also initiated if an LCS exceeds the acceptance limits. Sample results may be qualified and reported without reanalysis if:

- The analyte results are below the reporting limit and the LCS is above the upper control limit.
- The analyte results are above the relevant regulatory limit and the LCS is below the lower control limit (not applicable to the Ohio VAP).

See specific method SOPs for complete discussion of acceptance criteria and corrective action.

Note: Refer to the applicable SOP for approved corrective actions when analyzing samples for the Ohio VAP.

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 confirm matrix interference. A more detailed discussion of acceptance criteria and corrective action can be found in the laboratory's method SOPs and in Section 12.

24.6.4 Surrogate standard recoveries must be within acceptance limits for method blanks, LCSs, and CCVs.

24.6.5 If a surrogate standard recovery falls outside the acceptance limits in a client-supplied sample and there is not an obvious chromatographic matrix interference, reanalyze the sample to confirm a possible matrix effect. If the recoveries are confirmed or there was obvious chromatographic interference, results are reported from the original analysis and an appropriate qualifier is added. Dilutions or cleanup procedures may be required when obvious chromatographic interferences are present. If the reanalysis meets surrogate recovery criteria, the second run is reported (or both are reported if requested by the client). Under certain circumstances (and with exception of any samples analyzed for the Ohio VAP), 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. See specific method SOPs for a more detailed discussion of acceptance criteria and corrective action.

Note: For Ohio VAP, re-analyze a method blank or LCS to verify an outlier. If the same anomaly occurs upon re-analysis, then the entire batch must be re-prepared and re-analyzed. Exceptions include:

- Insufficient sample for re-preparation
- Holding time expired
- The LCS is biased high and the analytes in question are not detected in associated samples

24.7 Additional Procedures to Assure Quality Control

The laboratory has written and approved method SOPs, procedures to assure the accuracy of the test methods (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 and RLs can be found in Section 19.

- Use of formulae to reduce data is discussed in the method SOPs and in Section 20.
- Selection of appropriate reagents and standards is included in Section 9 and 21.

- A discussion on selectivity of the test is included in Section 5.
- Constant and consistent test conditions are discussed in Section 18.
- The laboratories sample acceptance policy is included in Section 23.

SECTION 25. REPORTING RESULTS

25.1 Overview

The results of each test are reported accurately, clearly, unambiguously, and objectively in accordance with State and Federal regulations as well as client requirements. Analytical results are issued in a format that is intended to satisfy customer and laboratory accreditation requirements as well as provide the end user with the information needed to properly evaluate the results. Where there is conflict between client requests and laboratory ethics or regulatory requirements, the laboratory's ethical and legal requirements are paramount and the laboratory must 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. Simplified reports must include information regarding any analytical results that are outside of QC limits and must include a reference to a full report that is made available to the client. Review of reported data is discussed 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 must contain the following information:

- A report title (e.g., Analytical Report for Samples) with a "sample results" column header.
- Each report page printed on company letterhead which includes the laboratory name, address, and telephone number.
- A unique identification of the report (e.g., work order number) and on each page an identification in order to ensure the page is recognized as part of the report and a clear identification of the end.
- **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. Pages are automatically paginated by LIMS when the report is created.
- A copy of the chain of custody (COC).

  - Any COCs involved with subcontracting are included.
  - There is a statement on the front of the report that says, “The Chain of Custody, X page(s), is included and is an integral part of this report." The number of pages of the COC (X) is calculated automatically by the number of pages that are scanned in the COC folder. Any pages scanned with the COC (i.e., non-conformances, emails, etc.) are included in the page count.
  - Any additional addenda to the report must be treated in a similar fashion, so it is a recognizable part of the report and cannot accidentally get separated from the report (e.g., sampling information).
- The name and address of client and a project name/number, if applicable.
- Client project manager or other contact
- Description and unambiguous identification of the tested sample(s) including the client identification code.
- Date of receipt of sample, date and time of collection, 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. The analysis time is always included on the report. The preparation date and time are included for those methods that require a separate preparation.
- Date reported or date of revision, if applicable.
- Method of analysis including method code (EPA, Standard Methods, etc).
- Reporting limit.
- Method detection limits (if requested)
- Definition of data qualifiers and reporting acronyms (e.g., ND).
- Sample results.
- QC data consisting of method blank, surrogate, LCS, and MS/MSD recoveries, duplicates, and control limits.
- Condition of samples at receipt including temperature (included on the Cooler Receipt Form, which is part of the COC).
- A statement to the effect that the results relate only to the items tested and the sample as received by the laboratory.
- A statement that the report shall not be reproduced except in full, without prior express written approval by the laboratory.
- A signature and title of the person(s) accepting responsibility for the content of the report and date of issue. Signatories are appointed by the Laboratory Director. For applying an electronic signature, refer to the Corporate SOP on *Electronic Reporting and Signature Policy* (No. CA-I-P-002).
- When TNI accreditation is required, the laboratory certifies that the test results meet all requirements of TNI or provides reasons and/or justification if they do not. Accreditations/certifications are included near the end of the report and are listed by method.
- The laboratory includes a cover letter.

- Where applicable, a narrative to the report that explains the issue(s) and corrective action(s) taken in the event that a specific accreditation or certification requirement was not met.
- When soil samples are analyzed, a specific identification as to whether soils are reported on a “wet weight” or “dry weight” basis.
- Appropriate laboratory certification number for the state of origin of the sample, if applicable.
- If only part of the report is provided to the client (client requests some results before all of it is complete), it must be clearly indicated on the report (e.g., partial report, draft report). A complete report must be sent once all of the work has been completed.
- Any non-TestAmerica subcontracted analysis results are provided as a separate report on the official letterhead of the subcontractor. All TestAmerica network subcontracting is clearly identified on the report as to which laboratory performed a specific analysis.
- 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.
- Reports for Ohio VAP work require a VAP affidavit be completed and included with the report.

25.3 Reporting Level or Report Type

TestAmerica Nashville offers three levels of quality control reporting. Each level, in addition to its own specific requirements, contains all the information provided in the preceding level. The packages provide the following information in addition to the information described above:

- Level II is a report plus summary information, including results for the method blank reported to the laboratory MDL, percent recovery for laboratory control samples and matrix spike samples, and the RPD values for all MSD and sample duplicate analyses.
- Level III contains all the information supplied in Level II, but presented on the CLP-like summary forms, and relevant calibration information. A Level II report is not included, unless specifically requested. No raw data is provided.
- Level IV is the same as Level III with the addition of all raw supporting data.

In addition to the various levels of QC packaging, the laboratory also provides reports in diskette deliverable form. Initial reports may be provided to clients by facsimile. All faxed reports are followed by hardcopy. Procedures used to ensure client confidentiality are outlined in Section 25.6.

25.3.1 Electronic Data Deliverables (EDDs)

EDDs are routinely offered as part of TestAmerica’s services. TestAmerica Nashville offers a variety of EDD formats including Environmental Restoration Information Management System (ERPIMS), New Agency Standard (NAS), Format A, Excel, Dbase, GISKEY, and Text Files.

EDD specifications are submitted to the IT department by the PM for review and undergo the contract review process. Once the facility has committed to providing data in a specific electronic format, the coding of the format may need to be performed. This coding is documented and validated. The validation of the code is retained by the IT staff coding the EDD.

EDDs shall be subject to a review to ensure their accuracy and completeness. If EDD generation is automated, review may be reduced to periodic screening if the laboratory can demonstrate that it

can routinely generate that EDD without errors. Any revisions to the EDD format must be reviewed until it is demonstrated that it can routinely be generated without errors. If the EDD can be reproduced accurately and if all subsequent EDDs can be produced error-free, each EDD does not necessarily require a review.

25.4 Supplemental Information for Tests

The laboratory identifies any unacceptable QC analyses or any other unusual circumstances/ observations (such as environmental conditions and any non-standard conditions) that may have affected the quality of a result in the form of a footnote, a qualifier, and/or a narrative explaining the discrepancy in the front of the report.

Analytical concentrations outside of the calibration range, either high or low, are qualified as estimated values.

Where quality system requirements are not met, a statement of compliance/non-compliance with requirements and/or specifications is included in the report and discusses identification of test results derived from any sample that did not meet TNI sample acceptance requirements (i.e., improper container, holding time, or temperature).

Where applicable, a statement on the estimated uncertainty of measurements is included. Information on uncertainty is included 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 must determine if a response can be prepared. If so, the Laboratory Director designates the appropriate member of the management team to prepare a response. The response must be fully documented, and reviewed by the Laboratory Director, before release to the client. There may be additional fees charged to the client at this time as this is a non-routine function of the laboratory.

Note: Review of data deliverable packages for submittal to regulatory authorities requires responses to non-conforming data concerning potential impact on data quality. This necessitates a limited scope of interpretation, and this work is performed by the QA Department. This is the only form of "interpretation" of data that is routinely performed by the laboratory.

When opinions or interpretations are included in the report, the laboratory provides an explanation as to the basis upon which the opinions and interpretations have been made. Opinions and interpretations are clearly noted as such and, where applicable, a comment must be added suggesting that the client verify the opinion or interpretation with their regulator.

Note: For Ohio VAP, a laboratory must narrate potential bias in sample results when requirements provided in a SOP cannot be met.

25.5 Environmental Testing Obtained from Subcontractors

If TestAmerica Nashville is not able to provide to the client the requested analysis, the samples are subcontracted following the procedures outlined in the Corporate SOP on *Subcontracting* (SOP No. CA-L-S-002).

Data reported from analyses performed by a subcontracting laboratory are clearly identified as such on the analytical report provided to the client. Results from a subcontracting laboratory outside of the TestAmerica network are reported to the client on the subcontracting 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: Confidentiality does not apply to the extent that the information is required to be disclosed by TestAmerica under the compulsion of legal process. TestAmerica does, to the extent feasible, provide reasonable notice to the client before disclosing the information.

Note: Authorized representatives of an accrediting authority are permitted to make copies of any analyses or records relevant to the accreditation process and copies may be removed from the laboratory for purposes of assessment.

25.6.1 Report deliverable formats are discussed with each new client. If a client requests that reports be faxed or e-mailed, the reports are faxed with a cover sheet or e-mailed with the following note that includes a confidentiality statement similar to the following:

This material is intended only for the use of the individual(s) or entity to whom it is addressed, and may contain information that is privileged and confidential. If you are not the intended recipient, or the employee or agent responsible for delivering this material to the intended recipient, you are hereby notified that any dissemination, distribution or copying of this communication is strictly prohibited. If you have received this communication in error, please notify us immediately by telephone at the 1-800-765-0980 (or for e-mails: please notify us immediately by e-mail or by phone (1-800-765-0980) and delete this material from any computer).

25.7 Formats of Reports

The format of reports is designed to accommodate each type of environmental test carried out and to minimize the possibility of misunderstanding or misuse.

25.8 Amendments to Test Reports

Corrections, additions, or deletions to reports are only made when justification arises through supplemental documentation. Justification is documented using the laboratory's NCM or CAR system (refer to Section 12). Revisions associated with technical issues must be reviewed by the Laboratory Director, Technical Director, and QA Manager.

The revised report is retained on the Archive data server along with the original report. When the PDF of a report is created, the PDF filename has the work order number and the date and time that

the PDF was created. The original report has the earliest date, and any revisions have later dates and times.

When the report is re-issued, a notation that the report has been revised is placed on the cover/signature page of the report along 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 11/3/07 to include toluene in sample NQA1504 per client's request. This final report replaces the final report generated on 10/27/07 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 the laboratory's QA Manager.

Appendix 1. Laboratory Floor Plan

Key Areas			
1. Project Management	7. Semivolatiles GC/MS	14. Volatiles GC/MS	20. Break room
2. Sample Control	8. Diesel Range Organics	15. HPLC	21. LIMS/IT
3. Organic Extractions	9. Gasoline Range Organics, GC Volatiles	16. Sample Storage	22. Balance room
4. TCLP	10. Inorganics	17. Waste Disposal	23. Data Packages
5. Shipping	11. Metals	18. Walk-in Cooler	24. Conference room
6. Pesticides/PCBs/Herbicides	12. ICP/MS	19. Archiving	25. Accounting
	13. TOC/TOX		26. Liquid Chromatography

TestAmerica
THE LEADER IN ENVIRONMENTAL TESTING

Appendix 2. Glossary/Acronyms

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)

Aliquot - A measured portion of a sample used for analysis.

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)

Assessment - The evaluation process used to measure or establish the performance, effectiveness, and conformance of an organization and/or its systems to defined criteria (to the standards and requirements of laboratory accreditation). (TNI)

Audit - A systematic and independent examination of facilities, equipment, personnel, training, procedures, record-keeping, data validation, data management, and reporting aspects of a system to determine whether QA/QC and technical activities are being conducted as planned and whether these activities will effectively achieve quality objectives. (TNI)

Batch - Environmental samples which are prepared and/or analyzed together with the same process and personnel, using the same lot(s) of reagents. A **preparation batch** is composed of one to 20 environmental samples of the same matrix, meeting the above mentioned criteria and with a maximum time between the start of processing of the first and last sample in the batch to be 24 hours. An **analytical batch** is composed of prepared environmental samples (extracts, digestates, or concentrates) and /or those samples not requiring preparation which are analyzed together as a group using the same calibration curve or factor. An analytical batch can include samples originating from various environmental matrices and can exceed 20 samples. (TNI)

Bias - The systematic or persistent distortion of a measurement process, which causes errors in one direction (i.e., the expected sample measurement is different from the sample's true value). (TNI)

Blank - A sample that has not been exposed to the analyzed sample stream in order to monitor contamination during sampling, transport, storage, or analysis. The blank is subjected to the usual analytical and measurement process to establish a zero baseline or background value and is sometimes used to adjust or correct routine analytical results. (ASQC)

Calibration - A set of operations that establish, under specified conditions, the relationship between values of quantities indicated by a measuring instrument or measuring system, or

values represented by a material measure or a reference material, and the corresponding values realized by standards. (TNI)

1) In calibration of support equipment the values realized by standards are established through the use of reference standards that are traceable to the International System of Units (SI).

2) In calibration according to methods, the values realized by standards are typically established through the use of Reference Materials that are either purchased by the laboratory with a certificate of analysis or purity, or prepared by the laboratory using support equipment that has been calibrated or verified to meet specifications.

Calibration Curve - The mathematical relationship between the known values, such as concentrations, of a series of calibration standards and their instrument response. (NELAC)

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)

Continuing Calibration Blank (CCB) - A solvent blank following a continuing calibration verification.

Continuing Calibration Verification (CCV) - A standard from the calibration curve that is used to verify that the instrument remains properly calibrated throughout the analytical run.

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)

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 is most frequently 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, processes, or procedure.

Data Audit - A qualitative and quantitative evaluation of the documentation and procedures associated with environmental measurements to verify that the resulting data are of acceptable quality (i.e., that they meet specified acceptance criteria).

Data Reduction - The process of transforming the number of data by arithmetic or statistical calculations, standard curves, and concentration factors and collation into a more useable form. (TNI)

Deficiency - An unauthorized deviation from acceptable procedures or practices, or a defect in an item. (ASQC)

Demonstration of Capability - A procedure to establish the ability of the analyst to generate analytical results of acceptable accuracy and precision. (TNI)

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.

Finding - An assessment conclusion that identifies a condition having a significant effect on an item or activity. An assessment finding is normally a deficiency and is normally accompanied by specific examples of the observed condition. (TNI)

Holding Times - The maximum times that samples may be held prior to analyses and still be considered valid or not compromised. (40 CFR Part 136)

Initial Calibration Blank (ICB) - A solvent blank immediately following an initial calibration verification.

Initial Calibration Verification (ICV) - A standard from a second source that is analyzed to verify the calibration curve.

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.

Instrument Response - Instrument response is normally expressed as either peak area or peak height; however, it may also reflect a numerical representation of some type of count on a detector (e.g., photomultiplier tube, or diode array detector) and is used in this document to represent all types.

Laboratory - A defined facility performing environmental analyses in a controlled and scientific manner. (TNI)

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 which is 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.

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 generates a correlation coefficient (r) or coefficient of determination (r^2) 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 0.990 for organics and 0.995 for inorganics.

Matrix - The component or substrate that contains the analyte of interest. For purposes of batch and QC requirement determinations, the following matrix distinctions are to be used:

Aqueous: Any aqueous sample excluded from the definition of drinking water matrix or saline/estuarine sources. 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.

Non-aqueous Liquid: Any organic liquid with <15% settleable solids. Described as Waste in LIMS.

Biological Tissue: Any sample of a biological origin such as fish tissue, shellfish, or plant material. Such samples must be grouped according to origin.

Solids: Includes soils, sediments, sludges, and other matrices with >15% settleable solids.

Air and Emissions: Whole gas or vapor samples including those contained in flexible or rigid wall containers and the extracted, concentrated analytes of interest from a gas or vapor that are collected with a sorbant tube, impinger solution, filter, or other device. (TNI)

Matrix Spike (spiked sample or fortified sample) - A sample prepared, taken through all sample preparation and analytical steps of the procedure unless otherwise noted in a referenced method, by adding a known amount of target analyte to a specified amount of sample for which an independent test result of target analyte concentration is available. Matrix spikes are used, for example, to determine the effect of the matrix on a method's recovery efficiency.

Matrix Spike Duplicate (spiked sample or fortified sample duplicate) - A replicate matrix spike prepared and analyzed to obtain a measure of the precision of the recovery for each analyte.

Method Blank - A sample of a matrix similar to the batch of associated samples (when available) that is free from the analytes of interest and is processed simultaneously with and under the same conditions as samples through all steps of the analytical procedures, and in which no target analytes or interferences are present at concentrations that impact the analytical results for sample analyses.

Method Detection Limit (MDL) - The minimum concentration of a substance (an analyte) that can be measured and reported with 99% confidence that the analyte concentration is greater than zero and is determined from analysis of a sample in a given matrix containing the analyte. (40 CFR Part 136, Appendix B)

Method Reporting Limit - The laboratory nominal Reporting Limit (RL) or the level of sensitivity required by the client but not lower than the method detection limit (MDL).

Negative Control - Measures taken to ensure that a test, its components, or the environment do not cause undesired effects or produce incorrect test results.

Non-Conformance - An indication, judgment, or state of not having met the requirements of the relevant specifications, contract, or regulation.

Performance Audit - The routine comparison of independently obtained qualitative and quantitative measurement system data with routinely obtained data in order to evaluate the proficiency of an analyst or laboratory.

Positive Control - Measures taken to ensure that a test and/or its components are working properly and producing correct or expected results from positive test subjects.

Precision - The degree to which a set of observations or measurements of the same property, obtained under similar conditions, conform to themselves; a data quality indicator. Precision is usually expressed as standard deviation, variance, or range in either absolute or relative terms. (TNI)

Preservation - Any conditions under which a sample must be kept in order to maintain chemical and/or biological integrity prior to analysis. (TNI)

Proficiency Testing - A means of evaluating a laboratory's performance under controlled conditions relative to a given set of criteria through analysis of unknown samples provided by an external source. (TNI)

Proficiency Testing Program - The aggregate of providing rigorously controlled and standardized environmental samples to a laboratory for analysis, reporting of results, statistical evaluation of the results, and the collective demographics and results summary of all participating laboratories. (TNI)

Proficiency Test Sample (PT) - A sample, the composition of which is unknown to the laboratory and is provided to test whether the laboratory can produce analytical results within specified acceptance criteria. (TNI)

Quality Assurance - An integrated system of management activities involving planning, implementation, assessment, reporting, and quality improvement to ensure that a process, item, or service is of the type of quality needed and expected by the client. (TNI)

Quality Assurance [Project] Plan (QAPP) - A formal document describing the detailed quality control procedures by which the quality requirements defined for the data and decisions pertaining to a specific project are to be achieved. (EAP-QAD)

Quality Control - The overall system of technical activities that measures the attributes and performance of a process, item, or service against defined standards to verify that they meet the stated requirements established by the customer; operational techniques and activities that are used to fulfill requirements for quality; also the system of activities and checks used to ensure that measurement systems are maintained within prescribed limits, providing protection against "out of control" conditions and ensuring that the results are of acceptable quality. (TNI)

Quality Control Sample - A sample used to assess the performance of all or a portion of the measurement system. One of any number of samples, such as Certified Reference Materials, a quality system matrix fortified by spiking, or actual samples fortified by spiking, intended to demonstrate that a measurement system or activity is in control. (TNI))

Quality Manual - A document stating the management policies, objectives, principles, organizational structure and authority, responsibilities, accountability, and implementation of an agency, organization, or laboratory, to ensure the quality of its product and the utility of its product to its users. (TNI)

Quality System - A structured and documented management system describing the policies, objectives, principles, organizational authority, responsibilities, accountability, and implementation plan of an organization for ensuring quality in its work processes, products (items), and services. The quality system provides the framework for planning, implementing, and assessing work performed by the organization and for carrying out required QA and QC activities. (TNI)

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 Method - A method of known and documented accuracy and precision issued by an organization recognized as competent to do so. (TNI)

Reference Standard - Standard used for the calibration of working measurement standards in a given organization or a given location. (TNI))

Requirement - Denotes a mandatory specification; often designated by the term “shall”. (NELAC)

Sampling - Activity related to obtaining a representative sample of the object of conformity assessment, according to a procedure.

Second Order Polynomial Curve (Quadratic Curve) - The second 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 second order regression generates a coefficient of determination (COD or r^2) that is a measure of the "goodness of fit" of the quadratic curvature to 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 0.990.

Selectivity - The ability to analyze, distinguish, and determine a specific analyte or parameter from another component that may be a potential interferent or that may behave similarly to the target analyte or parameter within the measurement system. (TNI)

Sensitivity - The capability of a method or instrument to discriminate between measurement responses representing different levels (e.g., concentrations) of a variable of interest. (NELAC)

Spike - A known mass of target analyte added to a blank, sample, or sub-sample; used to determine recovery efficiency or for other quality control purposes.

Standard - The document describing the elements of laboratory accreditation that has been developed and established within the consensus principles of standard setting and meets the approval requirements of standard adoption organization’s 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.

Supervisor (however named) - The individual(s) designated as being responsible for a particular area or category of scientific analysis. This responsibility includes direct day-to-day supervision of technical employees, supply and instrument adequacy and upkeep, quality assurance/quality control duties, and ascertaining that technical employees have the required balance of education, training, and experience to perform the required analyses. (NELAC)

Surrogate - A substance with properties that mimic the analyte of interest, but is unlikely to be found in environmental 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 is reported to the client whose sample produced poor recovery. (QAMS)

Systems Audit (also Technical Systems Audit) - A thorough, systematic, qualitative on-site assessment of the facilities, equipment, personnel, training, procedures, record keeping, data validation, data management, and reporting aspects of a total measurement system. (EPA-QAD)

Technical Director - Individuals(s) who has overall responsibility for the technical operation of the environmental testing laboratory. (NELAC)

Technology - A specific arrangement of analytical instruments, detection systems, and/or preparation techniques.

Test - A technical operation that consists of the determination of one or more characteristics or performance of a given product, material, equipment, organism, physical phenomenon, process, or service according to a specified procedure. The result of a test is normally recorded in a document sometimes called a test report or a test certificate. (ISO/IEC Guide 2-12.1, amended)

Test Method - An adoption of a scientific technique for a specific measurement problem, as documented in a laboratory SOP. (TNI)

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.

Weighting Factor – A mathematical factor used to improve linearity at the low end of a calibration curve. The square of the residuals is based on $1/x$ or $1/x^2$ rather than x (where x is the concentration value). This technique is preferred over the use of a quadratic regression.

Acronyms:

CAR	Corrective Action Report
CCB	Continuing Calibration Blank
CCV	Continuing Calibration Verification
CF	Calibration Factor
CFR	Code of Federal Regulations
COC	Chain of Custody
CQMP	Corporate Quality Management Plan
DOC	Demonstration of Capability
DQO	Data Quality Objectives
DUP	Duplicate
EHS	Environment, Health and Safety
EPA	Environmental Protection Agency
GC	Gas Chromatography
GC/MS	Gas Chromatography/Mass Spectrometry
HPLC	High Performance Liquid Chromatography
ICB	Initial Calibration Blank
ICP	Inductively Coupled Plasma Atomic Emission Spectroscopy
ICP/MS	ICP/Mass Spectroscopy
ICV	Initial Calibration Verification
IDL	Instrument Detection Limit
IH	Industrial Hygiene
IS	Internal Standard
LCS	Laboratory Control Sample
LCSD	Laboratory Control Sample Duplicate
LIMS	Laboratory Information Management System
MB	Method Blank
MDL	Method Detection Limit
MDLV	MDL Verification Check Standard
MS	Matrix Spike
MSD	Matrix Spike Duplicate
NCM	Non-Conformance Memo
NELAC	National Environmental Laboratory Accreditation Conference
NELAP	National Environmental Laboratory Accreditation Program
PT	Performance Testing
QAM	Quality Assurance Manual
QA/QC	Quality Assurance / Quality Control
QAPP	Quality Assurance Project Plan
RF	Response Factor
RL	Reporting Limit
RPD	Relative Percent Difference
RSD	Relative Standard Deviation
SD	Standard Deviation
SDS	Safety Data Sheet
SOP	Standard Operating Procedure
TAT	Turn-Around-Time
TNI	The NELAC Institute
VOA	Volatile Organic Analytes
VOC	Volatile Organic Compound

See Controlled Document QAF-45, TestAmerica Nashville Acronyms, Keywords, and Definitions.

Appendix 3. Laboratory Certifications, Accreditations, Validations

TestAmerica Nashville maintains accreditations, certifications, and approvals with numerous state and national entities. Programs vary but may include on-site audits, reciprocal agreements with another entity, performance testing evaluations, review of the QA Manual, Standard Operating Procedures, Method Detection Limits, training records, etc. At the time of this QA Manual revision, the laboratory has accreditation/certification/licensing with the following organizations:

Organization	Lab Number	Organization	Lab Number
A2LA	0453.07	New Jersey (2nd NELAP)	TN965
Alaska	UST-087	New York (2nd NELAP)	11342
Arkansas	88-0737	North Carolina	387
Arizona	AZ0473	North Dakota	R-146
California	01168CA	Ohio	CL0033
Connecticut	PH-0220	Oklahoma	9412
Florida (1o NELAP)	E87358	Oregon (2nd NELAP)	TN200001
Illinois (2nd NELAP)	200010	Pennsylvania (2nd NELAP)	68-00585
Iowa	131	Rhode Island	LAO00268
Kansas (2nd NELAP)	E-10229	South Carolina	84009001, 002
Kentucky	19	Tennessee DW	020 08
Louisiana (2nd NELAP)	01945	Tennessee Controlled Substances	5741902
Maryland	316	Texas (2nd NELAP)	T104704077
Massachusetts	M-TN032	Utah (2nd NELAP)	6157260177
Minnesota	047-999-345	Virginia (2nd NELAP)	460152
Mississippi	none	Washington	C789
Montana	none	West Virginia	219
TNI/NELAP	E87358	Wisconsin	998020430
Nevada	TN00032	Wyoming UST	A2LA 0453-07
New Hampshire (2nd NELAP)	2963		

Subject to Change

The certificates and accredited parameter lists are available for each State/Program at www.testamericainc.com under Analytical Services Search – Certifications.

Claims of Accreditation Status

TestAmerica Nashville has agreed to make only valid claims as to its accreditation/certification status by any authority by ensuring that the expiration dates are not exceeded and the method-specific scope or parameter lists are supportable, as required by each. Any false claims would be reported to that authority. The agreement covers the use of the authority's name, such as "Authority-Accredited," symbols, or certificate number. The only valid proof of accreditation/certification is the current certificate and scope of the authority. It is the responsibility of the laboratory to make these documents available to all staff and it is the staff's duty to reference only the current documents.

A report including scoped and non-scoped analytes may only be presented on the same report if the non-accredited results are clearly and unambiguously identified. No report with non-scope analytes may be associated with the symbol, "Authority accredited" phrase, or the certificate number. Only the analytes specified by a unique method are valid within the scope. There must be no intentional misleading of the users of the laboratory's services in this regard.

No opinions and/or interpretations based on results outside the laboratory's scope may be presented on a document referenced by "Authority-accredited, the symbol, or the certificate number. If these are made, they must be written in a separate letter which is not endorsed by the authority.

The "Authority-accredited" symbol may only be affixed to equipment calibrated by a laboratory that is accredited by the authority. If calibration labels contain the symbol, they must also show the calibration laboratory's name or its certificate number, the instrument's unique identification, the date of the last calibration, and a cross-reference to the last calibration certificate.

If the company decides to use the "Authority-accredited" symbol in marketing activities, no misrepresentation may occur. Only reference to the accredited scope at a specific laboratory site is allowed. If any "Authority-accredited" language is used in proposals or quotations, any non-scope analytes must be clearly denoted as not accredited by that authority. The same is true for any use of laboratory letterhead with the "Authority-accredited" wording or symbol. The symbol may not be affixed to any material, item, product, part, or packaging, thereby implying accreditation status to that piece. In literature, any use of the symbol must be positioned adjacent to the accredited laboratory's name and clearly state that the presence of the symbol does not imply certification/approval of the products tested. At no time may the symbol appear to suggest that a person is accredited. Misrepresentation of accreditation status is never allowed and must be reported if it occurs. If in doubt, the idea of the symbol's use may be presented to the authority for approval.

If accreditation is terminated or suspended, the laboratory must immediately cease to use the "Authority-accredited" wording, the symbol, or the certificate number reference in any way and inform clients impacted by the change.

Note: The use of the A2LA logo is reserved for A2LA only. TestAmerica Nashville may only use the A2LA accredited symbol so long as the accreditation remains current. Further information can be found in the A2LA document *P101 – Rules for Making Reference to A2LA Accredited Status*.

Appendix 4. Data Qualifiers

Flag	Flag Text	Conditions	Arizona	Florida	Comment
*	ICPMS Relative Intensity is outside the method limits.	ISTD Relative Intensity Failed High	E7		
*	ICPMS Relative Intensity is outside the method limits.	ISTD Relative Intensity Failed Low	E7		
*	ISTD response or retention time outside acceptable limits	ISTD Area High	E7		
*	ISTD response or retention time outside acceptable limits	ISTD Area Low	E7		
*	ISTD response or retention time outside acceptable limits	ISTD RT Fail			
*	LCS or LCSD exceeds the control limits	LCS Negative Recovery High			
*	LCS or LCSD exceeds the control limits	LCS Negative Recovery Low			
*	LCS or LCSD exceeds the control limits	LCS Recovery High	L3	J3	
*	LCS or LCSD exceeds the control limits	LCS Recovery Low	L4	J3	Data impacted; re-analysis should be performed
*	LCS or LCSD exceeds the control limits	LCS Recovery			
*	LCS or LCSD exceeds the control limits	LCSD Recovery High	L3	J3	
*	LCS or LCSD exceeds the control limits	LCSD Recovery Low	L4	J3	Data impacted; re-analysis should be performed
*	LCS or LCSD exceeds the control limits	LCSD Recovery			

Flag	Flag Text	Conditions	Arizona	Florida	Comment
*	RPD of the LCS and LCSD exceeds the control limits	LCSD Percent RPD	R6	J3	
^	ICV, CCV, ICB, CCB, ISA, ISB, CRI, CRA, DLCK or MRL standard: Instrument related QC exceeds the control limits.	CCB Result Detected	B1	V	Use only if affected samples are ND
^	ICV, CCV, ICB, CCB, ISA, ISB, CRI, CRA, DLCK or MRL standard: Instrument related QC exceeds the control limits.	CCV Recovery High	V1, V7		Use only if affected samples are ND
^	ICV, CCV, ICB, CCB, ISA, ISB, CRI, CRA, DLCK or MRL standard: Instrument related QC exceeds the control limits.	CCV Recovery Low	V8, V9		Data impacted; re-analysis should be performed
^	ICV, CCV, ICB, CCB, ISA, ISB, CRI, CRA, DLCK or MRL standard: Instrument related QC exceeds the control limits.	CCVL Recovery High			
^	ICV, CCV, ICB, CCB, ISA, ISB, CRI, CRA, DLCK or MRL standard: Instrument related QC exceeds the control limits.	CCVL Recovery Low			
^	ICV, CCV, ICB, CCB, ISA, ISB, CRI, CRA, DLCK or MRL standard: Instrument related QC exceeds the control limits.	CRA Fail			
^	ICV, CCV, ICB, CCB, ISA, ISB, CRI, CRA, DLCK or MRL standard: Instrument related QC exceeds the control limits.	CRA Recovery High	V1, N4, N5		Use only if affected samples are ND
^	ICV, CCV, ICB, CCB, ISA, ISB, CRI, CRA, DLCK or MRL standard: Instrument related QC exceeds the control limits.	CRA Recovery Low	V9		Data impacted; re-analysis should be performed

Flag	Flag Text	Conditions	Arizona	Florida	Comment
^	ICV, CCV, ICB, CCB, ISA, ISB, CRI, CRA, DLCK or MRL standard: Instrument related QC exceeds the control limits.	CRI Fail			
^	ICV, CCV, ICB, CCB, ISA, ISB, CRI, CRA, DLCK or MRL standard: Instrument related QC exceeds the control limits.	CRI Recovery High	V1, N4, N5		Use only if affected samples are ND
^	ICV, CCV, ICB, CCB, ISA, ISB, CRI, CRA, DLCK or MRL standard: Instrument related QC exceeds the control limits.	CRI Recovery Low	V9		Data impacted; re-analysis should be performed
^	ICV, CCV, ICB, CCB, ISA, ISB, CRI, CRA, DLCK or MRL standard: Instrument related QC exceeds the control limits.	ICB Result Detected	B1	V	Use only if affected samples are ND
^	ICV, CCV, ICB, CCB, ISA, ISB, CRI, CRA, DLCK or MRL standard: Instrument related QC exceeds the control limits.	ICSA Recovery High			Data impacted; re-analysis should be performed
^	ICV, CCV, ICB, CCB, ISA, ISB, CRI, CRA, DLCK or MRL standard: Instrument related QC exceeds the control limits.	ICSA Recovery Low			Data impacted; re-analysis should be performed
^	ICV, CCV, ICB, CCB, ISA, ISB, CRI, CRA, DLCK or MRL standard: Instrument related QC exceeds the control limits.	ICSAB Recovery High			Data impacted; re-analysis should be performed
^	ICV, CCV, ICB, CCB, ISA, ISB, CRI, CRA, DLCK or MRL standard: Instrument related QC exceeds the control limits.	ICSAB Recovery Low			Data impacted; re-analysis should be performed

Flag	Flag Text	Conditions	Arizona	Florida	Comment
^	ICV, CCV, ICB, CCB, ISA, ISB, CRI, CRA, DLCK or MRL standard: Instrument related QC exceeds the control limits.	ICSB Recovery			
^	ICV, CCV, ICB, CCB, ISA, ISB, CRI, CRA, DLCK or MRL standard: Instrument related QC exceeds the control limits.	ICV Recovery High	V1, V7		Use only if affected samples are ND
^	ICV, CCV, ICB, CCB, ISA, ISB, CRI, CRA, DLCK or MRL standard: Instrument related QC exceeds the control limits.	ICV Recovery Low	V8, V9		Data impacted; re-analysis should be performed
^	ICV, CCV, ICB, CCB, ISA, ISB, CRI, CRA, DLCK or MRL standard: Instrument related QC exceeds the control limits.	ICVL Recovery High			
^	ICV, CCV, ICB, CCB, ISA, ISB, CRI, CRA, DLCK or MRL standard: Instrument related QC exceeds the control limits.	ICVL Recovery Low			
^	ICVH recovery was above method acceptance limits	ICVH Recovery High			
^	ICVH recovery was below method acceptance limits	ICVH Recovery Low			
^	ICV, CCV, ICB, CCB, ISA, ISB, CRI, CRA, DLCK or MRL standard: Instrument related QC exceeds the control limits.	ISCA Recovery			
^	ICV, CCV, ICB, CCB, ISA, ISB, CRI, CRA, DLCK or MRL standard: Instrument related QC exceeds the control limits.	Linear Range Check High			Use only if affected samples are ND

Flag	Flag Text	Conditions	Arizona	Florida	Comment
^	ICV, CCV, ICB, CCB, ISA, ISB, CRI, CRA, DLCK or MRL standard: Instrument related QC exceeds the control limits.	Linear Range Check Low			Data impacted; re-analysis should be performed
^	ICV, CCV, ICB, CCB, ISA, ISB, CRI, CRA, DLCK or MRL standard: Instrument related QC exceeds the control limits.	MRL Fail High	V1, L3		Use only if affected samples are ND
^	ICV, CCV, ICB, CCB, ISA, ISB, CRI, CRA, DLCK or MRL standard: Instrument related QC exceeds the control limits.	MRL Fail Low	V9, L4		Data impacted; re-analysis should be performed
+	MSA correlation coefficient is less than 0.995.	MSA CC < 0.995			
<	Not detected at or above the reporting limit	Not Detected			
>	The analyte exceeded the indicated concentration	Other3 - refer to flag			
4	MS, MSD: The analyte present in the original sample is 4 times greater than the matrix spike concentration; therefore, control limits are not applicable.	Analyte 4X MS	M3	J3	
B	Compound was found in the blank and sample.	PB Result Detected	B1	V	Data impacted; re-analysis should be performed
b	Result Detected in the USB	USB Result Detected	K5		For BOD--unseeded blank
B7	Target analyte detected in method blank at or above method reporting limit. Concentration found in the sample was 10 times above the concentration found in the blank.	M1 Manual	B7		Not used for Florida. For Florida, do not flag where sample results are >10x the method blank.
BR	Analyte breached action limit	Result > Than Action Limit			

Flag	Flag Text	Conditions	Arizona	Florida	Comment
c	Compound exceeds routine control limits, but is within acceptable client specific calibration criteria	Exceeds lab limits meets client limits			
C	Pesticide identification was confirmed by GC/MS.	Confirmed GCMS			
C	See Case Narrative	See Case Narrative	N1		
CE1	1,4-DM-2,6-DNB & 1,4-DM-2,5-DNB Co-elute	Other3 - refer to flag			
cn	Refer to Case Narrative for further detail	See Case Narrative	N1		
D	Sample results are obtained from a dilution; the surrogate or matrix spike recoveries reported are calculated from diluted samples.	D1 Manual	S8		
D	Surrogate or matrix spike recoveries were not obtained because the extract was diluted for analysis; also compounds analyzed at a dilution may be flagged with a D.	From a Dilution	S8		
DW	Result has been dry weight corrected	Dry Weight			
E	Result exceeded calibration range.	Off scale high	N1	L	Data impacted; re-analysis should be performed
F	Duplicate RPD exceeds the control limit	Duplicate RPD	R9	J3	Specify if matrix interference
F	MS or MSD exceeds the control limits	MS Recovery High	M1, N1	J3	
F	MS or MSD exceeds the control limits	MS Recovery Low	M2, N1	J3	
F	MS or MSD exceeds the control limits	MS Recovery			
F	MS or MSD exceeds the control limits	MSD Recovery High	M1, N1	J3	

Flag	Flag Text	Conditions	Arizona	Florida	Comment
F	MS or MSD exceeds the control limits	MSD Recovery Low	M2, N1	J3	
F	MS or MSD exceeds the control limits	MSD Recovery			
F	RPD of the MS and MSD exceeds the control limits	MSD RPD	R4, Q11	J3	
FT	This analysis was performed in the field by the sampler whose name appears on the attached Chain of Custody form.	Other4 - refer to flag			
g	Result fails applicable drinking water standards	Other2 - refer to flag	T6		
h	Alternate peak selection upon analytical review.	Alternate Peak Selected			
H	Sample was prepped or analyzed beyond the specified holding time	Sample Analyzed out of HT	H1, H2, H3	Q	Specify if received past HT, approved by client, re-prep outside but original within HT, etc.
H	Sample was prepped or analyzed beyond the specified holding time	Sample Prepped out of HT	H1	Q	Specify if received past HT, approved by client, re-prep outside but original within HT, etc.
HF	Field parameter with a holding time of 15 minutes	Sample HT is Immediate and is flagged.	H5		
I	Indicates the presence of an interference, recovery is not calculated.	Not Reported - Interference		J4	
J	Result is less than the RL but greater than or equal to the MDL and the concentration is an approximate value.	Estimated Result	E4	I	
K	Benzo (b&k) fluoranthene are unresolved due to matrix, result is reported as Benzo(b)fluoranthene.	Other2 - refer to flag			

Flag	Flag Text	Conditions	Arizona	Florida	Comment
k	Benzo (b&k) fluoranthene are unresolved due to matrix, result is reported as Benzo(k)fluoranthene.	Other5 - refer to flag			
L	A negative instrument reading had an absolute value greater than the reporting limit	Absolute value of Negative Reading > RL			Metals--2x allowance for samples; remove if samples not more than -2x
LW	Quantitated against gasoline	Quantified as Gasoline			
N	Presumptive evidence of material.	Presumptive Evidence		N	
ND	Compound not detected.	Not Detected			
P	The %RPD between the primary and confirmation column/detector is >40%. The higher value has been reported	Report Higher if Dual Column RPD Difference > 40%	C6		
p	The %RPD between the primary and confirmation column/detector is >40%. The lower value has been reported.	Report Lower if Dual Column RPD Difference > 40%	C7		
Q	Result was qualitatively confirmed, but not quantitated.	Presented but not quantitated			
Q3	Sample received with improper chemical preservation.	Other - refer to flag	Q3		

Flag	Flag Text	Conditions	Arizona	Florida	Comment
R	The instrument was not calibrated for this compound. A non-detect indicates that the characteristic ions were not present and the compound was not qualitatively identified. No controls were present to determine either sample preparation efficiency or the instrument sensitivity for the compound. As a result, the limit of detection is not known and the reported concentrations are estimates.	Analyte Qualitatively Screened For	T4		
S	Result was determined by the Method of Standard Additions	Result Determined by MSA	M5		
s	SCB Recovery exceeds limits.	Seed Control Blank Recovery High	K4		Seed control blank for BOD
s	SCB Recovery exceeds limits.	Seed Control Blank Recovery Low	K4		Seed control blank for BOD
T	Result is a tentatively identified compound (TIC) and an estimated value.	Tentatively Identified Compound	T4		
V	Serial Dilution exceeds the control limits	SD% Difference Failure			
W	PS: Post-digestion spike was outside control limits	Post Spike High			
W	PS: Post-digestion spike was outside control limits	Post Spike Low			
X	Surrogate is outside control limits	Surrogate Recovery High	S10	J1	Use only if affected samples are ND
X	Surrogate is outside control limits	Surrogate Recovery Low	S7,S12	J1	Data impacted; re-extraction and/or analysis should be performed
X	Surrogate is outside control limits	Surrogate Recovery			
Y	The chromatographic response resembles a typical fuel pattern.	Resembles Fuel Pattern			

Flag	Flag Text	Conditions	Arizona	Florida	Comment
Z	The chromatographic response does not resemble a typical fuel pattern.	Does Not Resemble Fuel Pattern			

For Ohio VAP: The laboratory must implement corrective action procedures to resolve the deviation and limit qualification of the final results. The laboratory is not permitted to deviate from its VAP approved SOP if it intends to attest under affidavit that the "results" are VAP certified. When all corrective actions listed in the SOP have been exhausted, it may be necessary to use technical judgment in which case the decision process and rationale is presented in the final report and/or affidavit and the data will be noted as 'not VAP certified' on the affidavit. A discussion of bias is also required in the narrative.

Appendix 5. Federal Appendix I - Constituents for Assessment Monitoring (40 CFR Part 258)

Appendix I Constituents	CAS Registry Number	Maximum Contaminant Level (mg/L)	RCRA Method
VOLATILES			
Acetone	67-64-1		8260
Acrylonitrile	107-13-1		8260
Benzene	71-43-2	0.005	8260
Bromochloromethane; Chlorobromomethane	74-97-5		8260
Bromodichloromethane; Dichlorobromomethane	75-27-4	**	8260
Bromoform; Tribromomethane	75-25-5	**	8260
Carbon disulfide	75-15-0		8260
Carbon tetrachloride	56-23-5	0.005	8260
Chlorobenzene	108-90-7	0.1	8260
Chloroethane; Ethyl chloride	75-00-3		8260
Chloroform; Trichloro-methane	67-66-3	**	8260
Dibromochloromethane; Chlorodibromomethane	124-48-1	**	8260
1,2-Dibromo-3-chloropro-pane; DBCP	96-12-8	0.0002	8011
1,2-Dibromoethane; Ethylene dibromide; EDB	106-93-4	0.00005	8011
o-Dichlorobenzene; 1,2-Dichlorobenzene	95-50-1	0.6	8260
p-Dichlorobenzene; 1,4-Dichlorobenzene	106-46-7	0.075	8260
trans-1,4-Dichloro-2-butene	110-57-6		8260
1,1-Dichloroethane; Ethylidene chloride	75-34-3		8260
1,2-Dichloroethane; Ethylene dichloride	107-06-2	0.005	8260
1,1-Dichloroethylene; Vinylidene chloride; 1,1-Dichloroethene	75-35-4	0.007	8260
cis-1,2-Dichloroethylene; cis-1,2-Dichloroethene	156-59-2	0.07	8260
trans-1,2-Dichloroethylene; trans-1,2-Dichloroethene	156-60-5	0.1	8260
1,2-Dichloropropane; Propylene dichloride	78-87-5	0.005	8260
cis-1,3-Dichloropropene	10061-01-5		8260
trans-1,3-Dichloropropene	10061-02-6		8260
Ethylbenzene	100-41-4	0.7	8260
2-Hexanone; Methyl butyl ketone	591-78-6		8260
Methyl bromide; Bromomethane	74-83-9		8260
Methyl chloride; Chloromethane	74-87-3		8260
Methyl ethyl ketone; MEK; 2-Butanone	78-93-3		8260
Methyl iodide; Iodomethane	74-88-4		8260
4-Methyl-2-pentanone; Methyl isobutyl ketone	108-10-1		8260
Methylene chloride; Dichloromethane	75-09-2	0.005	8260
Styrene	100-42-5	0.1	8260
1,1,1,2-Tetrachloroethane	630-20-6		8260
1,1,2,2-Tetrachloroethane	79-34-5		8260
Tetrachloroethylene; Tetrachloroethene; Perchloroethylene	127-18-4	0.005	8260
Toluene	108-88-3	1	8260
1,1,1-Trichloroethane; Methyl chloroform	71-55-6	0.2	8260
1,1,2-Trichloroethane	79-00-5	0.005	8260
Trichloroethylene; Trichloroethene	79-01-6	0.005	8260
Trichlorofluoromethane	75-69-4		8260

Appendix I Constituents	CAS Registry Number	Maximum Contaminant Level (mg/L)	RCRA Method
1,2,3-Trichloropropane	96-18-4		8260
Vinyl acetate	108-05-4		8260
Vinyl chloride; Chloroethene	75-01-4	0.002	8260
Xylene (total)	96-47-6, 108-38-3, 106-42-3, 1330-20-7.	10	8260
METALS			
Antimony	7440-36-0	0.006	6010
Arsenic	7440-38-2	0.05	6010
Barium	7440-39-3	2.0	6010
Beryllium	7440-41-7	0.004	6010
Cadmium	7440-43-9	0.005	6010
Chromium	7440-47-3	0.1	6010
Cobalt	7440-48-4		6010
Copper	7440-50-8	1	6010
Lead	7439-92-1	0.015	6010
Nickel	7440-02-0	0.1	6010
Selenium	7782-49-2	0.05	6010
Silver	7440-22-4	0.1	6010
Thallium	7440-28-0	0.002	6010
Vanadium	7440-62-2		6010
Zinc	7440-66-6	5	6010

Note: Depending upon the state, additional constituents may be required.

Appendix 6. Federal Appendix II - Constituents for Assessment Monitoring (40 CFR Part 258)

Appendix II Constituents	CAS Registry Number	Maximum Contaminant Level (mg/L)	RCRA Method
VOLATILES			
Acetone	67-64-1		8260
Acetonitrile; Methyl cyanide	75-05-8		8260
Acrolein	107-02-8		8260
Acrylonitrile	107-13-1		8260
Allyl chloride	107-05-1		8260
Benzene	71-43-2	0.005	8260
Bromochloromethane; Chlorobromomethane	74-97-5		8260
Bromodichloromethane; Dichlorobromomethane	75-27-4	**	8260
Bromoform; Tribromomethane	75-25-5	**	8260
Carbon disulfide	75-15-0		8260
Carbon tetrachloride	56-23-5	0.005	8260
Chlorobenzene	108-90-7	0.1	8260
Chloroethane; Ethyl chloride	75-00-3		8260
Chloroform; Trichlorome-thane	67-66-3	**	8260
Chloroprene	126-99-8		8260
Dibromochloromethane; Chlorodibromomethane	124-48-1	**	8260
1,2-Dibromo-3-chloropro-pane; DBCP	96-12-8	0.0002	8011
1,2-Dibromoethane; Ethylene dibromide; EDB	106-93-4	0.00005	8011
o-Dichlorobenzene; 1,2-Dichlorobenzene	95-50-1	0.6	8260
m-Dichlorobenzene; 1,3-Dichlorobenzene	541-73-1		8260
p-Dichlorobenzene; 1,4-Dichlorobenzene	106-46-7	0.075	8260
trans-1,4-Dichloro-2-butene	110-57-6		8260
Dichlorodifluoromethane	75-71-8		8260
1,1-Dichloroethane; Ethylidene chloride	75-34-3		8260
1,2-Dichloroethane; Ethylene dichloride	107-06-2	0.005	8260
1,1-Dichloroethylene; Vinylidene chloride; 1,1-Dichloroethene	75-35-4	0.007	8260
cis-1,2-Dichloroethylene; cis-1,2-Dichloroethene	156-59-2	0.07	8260
trans-1,2-Dichloroethylene; trans-1,2-Dichloroethene	156-60-5	0.1	8260
1,2-Dichloropropane; Propylene dichloride	78-87-5	0.005	8260
1,3-Dichloropropane; Trimethylene dichloride	142-28-9		8260
2,2-Dichloropropane; Isopropylidene chloride	594-20-7		8260
1,1-Dichloropropene	563-58-6		8260
cis-1,3-Dichloropropene	10061-01-5		8260
trans-1,3-Dichloropropene	10061-02-6		8260
Ethylbenzene	100-41-4	0.7	8260
Ethyl methacrylate	97-63-2		8260
2-Hexanone; Methyl butyl ketone	591-78-6		8260
Isobutyl alcohol	78-83-1		8260
Methacrylonitrile	126-98-7		8260
Methyl bromide; Bromomethane	74-83-9		8260
Methyl chloride; Chloromethane	74-87-3		8260
Methyl ethyl ketone; MEK; 2-Butanone	78-93-3		8260
Methyl iodide; Iodomethane	74-88-4		8260
Methyl methacrylate	80-62-6		8260

Appendix II Constituents	CAS Registry Number	Maximum Contaminant Level (mg/L)	RCRA Method
4-Methyl-2-pentanone; Methyl isobutyl ketone	108-10-1		8260
Methylene bromide; Dibromomethane	74-95-3		8260
Methylene chloride; Dichloromethane	75-09-2	0.005	8260
Propionitrile; Ethyl cyanide	107-12-0		8260
Styrene	100-42-5	0.1	8260
1,1,1,2-Tetrachloroethane	630-20-6		8260
1,1,2,2-Tetrachloroethane	79-34-5		8260
Tetrachloroethylene; Tetrachloroethene; Perchloroethylene	127-18-4	0.005	8260
Toluene	108-88-3	1	8260
1,1,1-Trichloroethane; Methyl chloroform	71-55-6	0.2	8260
1,1,2-Trichloroethane	79-00-5	0.005	8260
Trichloroethylene; Trichloroethene	79-01-6	0.005	8260
Trichlorofluoromethane	75-69-4		8260
1,2,3-Trichloropropane	96-18-4		8260
Vinyl acetate	108-05-4		8260
Vinyl chloride; Chloroethene	75-01-4	0.002	8260
Xylene (total)	96-47-6, 108-38-3, 106-42-3, 1330-20-7.	10	8260
SEMIVOLATILES			
Acenaphthene	83-32-9		8270
Acenaphthylene	208-96-8		8270
Acetophenone	98-86-2		8270
2-Acetylaminofluorene; 2-AAF	53-96-3		8270
4-Aminobiphenyl	92-67-1		8270
Anthracene	120-12-7		8270
Benzo[a]anthracene; Benzathracene	56-55-3		8270
Benzo[b]fluoranthene	205-99-2		8270
Benzo[k]fluoranthene	207-08-9		8270
Benzo[ghi]perylene	191-24-2		8270
Benzyl alcohol	100-51-6		8270
Bis(2-chloroethoxy)methane	111-91-1		8270
Bis(2-chloroethyl)ether; Dichloroethyl ether	111-44-4		8270
Bis(2-chloroisopropyl)ether; Bis(2-chloro-1-methylethyl) ether; 2,2-Dichlorodiisopro-pyl ether; DCIP	108-60-1		8270
Bis(2-ethylhexyl) phthalate	117-81-7	0.006	8270
4-Bromophenyl phenyl ether	101-55-3		8270
Butyl benzyl phthalate	85-68-7		8270
p-Chloroaniline; 4-Chloro-aniline	106-47-8		8270
Chlorobenzilate	510-15-6		8270
p-Chloro-m-cresol; 4-Chloro-3-methylphenol	59-50-7		8270
2-Chloronaphthalene	91-58-7		8270
2-Chlorophenol	95-57-8		8270
4-Chlorophenyl phenyl ether	7005-72-3		8270
Chrysene	218-01-9		8270
m-Cresol; 3-Methylphenol	108-39-4		8270
o-Cresol; 2-Methylphenol	95-48-7		8270

Appendix II Constituents	CAS Registry Number	Maximum Contaminant Level (mg/L)	RCRA Method
p-Cresol; 4-Methylphenol	106-44-5		8270
Diallate	2303-16-4		8270
Dibenz[a,h]anthracene	53-70-3		8270
Dibenzofuran	132-64-9		8270
Di-n-butyl phthalate	84-74-2		8270
3,3'-Dichlorobenzidine	91-94-1		8270
2,4-Dichlorophenol	120-83-2		8270
2,6-Dichlorophenol	87-65-0		8270
Diethyl phthalate	84-66-2		8270
o,o-Diethyl-o-2-pyrazinyl phosphorothioate; Thionazin	297-97-2		8270
Dimethoate	60-51-5		8270
p-(Dimethylamino)azoben-zene	60-11-7		8270
7,12-Dimethylbenz[a]an-thracene	57-97-6		8270
3,3'-Dimethylbenzidine	119-93-7		8270
2,4-Dimethylphenol	105-67-9		8270
Dimethyl phthalate	131-11-3		8270
m-Dinitrobenzene	99-65-0		8270
4,6-Dinitro-o-cresol; 4,6-Dinitro-2-methylphenol	534-52-1		8270
2,4-Dinitrophenol	51-28-5		8270
2,4-Dinitrotoluene	121-4-2		8270
2,6-Dinitrotoluene	606-20-2		8270
Di-n-octyl phthalate	117-84-0		8270
Diphenylamine	122-39-4		8270
Disulfoton	298-04-4		8270
Ethyl methanesulfonate	62-50-0		8270
Famphur	52-85-7		8270
Fluoranthene	206-44-0		8270
Fluorene	86-73-7		8270
Hexachlorobenzene	118-74-1	0.001	8270
Hexachlorobutadiene	87-68-3		8270
Hexachlorocyclopentadiene	77-47-4	0.05	8270
Hexachloroethane	67-72-1		8270
Hexacloroproprene	1888-71-7		8270
Indeno[1,2,3-cd]pyrene	193-39-5		8270
Isodrin	465-73-6		8270
Isophorone	78-59-1		8270
Isosafrole	120-58-1		8270
Kepone	143-50-0		8270
Methapyrilene	91-80-5		8270
3-Methylcholanthrene	56-49-5		8270
Methyl methanesulfonate	66-27-3		8270
2-Methylnaphthalene	91-57-6		8270
Methyl parathion; Parathion methyl	298-00-0		8270
Naphthalene	91-20-3		8270
1,4-Naphthoquinone	130-15-4		8270
1-Naphthylamine	134-32-7		8270
2-Naphthylamine	91-59-8		8270
o-Nitroaniline; 2-Nitroaniline	88-74-4		8270

Appendix II Constituents	CAS Registry Number	Maximum Contaminant Level (mg/L)	RCRA Method
m-Nitroaniline;3-Nitroaniline	99-09-2		8270
p-Nitroaniline; 4-Nitroaniline	100-01-6		8270
Nitrobenzene	98-95-3		8270
o-Nitrophenol; 2-Nitrophenol	88-75-5		8270
p-Nitrophenol; 4-Nitrophenol	100-02-7		8270
N-nitrosodi-n-butylamine	924-16-3		8270
N-nitrosodiethylamine	55-18-5		8270
N-nitrosodimethylamine	62-75-9		8270
N-nitrosodiphenylamine	86-30-6		8270
N-nitrosodipropylamine; Di-n-propyl-nitrosamine; N-nitroso-n-dipropylamine	621-64-7		8270
N-nitrosomethylethylamine	1059-95-6		8270
N-nitrosopiperidine	100-75-4		8270
N-nitrosopyrrolidine	930-55-2		8270
5-Nitro-o-toluidine	99-55-8		8270
Parathion	56-38-2		8270
Pentachlorobenzene	608-93-5		8270
Pentachloronitrobenzene	82-68-8		8270
Phenacetin	62-44-2		8270
Phenanthrene	85-01-8		8270
Phenol	108-95-2		8270
p-Phenylenediamine	106-50-3		8270
Phorate	298-02-2		8270
Pronamide	23950-58-5		8270
Pyrene	129-00-0		8270
Safrole	94-59-7		8270
1,2,4,5-Tetrachlorobenzene	95-94-3		8270
2,3,4,6-Tetrachlorophenol	58-90-2		8270
o-Toluidine	95-53-4		8270
1,2,4-Trichlorobenzene	120-82-1		8270
2,4,5-Trichlorophenol	95-95-4		8270
2,4,6-Trichlorophenol	88-06-2		8270
o,o,o-Triethyl phosphoro-thioate; Terbufos	126-68-1		8270
sym-Trinitrobenzene; 1,3,5-Trinitrobenzene	99-35-4		8270
Benzo[a]pyrene	50-32-8	0.0002	8310
ORGANOCHLORINE PESTICIDES/PCBs			
Aldrin	309-00-2		8081
alpha-BHC; alpha-Benzene hexachloride	319-84-6		8081
beta-BHC; beta-Benzene hexachloride	309-85-7		8081
delta-BHC;delta-Benzene hexachloride	319-86-8		8081
gamma-BHC; gamma-Benzene hexachloride; Lindane	58-89-9	0.0002	8081
alpha-Chlordane	5103-71-9	0.002	8081
gamma-Chlordane	5103-74-2	0.002	8081
4,4'-DDD	72-54-8		8081
4,4'-DDE	72-55-9		8081
4,4'-DDT	50-29-3		8081
Dieldrin	60-57-1		8081
Endosulfan I	959-98-8		8081

Appendix II Constituents	CAS Registry Number	Maximum Contaminant Level (mg/L)	RCRA Method
Endosulfan II	33213-65-9		8081
Endosulfan sulfate	1031-07-8		8081
Endrin	72-20-8	0.002	8081
Endrin aldehyde	7421-93-4		8081
Heptachlor	76-44-8	0.0004	8081
Heptachlor epoxide	1024-57-3	0.0002	8081
Methoxychlor	72-43-5	0.04	8081
Toxaphene	8001-35-2	0.003	8081
Polychlorinated biphenyls; PCBs; Aroclors	1336-36-3, 12674-11-2, 11104-28-2, 11141-16-5, 53469-21-9, 12672-29-6, 11097-69-1, 11096-82-5	0.0005	8082
HERBICIDES			
2,4-D; 2,4-Dichlorophenoxy-acetic acid	94-75-7	0.02	8151
2,4,5-T; 2,4,5-Trichlorophen-oxyacetic acid	93-76-5		8151
2,4,5-TP; 2,4,5-Trichloro-phenoxypropanoic acid; Silvex	93-72-1	0.05	8151
Dinoseb; DNBP; 2-sec-Butyl-4,6-dinitrophenol	88-85-7		8151
Pentachlorophenol	87-86-5	0.001	8151
METALS			
Antimony	7440-36-0	0.006	6010
Arsenic	7440-38-2	0.05	6010
Barium	7440-39-3	2.0	6010
Beryllium	7440-41-7	0.004	6010
Cadmium	7440-43-9	0.005	6010
Chromium	7440-47-3	0.1	6010
Cobalt	7440-48-4		6010
Copper	7440-50-8	1	6010
Lead	7439-92-1	0.015	6010
Nickel	7440-02-0	0.1	6010
Selenium	7782-49-2	0.05	6010
Silver	7440-22-4	0.1	6010
Thallium	7440-28-0	0.002	6010
Tin	7440-31-5		6010
Vanadium	7440-62-2		6010
Zinc	7440-66-6	5	6010
Mercury	7439-97-6	0.002	7470
OTHER INORGANICS			
Cyanide	57-12-5	0.2 as Free	9012
Sulfide	18496-25-8		9034

Appendix 7. Federal Appendix IX – Groundwater Monitoring List (40 CFR Part 264)

Appendix IX Constituents	CAS Registry Number	RCRA Method
VOLATILES		
Acetone	67-64-1	8260
Acetonitrile; Methyl cyanide	75-05-8	8260
Acrolein	107-02-8	8260
Acrylonitrile	107-13-1	8260
Allyl chloride	107-05-1	8260
Benzene	71-43-2	8260
Bromodichloromethane; Dichlorobromomethane	75-27-4	8260
Bromoform; Tribromomethane	75-25-5	8260
Carbon disulfide	75-15-0	8260
Carbon tetrachloride	56-23-5	8260
Chlorobenzene	108-90-7	8260
Chloroethane; Ethyl chloride	75-00-3	8260
Chloroform; Trichloro-methane	67-66-3	8260
Chloroprene	126-99-8	8260
Dibromochloromethane; Chlorodibromomethane	124-48-1	8260
1,2-Dibromo-3-chloropro-pane; DBCP	96-12-8	8260
1,2-Dibromoethane; Ethylene dibromide; EDB	106-93-4	8260
o-Dichlorobenzene; 1,2-Dichlorobenzene	95-50-1	8260
m-Dichlorobenzene; 1,3-Dichlorobenzene	541-73-1	8260
p-Dichlorobenzene; 1,4-Dichlorobenzene	106-46-7	8260
trans-1,4-Dichloro-2-butene	110-57-6	8260
Dichlorodifluoromethane	75-71-8	8260
1,1-Dichloroethane; Ethylidene chloride	75-34-3	8260
1,2-Dichloroethane; Ethylene dichloride	107-06-2	8260
1,1-Dichloroethylene; Vinylidene chloride; 1,1-Dichloroethene	75-35-4	8260
trans-1,2-Dichloroethylene; trans-1,2-Dichloroethene	156-60-5	8260
1,2-Dichloropropane; Propylene dichloride	78-87-5	8260
cis-1,3-Dichloropropene	10061-01-5	8260
trans-1,3-Dichloropropene	10061-02-6	8260
1,4-Dioxane	123-91-1	8260
Ethylbenzene	100-41-4	8260
Ethyl methacrylate	97-63-2	8260
Hexachlorobutadiene	87-68-3	8260
2-Hexanone; Methyl butyl ketone	591-78-6	8260
Isobutyl alcohol	78-83-1	8260
Methacrylonitrile	126-98-7	8260
Methyl bromide; Bromomethane	74-83-9	8260
Methyl chloride; Chloromethane	74-87-3	8260
Methyl ethyl ketone; MEK; 2-Butanone	78-93-3	8260
Methyl iodide; Iodomethane	74-88-4	8260
Methyl methacrylate	80-62-6	8260
4-Methyl-2-pentanone; Methyl isobutyl ketone	108-10-1	8260

Appendix IX Constituents	CAS Registry Number	RCRA Method
Methylene bromide; Dibromomethane	74-95-3	8260
Methylene chloride; Dichloromethane	75-09-2	8260
Propionitrile; Ethyl cyanide	107-12-0	8260
Styrene	100-42-5	8260
1,1,1,2-Tetrachloroethane	630-20-6	8260
1,1,2,2-Tetrachloroethane	79-34-5	8260
Tetrachloroethylene; Tetrachloroethene; Perchloroethylene	127-18-4	8260
Toluene	108-88-3	8260
1,1,1-Trichloroethane; Methyl chloroform	71-55-6	8260
1,1,2-Trichloroethane	79-00-5	8260
Trichloroethylene; Trichloroethene	79-01-6	8260
Trichlorofluoromethane	75-69-4	8260
1,2,3-Trichloropropane	96-18-4	8260
Vinyl acetate	108-05-4	8260
Vinyl chloride; Chloroethene	75-01-4	8260
Xylenes (total)	96-47-6, 108-38-3, 106-42-3, 1330-20-7.	8260
SEMIVOLATILES		
Acenaphthene	83-32-9	8270
Acenaphthylene	208-96-8	8270
Acetophenone	98-86-2	8270
2-Acetylaminofluorene; 2-AAF	53-96-3	8270
4-Aminobiphenyl	92-67-1	8270
Aniline	62-53-3	8270
Anthracene	120-12-7	8270
Aramite	140-57-8	8270
Benzo(a)anthracene; Benzathracene	56-55-3	8270
Benzo(a)pyrene	50-32-8	8270
Benzo(b)fluoranthene	205-99-2	8270
Benzo(k)fluoranthene	207-08-9	8270
Benzo(ghi)perylene	191-24-2	8270
Benzyl alcohol	100-51-6	8270
Bis(2-chloroethoxy)methane	111-91-1	8270
Bis(2-chloroethyl)ether; Dichloroethyl ether	111-44-4	8270
Bis(2-chloro-1-methylethyl) ether; 2,2-Dichlorodiiso-propyl ether; DCIP	108-60-1	8270
Bis(2-ethylhexyl) phthalate	117-81-7	8270
4-Bromophenyl phenyl ether	101-55-3	8270
Butyl benzyl phthalate	85-68-7	8270
p-Chloroaniline; 4-Chloro-aniline	106-47-8	8270
Chlorobenzilate	510-15-6	8270
p-Chloro-m-cresol; 4-Chloro-3-methylphenol	59-50-7	8270
2-Chloronaphthalene	91-58-7	8270
2-Chlorophenol	95-57-8	8270
4-Chlorophenyl phenyl ether	7005-72-3	8270

Appendix IX Constituents	CAS Registry Number	RCRA Method
Chrysene	218-01-9	8270
m-Cresol; 3-Methylphenol	108-39-4	8270
o-Cresol; 2-Methylphenol	95-48-7	8270
p-Cresol; 4-Methylphenol	106-44-5	8270
Diallate	2303-16-4	8270
Dibenz(a,h)anthracene	53-70-3	8270
Dibenzofuran	132-64-9	8270
Di-n-butyl phthalate	84-74-2	8270
3,3'-Dichlorobenzidine	91-94-1	8270
2,4-Dichlorophenol	120-83-2	8270
2,6-Dichlorophenol	87-65-0	8270
Diethyl phthalate	84-66-2	8270
o,o-Diethyl-o-2-pyrazinyl phosphorothioate; Thionazine	297-97-2	8270
Dimethoate	60-51-5	8270
p-(Dimethylamino)azobenzene	60-11-7	8270
7,12-Dimethylbenz(a)anthracene	57-97-6	8270
3,3'-Dimethylbenzidine	119-93-7	8270
2,4-Dimethylphenol	105-67-9	8270
Dimethyl phthalate	131-11-3	8270
a,a-Dimethylphenethylamine	122-09-8	8270
m-Dinitrobenzene; 1,3-Dinitrobenzene	99-65-0	8270
4,6-Dinitro-o-cresol; 4,6-Dinitro-2-methylphenol	534-52-1	8270
2,4-Dinitrophenol	51-28-5	8270
2,4-Dinitrotoluene	121-4-2	8270
2,6-Dinitrotoluene	606-20-2	8270
Di-n-octyl phthalate	117-84-0	8270
Dinoseb; DNBP; 2-sec-Butyl-4,6-dinitrophenol	88-85-7	8270
Diphenylamine	122-39-4	8270
Disulfoton	298-04-4	8270
Ethyl methanesulfonate	62-50-0	8270
Famphur	52-85-7	8270
Fluoranthene	206-44-0	8270
Fluorene	86-73-7	8270
Hexachlorobenzene	118-74-1	8270
Hexachlorobutadiene	87-68-3	8270
Hexachlorocyclopentadiene	77-47-4	8270
Hexachloroethane	67-72-1	8270
Hexachlorophene	70-30-4	8270
Hexacloroproprene	1888-71-7	8270
Indeno(1,2,3-cd)pyrene	193-39-5	8270
Isodrin	465-73-6	8270
Isophorone	78-59-1	8270
Isosafrole	120-58-1	8270
Kepone	143-50-0	8270
Methapyrilene	91-80-5	8270
3-Methylcholanthrene	56-49-5	8270
Methyl methanesulfonate	66-27-3	8270

Appendix IX Constituents	CAS Registry Number	RCRA Method
2-Methylnaphthalene	91-57-6	8270
Methyl parathion; Parathion methyl	298-00-0	8270
Naphthalene	91-20-3	8270
1,4-Naphthoquinone	130-15-4	8270
1-Naphthylamine	134-32-7	8270
2-Naphthylamine	91-59-8	8270
o-Nitroaniline; 2-Nitroaniline	88-74-4	8270
m-Nitroaniline;3-Nitroaniline	99-09-2	8270
p-Nitroaniline; 4-Nitroaniline	100-01-6	8270
Nitrobenzene	98-95-3	8270
5-Nitro-o-toluidine	99-55-8	8270
o-Nitrophenol; 2-Nitrophenol	88-75-5	8270
p-Nitrophenol; 4-Nitrophenol	100-02-7	8270
4-Nitroquinoline 1-oxide	56-57-5	8270
N-nitrosodi-n-butylamine	924-16-3	8270
N-nitrosodiethylamine	55-18-5	8270
N-nitrosodimethylamine	62-75-9	8270
N-nitrosodiphenylamine	86-30-6	8270
N-nitrosodipropylamine; Di-n-propyl-nitrosamine; N-nitroso-n-dipropylamine	621-64-7	8270
N-nitrosomethylethylamine	1059-95-6	8270
N-nitrosomorpholine	59-89-2	8270
N-nitrosopiperidine	100-75-4	8270
N-nitrosopyrrolidine	930-55-2	8270
Parathion	56-38-2	8270
Pentachlorobenzene	608-93-5	8270
Pentachloroethane	76-01-7	8270
Pentachloronitrobenzene	82-68-8	8270
Pentachlorophenol	87-86-5	8270
Phenacetin	62-44-2	8270
Phenanthrene	85-01-8	8270
Phenol	108-95-2	8270
p-Phenylenediamine; 1,4-Phenylenediamine	106-50-3	8270
Phorate	298-02-2	8270
2-Picoline	109-06-8	8270
Pronamide	23950-58-5	8270
Pyrene	129-00-0	8270
Pyridine	110-86-1	8270
Safrole	94-59-7	8270
1,2,4,5-Tetrachlorobenzene	95-94-3	8270
2,3,4,6-Tetrachlorophenol	58-90-2	8270
Tetraethylpyrophosphate, Sulfotep	3689-24-5	8270
o-Toluidine	95-53-4	8270
1,2,4-Trichlorobenzene	120-82-1	8270
2,4,5-Trichlorophenol	95-95-4	8270
2,4,6-Trichlorophenol	88-06-2	8270
o,o,o-Triethyl phosphoro-thioate; Terbufos	126-68-1	8270

Appendix IX Constituents	CAS Registry Number	RCRA Method
sym-Trinitrobenzene; 1,3,5-Trinitrobenzene	99-35-4	8270
ORGANOCHLORINE PESTICIDES/PCBs		
Aldrin	309-00-2	8081
alpha-BHC; alpha-Benzene hexachloride	319-84-6	8081
beta-BHC; beta-Benzene hexachloride	309-85-7	8081
delta-BHC;delta-Benzene hexachloride	319-86-8	8081
gamma-BHC; gamma-Benzene hexachloride; Lindane	58-89-9	8081
alpha-Chlordane	5103-71-9	8081
gamma-Chlordane	5103-74-2	8081
4,4'-DDD	72-54-8	8081
4,4'-DDE	72-55-9	8081
4,4'-DDT	50-29-3	8081
Dieldrin	60-57-1	8081
Endosulfan I	959-98-8	8081
Endosulfan II	33213-65-9	8081
Endosulfan sulfate	1031-07-8	8081
Endrin	72-20-8	8081
Endrin aldehyde	7421-93-4	8081
Heptachlor	76-44-8	8081
Heptachlor epoxide	1024-57-3	8081
Methoxychlor	72-43-5	8081
Toxaphene	8001-35-2	8081
Polychlorinated biphenyls; PCBs; Aroclors	1336-36-3, 12674-11-2, 11104-28-2, 11141-16-5, 53469-21-9, 12672-29-6, 11097-69-1, 11096-82-5	8082
HERBICIDES		
2,4-D; 2,4-Dichlorophenoxy-acetic acid	94-75-7	8151
2,4,5-T; 2,4,5-Trichlorophen-oxyacetic acid	93-76-5	8151
2,4,5-TP; 2,4,5-Trichloro-phenoxypropanoic acid; Silvex	93-72-1	8151
METALS		
Antimony	7440-36-0	6010
Arsenic	7440-38-2	6010
Barium	7440-39-3	6010
Beryllium	7440-41-7	6010
Cadmium	7440-43-9	6010
Chromium	7440-47-3	6010
Cobalt	7440-48-4	6010
Copper	7440-50-8	6010
Lead	7439-92-1	6010
Nickel	7440-02-0	6010
Selenium	7782-49-2	6010
Silver	7440-22-4	6010

Appendix IX Constituents	CAS Registry Number	RCRA Method
Thallium	7440-28-0	6010
Tin	7440-31-5	6010
Vanadium	7440-62-2	6010
Zinc	7440-66-6	6010
Mercury	7439-97-6	7470,7471
OTHER INORGANICS		
Cyanide	57-12-5	9012
Sulfide	18496-25-8	9034
DIOXINS		
2,3,7,8-Tetrachlorodibenzo-o-dioxin	1746-01-6	8280 / 8290
Polychlorinated dibenzo-furans; PCDFs	NA	8280 / 8290
Polychlorinated dibenzo-p-dioxins; PCDDs	NA	8280 / 8290

Appendix 8. Federal Target Compound and Analyte List

TCL/TAL Constituents	CAS Registry Number	RCRA Method
VOLATILES		
Acetone	67-64-1	8260
Benzene	71-43-2	8260
Bromochloromethane	74-97-5	8260
Bromodichloromethane; Dichlorobromomethane	75-27-4	8260
Bromoform; Tribromomethane	75-25-5	8260
Carbon disulfide	75-15-0	8260
Carbon tetrachloride	56-23-5	8260
Chlorobenzene	108-90-7	8260
Chloroethane; Ethyl chloride	75-00-3	8260
Chloroform; Trichlorome-thane	67-66-3	8260
Cyclohexane	110-82-7	8260
Dibromochloromethane; Chlorodibromomethane	124-48-1	8260
1,2-Dibromomethane	106-93-4	8260
1,2-Dibromo-3-chloropropane	96-12-8	8260
o-Dichlorobenzene; 1,2-Dichlorobenzene	95-50-1	8260
m-Dichlorobenzene; 1,3-Dichlorobenzene	541-73-1	8260
p-Dichlorobenzene; 1,4-Dichlorobenzene	106-46-7	8260
Dichlorodifluoromethane	75-71-8	8260
1,1-Dichloroethane; Ethylidene chloride	75-34-3	8260
1,2-Dichloroethane; Ethylene dichloride	107-06-2	8260
1,1-Dichloroethylene; Vinylidene chloride; 1,1-Dichloroethene	75-35-4	8260
cis-1,2-Dichloroethylene; cis-1,2-Dichloroethene	156-59-2	8260
trans-1,2-Dichloroethylene; trans-1,2-Dichloroethene	156-60-5	8260
1,2-Dichloropropane; Propylene dichloride	78-87-5	8260
cis-1,3-Dichloropropene	10061-01-5	8260
trans-1,3-Dichloropropene	10061-02-6	8260
1,4-Dioxane	123-91-1	8260
Ethylbenzene	100-41-4	8260
2-Hexanone; Methyl butyl ketone	591-78-6	8260
Isopropylbenzene	98-82-8	8260
Methyl acetate	79-20-9	8260
Methyl bromide; Bromomethane	74-83-9	8260
Methyl chloride; Chloromethane	74-87-3	8260
Methylcyclohexane	108-87-2	8260
Methyl ethyl ketone; MEK; 2-Butanone	78-93-3	8260
Methyl tert-butyl ether	1634-04-4	8260
4-Methyl-2-pentanone; Methyl isobutyl ketone	108-10-1	8260
Methylene chloride; Dichloromethane	75-09-2	8260
Styrene	100-42-5	8260
1,1,2,2-Tetrachloroethane	79-34-5	8260
Tetrachloroethylene; Tetrachloroethene; Perchloroethylene	127-18-4	8260
Toluene	108-88-3	8260
1,2,3-Trichlorobenzene	87-61-6	8260
1,2,4-Trichlorobenzene	120-82-1	8260
1,1,1-Trichloroethane; Methyl chloroform	71-55-6	8260

TCL/TAL Constituents	CAS Registry Number	RCRA Method
1,1,2-Trichloroethane	79-00-5	8260
Trichloroethylene; Trichloroethene	79-01-6	8260
Trichlorofluoromethane	75-69-4	8260
1,1,2-Trichloro-1,2,2-trifluoroethane	76-13-1	8260
Vinyl chloride; Chloroethene	75-01-4	8260
m-,p-Xylene	179601-23-1	8260
o-Xylene	95-47-6	8260
Xylene (total)	96-47-6, 108-38-3, 106-42-3, 1330-20-7.	8260
SEMIVOLATILES		
Acenaphthene	83-32-9	8270
Acenaphthylene	208-96-8	8270
Acetophenone	98-86-2	8270
Anthracene	120-12-7	8270
Atrazine	1912-94-9	8270
Benzaldehyde	100-52-7	8270
Benzo(a)anthracene; Benzathracene	56-55-3	8270
Benzo(a)pyrene	50-32-8	8270
Benzo(b)fluoranthene	205-99-2	8270
Benzo(k)fluoranthene	207-08-9	8270
Benzo(ghi)perylene	191-24-2	8270
1,1'-Biphenyl	92-52-4	8270
Bis(2-chloroethoxy)methane	111-91-1	8270
Bis(2-chloroethyl)ether; Dichloroethyl ether	111-44-4	8270
Bis(2-chloro-1-methylethyl) ether; 2,2-Dichlorodiisopropyl ether; DCIP; 2,2'-Oxybis(1-chloropropane)	108-60-1	8270
Bis(2-ethylhexyl) phthalate	117-81-7	8270
4-Bromophenyl phenyl ether	101-55-3	8270
Butyl benzyl phthalate	85-68-7	8270
Caprolactam	105-60-2	8270
Carbazole	86-74-8	8270
4-Chloroaniline	106-47-8	8270
p-Chloro-m-cresol; 4-Chloro-3-methylphenol	59-50-7	8270
2-Chloronaphthalene	91-58-7	8270
2-Chlorophenol	95-57-8	8270
4-Chlorophenyl phenyl ether	7005-72-3	8270
Chrysene	218-01-9	8270
m-Cresol; 3-Methylphenol	108-39-4	8270
o-Cresol; 2-Methylphenol	95-48-7	8270
p-Cresol; 4-Methylphenol	106-44-5	8270
Dibenz(a,h)anthracene	53-70-3	8270
Dibenzofuran	132-64-9	8270
Di-n-butyl phthalate	84-74-2	8270
3,3'-Dichlorobenzidine	91-94-1	8270
2,4-Dichlorophenol	120-83-2	8270
Diethyl phthalate	84-66-2	8270
2,4-Dimethylphenol	105-67-9	8270
Dimethyl phthalate	131-11-3	8270

TCL/TAL Constituents	CAS Registry Number	RCRA Method
4,6-Dinitro-o-cresol; 4,6-Dinitro-2-methylphenol	534-52-1	8270
2,4-Dinitrophenol	51-28-5	8270
2,4-Dinitrotoluene	121-4-2	8270
2,6-Dinitrotoluene	606-20-2	8270
Di-n-octyl phthalate	117-84-0	8270
Fluoranthene	206-44-0	8270
Fluorene	86-73-7	8270
Hexachlorobenzene	118-74-1	8270
Hexachlorobutadiene	87-68-3	8270
Hexachlorocyclopentadiene	77-47-4	8270
Hexachloroethane	67-72-1	8270
Indeno(1,2,3-cd)pyrene	193-39-5	8270
Isophorone	78-59-1	8270
2-Methylnaphthalene	91-57-6	8270
Naphthalene	91-20-3	8270
o-Nitroaniline; 2-Nitroaniline	88-74-4	8270
m-Nitroaniline;3-Nitroaniline	99-09-2	8270
p-Nitroaniline; 4-Nitroaniline	100-01-6	8270
Nitrobenzene	98-95-3	8270
o-Nitrophenol; 2-Nitrophenol	88-75-5	8270
p-Nitrophenol; 4-Nitrophenol	100-02-7	8270
n-Nitrosodiphenylamine	86-30-6	8270
n-Nitrosodipropylamine; Di-n-propyl-nitrosamine; N-nitroso-n-dipropylamine	621-64-7	8270
Pentachlorophenol	87-86-5	8270
Phenanthrene	85-01-8	8270
Phenol	108-95-2	8270
Pyrene	129-00-0	8270
1,2,4,5-Tetrachlorobenzene	95-94-3	8270
2,3,4,6-Tetrachlorophenol	58-90-2	8270
2,4,5-Trichlorophenol	95-95-4	8270
2,4,6-Trichlorophenol	88-06-2	8270
ORGANOCHLORINE PESTICIDES/PCBs		
Aldrin	309-00-2	8081
alpha-BHC; alpha-Benzene hexachloride	319-84-6	8081
beta-BHC; beta-Benzene hexachloride	309-85-7	8081
delta-BHC;delta-Benzene hexachloride	319-86-8	8081
gamma-BHC; gamma-Benzene hexachloride; Lindane	58-89-9	8081
alpha-Chlordane	5103-71-9	8081
gamma-Chlordane	5103-74-2	8081
4,4'-DDD	72-54-8	8081
4,4'-DDE	72-55-9	8081
4,4'-DDT	50-29-3	8081
Dieldrin	60-57-1	8081
Endosulfan I	959-98-8	8081
Endosulfan II	33213-65-9	8081
Endosulfan sulfate	1031-07-8	8081
Endrin	72-20-8	8081
Endrin aldehyde	7421-93-4	8081

TCL/TAL Constituents	CAS Registry Number	RCRA Method
Endrin ketone	53494-70-5	8081
Heptachlor	76-44-8	8081
Heptachlor epoxide	1024-57-3	8081
Methoxychlor	72-43-5	8081
Toxaphene	8001-35-2	8081
Polychlorinated biphenyls; PCBs; Aroclor 1016, 1221, 1232, 1242, 1248, 1254, 1260, 1262, 1268	12674-11-2, 11104-28-2, 11141-16-5, 53469-21-9, 12672-29-6, 11097-69-1, 11096-82-5, 37324-23-5, 11100-14-4	8082
METALS		
Aluminum	7429-90-5	6010
Antimony	7440-36-0	6010
Arsenic	7440-38-2	6010
Barium	7440-39-3	6010
Beryllium	7440-41-7	6010
Cadmium	7440-43-9	6010
Calcium	7440-70-2	6010
Chromium	7440-47-3	6010
Cobalt	7440-48-4	6010
Copper	7440-50-8	6010
Iron	7439-89-6	6010
Lead	7439-92-1	6010
Magnesium	7439-95-4	6010
Manganese	7436-96-5	6010
Nickel	7440-02-0	6010
Potassium	7440-09-7	6010
Selenium	7782-49-2	6010
Silver	7440-22-4	6010
Sodium	7440-23-5	6010
Thallium	7440-28-0	6010
Vanadium	7440-62-2	6010
Zinc	7440-66-6	6010
Mercury	7439-97-6	7470/7471
MISCELLANEOUS		
Cyanide	57-12-5	9012

Appendix 9. Federal Priority Pollutant List

PPL Constituents	CAS Registry Number	CWA Method
VOLATILES		
Acrolein	107-02-8	624
Acrylonitrile	107-13-1	624
Benzene	71-43-2	624
Bromodichloromethane; Dichlorobromomethane	75-27-4	624
Bromoform; Tribromomethane	75-25-5	624
Carbon tetrachloride	56-23-5	624
Chlorobenzene	108-90-7	624
Chloroethane; Ethyl chloride	75-00-3	624
2-Chloroethyl vinyl ether	110-75-8	624
Chloroform; Trichloromethane	67-66-3	624
Dibromochloromethane; Chlorodibromomethane	124-48-1	624
o-Dichlorobenzene; 1,2-Dichlorobenzene	95-50-1	624
m-Dichlorobenzene; 1,3-Dichlorobenzene	541-73-1	624
p-Dichlorobenzene; 1,4-Dichlorobenzene	106-46-7	624
1,1-Dichloroethane; Ethylidene chloride	75-34-3	624
1,2-Dichloroethane; Ethylene dichloride	107-06-2	624
1,1-Dichloroethylene; Vinylidene chloride; 1,1-Dichloroethene	75-35-4	624
trans-1,2-Dichloroethylene; trans-1,2-Dichloroethene	156-60-5	624
1,2-Dichloropropane; Propylene dichloride	78-87-5	624
cis-1,3-Dichloropropene	10061-01-5	624
trans-1,3-Dichloropropene	10061-02-6	624
Ethylbenzene	100-41-4	624
Methyl bromide; Bromomethane	74-83-9	624
Methyl chloride; Chloromethane	74-87-3	624
Methylene chloride; Dichloromethane	75-09-2	624
1,1,2,2-Tetrachloroethane	79-34-5	624
Tetrachloroethylene; Tetrachloroethene; Perchloroethylene	127-18-4	624
Toluene	108-88-3	624
1,1,1-Trichloroethane; Methyl chloroform	71-55-6	624
1,1,2-Trichloroethane	79-00-5	624
Trichloroethylene; Trichloroethene	79-01-6	624
Vinyl chloride; Chloroethene	75-01-4	624
SEMIVOLATILES		
Acenaphthene	83-32-9	625
Acenaphthylene	208-96-8	625
Anthracene	120-12-7	625
Benzidine	92-87-5	625
Benzo(a)anthracene; Benzathracene	56-55-3	625
Benzo(a)pyrene	50-32-8	625
Benzo(b)fluoranthene	205-99-2	625
Benzo(k)fluoranthene	207-08-9	625
Benzo(ghi)perylene	191-24-2	625
Bis(2-chloroethoxy)methane	111-91-1	625
Bis(2-chloroethyl)ether; Dichloroethyl ether	111-44-4	625
Bis(2-chloro-1-methylethyl) ether; 2,2-Dichlorodiisopropyl ether; DCIP; 2,2'-Oxybis(1-chloropropane)	108-60-1	625

PPL Constituents	CAS Registry Number	CWA Method
Bis(2-ethylhexyl) phthalate	117-81-7	625
4-Bromophenyl phenyl ether	101-55-3	625
Butyl benzyl phthalate	85-68-7	625
p-Chloro-m-cresol; 4-Chloro-3-methylphenol	59-50-7	625
2-Chloronaphthalene	91-58-7	625
2-Chlorophenol	95-57-8	625
4-Chlorophenyl phenyl ether	7005-72-3	625
Chrysene	218-01-9	625
Dibenz(a,h)anthracene	53-70-3	625
Di-n-butyl phthalate	84-74-2	625
3,3'-Dichlorobenzidine	91-94-1	625
2,4-Dichlorophenol	120-83-2	625
Diethyl phthalate	84-66-2	625
2,4-Dimethylphenol	105-67-9	625
Dimethyl phthalate	131-11-3	625
4,6-Dinitro-o-cresol; 4,6-Dinitro-2-methylphenol	534-52-1	625
2,4-Dinitrophenol	51-28-5	625
2,4-Dinitrotoluene	121-4-2	625
2,6-Dinitrotoluene	606-20-2	625
Di-n-octyl phthalate	117-84-0	625
1,2-Diphenylhydrazine	122-66-7	625
Fluoranthene	206-44-0	625
Fluorene	86-73-7	625
Hexachlorobenzene	118-74-1	625
Hexachlorobutadiene	87-68-3	625
Hexachlorocyclopentadiene	77-47-4	625
Hexachloroethane	67-72-1	625
Indeno(1,2,3-cd)pyrene	193-39-5	625
Isophorone	78-59-1	625
Naphthalene	91-20-3	625
Nitrobenzene	98-95-3	625
o-Nitrophenol; 2-Nitrophenol	88-75-5	625
p-Nitrophenol; 4-Nitrophenol	100-02-7	625
N-nitrosodimethylamine	62-75-9	625
N-nitrosodiphenylamine	86-30-6	625
N-nitrosodipropylamine; Di-n-propyl-nitrosamine; N-nitroso-n-dipropylamine	621-64-7	625
Pentachlorophenol	87-86-5	625
Phenanthrene	85-01-8	625
Phenol	108-95-2	625
Pyrene	129-00-0	625
1,2,4-Trichlorobenzene	120-82-1	625
2,4,6-Trichlorophenol	88-06-2	625
ORGANOCHLORINE PESTICIDES/PCBs		
Aldrin	309-00-2	608
alpha-BHC; alpha-Benzene hexachloride	319-84-6	608
beta-BHC; beta-Benzene hexachloride	309-85-7	608
delta-BHC;delta-Benzene hexachloride	319-86-8	608
gamma-BHC; gamma-Benzene hexachloride; Lindane	58-89-9	608

PPL Constituents	CAS Registry Number	CWA Method
alpha-Chlordane	5103-71-9	608
gamma-Chlordane	5103-74-2	608
4,4'-DDD	72-54-8	608
4,4'-DDE	72-55-9	608
4,4'-DDT	50-29-3	608
Dieldrin	60-57-1	608
Endosulfan I	959-98-8	608
Endosulfan II	33213-65-9	608
Endosulfan sulfate	1031-07-8	608
Endrin	72-20-8	608
Endrin aldehyde	7421-93-4	608
Endrin ketone	53494-70-5	608
Heptachlor	76-44-8	608
Heptachlor epoxide	1024-57-3	608
Methoxychlor	72-43-5	608
Toxaphene	8001-35-2	608
Polychlorinated biphenyls; PCBs; Aroclor 1016, 1221, 1232, 1242, 1248, 1254, 1260	12674-11-2, 11104-28-2, 11141-16-5, 53469-21-9, 12672-29-6, 11097-69-1, 11096-82-5	608
METALS		
Antimony	7440-36-0	200.7
Arsenic	7440-38-2	200.7
Beryllium	7440-41-7	200.7
Cadmium	7440-43-9	200.7
Chromium	7440-47-3	200.7
Copper	7440-50-8	200.7
Lead	7439-92-1	200.7
Nickel	7440-02-0	200.7
Selenium	7782-49-2	200.7
Silver	7440-22-4	200.7
Thallium	7440-28-0	200.7
Zinc	7440-66-6	200.7
Mercury	7439-97-6	245.1
DIOXIN		
2,3,7,8-Tetrachlorodibenzo-p-dioxin (TCDD)	1746-01-6	8280/ 8290
MISCELLANEOUS		
Asbestos		
Cyanide	57-12-5	SM4500-CN C, E
Phenols	NA	420.1& 420.4

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