

Part II

Quality Assurance Project Plan

FINAL

**Quality Assurance Project Plan
Non-Time-Critical Removal Action
at the
Municipality of Culebra, PR**

US Army Engineering & Support Center, Huntsville
Contract # W912DY-05-D-0007, TO #0001

**Independent Technical Review
Certification**

**Responsibility
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Abbreviations & Acronyms

%R	percent recovery
°C	degrees Celsius
AA	atomic absorption
ADR	Analytical Data Review
AES	atomic emission spectroscopy
BS	Bachelor of Science
CB	calibration blank
CCB	continuing calibration blank
CCV	continuing calibration verification
CEHNC	United States Army Engineering and Support Center, Huntsville
CFR	Code of Federal Regulations
CVAA	cold-vapor atomic absorption
DoD	Department of Defense
DQO	data quality objectives
EDD	electronic data deliverable
EEG	Ellis Environmental Group, LC
EM	Engineer Manual
FSP	Field Sampling Plan
HMX	octahydro-1,3,5-tetranitro-1,3,5,7-tetrazocine
HPLC	high-performance liquid chromatography
HTRW	hazardous, toxic, and radioactive waste
IC	ion chromatography
ICB	initial calibration blank
ICAP	inductively coupled argon plasma
ICV	initial calibration verification
ID	identification
LCS	laboratory control sample
LIMS	Laboratory Information Management System
LQM	laboratory quality manual
MB	method blank
MEC	munitions and explosives of concern
MD	matrix duplicate
MDL	method detection limit
mL	milliliter
MS	matrix spike
MSA	Method of Standard Additions
MSD	matrix spike duplicate
NG	nitroglycerin

NIST	National Institute of Standards and Technology
nm	nanometer
PE	professional engineer
PETN	pentaerythritol tetranitrate
PG	professional geologist
ppb	parts per billion
PQL	practical quantitation limit
QA	quality assurance
QAPP	Quality Assurance Project Plan
QC	quality control
RCRA	Resource Conservation and Recovery Act
RPD	relative percent difference
SDR	Sample Discrepancy Report
SAP	Sampling and Analysis Plan
SOP	standard operating procedure
STL	Severn Trent Laboratories
USACE	United States Army Corps of Engineers
USEPA	United States Environmental Protection Agency
VOC	volatile organic compound

1.0 Introduction

1.0.01 Ellis Environmental Group, LC (EEG) has been contracted by the United States Army Engineering and Support Center, Huntsville (CEHNC) to conduct munitions removal and other munitions-related services on Culebra Island, Puerto Rico. This project includes the removal of munitions, and soil sampling and analyses for explosive compounds, metals, and perchlorate. This project is being conducted under Contract Number W912DY-05-D-0007.

1.0.02 The purpose of this Quality Assurance Project Plan (QAPP) is to define the laboratory requirements for the munitions and explosives of concern (MEC) removal action at Culebra Island and its surrounding cays, and it strives to be consistent with the EEG Field Sampling Plan (FSP) and the following referenced analytical methods:

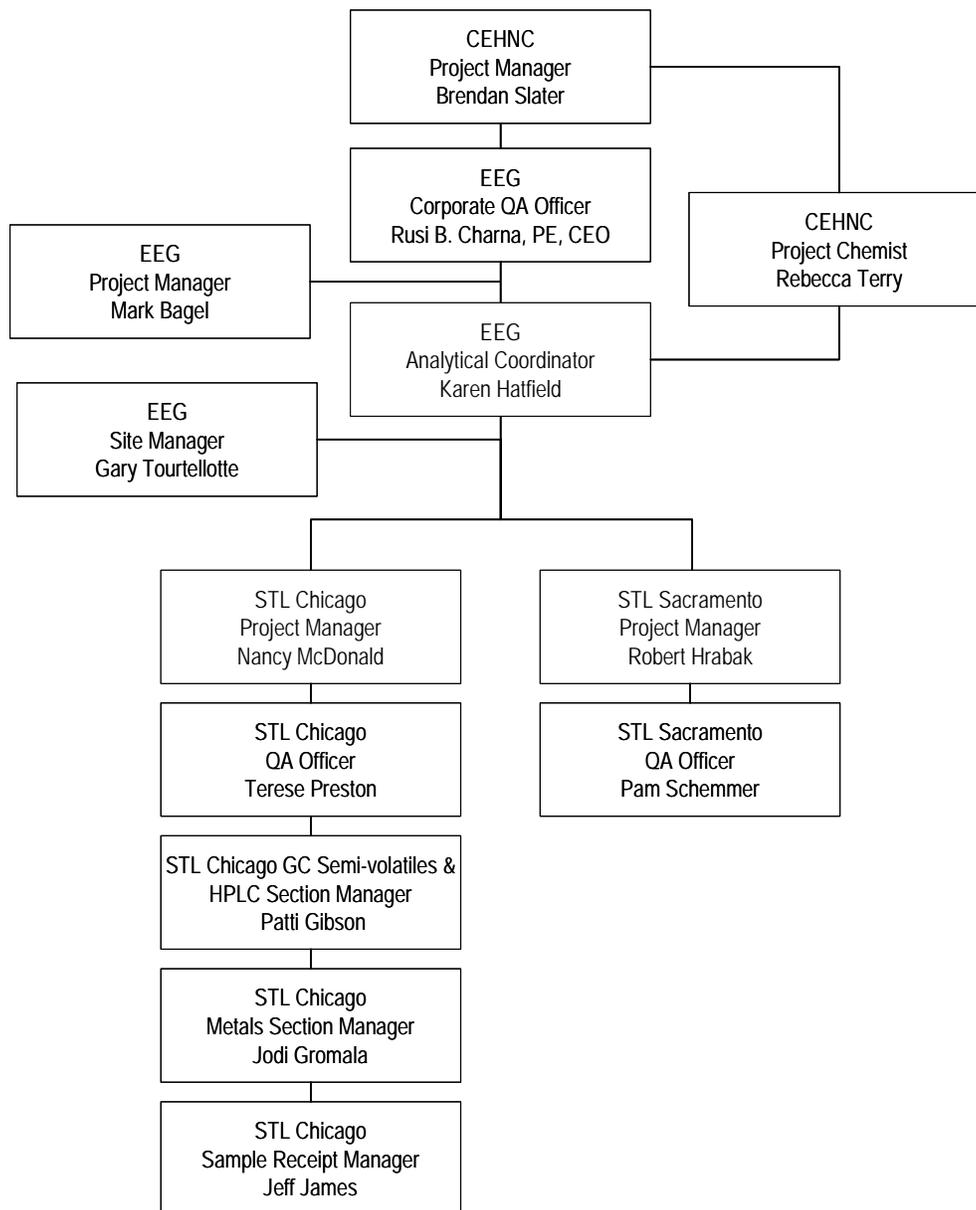
- 40 Code of Federal Regulations (CFR) Part 40, Appendix B
- Department of Defense (DoD) Quality Systems Manual for Environmental Laboratories, Version 1, October 2000
- Test Methods for Evaluation of Solid Waste (SW-846) Update IIIA, Office of Solid Waste and Emergency Response, United States Environmental Protection Agency (USEPA), 1999
- USEPA Region 5, Resource Conservation and Recovery Act (RCRA) QAPP, April 1998
- USEPA QA/R-5, EPA Requirements for Quality Assurance Plans, March 2001
- United States Army Corps of Engineers (USACE) Shell – Part of Engineering and Design-Requirements for the Preparation of Sampling and Analysis Plans (EM [Engineer Manual] 200-1-3), February 2001
- Chemical Quality Assurance for Hazardous, Toxic, and Radioactive Waste (HTRW) Projects, EM 200-1-6, October 1997
- USACE Chemical Quality Management Procedures and Notifications (see Attachment A)

1.0.03 Adherence to the procedures described in this QAPP should ensure data that are scientifically sound, valid, defensible, and of known, acceptable, and documented quality.

2.0 Project Laboratory Organization and Responsibilities

This chapter gives an overview of the quality assurance (QA) organization for this project and the lines of communication among key personnel. Severn-Trent Laboratory (STL) Chicago is the project laboratory, with STL Sacramento to provide analysis for perchlorate. The organization chart in **Figure 2-1** shows key personnel, and the subchapters following provide brief descriptions of their responsibilities. Qualifications for key personnel are in Attachment B. Laboratory certifications held by STL are included in Attachment C.

Figure 2-1. Chemical Quality Organization Chart



2.1 Project Organization and Responsibilities

2.1.1 EEG Project Quality Assurance Officer

Rusi B. Charna, professional engineer (PE) and EEG's CEO, is an experienced chemical engineer with over 30 years in the environmental field. He holds a Bachelor of Science (BS) degree in chemical engineering and a BS degree in chemistry. He will be ultimately responsible for the chemical QA system for this project. He will perform appropriate checks on the chemical quality organization to ensure that the QA system is being implemented properly.

2.1.2 EEG Project Manager

Mark Bagel, professional geologist (PG), is the EEG project manager. He is ultimately responsible for the successful and timely completion of the project and is also responsible for:

- Allocating and directing resources
- Assigning technical staff
- Ensuring the completion of all quality control (QC) requirements by team members
- Supervising the document control process
- Approving all deliverables and associated documents prior to transmittal
- Establishing and maintaining communication between the technical staff, project managers, QA officer, health and safety coordinator, and regulatory agencies
- Implementing all programs and protocols related to the project

2.1.3 EEG Analytical Coordinator / Chemistry Quality Assurance / Project Chemist

Karen Hatfield is EEG's analytical coordinator. Her responsibilities include:

- Ensuring that the laboratory implements the requirements of the project Work Plan
- Coordinating with the laboratory on QA/QC matters
- Coordinating the review of laboratory data
- Coordinating data validation activities
- Providing updates to the project manager with regard to laboratory performance
- Documenting changes to this QAPP

2.1.4 STL Chicago Project Manager

Nancy McDonald is the STL Chicago project manager. She will serve as the primary contact person for EEG, and she will ensure that the laboratory meets the project requirements. She will coordinate sampling schedules between the laboratory and EEG, proactively communicate with

EEG, ensure laboratory adherence to contract and QAPP requirements, monitor the progress and timeliness of the work, review work orders and laboratory reports, and process any changes in the QAPP. Along with the laboratory QA officer, she will ensure that project-specific corrective action is taken to address problems identified by audits or QC results.

2.1.5 STL Chicago Quality Assurance Officer

Terese Preston is the STL Chicago project QA officer. She is responsible for the development and administration of this QAPP. This role includes preparation and review of written documents defining QC procedures, review and approval of laboratory QC procedures, and development and implementation of corrective actions.

2.1.6 STL Sacramento Project Manager

Robert Hrabak is the STL Sacramento project manager. He will ensure that the laboratory meets the project requirements for perchlorate analysis. He will also coordinate sampling schedules with the STL Chicago project manager, ensure laboratory adherence to contract and QAPP requirements, monitor the progress and timeliness of the work, review work orders and laboratory reports, and process any changes in the QAPP. Along with the laboratory QA officer, he will ensure that project-specific corrective action is taken to address problems identified by audits or QC results.

2.1.7 STL Sacramento Quality Assurance Officer

Pam Schemmer is the STL Sacramento project QA officer. She is responsible for the development and administration of this QAPP for perchlorate analysis. This role includes preparation and review of written documents defining QC procedures, review and approval of laboratory QC procedures, and development and implementation of corrective actions.

2.2 Key Personnel

Contact information for key personnel is provided in the **Table 2-1**.

Table 2-1. Points of Contact

Function	Name	Location	Telephone
Prime Contractor – EEG			
Project Manager	Mark Bagel, PG	414 SW 140th Terrace Newberry, FL 32669	(352) 332-3888
Corporate QA Officer	Rusi B. Charna, PE		
Site Manager	Gary Tourtellotte		
Project Chemist / Analytical Coordinator / Chemistry QA	Karen Hatfield		
Analytical Laboratory – STL Chicago			
Project Manager	Nancy McDonald	2417 Bond St. University Park, IL 60466-3182	(708) 534-5200
Project Manager – Backup	Eric Lang		
Project QA Officer	Terese Preston		
GC Semi-volatiles & HPLC Section Manager	Patti Gibson		
Metals Section Manager	Jodi Gromala		
Sample Receipt Manager	Jeff James		
Analytical Laboratory – STL Sacramento			
Project Manager	Robert Hrabak	880 Riverside Parkway West Sacramento, CA 95605	(916) 373-5600
Project QA Manager	Pam Schemmer		
Regulatory Agency			
Puerto Rico Department of Environmental Quality	Yarissa Martinez, Project Manager	PO Box 11488 San Juan, PR 00910-1498	(787) 767-8056
CEHNC Personnel			
Project Manager	Brendan Slater	US Army Engineering & Support Center Attn: CEHNC-CT-E 4820 University Square Huntsville, AL 35807	(256) 895-1507
Project Chemist	Rebecca Terry		(256) 895-1460
Environmental Chemistry Branch Laboratory			
QA Samples	Laura Percefield	420 S. 18th St. Omaha, NE 68102	(402) 444-4302

3.0 Data Assessment Organization and Responsibilities

The data assessment organization is shown on **Figure 2-1**. Data assessment is based on the review of the data quality indicators, laboratory operations documentation, and data validation. These activities will be performed by the laboratory project managers, the laboratory QA officers, the EEG analytical coordinator, and the CEHNC project chemist. Data assessment is discussed in detail in Chapters 8 and 9.

4.0 Data Quality Objectives

4.1 Data Use Background

The data from this project will determine if any residues from destruction of MEC have contaminated the surrounding soil.

4.2 Measurement Quality Objectives

Data quality objectives (DQOs) will be based on the analytical reporting limits, precision, accuracy, and completeness discussed in Chapter 7.

5.0 Sample Receipt, Handling, Custody and Holding Time Requirements

5.1 Verification / Documentation of Cooler Receipt Condition

5.1.01 Sample custody starts in the field as the samples are taken, but sample container custody records, in preparation of sampling, start at the bottle manufacturer and the laboratory. The integrity of the sample containers depends on the proper cleaning, preparation, storage, shipment, and documentation by the bottle manufacturer. The laboratory documentation of custody starts when cleaned and preservative-prepared sample containers are shipped to the field under custody. Successful sample custody is initiated by field personnel using traceable containers and relies on the fastidious completion of field custody protocols.

5.1.02 EEG will transfer the samples under chain of custody from Puerto Rico to STL Chicago. A copy of the chain of custody form will be kept by EEG. The sample shipping manifests will include a copy of the United States Department of Agriculture soil permit (see Attachment D) along with the manifest stating that these are soil samples for analytic purposes.

5.1.03 The sample custodian receives samples shipped or delivered to STL. The sample custodian inspects the shipping container and samples for integrity and custody seals. The samples are checked for breakage, leakage, damage, and preservatives. The Job Sample Receipt Checklist Report (Attachment E) is used to check sample receipt condition, including temperature. A temperature blank is used to measure the temperature inside the cooler. The contents of the shipping container are verified against the chain of custody documentation. The chain of custody form is in Attachment E. Documentation of custody seal integrity, temperature, and sample preservations are made on the Job Sample Receipt Checklist Report. Any problems are documented on the chain of custody or in a sample control communication form. The STL project manager will either resolve the problem internally or contact EEG's analytical coordinator for resolution.

5.1.04 If the samples and documentation are acceptable, each sample is assigned a unique laboratory identification (ID) number from STL's Laboratory Information Management System (LIMS). When the LIMS log has been completed, the samples are transferred to the appropriate refrigerators. Separate refrigerators are used for samples suspected to contain high levels of organic compounds and for samples receiving analysis for volatile compounds. The sample

refrigerators are kept at 4 ± 2 degrees Celsius ($^{\circ}\text{C}$). The refrigerators storing samples for volatile analysis are monitored for contamination with refrigerator blanks.

5.1.05 Sample distribution is controlled and described in facility-specific standard operating procedures (SOPs). Thirty days after a final laboratory report has been generated and mailed to EEG, the samples are transferred from cold storage to the sample disposal area.

5.1.06 Holding times for samples are identified in the FSP.

5.2 Corrective Action for Incoming Samples

5.2.01 QC elements are used to monitor and assess the validity of sampling and analysis activities. Formal corrective actions (see Attachment F, SOP UQA-029) will be initiated in each subcontracted laboratory if data are determined to be of questionable validity, if QC elements are not within required limits, or if a performance trend develops. For routine problems, the analysts correct the problem and document such activity in the analytical run log or worksheet, and a formal corrective action report is not required.

5.2.02 Within each subcontracted laboratory, any employee aware of a problem related to one or more samples is responsible for initiating a Sample Discrepancy Report (SDR) (see Attachment F).

5.2.1 Internal Corrective Actions

Examples of QC elements generally monitored by each subcontractor laboratory are listed in Chapter 7 and Chapter 8. Other method-specific QC elements are also monitored during routine operations. See Attachment F for the corrective actions for each method.

5.2.2 External Corrective Actions

Any actions deemed necessary by EEG, CEHNC, or any other external regulatory or certifying agencies will be taken by the affected subcontractor laboratory as necessary. These actions are most likely to arise from a systems or performance audit, or from data review conducted by the agency.

5.2.3 Documentation

5.2.3.01 An SDR will be used by the subcontractor laboratories to document deficiencies and exceptions that may impact data quality, production, efficiency, or relations with STL or USACE.

To the extent possible, the laboratories will resolve all situations that require corrective action before data quality is compromised. These non-conformance actions do not require documentation in a formal SDR.

5.2.3.02 The following standards apply to corrective actions.

- The Job Sample Receipt Checklist Report (Attachment E) is a form of corrective action report. It documents problems encountered during sample receipt.
- If a critical problem requires immediate action in consultation with EEG (e.g., samples received after holding time expired, holding time missed during the analytical process, insufficient sample volume), the laboratory will notify EEG's analytical coordinator immediately and the corrective action designed in consultation with the USACE.
- If the laboratory reports data whose QC elements are not within criteria, the exceptions are noted in the case narrative.
- If the laboratory discovers any problems after the report has been sent to the client (e.g., after system or data audit, client inquiries, external review), a formal SDR will be initiated.

5.2.4 Sample Discrepancy Report Responsibility

5.2.4.01 SDRs are the responsibility of the laboratory staff. Any laboratory employee who becomes aware of a problem with any aspect of reported data is responsible for initiating an SDR. In most cases, this will be primarily the analyst's responsibility, but any reviewer or person in contact with the client that becomes aware of a problem must initiate a formal corrective action report.

5.2.4.02 The laboratory's project manager or point of contact as shown in Chapter 2 is responsible for reporting to the EEG analytical laboratory coordinator and to EEG's project chemist all corrective actions taken by the laboratory for this project. The laboratory project manager is responsible for ensuring that the action is implemented and documented in the case narrative. EEG's project chemist is responsible for reporting the action to EEG's project manager and to the USACE project chemist.

5.2.5 Sample Discrepancy Report Approval

SDRs are approved by the originating laboratory's project manager and QA officer, and completed SDRs are filed in the project file(s).

6.0 Analytical Procedures

6.1 Explosives Preparation and Analysis

6.1.1 Method 8330 – Explosives Residues by HPLC

6.1.1.01 Method 8330 provides high-performance liquid chromatography (HPLC) conditions for the detection of parts per billion (ppb) levels of certain explosives residues in water, soil, and sediment matrix. Prior to use of this method, appropriate sample preparation techniques must be used. These techniques are as follows.

6.1.1.02 Sample Homogenization: Soil samples are dried in air at room temperature or colder to a constant weight, taking care not to expose the samples to direct sunlight. The dried samples are ground and homogenized thoroughly in an acetonitrile-rinsed mortar to pass a 30-mesh sieve.

6.1.1.03 Explosives will be analyzed on the primary column and all positive results will be confirmed on the secondary column.

6.1.1.04 The laboratory will follow the SOP in Attachment F, which includes surrogates.

6.1.1.05 Low-Level Salting-Out Method With No Evaporation: Aqueous samples of low concentration are extracted by a salting-out extraction procedure with acetonitrile and sodium chloride. The small volume of acetonitrile that remains undissolved above the salt water is drawn off and transferred to a smaller volumetric flask. It is back-extracted by vigorous stirring with a specific volume of salt water. After equilibration, the phases are allowed to separate and the small volume of acetonitrile, residing in the narrow neck of the volumetric flask, is removed using a Pasteur pipette. The concentrated extract is diluted 1:1 with reagent grade water. An aliquot is separated on a C-18 reverse phase column, determined at 265 nanometers (nm), and confirmed on a CN reverse-phase column.

6.1.1.06 High-Level Direct-Injection Method: Aqueous samples of higher concentration can be diluted 1:1 volume:volume (v:v) with methanol or acetonitrile, filtered, separated on a C-18 reverse-phase column, determined at 254 nm, and confirmed on a CN reverse-phase column. If octahydro-1,3,5-tetranitro-1,3,5,7-tetrazonine (HMX) is a target analyte, methanol is preferred.

6.1.1.07 Soil and sediment samples are extracted using acetonitrile in an ultrasonic bath, filtered, and analyzed by either the low level salting-out method or the high-level direct-injection method.

6.1.2 Method 8330M – NG and PETN by HPLC

Nitroglycerin (NG) and pentaerythritol tetranitrate (PETN) are analyzed as described in Method 8330, except the wavelength is 210 nm.

6.2 Metals Preparation Procedures and Analysis

Two techniques—inductively coupled argon plasma (ICAP) atomic emission spectroscopy (AES) and atomic absorption (AA) spectroscopy—will be employed to measure levels of specified metals in the samples. Sample digestion is required prior to most ICAP and AA analyses.

6.2.1 Method 3050A — Acid Digestion of Sediments, Sludges, and Solids

This digestion method is used to prepare sediment and soil samples for analysis by ICAP. A portion of the sample is digested with nitric acid. A final reflux procedure is performed using concentrated hydrochloric acid or concentrated nitric acid based on the SW 6010B method for ICAP. The final volume is adjusted to 50 milliliters (mL).

6.2.2 Method 6010B — Inductively Coupled Argon Plasma Procedures

6.2.2.01 Method 6010B is a procedure for determining elements in solutions using ICAP AES samples. Soils require digestion by Method 3050A prior to analysis.

6.2.2.02 Method 6010B provides a simultaneous multi-element determination by ICAP. Elements for this project are arsenic, barium, cadmium, chromium, lead, selenium, silver, aluminum, antimony, beryllium, calcium, cobalt, copper, iron, magnesium, manganese, nickel, potassium, sodium, thallium, vanadium, zinc, and strontium. Samples are nebulized, and the resulting aerosol is transported to the plasma. Radio frequency ICAP produces element-specific atomic line emission spectra. The spectra are dispersed and the lines monitored by photo-multiplier tubes. The background will be measured and the results corrected for background levels.

6.2.3 Method 7471A — Mercury Procedure by Cold-Vapor Atomic Absorption

Method 7471A is the procedure for determining mercury in soil samples. Method 7471A is done by cold-vapor atomic absorption (CVAA) procedures for determining the concentration of mercury. Sample preparation is specified in the method. Following dissolution, mercury in the sample is reduced to the elemental state, separated from solution, and passed through a cell positioned in the light path of an AA spectrometer or mercury-specific analyzer.

6.2.4 Method EPA 314.0 (modified) — Perchlorate

6.2.4.01 A portion of homogenized sample is leached with deionized water for one hour, centrifuged, and filtered. A 1.0 mL volume of sample is introduced into an ion chromatograph. Perchlorate is separated and measured using a system comprised of an ion chromatograph pump, sample injection valve, guard column, analytical column, suppressor device, and conductivity detector.

6.2.4.02 The complete SOPs for the above methods are found in Attachment F.

6.3 Analytical Detection Limits

6.3.01 Various terms are used to express detection and reporting limits in environmental chemistry. The terms used for the work performed under this QAPP will be “method detection limit” (MDL) and “reporting limit.”

6.3.02 The MDL is an empirically derived value used to estimate the lowest concentration that a method can detect in a matrix-free environment. SW-846 defines the MDL as the minimum concentration of a substance that can be measured and reported with 99 percent confidence level and where the analyte concentration is greater than zero. The MDL is determined from the analysis of replicate samples of a given matrix containing analytes that have been processed through the preparation or extraction procedure. The guidance in 40 CFR 136, Appendix B, with additional laboratory-specific requirements, is used to produce MDL and is then annually updated by the laboratory. The latest values for the MDL are presented in Attachment G, and they may be updated during the course of the project as required by methods and regulatory agencies.

6.3.03 The reporting limit is a uniform reporting limit based on method practical quantitation limits (PQLs), actual performance at STL Chicago and STL Sacramento laboratories, and expected method performance in routine water and soil samples. The PQL is the lowest concentration that a method can reliably achieve within limits of precision and accuracy. Although the reporting limit is primarily based on the PQL, the reporting limit also evaluates empirical data for soil and water methods. The SW-846 PQLs often extrapolate soil PQL from water PQL, and they are not strictly based on the determinant method. Reporting limits are highly matrix-dependent. The latest values for the reporting limit are found in Attachment G.

6.4 Preventive Maintenance

6.4.01 The laboratory is equipped with sophisticated instrumentation needed to ensure successful completion of this project. A preventive maintenance schedule is in place in each laboratory to minimize instrument downtime and to obtain reliable data over the life of the instrument. Analysts and supervisors are primarily responsible for routine maintenance and repair of the instruments. Service agreements are kept for some major instruments in the each laboratory. Major repairs that go beyond the expertise of the analysts and supervisors are contracted to external specialists.

6.4.02 The preventive maintenance schedules are based primarily on manufacturer guidance, recommendations in the literature, and the experience of the analysts and supervisors. Some of the items will be performed as an integral part of each procedure (e.g., changing the injection port septum in gas chromatographs). Others will be followed as closely as possible, balancing to the extent possible the workload and the urgency of the need for preventive maintenance (e.g., clean and realign torch on ICAPs). Common sense and familiarity with the performance of each instrument will dictate whether the preventive maintenance schedule needs to be accelerated or delayed for that instrument. Trends and excursions from accepted limits for QC sample results are monitored to determine if there is instrument malfunction, and in such cases preventive maintenance is provided on an as-needed basis.

6.4.1 Routine Maintenance Activities

Preventive maintenance schedules for explosives and metals equipment are listed in **Table 6-1**.

Table 6-1. Preventive Maintenance for Laboratory Instruments

Instrument	Activity	Frequency
High-pressure liquid chromatograph	Check solvents in reservoirs	Daily
	Check gas supply	Daily
	Flush system with solvent to remove bubbles	Daily
	Pre-filter all samples	Daily
	Change pump seals when flow becomes inconsistent	As needed
	Change guard column	As needed
	Backflush column	As needed

Instrument	Activity	Frequency
Inductively coupled plasma atomic emission spectrometer	Check aspiration tubing	Daily
	Clean torch assembly	Monthly
	Clean spray chamber	Monthly
	Check gases	Daily
	Clean, lubricate pump rollers	As needed
	Check O-rings	Monthly
Cold-vapor atomic absorption spectrometerr	Check tubing	Daily
	Clean sparger	After each sample
	Clean windows	Monthly
	Change source lamp	As needed
Ion chromatograph	Inspect all itelium connections	Before each run
	Calibrate conductivity meter	Before each run
	Prime pump	Before each run

6.4.2 Contingency Plan

6.4.2.01 The laboratory has several pieces of analytical equipment in duplicate. This redundancy allows the laboratory to keep performing critical analyses on one instrument should the other be out of service.

6.4.2.02 In the event of instrument failure, or if critical holding times are approaching on a number of samples, these samples may be diverted to another laboratory, provided that they are properly certified for the project. This will be done in consultation with the USACE project chemist. When shipping samples to another laboratory, chain-of-custody procedures are maintained as described in Chapter 5.

6.4.2.03 As a further precaution, the laboratory keeps its major instrumentation connected to an uninterruptible power supply, which provides line conditioning and backup power.

6.4.3 Periodic Equipment Calibration

Balances are checked every day before the first use with a weight set traceable to Class S weights. Temperature in incubators, ovens, and refrigerators are monitored daily using thermometers that are calibrated against a National Institute of Standards and Technology (NIST)-traceable thermometer. All thermometers in use in the laboratory are verified for accuracy against an NIST-traceable thermometer at least every 12 months and when they are first placed into service. All

mechanical pipettes and other devices used to deliver accurate volumes during the analytical process are verified every 12 months. **Table 6-2** presents a summary of calibration requirements for equipment that is used periodically.

Table 6-2. Periodic Equipment Calibrations

Type of Equipment	Calibration Requirements
Balances	<p>Serviced and calibrated annually by an approved vendor.</p> <p>Calibration checked daily or before use by analyst with weight(s) classified as Class S by NIST or Class 1 traceable. Acceptance criteria vary according to weight used and accuracy of balance. Acceptance criteria are documented in the laboratory logbook.</p> <p>All Class 1 weights are certified by an outside vendor every 3 years.</p> <p>All non-Class 1 weights are checked annually against NIST Class 1 weights annually.</p> <p>Acceptance criteria is 1 percent for top-loading and 0.1 percent for analytical balances.</p>
Thermometers	<p>Working glass thermometers are calibrated against a certified NIST thermometer at least annually as described in operation-specific SOPs.</p> <p>Working non-glass thermometers are calibrated against a certified NIST thermometer at least annually as described in operation-specific SOPs.</p> <p>The NIST thermometer is re-certified every 3 years.</p> <p>Acceptance criteria is $\pm 2^{\circ}\text{C}$.</p>
Refrigerators / Freezers	<p>Thermometers are immersed in a liquid such as mineral oil or glycerol for calibration and placed in all refrigerators and freezers..</p> <p>Temperature of units used for sample or standard storage are checked daily as described in operation-specific SOPs. Refrigerator acceptance limits: $4\pm 2^{\circ}\text{C}$; freezer acceptance limits: $\pm 10^{\circ}\text{C}$.</p>
Ovens	<p>Temperature of units is checked daily or prior to use.</p> <p>Acceptance limits vary according to use as described in operation-specific SOPs and must be documented in the temperature logbook.</p> <p>Acceptance criteria is $\pm 2^{\circ}\text{C}$.</p>
Micropipettors	<p>Calibrations are checked gravimetrically as required by the operation-specific SOP.</p> <p>Calibrated at the frequency (normally quarterly) required by the manufacturer at a minimum.</p> <p>Acceptance criteria is ± 1 percent.</p>
Syringes, Volumetric Glassware, and Graduated Glassware	<p>Syringes and volumetric glassware are purchased as Class A.</p> <p>Class A items are certified by the manufacturer to be within ± 1 percent of the measured volume; therefore, calibration of these items by the laboratories is not required.</p> <p>Analysts are trained in the proper use and maintenance of measuring devices to ensure the measurement of standards, reagents and sample volumes are within method tolerances.</p> <p>The accuracy of Class A volumetric glassware will be checked when first received at a rate of one per lot.</p> <p>Acceptance criteria is ± 1 percent.</p>

6.5 Calibration Procedures and Frequency

6.5.01 This subchapter discusses general requirements for instrument calibration, standard preparation, and traceability.

6.5.02 Instrument calibration is necessary for accurate sample quantitation. Calibrations establish the dynamic range of an instrument and response factors to be used for quantitation, and they demonstrate instrument sensitivity. Accurate sample quantitation also relies on accurate standards. Standard accuracy may be established by tracing the quantitation standard to a source of known and documented quality or by the comparison of standards from different sources. Instrument calibrations and standards are unambiguously documented so that the process of calibration can be re-created.

6.5.1 Standards

6.5.1.01 The accuracy of sample target analytes quantitation is directly related to the accuracy of the standards used for instrument calibration. To ensure the highest quality standard, primary reference standards used by STL are obtained from the NIST or reliable commercial sources. When standards are received at the laboratory, the date received, supplier, lot number, purity and concentration, and expiration date are recorded in a standard logbook. Vendor certifications sent with the standards are also filed.

6.5.1.02 Standards purchased by STL may be in a pure form or in a stock or working standard solution. Often dilutions are made from vendor standards. All standards made are given a standard identification number and have the following information recorded in a standard logbook:

- Source of standard used to prepare dilution
- Preparer's initials
- Initial concentration
- Final concentration
- Solvent source and lot number of solvent
- Volume of final solution
- Volume of standard diluted

6.5.1.03 After preparation and before routine use, standards are validated. Validation procedures range from a check for chromatographic purity to verification of the concentration of the standard using a standard prepared at a different time or obtained from a different source. Reagents are also examined for purity by subjecting an aliquot or sub-sample to the analytical method in which it will be used. For example, every lot of dichloromethane (for organic extractable) is analyzed for undesirable contaminants prior to use in the laboratory. Standards are

routinely checked for signs of deterioration (e.g., discoloration, formation of precipitates, and changes in concentration), and they are discarded if deterioration is suspected or their expiration date has passed. Expiration dates may be taken from vendor recommendations, analytical methods, or internal research.

6.5.2 Explosives Method Calibration

6.5.2.01 The field of chromatography involves a variety of instrumentation and detection systems. While calibration requirements vary depending on the type of analytical system and methodology, the following principles of calibration generally apply.

- Calibration occurs before any sample quantitation.
- Initial five-point calibrations are performed periodically, which encompass the reporting limit.
- Daily standards (initial calibration verification [ICV] standards) are analyzed prior to sample analysis.
- Continuing calibration verification (CCV) standards are analyzed at a specific frequency throughout the sample analysis.

6.5.2.02 Sample quantitation is with an external calibration technique.

6.5.2.03 The laboratory will meet the requirements in Attachment H.

6.5.2.04 See Attachment F for STL Chicago's explosive calibration and corrective actions.

6.5.3 Metals Method Calibration

Twenty-three metals listed in Subchapter 6.2.2 will be analyzed by ICAP, and mercury will be analyzed by CVAA. Both techniques are discussed below. The laboratory will follow the SOPs in Attachment F, and calibration and corrective actions are described there.

6.5.3.1 Inductively Coupled Argon Plasma

6.5.3.1.01 Prior to any sample analyses, the ICAP is calibrated daily using criteria prescribed in the analytical method. The calibration is then verified using a standard from an independent source ICV. The working range of the instrument is established each quarter-year with a linear range verification check standard. Sample quantitation may not be performed outside the linear range.

6.5.3.1.02 An initial instrument calibration is established daily by analyzing a minimum of two standards, one of which is a calibration blank (CB). The calibration is monitored throughout the day by analyzing a continuing calibration blank (CCB) and a CCV after every 10 samples. The CCV is a standard at the mid-range of the calibration. If the verification standard and blank do not meet established criteria, an SDR must be completed. The SDR procedures include examination of instrument performance and analysis information, consultation with the group leader, and a decision path to determine if re-calibration and re-analysis of samples back to the previously acceptable calibration check is warranted.

6.5.3.1.03 An inter-element check standard is analyzed at the beginning and end (or after 8 hours) of each analytical run on the ICAP to verify that inter-element and background correction factors have remained constant. Results outside of the established criteria require re-analysis of samples.

6.5.3.2 Mercury Cold-Vapor Atomic Absorption

Each AA unit is calibrated prior to any analyses being conducted. A calibration curve is prepared with a minimum of a CB and three standards, and it is then verified with a standard that has been prepared from an independent source. The calibration is then verified on an ongoing basis with a CCB and a CCV. If the ongoing calibration standard and blank do not meet established acceptance criteria, the SDR form must be completed describing what action should be taken.

6.6 Laboratory Quality Control Procedures

6.6.1 Analytical Sequence Quality Control

6.6.1.1 Metals by ICAP and Mercury

1. Initial calibration (daily)
2. ICV (after initial calibration)
3. Initial calibration blank (ICB) (after initial calibration)
4. Inter-element check (beginning of analytical sequence)
5. CCB (every 10 samples and end of analytical sequence)
6. CCV (every 10 samples and end of analytical sequence)
7. Method blank (MB) (1 per sample batch)
8. Laboratory control sample (LCS) (1 per sample batch)
9. Matrix spike (MS) (1 per sample batch)
10. Matrix duplicate (MD) (1 per sample batch)

11. Post digestion spike (as needed)
12. Serial dilution (as needed)
13. Method of Standard Additions (MSA) (as needed with samples with matrix effects)

6.6.1.2 Explosives by HPLC

1. Initial calibration (daily)
2. ICV (after initial calibration)
3. CCV (every 10 samples and end of analytical sequence)
4. MB (1 per sample batch)
5. LCS (1 per sample batch)
6. MS/matrix spike duplicate (MSD) (1 per sample batch)
7. Surrogates (on each sample, standard, blank, and QC sample)
8. Confirmation (on all positive results)

6.6.1.3 Perchlorate by Ion Chromatography (IC)

1. Initial calibration (daily)
2. ICV (after initial calibration)
3. CCV (every 10 samples and end of analytical sequence)
4. MB (1 per sample batch)
5. LCS (1 per sample batch)
6. MS/MSD (1 per sample batch)

6.6.2 Batch / Matrix-Specific / Performance-Based Quality Control

Laboratory performance QC is required to ensure that the laboratory systems (instrumentation, sample preparation, analysis, data reduction, etc.) are operating within acceptable QC guidelines during data generation. Laboratory QC samples consist of MBs, instrument blanks, and LCSs. In addition to laboratory performance QC, matrix-specific QC is utilized to determine the effect of the sample matrix on the data being generated. Typically, this includes the use of MSs, MSDs, sample duplicates, and surrogate compounds.

6.6.2.1 Quality Control Project Batch

6.6.2.1.01 The QC batch consists of a set of up to 20 field samples from this project with the same matrix (e.g., aqueous, solid, waste) that are processed using the same procedures, reagents,

and standards within the same time period. The subcontractor laboratories for this project will utilize this definition of a QC batch.

6.6.2.1.02 In addition to the up to 20 non-QC samples, an analytical batch includes the following QC samples: MB, MS, MSD, and LCS.

6.6.2.2 Method Blanks

6.6.2.2.01 The MB is an Ottawa sand for solid samples and measures laboratory-introduced contamination for the batch. The MB is carried through every aspect of the procedure followed for samples, including preparation, cleanup, and analysis, and is analyzed with each QC batch processed.

6.6.2.2.02 The MB is used to identify any interferences or contamination of the analytical system that may lead to the reporting of elevated analyte concentrations or false positive data. Potential sources of contamination include solvent, reagents, glassware, other sample processing hardware, or the laboratory environment.

6.6.2.2.03 Typically, the requirements for MBs are that any analytes detected must be below half of the reporting limit. If there are any positive results for a MB (above or below the reporting limit), the data are evaluated to determine impacts and whether the associated sample results are adversely impacted.

6.6.2.2.04 It is a goal to have no detected target analytes in the MBs, but analytes may be periodically detected in blanks due to the nature of the analysis or the reporting limit of the analyte. For ICAP metals analyses, copper, zinc, and iron may sometimes be found in MBs. For these common laboratory contaminants, data may be reported with qualifiers if the concentration of the analyte is less than five times the MDL. Any laboratory contaminants found in the MB will be discussed in the report narrative.

6.6.2.2.05 Blank subtraction shall not be performed for this project.

6.6.2.3 Instrument Blank

6.6.2.3.01 The instrument blank is an unprocessed aliquot of reagent used to monitor the contamination of the analytical system at the instrument. Instrument blanks are typically analyzed on each day the instrument is used, and can be replaced by an MB.

6.6.2.3.02 System contamination may lead to the reporting of elevated analyte concentrations or false positive data. The instrument blank does not undergo the entire sample preparation process and generally consists of an aliquot of the same reagent(s) used for a sample dilution.

6.6.2.3.03 If an instrument blank shows any positive results, analysis is halted and corrective action implemented to remove the contamination. If the instrument blank was a part of an automatic run, the same criteria and evaluation process is used as for an MB.

6.6.2.4 Laboratory Control Samples

6.6.2.4.01 Ottawa sand fortified with known amounts of selected target analytes is used for the LCS for solid samples. The LCS is carried through every aspect of the procedure, including preparation, cleanup, and analysis of the samples. An LCS is prepared and analyzed with each QC project batch processed.

6.6.2.4.02 Review of the LCS recovery data is used to monitor the performance of the analytical methods. Day-to-day performance is characterized by evaluation of the accuracy of the results. Ongoing monitoring of the LCS results provides evidence that the laboratory is performing the method within both acceptable accuracy and precision guidelines.

6.6.2.4.03 The recoveries of spiked analytes, LCS, are compared to control limits generated from historical data. If any analyte is not within control limits, the data are evaluated to determine the severity of the impact on sample data quality. See Attachment H for the corrective action that will be taken when data exceed the control limits, and see Attachment F for the SDR form.

6.6.2.5 Matrix Spike

6.6.2.5.01 The MS is an environmental sample to which known concentrations of selected target analytes have been added. MSs are analyzed to evaluate the effect of the sample matrix on the analytical methodology. The MS undergoes the same extraction and analytical procedures as the unfortified client sample. An MS is prepared and analyzed for each 20 samples processed where appropriate.

6.6.2.5.02 Evaluation of MS recovery data is used to monitor the effects that the sample matrix may have had on the performance of the analytical method. Due to the potential variability of the matrix of each sample, these results may have immediate bearing only on the specific sample spiked and not on all samples in the QC batch.

6.6.2.6 Matrix Spike Duplicate

6.6.2.6.01 The MSD is a second aliquot of the same sample used for the MS that is spiked with known concentrations of selected target analytes. The MSD is analyzed with the associated sample and MS. The MSD undergoes the same extraction and analytical procedures as the unfortified client sample. An MSD is prepared and analyzed with each QC batch processed where appropriate.

6.6.2.6.02 The results of the MSD by itself are evaluated in the same manner as the MS. The results of the MS and MSD are compared to determine the effect of the matrix on the precision of the analytical process. Due to the potential variability of the matrix of each sample, the MS/MSD results may have immediate bearing only on the specific sample spiked and not on all samples in the QC batch.

6.6.2.6.03 The relative percent difference (RPD) between the duplicate MSs is compared to precision control limits. If any analyte is not within precision control limits, the failure is ascribed to matrix effects (usually sample non-homogeneity) and the data reported with narration.

6.6.2.7 Sample Duplicate

6.6.2.7.01 A sample duplicate is a second aliquot of an environmental sample taken from the same sample container. It is processed in the same manner and at the same time as the first aliquot of the sample. For most projects, a duplicate is prepared and analyzed only when MS/MSDs are not possible.

6.6.2.7.02 The results of the sample and its duplicate are compared to determine the effect of the matrix on the precision of the analytical process. Due to the potential variability of the matrix of each sample, the sample duplicate results may have immediate bearing only on the sample analyzed in duplicate.

6.6.2.7.03 The RPD between the duplicates is compared to in-house-generated precision control limits. If any analyte is not within precision control limits, the failure is ascribed to matrix effects (usually sample non-homogeneity) and the data reported with narration.

6.6.2.8 Surrogates

6.6.2.8.01 Surrogates will be used by STL Chicago for Method 8330 (explosives). Surrogates are organic compounds that are similar in chemical composition and behavior to the target

analytes but are not normally found in environmental samples. Known amounts of the surrogates are added to samples and QC samples being tested for organic analytes.

6.6.2.8.02 Review of surrogate data is used to monitor the effect of the sample matrix and the accuracy of the analysis.

6.6.2.8.03 The recoveries of spiked surrogates are compared to control limits generated from historical data. If any analyte is not within control limits, the data are evaluated to determine the severity of the impact on sample data quality.

6.6.2.8.04 Surrogates are analyzed only with organic analyses such as the analysis of explosives.

6.6.2.9 Interference Check Sample

6.6.2.9.01 An interference check sample is a solution containing known concentrations of both interfering and analyte elements. Analysis of this sample will be used to verify background and inter-element correction factors (metals analyses only).

6.6.2.9.02 A minimum of one set of interference check sample solutions will be analyzed at the beginning of each ICAP sequence. See the ICAP SOP in Attachment F for the specific criteria for the interference check sample.

6.7 Performance and System Audits

6.7.01 Internal and external audits are conducted regularly at the laboratory to ensure that the guidance provided in this document and in project-specific documents is followed. Internal audits are performed by each laboratory's QA department, which is responsible for all QA/QC functions in that laboratory, and/or members of the professional laboratory staff who do not normally work in the section being audited.

6.7.02 To provide an independent and unbiased review of laboratory operation, the laboratory participates in external audits conducted by persons who are not direct employees of the laboratory. Two types of audits are performed in each laboratory. Performance audits require the analysis of blind samples or other samples whose values are not known to the analytical areas. These results are used to evaluate the accuracy of the laboratory analytical system. Systems audits involve an in-depth review and evaluation of some or all components of the analytical laboratory

to determine the proper application of guidelines listed in this document and/or each laboratory's laboratory quality manual (LQM).

6.7.1 Internal Audits

6.7.1.01 The QA department at the laboratory conducts several audits (systems, data) during the course of each calendar year. During these audits, one or more components of the laboratory are reviewed to determine if that part is functioning in compliance with the requirements specified in the LQM, the approved SOP, and approved methodology. An audit report, listing deficiencies that must be addressed in order to correct or improve the laboratory operations, is prepared.

6.7.1.02 The laboratory performs an annual double blind performance evaluation study in which all systems to which a client is normally exposed are evaluated, including customer service and turnaround time. The analytical and subjective results of the study are distributed to the analytical department within the laboratory for corrective action when applicable.

6.7.2 External Audits

6.7.2.01 Each subcontractor laboratory undergoes systems audits as needed to satisfy certification or project requirements. These audits are conducted by the certifying agency or contractor with the full cooperation of the laboratory staff and management.

6.7.2.02 Each subcontractor laboratory also regularly participates in three semiannual performance testing studies: water supply, water pollution, and soil studies.

6.8 Non-Conformance and Corrective Actions

6.8.01 QC elements are used to monitor and assess the validity of sampling and analysis activities. Formal corrective actions (see Attachment F, SOP UQA-029) will be initiated in each subcontractor laboratory if (a) data are determined to be of questionable validity, or (b) if QC elements are not within required limits, or (c) if a performance trend develops. For routine problems, the analysts correct the problem and document such activity in the analytical run log or worksheet, and a formal corrective action report is not required.

6.8.02 Within each subcontractor laboratory, any employee aware of a problem related to one or more samples is responsible for initiating an SDR (Attachment F).

6.8.1 Internal Corrective Actions

Examples of QC elements generally monitored by each subcontractor laboratory are listed in Subchapter 6.5 and Subchapter 6.6. Other method-specific QC elements are also monitored during routine operations. See Attachment F for the corrective actions for each method.

6.8.2 External Corrective Actions

Any actions deemed necessary by EEG, USACE, or any other external regulatory or certifying agencies will be taken by the affected subcontractor laboratory as necessary. These actions are most likely to arise from a systems or performance audit, or from data review conducted by the agency.

6.8.3 Documentation

6.8.3.01 SDRs will be used by the subcontractor laboratories to document deficiencies and exceptions that may impact data quality, production, efficiency, or relations with STL Chicago or USACE. To the extent possible, the laboratories will resolve all situations that require corrective action before data quality is compromised. These non-conformance actions do not require documentation in a formal SDR.

6.8.3.02 The following standards apply to corrective actions:

- The Job Sample Receipt Checklist Report (Attachment E) is a form of corrective action report. It documents problems encountered during sample receipt.
- If there is a critical problem that requires immediate action (e.g., samples received after holding time expired, holding time missed during the analytical process, insufficient sample volume), the laboratory will notify EEG's laboratory coordinator immediately and the corrective action designed in consultation with USACE.
- If the laboratory reports data whose QC elements are not within criteria, the exceptions are noted in the case narrative.
- If the laboratory discovers any problems after the report has been sent to the client (e.g., after system or data audit, client inquiries, external review), a formal SDR will be initiated.

6.8.4 Sample Discrepancy Report Responsibility

6.8.4.01 SDRs are the responsibility of the laboratory staff. Any laboratory employee who becomes aware of a problem with any aspect related to reported data is responsible for initiating

an SDR. In most cases, this will be primarily the analysts' responsibility, but any reviewer or person in contact with the client that becomes aware of a problem must initiate a formal corrective action report.

6.8.4.02 The laboratory's project manager or point of contact as shown in Chapter 2 is responsible for reporting to the laboratory project manager and to EEG's project chemist all corrective actions taken by the laboratory for this project. The laboratory project manager is responsible for ensuring that the action is implemented and documented in the case narrative. EEG's project chemist is responsible for reporting the action to EEG's project manager and to the USACE project chemist.

6.8.5 Sample Discrepancy Report Approval

SDRs (see Attachment F) are approved by the originating laboratory's project manager and QA manager, and completed SDRs are filed in the affected project file(s).

7.0 Data Reduction / Calculation of Data Quality Indicators

Project quality indicator, precision, and accuracy measurements employed by the subcontracted laboratories to support this project are summarized in Attachment H.

7.1 Precision

7.1.01 Precision is an estimate of variability, i.e., it is an estimate of agreement among individual measurements of the same physical or chemical property, under prescribed similar conditions. The precision of a measurement system is generally affected by random errors (e.g., sample non-homogeneity). For this project, precision will be expressed as RPD between duplicate measurements.

7.1.02 Calculation of RPD between duplicates:

$$RPD = \frac{|Value\ 1 - Value\ 2|}{Value\ 1 + Value\ 2} \times 100$$

7.2 Accuracy (Bias)

7.2.01 Accuracy is a measure of the agreement of an analysis result and a true or expected value, or between the average of a number of measurements and the true or expected value. Systematic errors affect accuracy. For chemical properties, accuracy is expressed as a percent recovery (%R) or as percent bias (100 – %R).

7.2.02 For this project, accuracy will be measured by analyzing spiked samples (e.g., MS or LCS), or by adding surrogate compounds for organic tests. Percent recovery is calculated using the following equations. When measured using an MS, accuracy measurements are specific to the sample used and may not reflect on the accuracy of associated samples.

7.2.03 Calculation of %R for MSs:

$$\% R = \frac{\text{Amount found in spiked sample} - \text{Amount found in unspiked (native) sample}}{\text{Amount spiked}} \times 100$$

7.2.04 Calculation of %R for LCSs and surrogates:

$$\% R = \frac{\text{Amount found}}{\text{Amount spiked}} \times 100$$

7.3 Sample Quantitation / Reporting Limits (Limit of Detection)

7.3.1 Calculation of Control Limits

RPDs are statistical control limits based on laboratory historical data and derived on an annual basis. For accuracy, the QC analytes in a given matrix are tabulated over time and a mean recovery is established, as is the standard deviation(s) of those recoveries.

7.3.2 Method Detection Limit

7.3.2.01 The subcontractor laboratories use MDLs that are verified annually (or more frequently) as described below. The MDL is three to five times lower than the reporting limit, which is included in most calibration curves and verified daily. The MDL therefore represents a value that can be reliably detected and distinguished from noise levels.

7.3.2.02 Annual verification of the MDLs is performed according to procedures described in 40 CFR Part 136, Appendix B.

- MDLs are calculated for each analyte (provided it can be spiked) and matrix type (aqueous, solid).
- An MDL study is performed whenever a new instrument is placed on line, if the configuration of any one instrument is changed, or if the sample preparation method or technique is changed.
- The mean of the seven measured concentrations of the MDL spikes, divided by the empirically determined MDL, should be between 1 and 5 for reagent water matrix, and between 1 and 10 for other matrices. Otherwise, the spike concentrations should be adjusted and the MDL studies repeated.
- If multiple instruments with identical configurations are used, the MDL study is performed using one of the instruments. An MDL verification check sample (see below) is analyzed on the other instruments to verify sensitivity.
- If a method is performed using multiple instruments with similar configurations, the MDL study will be performed on the least sensitive instrument; however, the MDL verification check sample will be analyzed on all instruments.

- MDL samples are prepared using all preparation and cleanup techniques routinely used on samples.
- The static MDL is verified if it is equal to or higher than the measured MDL.
- If the static MDL is not verified by the measured MDL, the MDL can be verified by an MDL check sample that meets criteria.
- Each MDL study is followed by an MDL verification check sample (see below).

7.3.2.03 If the annual MDL study is delayed, the previous or theoretical MDL is verified on a quarterly basis until the MDL study can be completed. The MDL verification check sample:

- Consists of a blank (deionized water, Ottawa sand) spiked with the target analytes at a concentration up to two times the static MDL
- Is prepared using all preparation and cleanup techniques routinely used on samples
- Is analyzed on all instruments routinely used for that method/technique
- Must have a response that is readily distinct from the instrument's noise level (signal-to-noise ratio is equal to or greater than 3) or the analyte can be readily identified and quantified (i.e., detected)

7.3.2.04 The primary evaluation feature of MDL study results is the spike-to-MDL ratio. The ideal ratio between the spike level and the MDL value is 5, for at this spike level the random effects of analytical variability (i.e., noise) are not overshadowed by the analyte signal. MDLs are always adjusted to reflect dilutions and, in the case of solid samples, moisture content. Dilutions required to analyze samples within instrument or calibration constraints and the presence of moisture in soil samples always results in increased reporting limits.

7.4 Data Completeness

7.4.01 Data completeness for acceptable data will be calculated as a percentage of acceptable data out of the total amount of data generated. For this project, acceptable data includes both data that passed all QC criteria and data that may not have passed all criteria but that had appropriate corrective action taken.

7.4.02 The formula for calculation of data completeness for acceptable data is:

$$\%Data\ completeness\ for\ acceptable\ data = \frac{\textit{number of acceptable data}}{\textit{number of possible results}}$$

7.4.03 Data completeness for acceptable data is calculated and reported for each method, matrix, and analyte combination. For completeness requirements, acceptable data are all results not qualified with an R flag. The requirement for data completeness for acceptable data for this project is 90 percent for each individual analytical method.

7.4.04 Data completeness for quality data will be calculated as a percentage of quality data out of the total amount of data generated. For this project, quality data is only that data that has passed all QC criteria described in this QAPP.

7.4.05 The formula for calculation of data completeness for quality data is:

$$\%Data\ completeness\ for\ quality\ data = \frac{\textit{number of acceptable data}}{\textit{number of possible results}}$$

7.4.06 The requirement for data completeness for quality data is 80 percent for each individual analytical method.

8.0 Laboratory Operations Documentation

8.0.01 The goal of the laboratory is to provide complete, accurate, and verifiable data. To meet this goal, procedures for ensuring the correctness of the data must be followed. Subchapter 6.5 discusses the key elements of the calibration procedures followed by the laboratory to ensure traceability of the results.

8.0.02 Documentation of data reduction requirements ensures that all calculations were performed according to method requirements, and that minimum guidelines are followed in generating the final sample results.

8.0.03 The objective of data verification is to provide results of a verifiable and acceptable quality whose validity is not jeopardized. The data verification process ensures that:

- The correct samples are reported
- No systematic errors were made in calculating the final results
- Samples were analyzed within calibration criteria
- Samples were analyzed within holding times
- QC elements monitored were within known acceptable limits

8.0.04 The purpose of data reporting is to pass on the analytical information to the user. The information must be complete according to the user's needs and in a format that meets the user's requirements.

8.1 Sample Management Records

8.1.01 Data reduction is the first step to sample management records and refers to all activities that convert analytical values into final sample concentrations of the target analytes. These activities may involve analyte ID, mathematical calculations, and summary statistics. The laboratory will calculate results as described in the SOPs (Attachment F).

8.1.02 Initial data reduction is the responsibility of the analyst who performs the analysis. The analyst is responsible for:

- Ensuring that samples are analyzed only when the instrument is calibrated according to the method
- Ensuring that QC results (spike recoveries, precision for duplicates) are calculated correctly and within criteria, and, if not, initiating corrective actions
- Identifying QC results for review by the responsible person(s)

- Documenting sample preparation and analysis, and the conditions under which they were performed, in a bound laboratory notebook
- Ensuring that the laboratory sample ID is correctly transcribed into all analytical records
- Correctly entering all of the parameters needed for final result calculation, if the data reduction will be performed using computer-controlled data acquisition and data reduction
- Performing the calculations according to the method requirements, if data reduction will be performed using a pocket calculator
- Ensuring that the entry is made correctly, if the result is transcribed
- Performing data review on their own or on peer data
- Alerting a supervisor about any problems that the analyst believes may affect the quality of the data

8.1.1 Manual Data Reduction

8.1.1.01 Manual data reduction refers to those activities in which analytical output is converted to analyte concentration in samples by calculations performed manually. The analyst:

- Ensures that all data are correctly transcribed into worksheets, forms, or computer applications
- Keeps raw data as a part of the analysis records, if the analytical instrument used generates hardcopy reports (strip charts, tabular reports, etc.)
- Selects the appropriate, method-specified formulae for calculating results (the formulae used are written in the SOPs)
- Proofreads computer-generated reports to ensure that the raw data manually entered into the computer application is correct

8.1.1.02 Raw data hardcopy reports are identified with date of analysis, laboratory sample ID, analyst, and referenced method or SOP.

8.1.2 Computer Data Reduction

8.1.2.01 Computer data reduction refers to those activities in which analytical acquisition and initial calculations are performed automatically by validated computer applications. Appropriate to the method used, the analyst will:

- Ensure that all variables required for final calculations (sample amount, dilution factor, extract volume, percent solids, surrogate amount, etc.) are entered correctly

- Verify that computer IDs are correctly made
- Calculate surrogate recoveries and verify that internal standard responses are acceptable
- Verify that target compounds analyzed by chromatographic methods are within the appropriate retention time or relative retention time windows

8.1.2.02 Raw data files are assigned a unique filename by the analyst performing the analyses. In some instances, the computer performs the filename assignment using rules that ensure that filenames will not be repeated. Files containing sample-specific information (laboratory sample ID, sample amount, dilution factor, etc.) are cross-referenced to each raw data file using application functionality. The sample analysis logbooks can be used as an alternative cross-reference between the laboratory sample ID and the raw data file name.

8.1.3 Significant Figures

8.1.3.01 All organic results are rounded to two significant digits. Inorganic and geotechnical results are reported to two significant figures if the value is less than 10, and to three significant figures if greater than or equal to 10.

8.1.3.02 Whenever data is reduced using computer applications, the rounding rules used are those provided with the operating software. During manual calculations, the following rounding rules are followed.

- If the digit to be dropped is less than 5, do not change the last digit to be retained (e.g., 2.23 rounds off to 2.2).
- If the digit to be dropped is greater than 5, increase the last digit to be retained by one (e.g., 2.26 rounds to 2.3).
- If the digit to be dropped is equal to 5, increase the last digit to be retained by one if it is odd (e.g., 2.35 rounds to 2.4) or do not change the last digit to be retained if it is even (e.g., 2.45 rounds to 2.4).

8.1.4 Data Review

8.1.4.01 All analytical data generated at each laboratory are extensively checked for accuracy and completeness. The laboratory is responsible for ensuring that valid data includes several levels of review. Each level demands specific action to prevent unqualified release of erroneous data and to correct problems discovered during the review process. Each subcontractor laboratory data validation process will include data generation, reduction, and three levels of review.

8.1.4.02 The Level 1 review is performed by the analyst who generates the analytical data.

The analyst reviews the data package to ensure that:

- Sample preparation information is correct and complete
- Analysis information is correct and complete
- The appropriate SOPs have been followed
- Analytical results are correct and complete
- QC samples are within established control limits (blanks are acceptable)
- Special sample preparation and analytical requirements have been met
- Documentation is complete (e.g., all anomalies in the preparation and analysis have been documented; out-of-control forms, if required, are complete; holding times are documented, etc.)

8.1.4.03 Level 2 review is performed by the laboratory QA officer, whose function is to provide an independent review of the data package. This review is structured to ensure that:

- Calibration data are scientifically sound, appropriate to the method, and completely documented
- QC samples are within established guidelines
- Qualitative ID of sample components is correct
- Quantitative results are correct
- Documentation is complete and correct (e.g., anomalies in the preparation and analysis have been documented; out-of-control forms, if required, are complete; holding times are documented, etc.)
- The data are ready for incorporation into the final report
- The data package is complete and ready for data archive

8.1.4.04 Level 2 review is structured so that all calibration data and QC sample results are reviewed, and all of the analytical results from 10 percent of the samples are checked back to the bench sheet. If no problems are found with the data package, the review is considered complete. If any problems are found with the data package, an additional 10 percent of the samples are checked to the bench sheet. The process continues until no errors are found, or until the data package has been reviewed in its entirety. Level 2 data review is documented, and the signature of the reviewer and the date of review recorded. The reviewed data are then approved for release and a final report is prepared.

8.1.4.05 Before the report is released to EEG, the laboratory project manager reviews the report to verify the accuracy and completeness of the Level 2 review and ensure that the data meets the overall objectives of the project. This review is the Level 3 review.

8.1.4.06 Each step of this review process involves evaluation of data quality based on both the results of the QC data and the professional judgment of those conducting the review. This application of technical knowledge and experience to the evaluation of the data is essential in ensuring that data are of consistently of high quality.

8.1.5 Other Review

The data derived from this project must be evaluated and approved by a Puerto Rico licensed chemist in accordance with Puerto Rico Department of Environmental Quality guidelines.

8.1.6 Procedures for Handling Unacceptable Data

All QC information will be recorded in the notebooks and printouts. It is the analyst's responsibility to check the QC information against limits for the analysis. When analysis of a QC sample (blank, spike, check standard, replicate, or similar sample) shows that the analysis of that batch of samples is not in control, the analyst will perform corrective action or bring the matter to the attention of the group leader. The group leader will, if necessary, consult with the laboratory QA officer or the laboratory project manager to determine whether the analysis can proceed, whether selected samples should be rerun, or whether specific corrective action needs to be taken before analyzing additional samples. Out-of-control analyses must be documented. The analyst or group leader will file an SDR with the laboratory QA officer for laboratory analysis out-of-control events that require documentation.

8.2 Data Reporting Procedures

8.2.1 Data Package Format and Contents

The laboratory will supply a definitive data package. The definitive data package format allows for the review of the data by an independent organization but the data package does not allow for complete independent reconstruction of the analytical data. Definitive data are produced using rigorous analytical methods, such as USEPA standard reference methods (e.g., SW-846, Contract Laboratory Program). Analyte presence and quantitation are confirmed through extensive QC procedures at the laboratory, which may be on site or off site. As discussed in more detail in the following chapters, the definitive data package will include a cover sheet, table of contents, case

narrative, the analytical results, laboratory reporting limits, sample management records, and internal laboratory QA/QC information. The laboratory data package will be organized such that the analytical results are reported on a per-batch basis unless otherwise specified.

8.2.1.1 Cover Sheet

8.2.1.1.01 The cover sheet will specify the following information:

- Title of report (i.e., Test Report, Test Certificate)
- Name and location of laboratory (to include a point of contact, with telephone and fax numbers)
- Name and location of subcontractor laboratories, and appropriate test method performed
- Contract number
- Client name and address
- Project name and site location
- Statement of data authenticity and official signature and title of person authorizing report release

8.2.1.1.02 Amendments to previously released reports shall clearly identify the serial number for the previous report and state the reason(s) for reissuance of the report.

8.2.1.2 Table of Contents

Laboratory data packages will be organized in a format that allows for easy identification and retrieval of information. An index or table of contents will be included for this purpose.

8.2.1.3 Case Narrative

A case narrative will be included in each report. The case narrative will contain a table or tables summarizing samples received, providing a correlation between field sample numbers and laboratory sample numbers, and identifying which analytical test methods were performed and by which laboratories. Samples that were received but not analyzed will also be identified.

Extractions or analyses that are performed out of holding times will be appropriately noted. The case narrative will define all data qualifiers or flags used. Deviations of any calibration standards or QC sample results from appropriate acceptance limits will be noted, and associated corrective actions taken by the laboratory will be discussed. Any other factors that could affect the sample results (e.g., air bubbles in volatile organic compound [VOC] sample vials, excess headspace in

soil VOC containers, the presence of multiple phases, sample temperature and sample potential of hydrogen (pH) excursions, container type or volume, etc.) will be noted.

8.2.1.4 Analytical Results

The results for each sample will contain the following information, at minimum (information need not be repeated if noted elsewhere in the data package):

- Laboratory name and location (city and state)
- Project name and unique ID number
- Field sample ID number as written on custody form
- Laboratory sample ID number
- Matrix (soil, water, oil, etc.)
- Sample description
- Sample preservation or condition at receipt
- Date sample collected
- Date sample received
- Date sample extracted or prepared
- Date sample analyzed
- Analysis time when holding time limit is less than 48 hours
- Method (and SOP) numbers for all preparation, cleanup, and analysis procedures used
- Preparation, analysis, and other batch numbers
- Analyte or parameter
- Method reporting limits adjusted for sample-specific factors (e.g., aliquot size, dilution/concentration factors, moisture content)
- Method quantitation limits (low-level standard concentration)
- MDLs
- Analytical results with correct number of significant figures
- All confirmation data
- Any data qualifiers assigned
- Concentration units
- Dilution factors (All reported data shall reflect any dilutions or concentrations. The dilution factor, if applicable, will be noted on the analytical report. If neat and/or diluted results are available, data from all runs will be recorded and reported.)

- Percent moisture or percent solids (all soils, sediments, sludges, etc., are to be reported on a dry weight basis)
- Chromatograms, as needed
- Sample aliquot analyzed
- Final extract volume

8.2.1.5 Laboratory Reporting Limits

The laboratory may use a reporting limit expressed in terms of detection limit, quantitation limit, regulatory action level, or project-specific threshold limits; however, the laboratory's use of these terms must be well-defined. In addition, the "<" (less than) reporting convention must be used in accordance with the requirements established in Subchapter 7.3.

8.2.1.6 Sample Management Records

These types of records include the documentation accompanying the samples (i.e., original chain-of-custody record, shipping documents, laboratory notification sheets), records generated by the laboratory that detail the condition of the samples upon receipt at the laboratory (i.e., sample cooler receipt forms, any telephone conversation records, etc.), and any records generated to document sample custody, transfer, analysis, and disposal.

8.2.1.7 Quality Assurance / Quality Control Information

The minimum data package must include the calibration, calibration verification, and internal laboratory QA/QC data with their respective acceptance criteria. The data package will also include the laboratory's method quantitation and reporting limits for project-specific parameters. The calibration data shall include a summary of the ICV, all calibration verification standards, and any performance standards analyzed in conjunction with the test method. All calibration deviations shall be discussed within the case narrative. The data package will correlate the method QC data with the corresponding environmental samples on a per-preparation batch basis with batch numbers clearly shown. Method QC data must include all spike target concentration levels, the measured spike concentration and calculated recoveries, all measures of precision, including RPD, and all control limits for bias and precision. This would include laboratory performance information such as results for MB, recoveries for LCSs, and recoveries for QC sample surrogates; and matrix-specific information such as MD RPDs, MS and MSD recoveries, MS/MSD RPDs, field sample surrogate recoveries, serial dilutions, and post digestion spike, etc. At minimum, internal QC samples will be analyzed and reported at rates specified in the specific

methods, within USACE guidance, or as specified in the contract, whichever is greater. Any deviations from the measurement quality objectives will be noted. The data package will also include any data review, non-conformance, or corrective action forms.

8.2.2 Electronic Deliverables

8.2.2.01 Electronic chemical data will be provided to CEHNC in the Analytical Data Review (ADR) format. STL will develop a comprehensive library file for all of the methods to be analyzed under this Scope of Work. The library file will accurately reflect all of the analytical quality requirements as documented in the final Sampling and Analysis Plan for this project and will be provided to CEHNC for use in screening electronic data deliverable (EDD) submittals.

8.2.2.02 All electronic data submitted by STL will be error-free and in complete agreement with the hardcopy data. Data files are to be delivered both by e-mail and on high-density compact disk accompanying the hardcopy data reports. The disk must be submitted with a transmittal letter from the laboratory that certifies that the file is in agreement with hardcopy data reports and has been found to be free of errors using the latest version of the ADR evaluation software provided to the laboratory. STL will archive the electronic raw data and sufficient associated hardcopy data (e.g., sample log-in sheets and sample preparation log sheets) to completely reconstruct the analyses that were performed for a period of 10 years after completion of this contract.

8.3 Data Management Procedures

8.3.1 Laboratory Turnaround Time

The laboratory turnaround time will be 28 days for the complete data package. Copies of the results only can be received after 14 days

8.3.2 Data Archive / Retention Requirements

The laboratory will retain all records that pertain to this project for a minimum of five years from the date the records are formally archived. Archived record indexes are maintained in a database, which allows rapid retrieval of the archives. Archives are stored on site and are protected against fire, theft, loss, deterioration and vermin. Electronic records are protected from deterioration caused by magnetic fields and/or electronic deterioration.

9.0 Data Assessment Procedures

9.1 Data Quality Control Review

Data QC review is discussed in Subchapter 8.2.

9.2 Data Verification / Validation

9.2.01 EEG’s project chemist will conduct an independent data validation prior to data acceptance. All samples for all methods and analytes in this project will be validated. This data validation shall include all data documentation from raw data to the reported results in accordance with the requirements specified in the following documents:

- Project FSP
- Project QAPP
- Subcontractor laboratories’ SOPs and LQMs
- USEPA SW-846 Update IIIA, 1999
- DQOs in Attachment H

9.2.02 A thorough review of all data documentation from the raw data to the reported results will be performed. For each method, the following types of data will be reviewed to verify that they are complete and support the reported values.

9.2.1 Method 6010B – Metals

- Case narrative
- Sample IDs
- Chain of custody
- Holding time
- Initial calibration
- Instrument precision
- ICV
- ICB
- Inter-element check standards
- CCB
- CCV
- MB
- LCS

- MS
- MSD
- Post digestion spike
- Serial dilution
- MSA

9.2.2 Method 7471A – Mercury

- Case narrative
- Sample IDs
- Chain of custody
- Holding time
- Initial calibration
- Instrument precision
- ICV
- ICB
- CCB
- CCV
- MB
- LCS
- MS
- MSD
- Post digestion spike
- Serial dilution
- MSA

9.2.3 Method 8330 – Explosives

- Case narrative
- Sample IDs
- Chain of custody
- Holding time
- Initial calibration
- ICV
- CCV

- MB
- LCS
- MS
- MSD
- Surrogates
- Target analyte confirmation

9.2.4 Method 314 – Perchlorate

- Case narrative
- Sample IDs
- Chain of custody
- Holding time
- Initial calibration
- Second source verification
- Instrument performance check
- ICV
- CCV
- MB
- Pretreated laboratory blank
- LCS
- MS
- MSD

9.2.4.01 Following completion of this review, the project chemist will prepare a narrative report describing the data validation process and its results. Data qualifiers will be added to the analytical results report following USACE guidelines if the subcontractor laboratory did not already flag them. If data reported by the subcontractor laboratory are rejected, EEG will consult with the contracting officer regarding appropriate corrective actions.

9.3 Data Quality Objectives Reconciliation

The project chemist will determine if the DQOs summarized in the Sampling and Analysis Plan were attained. Contract compliance is assessed to ensure that stated requirements for daily QC have been met. The daily quality report, the contractor's data validation report, results from

performance evaluation samples, field oversight findings, and/or project-specific laboratory audits will all be reviewed to assure that the DQOs have been met.

9.4 Project Completeness Assessment

9.4.01 Data completeness for acceptable data is calculated as a percentage of acceptable data out of the total amount of data generated. The formula is number of acceptable data divided by number of possible results. For this project, acceptable data includes both data that passed all QC criteria and data that may not have passed all criteria but had appropriate corrective action taken.

9.4.02 Data completeness for acceptable data is calculated and reported for each method, matrix, and analyte combination. For completeness requirements, acceptable data are all results not qualified with a rejected (R) flag. The requirement for data completeness for acceptable data for this project is 90 percent for each individual analytical method.

9.4.03 Data completeness for quality control data will be calculated as a percentage of quality data out of the total amount of data generated. For this project, quality data is only that data which has passed all QC criteria described in this QAPP.

9.4.04 The formula for percent completeness is:

$$\% \text{ complete} = \frac{A - B}{A} \times 100$$

Where:

A = Total number of measurements

B = Total number of unacceptable measurements

ATTACHMENT A

**USACE Chemical Data Quality Management
Procedures and Notification**

Chapter 1

U.S. ARMY CORPS OF ENGINEERS CHEMICAL DATA QUALITY MANAGEMENT PROCEDURES AND NOTIFICATIONS

1-1. Introduction. Execution of the USACE Chemical Data Quality Management (CDQM) program for HTRW contamination requires the interface and coordination of several Corps personnel. Procedures and responsibilities for USACE staff performing government CDQM activities are defined and detailed in this Chapter. The USACE project manager (PM) is responsible for initiating and coordinating the defined CDQM activities.

1-2. Goals of the CDQM Program. The goals of the USACE CDQM program are to: 1) generate data of acceptable quality for the intended use; 2) satisfy the needs of the customer and the regulators; 3) generate sufficient data of known quality on the first attempt; and 4) provide an historical record for potential future use. When CDQM is used properly, the PM can readily measure the success of the team in meeting the project-specific DQOs. The USACE CDQM program consists of activities presented in ER 1110-1-263, CDQM for Hazardous Toxic and Radioactive Waste Remedial Activities, Engineer Manual (EM) 200-1-1, Validation of Analytical Chemistry Laboratories, EM 200-1-2, Technical Project Planning Guidance for HTRW Data Quality Design, and EM 200-1-3, Requirements for the Preparation of Sampling and Analysis Plans (SAPs).

1-3. Technical Project Planning. Each district is responsible for assessment of chemical data quality, including determination of data useability and DQO attainment. The district project chemist is a critical team member for this effort, and must be involved in preparation and review of project documents including scopes of work, SAPs, contract specifications, and final chemical data reports. The district project chemist must be involved at each step of an HTRW project, so that adequate data quality is maintained. The TPP process for design of DQOs is described in EM 200-1-2.

1-4. CDQM Activities. All HTRW projects require a comprehensive and multifaceted approach to QC and QA in order to achieve and document attainment of appropriate quality for the intended data usage. The district project chemist is the focal point to ensure that chemical data meet DQOs for each HTRW project. The district project chemist has several techniques to monitor and ensure the quality of chemical data. The district project chemist in conjunction with other members of the TPP team determine the appropriate level of compliance monitoring as discussed in ER 1110-1-263, Appendix A. This determination should be based upon the intended use of the data and the degree of confidence needed in the quality of the data. Compliance monitoring may consist of a combination of activities. Described below are twelve (12) activities that may be applied on a project-specific basis to assist in generating data of known quality. The twelve CDQM activities, their relative cost, and typical use are summarized in Table 1-1.

a. Validation of Primary and QA Laboratories. In general, commercial and government laboratories that support the USACE HTRW program should obtain a USACE laboratory validation prior to field studies or sample analysis. The QA laboratory is defined as the Chemistry and Materials Quality Assurance Laboratory (CMQAL), located in Omaha, Nebraska or a subcontracted agent that is responsible for analysis of the project QA samples. For some data uses, other programs (*i.e.*, State Fuel Storage Tank Program, A2LA, Navy and Air Force Installation Restoration Program (IRP) Audits) can be utilized. Projects should not be implemented without utilization of information from some accreditation authority. Validation should be maintained throughout the duration of the project. The USACE laboratory validation program is project specific. The validation is a parameter, method, and matrix-specific approval. For each new contract or delivery order awarded during the validation period, a project-specific request for validation should be sent to CENWO-HX-C (Corps of Engineers, Northwestern Division, Missouri River Region, HTRW-Center of Expertise, Chemical Data Quality Management Branch) for verification of laboratory status regardless of their expiration date on the list of validated laboratories. The primary objectives of the USACE laboratory validation program are to communicate to analytical service providers the USACE QC/QA requirements, verify the laboratories are performing specified analytical methods, and to ensure these laboratories meet the USACE requirements prior to sample analysis. Laboratory validations are performed under the administration of the HTRW-CX applying guidance outlined in EM 200-1-1. The USACE validation program is primarily based on SW-846 methods. The first step of the validation program is a paper review of the laboratory's capabilities to ensure that the proposed laboratory has the facility, equipment and personnel to meet the project required analyses. The laboratory must demonstrate capabilities by providing acceptable standard operating procedures (SOP) and successfully analyzing project required performance evaluation (PE) samples. The final step of the validation program is an on-site inspection of the laboratory's facility. Validation can be terminated at any step of the process due to inadequate laboratory documentation performance and/or execution. No notice or short notice on-site audits of facilities listed as USACE validated are available, but require the participation of at least one member of the project planning team.

b. Technical Document Review. The roles and responsibilities for document review are defined in the Environmental Cleanup and Protection Management Plan for Military Programs, 17 January 1996 and Corps of Engineers, Military Programs Directorate, Environmental Division, Policy and Technology Branch (CEMP-RT) Memoranda: 1) Environmental Cleanup and Protection Management Plan for Military programs, 17 January 1996; and 2) Technical Roles and Responsibilities for the USACE HTRW Program, 23 September 1997 (herein referred to as the HTRW Management Plan).

(1) HTRW Project Technical Verification Process. It is the responsibility of the contractor and the district to produce a quality product. Rather than employing multiple levels of detailed document review to ensure quality, the technical verification process transfers project

responsibility to the district and its contractors. In general, the HTRW design district is responsible for a QC review of the prime contractor's QC Plan and all project-specific deliverables. QC Plans, scopes of work, and other project documents completed in-house should be reviewed by an independent technical review function established by the design district. The Major Subordinate Command (MSC) will provide oversight of the district's QC process. Only inventory project reports for the FUDS program require approval at the division level. Districts may request HTRW-CX participation in a design district's independent technical review process. The MSCs may request HTRW-CX support in performing QA oversight and audits of HTRW design districts QC processes. HTRW-CX review is required on Category B projects (see below).

(2) HTRW Project Technical Categories. The HTRW design district screens each HTRW project against the decision tree criteria provided in Attachments 1 and 2 of the Management Plan to determine the appropriate review process. Category A includes all routine HTRW (as defined in the Management Plan), and all projects in the Preliminary Assessment(PA) phase and those beyond the Site Inspection (SI) or Resource Conservation Recovery Act (RCRA) Facility Assessment (RFA) phase. Category A excludes, however, National Priorities List (NPL) sites, Base Realignment and Closure (BRAC) sites, sites where innovative technologies are used, and sites with construction estimates greater than \$5 million. Category B includes all projects not in Category A, and any projects of special district, MSC, or HQ concern.

(3) Roles and Responsibilities for Review of Specific HTRW Products. Review responsibilities will vary depending on the category (Category A or Category B) of projects. The HTRW design district is responsible for all reviews of projects in Category A (Attachments 1, 2, and 3 of the Management Plan). Key documents for projects in Category B will be reviewed and approved by the HTRW design district and reviewed by the HTRW-CX. The PM provides appropriate technical documents to the HTRW-CX and QA laboratory for their information or review. Technical chemistry review by the HTRW-CX will be completed within two weeks for a Scope of Work and within three weeks for all other documents from time of receipt. If shorter review times are required, the PM coordinates with the Technical Liaison Manager (TLM) at the HTRW-CX. Comments from the HTRW-CX will be provided to the PM for all projects reviewed. A copy of all review comments and responses is placed in the permanent project file. Districts/centers with insufficient staff chemist resources to provide in-house review should rely upon the military design district, CMQAL or the HTRW-CX for document review. Note only certain key documents have been identified for HTRW-CX review as Category B projects; these are identified in Table 2 of the Management Plan. In addition, Chemical Quality Assurance Reports (CQARs)(Chapter 4) and Chemical Data Quality Assessment Reports (CDQARs) (Chapter 5) from all projects will be sent to the HTRW-CX. The HTRW-CX is responsible for 10% review of both CQARs and CDQARs. A summary of the reviews will be sent quarterly to CEMP-RT by the HTRW-CX.

c. Sample Handling Quality Assurance. The QA laboratory provides quick feedback regarding problems with sample shipments. The QA laboratory is responsible for checking the sample shipment for temperature, proper preservatives, correct containers *etc.* The Technical Manager (TM) or district project chemist is then notified within 24 hours regarding the status of the sample shipment via facsimile, electronic mail or telephone call. For most projects, this is beneficial because problems are detected and resolved while the sampling team is still in the field. This approach reduces the re-mobilizations to the field. The CMQAL or contract QA laboratory, and the primary laboratory complete and report a "Cooler Receipt Checklist" for all shipments sent to the laboratory. An example cooler receipt checklist is found in EM 200-1-1. A chain-of-custody (CoC) record must be initiated at the sampling stage and maintained throughout the analysis and reporting stages of the process. Sample reports must be easily traceable to CoC records. All documentation pertaining to sample receipt or analysis should be included in the laboratory's data report. If this function is performed without analysis of QA samples, samples must either be shipped back to the project site or additional funds provided to properly dispose of samples.

d. QA Sample Collection and Analysis. QA sample collection and analysis is the main tool to determine that the data generated by primary laboratories is technically valid and of adequate quality for the intended data usage. Based on the needs of the project, a percentage of samples are homogenized (except samples for volatiles testing, which are co-located), split, given a unique sample identification (ID) and sent to a primary contract laboratory and to a QA laboratory for analysis. QA sample collection does not have to be performed at the same frequency or rate for all test parameters, on all matrices, during all project phases, nor for any one type of project. General considerations should include: 1) the data use and users as defined by the project-specific DQOs; 2) the total number of samples being generated (*e.g.*, a larger number of total samples collected may lower the percentage of QA samples needed); and 3) the need for statistically significant information from QA sample data. Ideally, the USACE QA sample collection and analysis program is an interactive process whereby the QA laboratory in conjunction with the TM or district project chemist detects and solves problems as sampling and analysis occurs to ensure that the data generated for the project meets the project DQOs. The "value added" by this program can be divided into two areas.

(1) Detecting Analytical Problems. A primary function of the QA laboratory is to analyze samples as prescribed by the project and produce a data package that is reviewed real-time (at the bench during the time of analysis) for later comparison to the primary laboratory's data. Analysis and comparison of the QA sample data to the primary sample data can reveal problems with primary laboratory data even when all other data quality measurements are in control. A common problem is over-dilution of semi-volatile organic analytes by the contract laboratories. Analysis by the QA laboratory can help in deciding whether this was due to actual matrix effect or due to inadequate sample cleanup by the primary laboratory.

(2) Salvaging Data Useability. When the data comparison shows good correlation between the QA laboratory and primary laboratory data, this may bolster the credibility and useability of the data generated by the primary laboratory. This is especially true in cases where primary laboratory data comes under close scrutiny and fails some data quality criteria. Good correlation also reflects consistency in the sampling process, the lack of which is a major source of error or variation. The criteria that establish acceptable correlation between project, QC and QA sample results are described in Chapter 4.

e. Chemical Quality Assurance Reports (CQARs). CQARs are usually prepared by the CMQAL. The CQAR documents review of the QA laboratory data and the corresponding primary laboratory data. Data for project samples, QC samples and QA samples are compared, and the impact on the primary laboratory's data is documented. CQAR format is discussed in Chapter 4.

f. Chemical Data Quality Assessment Reports (CDQARs). CDQARs are prepared by the district project chemist. The CDQAR documents data useability, DQO attainment, and contract compliance. CDQAR format is discussed in Chapter 5.

g. Single or Double Blind PE Sample Analysis. Another means of testing the analyst's proficiency in identifying and quantifying analytes of interest is the use of single or double blind PE samples. The composition of PE samples is known to the originator, but not the analyst. In a single blind PE sample, both the originator and the analyst know that the sample is a PE sample. The USACE uses single blind PE samples as part of the process to validate laboratories. In a double blind PE, the sample is containerized, labeled, and submitted as an environmental sample. The analyst does not know that the sample is a PE sample; ideally, the PE sample will be indistinguishable from the other project samples. The use of double blind PE samples is considered a more effective way of detecting problems, since the laboratory would not be aware that it was being evaluated. However, it may be difficult to disguise a standard reference sample as a project sample. PE sample data are evaluated for compound ID, quantitation, and sample contamination. PE samples are recommended for sites that have the potential for a majority of non-detects, or for sites where the contaminants of concern have already been identified. Currently, the complete range of organic and inorganic PE samples are available for water only. Selected organic and inorganic PE samples are available for soil.

h. Review of Primary Laboratory Data. An independent data review of the entire primary data set should be performed by the prime contractor for contracted projects. In addition, the district project chemist or QA laboratory should review a portion of the primary laboratory data. The percentage of primary laboratory data reviewed by the government depends upon the project-specific DQOs. The district project chemist or CMQAL should review all the primary laboratory data for in-house projects. Data review is conducted to ensure that: 1) QC data provided in the laboratory deliverables are scientifically sound, appropriate to the method, and

completely documented; 2) QC samples are within established guidelines; 3) data were appropriately flagged by the laboratory; 4) documentation of all anomalies in sample preparation and analysis is complete and correct; 5) corrective action forms, if required, are complete; 6) holding times and preservation are documented; 7) data are ready for incorporation into the final report; and 8) data package is complete and ready for data archive. Details of the data review process are described in Chapter 3.

i. Validation of Data. Data validation is the process of data assessment in accordance with EPA regional or national functional guidelines or project-specific guidelines. Data validation includes assessment of the whole raw data package from the laboratory.

j. Field Audits. Sample collection field oversight is discussed in detail in Chapter 6. Audits should be performed on both an announced and unannounced basis, and should be coordinated with government geotechnical personnel, as appropriate. Audits may be performed during any stage of the project.

(1) Procedures. The auditor is responsible for checking that samples are collected and handled in accordance with the approved project plans and for confirming that documentation of work is adequate and complete. Specifically, the auditor should ensure that performance of field activities satisfies the project DQOs. Original records generated for all audits are retained within permanent project files. Records may include audit reports, written responses, record of the completed corrective actions, and documents associated with the conduct of audits that support audit findings and corrective actions. Checklists included in Chapter 6 can be used to guide performance of a field audit. For construction activities, the audit should assess the prime contractor's implementation of the three-phase chemical data control process. Details on contractor QC of field activities are found in EM 200-1-3.

(2) Personnel. Trained and experienced personnel should perform the field audits. These personnel should be knowledgeable in the subjects necessary for assessing the quality of the work being observed, including thorough knowledge of the contractual requirements. Preferably, field audits should be carried out by government personnel. The field audits may be performed by contract personnel with some objective relationship to the work being conducted in the field (*e.g.*, a prime contractor auditing its subcontractors).

(3) Desk Audit of Field Activities. Another mechanism for auditing field activities as they occur is to include government technical review of Daily QC Reports and field logs while the contractor is in the field. Desk audits of field activities require that these reports be supplied on a periodic basis (*e.g.*, daily or weekly) to the USACE technical staff. The requirement for periodic reporting must be included in the contract specifications or project delivery order, as well as in the project work plans. Since the contractor knows of this reporting requirement, it is not possible to perform an unannounced desk audit of field work.

k. Laboratory Audits. The primary and QA laboratories are responsible for maintaining detailed procedures to support the validity of all analytical work. Laboratory audits may consist of on-site inspections and/or analysis of PE samples. The audit verifies the laboratory's continuing ability to produce acceptable analytical data. If a performance problem is identified for sample analysis or data reporting, the HTRW-CX reserves the right to audit the laboratory anytime during the eighteen month period of validation. Laboratory audits may be carried out on either an announced or unannounced basis. More detail on this type of audit is found in EM 200-1-1.

l. Tape Audits. The purpose of a raw data review (tape audit) is to assess the quality of the data and to evaluate the overall laboratory performance. This information is then used by the data user to evaluate data quality and make a determination on the acceptability and the useability of the data. The tape audit is designed to independently verify the data reduction practices of an individual laboratory. All of the raw data from a given batch is recalculated by the evaluator and is compared to the results reported by the laboratory. The data quality is measured by laboratory compliance with the required methods and acceptable laboratory practices for analysis and for data reduction. Tape audits can only be performed when a specific analytical instrumental raw data output has been stored electronically. To implement this type of audit the contract must require the laboratory to provide electronic data (*i.e.*, magnetic tapes) needed to perform the audit. In addition, a means to read the data must be made available.

1-5. Primary CDQM Activities. While all twelve of the CDQM activities discussed in the previous section may be used on a project, six of the twelve should be used on most projects. The six primary CDQM activities for USACE HTRW projects are 1) validation of primary and QA laboratories, 2) technical document review, 3) sample handling QA, 4) QA sample collection and analysis, 5) preparation of CQARs by a qualified entity, and 6) preparation of CDQARs by the district project chemist. These elements should routinely be considered as candidates for inclusion in each project's set of CDQM activities.

a. Documentation of Selected CDQM Activities. The CDQM activities selected for each project shall be documented in the project-specific DQOs. A recommended procedure for documentation of the CDQM process is presented in American National Standard, Specifications and Guidelines for Quality Systems for Environmental Data Collection and Environmental Technology Programs (ANSI/ASQC E-4-1994).

b. Waiver of CDQM Activities. ER 1110-1-263 allows for any aspect of the program to be waived except for the DQO element specified in ER 1110-1-263 Section 7.b. ER 1110-1-263 states that all other CDQM elements may be waived for a specific project by the district PM with concurrence from the technical project team as defined in EM 200-1-2. The intent of ER 1110-1-263 is to provide a flexible CDQM program that produces data of known quality to satisfy the project-specific DQOs.

c. Documentation of Waiver. If the district project chemist in conjunction with the PM and technical project team decides not to use all of the six primary CDQM elements discussed above, a memorandum for record (MFR) is required. The district PM must document in the MFR what procedures will replace the waived compliance monitoring activity and demonstrate the concurrence of the technical project team including the district project chemist. The district project chemist will typically be tasked by the PM to prepare this documentation. The MFR should include the PM's signature and the project team's concurrence along with the following elements: 1) brief description of the project; 2) summary of the project objective; 3) description of the waived CDQM activities; and 4) description of alternate procedures to ensure data quality. Districts with insufficient staff chemist resources to provide technical team support should rely upon other HTRW design districts, the CMQAL, or the HTRW-CX for chemistry support.

1-6. Use of QA Samples by Project Phase. The use of QC and QA samples is a particularly powerful tool for maintenance of data quality. With primary, QC and QA data for a single sampling point one may perform both inter-laboratory and intra-laboratory data comparisons. In addition, QA samples may provide unique indications about the quality of the primary laboratory's data. The following sections describe the use of QA samples in various project phases.

a. Investigative Phase. The use of QA samples during the investigative phase adds value by verifying the analytes of concern and quantifying the levels of contamination. In general, QA samples are targeted in locations of known or expected contamination. If the primary and QA laboratory data are comparable, then this provides an additional level of confidence that the correct action was taken. If the primary laboratory data does not compare with the associated QA laboratory data, then this assures that the data from the site will be completely evaluated prior to a decision. In addition, the QA laboratory data yields information regarding the spatial heterogeneity of the soil contamination.

b. Pre-Design Phase. The pre-design phase of the HTRW program consists of bench and pilot scale studies. If data generated from these activities are used to size the system, accuracy of results is critical. Any false positive or false negative from the bench or pilot study could result in costly changes following construction of the completed system. QA sample collection provides a verification of the prime contractor's results for use in their design.

c. Remedial Action Phase. The remedial action phase of the HTRW program consists of treatment system analytical support. Verification of results from the actual treatment operations is a critical check for long-term operation of the system. QA samples would be useful during the early stages of the project when the system is optimized or at stages of major equipment changes. Many treatment systems focus on discharge quality, and verification of the results aids in the acceptability by the regulators.

d. **Post-Remedial Action Monitoring.** The post-remedial action phase of the HTRW program typically includes post-excavation confirmation sampling and/or treatment system analytical support. QA sample checks on post-excavation samples can bolster regulator's confidence in the effectiveness of remediation. Analytical support during the operation and maintenance (O&M) phase can last up to thirty years in the case of long-term monitoring. In all likelihood, the primary laboratory would change several times during the course of a long-term monitoring project. Use of the same QA laboratory would be instrumental in providing continuity from one laboratory's results to another and for resolving problems that inevitably arise when a large volume of data is collected over a long period of time.

1-7. **Omission of QA Samples.** For certain projects, QA samples may not be the best method of ensuring attainment of DQOs. The decision to omit QA samples for a given project must be made by the district project chemist in conjunction with the PM and technical project team. Omission of QA samples should be based on meeting project objectives and goals, rather than simply to reduce cost. The district chemist must balance the need to maintain quality with the need to perform work for a reasonable cost. The project categories that may not be good candidates for QA sample collection are described below.

a. **Underground Storage Tank (UST) Removals.** Samples collected to meet state or federal requirements pertaining to UST removals may omit QA samples if regulatory deadlines preclude the QA process.

b. **Lead Paint Testing.** Construction building material and debris sampling to test for leaded paint is not generally considered to be HTRW work. Samples of building materials or debris collected solely to test for the presence of leaded paint will not typically benefit from use of QA samples.

c. **Asbestos Testing.** Construction building material and debris sampling to test for asbestos is not generally considered to be HTRW work. Samples of building materials or debris collected solely to test for the presence of asbestos will not typically benefit from use of QA samples.

d. **Process Monitoring.** Samples collected to demonstrate the day-to-day efficacy of intermediate steps during a treatment process will not typically employ QA samples. However, collection of QA samples from the treatment system influent and discharge locations is recommended on an occasional basis.

e. **Waste Characterization.** Samples collected of drummed materials, tank contents, barrels, and similar materials for hazardous waste profiling do not usually employ QA samples.

f. **Treatability Studies.** Samples collected as part of a treatability study to demonstrate the efficacy of a remedial process do not usually employ QA samples. QA samples are

recommended for optimization studies.

g. Air Samples. Samples collected as part of an ambient air monitoring program usually do not employ QA sample collection. Specifically, this would apply to co-located air samples for both gas phase and particulate related components since co-located samples are not homogeneous. Gas phase samples collected with a split sampling device are likely to be homogeneous, and QA samples may provide added value.

h. Wipe Samples. Wipe samples (*i.e.*, for polychlorinated biphenyls (PCB) analysis) will not usually benefit from QA sample collection since co-located wipe samples are not identical.

i. Non-routine Methods. Certain methods are experimental, or laboratory-specific, and it is not possible to replicate them in a QA laboratory. If duplication of the method is difficult, QA samples are not usually employed.

j. Screening Data. Samples collected as part of a screening program usually do not employ QA sample collection. This would include screening data generated from immunoassay test kits, x-ray fluorescence, colorimetric, or field gas chromatography analyses.

1-8. Fraud Deterrence. Although not specifically designed to detect fraud, the USACE QC/QA program of laboratory validation, auditing (laboratory and field), sample receipt inspections, and review, verification, and/or validation of project, QC and QA data serves as a creditable deterrent to fraud.

1-9. Training. A number of training sessions are available (both internal and external to USACE) to provide the needed understanding of the principles and proper execution of the USACE CDQM program. USACE staff are encouraged to avail themselves of this training as appropriate.

1-10. Procedures for CDQM by Project Phase. The following outlines the procedures for CDQM for the investigative, pre-design and design, and remedial or removal action phases of the USACE HTRW program. The outlined activities demonstrate use of the six primary CDQM activities described in Section 1-5 and the technical document review process for Category A projects described in Section 1-4.b.

a. Investigative Phase. The investigative phase of the HTRW program consists of site characterization, engineering analysis, risk assessment, potentially responsible party (PRP) data gathering, and regulatory analysis. The investigative phases from the CERCLA process are the PA/SI and the Remedial Investigation/Feasibility Study (RI/FS). The investigative phase from the RCRA process are the RCRA Facility Assessment (RFA), RCRA Facility Investigation (RFI) and the Corrective Measures Study (CMS). The investigative phase of the FUDS program is

executed consistent with, but not identical to, the CERCLA process. For non-time critical removal actions, a PA/SI is performed initially and is followed by an Engineering Evaluation/Cost Analysis (EE/CA). The EE/CA takes the place of the RI/FS.

- (1) HTRW design district writes Scope of Services. For Category B projects (see paragraph 1-4.b.(2)), the HTRW design district submits Scope of Services to HTRW-CX for review.
- (2) HTRW design district solicits prime contractor services.
- (3) HTRW design district negotiates and awards contract or delivery order.
- (4) Prime contractor identifies primary laboratory to the district.
- (5) The PM, TM or district project chemist requests validation of the primary laboratory by the HTRW-CX via electronic mail or facsimile.
- (6) The HTRW-CX follows the process described in EM 200-1-1 to validate the laboratory. If the laboratory has not previously been validated by the HTRW-CX, the district project chemist should screen the laboratory to determine if its technical capabilities merit validation. Depending on the laboratory's validation status, some or all of the following procedures may be omitted. If requested by the HTRW-CX, the primary laboratory submits its Laboratory Quality Management Manual (LQMM) or Quality Assurance Plan (QAP), a representative SOP; to demonstrate the laboratory has the capability to run the required methods, and petroleum hydrocarbon SOPs (if necessary) to the HTRW-CX. Based on satisfactory review of the QAP and SOPs, PE samples are sent if available. The laboratory is then inspected by HTRW-CX. Personnel from the HTRW design district and CMQAL will be notified of a scheduled inspection and may assist with this process. If the laboratory fails to become validated, another laboratory should be selected.
- (7) The prime contractor submits the SAP, consisting of a Quality Assurance Project Plan (QAPP) and a Field Sampling Plan (FSP), for HTRW design district's approval. Other environmental regulatory programs may require different documentation than a SAP. For Category B projects (see paragraph 1-4.b.(2)), the HTRW design district sends SAP to HTRW-CX and HTRW-CX reviews the SAP and makes recommendations to HTRW design district.
- (8) From the SAP, the HTRW design district or the CMQAL makes an estimate of the cost of QA sample analysis. The budgeted amount must be funded by the HTRW design district to the CMQAL prior to sending samples for QA analysis. The QA laboratory must also be notified that QA samples will be sent. The HTRW design district must provide the QA laboratory with the following information: 1) project name; 2) approximate sampling dates; 3) number of samples; 4) matrix (matrices); 5) analyses; 6) DQOs; and 7) turnaround time. An example checklist to

submit this information is included as Figure 1-1.

(9) Field work begins after SAP is approved by the HTRW design district.

(10) The TM or district project chemist coordinates with the prime contractor for field and laboratory activities. Samples are collected in the field with project and QC samples sent to the primary laboratory and QA samples sent to the QA laboratory. QA samples are sent to the QA laboratory throughout the duration of the sampling effort or as defined by the project objectives.

(11) The primary and QA Labs should be notified upon final shipment of project samples.

(12) Prime contractor's analytical results are submitted to the HTRW design district within the time frame identified in the contract. The analytical results that correlate with the QA samples are sent to the CMQAL at the same time.

(13) The QA laboratory or another qualified entity prepares the CQAR and submits it to the HTRW design district and the HTRW-CX. The HTRW design district provides the CQAR to the prime contractor for inclusion in the project report.

(14) Prime contractor prepares the draft project report and submits it to the HTRW design district. The project report should include the CQAR, as well as the contractor's assessment of the primary laboratory data. The report is reviewed by the same office(s) that reviewed the SAP.

(15) District project chemist writes the CDQAR addressing data useability and DQO attainment from information received from the prime contractor and the CQAR. CDQARs must be prepared for all in-house and contractor executed projects. CDQARs will be sent by the HTRW design district to the HTRW-CX for all projects.

b. Pre-Design and Design Phase. The pre-design and design phase of the HTRW program consists of remedial action selection and design. The CERCLA design phase is remedial design (RD). The corresponding RCRA phase is called the Corrective Measures Design (CMD). The following outline applies when the design is prepared by a contractor. Modifications will be required if the design is performed in-house.

(1) Design district writes Scope of Services. For Category B projects (see paragraph 1-4.b.(2)), the HTRW design district submits Scope of Services to HTRW-CX for review.

(2) Design district solicits prime contractor services.

(3) Design district negotiates and awards prime contractor design contract or delivery order.

(4) If investigative activities are included in the design contract, steps 4-15 of paragraph 1-10.a. should be followed.

(5) Prime contractor submits Design Analysis Reports that contains a section that specifically addresses chemical quality management concerns. The prime contractor also submits plans and specifications which include chemical quality management at the preliminary, intermediate, and final phases. For the Total Environmental Restoration Contract (TERC), the prime contractor submits a Work Plan for each delivery order. All these documents are submitted by the prime contractor for HTRW design district's approval. The chemical section of the plans and specifications or work plan should give the construction contractor instructions for writing the SAP in addition to including all necessary site-specific chemical detail. For Category B projects (see paragraph 1-4.b.(2)), the HTRW design district submits these documents (to include the design analysis, plans and specifications, and the work plan) to the HTRW-CX for technical review, and comments are sent back to the design district.

(6) Design district assures that appropriate comments are addressed and incorporated into the documents. Revised documents and annotated comments are sent to the offices generating comments at the next submittal stage.

(7) Final (100%) plans and specifications are approved by the design district. From the contract specifications, a preliminary estimate is made of the funding required to support specified QA activities. The district advertises and awards the construction contract. For a Request for Proposal (RFP), the district solicits proposals from construction contractors. The district technical team evaluates the proposals and selects a contractor. Several other contracting mechanisms (*i.e.*, Invitation for Bid (IFB), cost-plus, *etc.*) exist that could be used instead of the RFP.

c. Remedial or Removal Action Phase. Many construction offices do not have sufficient chemistry training to make the decisions necessary to support the HTRW program. These construction offices should rely on basic chemistry support from resources at their HTRW design district, CMQAL or the HTRW-CX. Several guidance documents integrate chemical data QA for remedial actions into existing QA procedures for construction:

ER 415-1-10 Contractor Submittal Procedures

ER 1180-1-6 Quality Management

EP 715-1-2 A Guide to Effective Contractor Quality Control

CEGS 01451 Contractor Quality Control

CEGS 01450 Chemical Data Quality Control

- (1) District representative requests validation of the primary laboratory by the HTRW-CX via electronic mail or facsimile.
- (2) See paragraph 1-10.a(6) for the process and procedures for laboratory validation.
- (3) The designated HTRW design district, CMQAL or HTRW-CX (depending upon which organization is providing the basic chemistry support for the project) assists the Construction District in reviewing the SAP and makes recommendations to the construction district. Construction district approves or disapproves the prime contractor's SAP.
- (4) See paragraph 1-10.a.(8) for estimating and funding QA analysis.
- (5) Construction begins after SAP and prime contractor's laboratory are approved.
- (6) The construction representative coordinates with the prime contractor for field and laboratory activities. See paragraph 1-10.a.(10) for laboratory coordination and shipment. QA samples are sent to the QA laboratory throughout the duration of the sampling effort or as defined by the contract specifications.
- (7) Prime contractor notifies the primary laboratory and the CMQAL when the final project samples have been sent.
- (8) Prime contractor's analytical results are submitted to the construction office for transmittal to the CMQAL within the time frame identified in the contract.
- (9) The QA laboratory or another qualified entity prepares the CQAR and submits it to the construction district, associated HTRW design district and the HTRW-CX. The construction district provides the CQAR to the prime contractor for inclusion in the project report.
- (10) The prime contractor submits the project report to the construction district. The project report includes the CQAR, as well as the contractor's evaluation of the primary laboratory data. The report is reviewed by the construction representative with assistance from HTRW design district, CMQAL, or HTRW-CX staff, if requested.
- (11) Construction district writes the CDQAR addressing contract compliance, data useability and DQO attainment from information provided by the construction contractor and the CQAR. CDQARs will be sent by the construction district to the associated HTRW design district, and HTRW-CX for all projects.

1-11. Data Management and Archive Process. The prime contractor and laboratories are responsible for generating, controlling and archiving laboratory and field records for all projects. This information should be maintained with a system that is effective for retrieval of any documentation that affects the reported results. The TM determines whether supporting data should be transferred from the prime contractor to the USACE upon contract completion or remain the prime contractor's responsibility for archiving the data. This includes record generation and control, security, and maintenance of all project related documents. The duration of laboratory data and field record retention should be specified as part of the project DQOs.

a. Laboratory. The laboratory prepares and retains full analytical and QC documentation that can be tracked from initiation to disposal for each sample. The following minimum records should be stored for each project: 1) original work order, CoC, and other pertinent documents received with the samples, 2) communications between the laboratory, field, and the customer, 3) any associated corrective actions, 4) laboratory data packages, 5) finalized data report, 6) laboratory log books, and 7) electronic data. The laboratory should also maintain its QAP and relevant SOPs for the methods performed.

b. Field. Project-specific records that relate to field work performed should also be retained. These records may include correspondence, CoC records, field notes, and reports issued as a result of the work. In addition, records that document all field operations should be retained. This may include equipment performance records, maintenance logs, personnel files, general field procedures, and corrective action reports. For field operations hard copy records are acceptable.

Laboratory Notification Information Checklist

- project name
- project location
- general project objectives
- intended use(s) of data
- name and address of sampler's firm
- approximate sampling dates
- approximate number of samples, by matrix
- required data package turnaround time
- funding source (contract number and/or MIPR number)
- name, phone and facsimile numbers for person to be contacted by the laboratory if there are problems with the sample shipment
- name and address of primary (contractor's) laboratory (to be included in notification to CMQAL)
- project specific requirements
 - analysis method(s)
 - matrices
 - extraction method(s)
 - required sensitivity (reporting limits)
 - required precision
 - required accuracy
 - required comparability
- sample retention after analysis is complete
- disposition of samples after required retention time
- special data reporting requirements
- any special needs or comments (*i.e.*, unusual target analytes)
- revision number of notification

Figure 1-1

Table 1-1 CDQM Activities

QA Activity	Characteristics	Cost	Project Phase(s) In Which Commonly Used					
			PA	SI or RFA	RI/PS or RFI/ CMS	RD or CMD	RA or CMI	O&M
Lab Validation	Provides assurance that lab has necessary personnel & equipment to produce data of known and adequate quality	*	X	X	X	X	X	X
Document Review	Checks technical adequacy of project documents and monitors program compliance	\$ to \$\$	X	X	X	X	X	X
Sample Handling QA	Quick feedback regarding problems with sample shipments	\$	X	X	X		X	X
QA Sample Collection & Analysis	Detects analytical problems and may salvage data usability	\$\$ to \$\$\$\$\$	X	X	X		X	X
CQAR Preparation	Monitors intra- and inter-laboratory data comparability	\$ to \$\$\$\$	X	X	X		X	X
CDQAR Preparation	Checks contract compliance, data usability, and DQO attainment	\$ to \$\$	X	X	X		X	X
Performance Evaluation Samples	Provides assurance that lab correctly identifies and quantitates analytes of interest	\$ to \$\$\$\$\$	X		X			
Primary Lab Data Review	Monitors precision, accuracy, completeness, reproducibility, and sensitivity of primary data	\$ to \$\$\$	X	X	X		X	
Data Validation	Rigorous evaluation of data according to explicit EPA or other agency guidelines	\$\$ to \$\$\$\$	X		X			
Field Audits	Real-time oversight of accuracy & adequacy of field activities	\$ to \$\$	X		X		X	
Laboratory Audits	Unannounced audits verify lab's ability to produce acceptable data	\$ to \$\$			X			X
Tape Audits	Raw data review verifies data reduction procedures of lab	\$\$\$\$ to \$\$\$\$\$			X			

Cost ratings range from \$ to \$\$\$\$\$. \$ corresponds to <\$1000, while \$\$\$\$\$ corresponds to >\$10,000.

* For most programs, the cost of laboratory validation is funded by the HTRW-CX, not by the district or division. If laboratory validation is requested for a project that is outside those programs for which there is validation funding by the HTRW-CX, validation costs would typically be in the range \$\$ to \$\$\$.

ATTACHMENT B

Personnel Qualifications

Mark Bagel, PG

Experience

Mr. Bagel has 26 years of professional experience in environmental and engineering-related investigations in the following areas: contamination assessments of petroleum-contaminated, Superfund, and Resource Conservation and Recovery Act (RCRA) sites; solid waste and industrial waste landfill permitting and contamination assessments; stabilization study of organic sludges; oversight and evaluation of landfill liner and cover constructions; management and QC coordinator of ordnance-contaminated sites; supervision of geotechnical soils and materials testing laboratory; soil sampling and laboratory analysis for geotechnical soils investigations; preparation of Comprehensive Quality Assurance Plan and other QC and safety programs and plans, construction materials testing and analysis, and construction/demolition site QC manager.

Education

BA, Geology, State University of New York, 1978

- Geologic Studies, University of Houston, 1984
- Engineering Studies, University of Houston, 1986
- Engineering Studies, University of Florida, 1990 to 1994

Karen Hatfield, MS

Areas of Specialization

- Environmental Chemistry
- Project Management
- Data Validation
- Quality Control / Quality Assurance

Experience

Ms. Hatfield has 32 years of environmental and chemistry consulting experience in project management, environment chemistry, and environmental quality control. Her experience includes data validation, completion of Laboratory Quality Assurance Plans, data validation, and preparation of Quality Control Summary Reports. Her experience and insight has provided excellent and timely reports and plans to clients, meeting the client requirements within budget and on schedule.

Education

MS, Environmental Engineering Sciences, University of Florida
BS, Chemistry, University of Florida, 1971

Gary H. Tourtellotte, MS

Areas of Specialization

- NEPA Documentation
- Natural Resources Evaluations and Management
- Ecology of Marine and Freshwater Aquatic Communities
- Water Quality
- Design of Sampling Programs
- Data Analysis / Statistics

Experience

Mr. Tourtellotte has 25 years of experience in environmental consulting. His experience includes coastal resource evaluations and management, ecology of marine and freshwater aquatic communities, water quality, design of sampling programs, data analysis / statistics, NEPA documentation, power plant siting and impact analysis, phosphate mine impact analysis, and dredge and fill permitting. He has performed numerous studies of estuarine/marine and freshwater systems along the East Coast of the U.S. and the Gulf of Mexico. These studies have been in support of NPDES permitting, dredge and fill projects, siting of industrial facilities, environmental impact statements, environmental assessments, biological assessments, contamination and baseline surveys, and ecological risk assessment.

Education

MS, Oceanography, Old Dominion University, Norfolk, VA, 1979

BS, Biology, University of Miami, 1974

Environmental Toxicology and Risk Assessment Short Course, Duke University, School of the Environment, 1992

STL Personnel Profiles

Prepared for Ellis-Culebra Project

STL Chicago

Project Manager, Nancy S. McDonald

Ms. McDonald holds a B.A. in Biology from Augustana College. She has 16 years environmental laboratory experience. Her experience includes project management for industrial and municipal clients for wastewater discharge (NPDES), groundwater monitoring, soil and waste characterization. Ms. McDonald also provides support for engineering consultants contracted by the US Army Corps of Engineers (USACE) and US Navy. She has experience meeting the criteria in the USACE Louisville Chemistry Guidelines (LCG) and various state programs, including the Illinois EPA's Tiered Approach to Cleanup Objectives (TACO). Ms. McDonald previously served as the laboratory's proposal coordinator and initially worked in the field sampling department.

Quality Manager, Terese A. Preston

Ms. Preston has a B.A. degree in Biology from Jamestown College, Jamestown, ND. Ms. Preston is STL Chicago's Quality Manager. She has 21 years experience in the environmental laboratory industry and has been with the Chicago laboratory since 1984. She is experienced in environmental laboratory quality assurance practices, management, communications and analytical chemistry. Ms. Preston is responsible for the development and management of the laboratory's quality assurance program. She has considerable experience in preparing and implementing laboratory quality assurance and project specific plans, which include RI/FS and other projects for both the IEPA and U.S. EPA Region V contracts. She is an experienced laboratory data auditor, and performs contract and method compliance monitoring.

Customer Service Manager, Eric A. Lang

Mr. Lang has a M.B.A. with a concentration in Project Management from Keller Graduate School of Management and a B.S. in Biology/Chemistry from Eastern Michigan University. He has 21 years experience in the environmental laboratory industry, with 13 years experience as a project manager. Mr. Lang serves as project manager for several large national industrial corporations and engineering clients, including work performed under U.S. Navy and U.S. Army Corps of Engineers contracts. He previously managed the laboratory's metals section where his responsibilities included implementation of quality control procedures, data package review, training of analysts in instrument operation, supervision of metals personnel and method development for metals analyses. Mr. Lang initially served as a chemist, performing inorganic analyses on water and waste samples.

Chromatography Laboratory Section Manager, Patti Gibson

Ms. Gibson has a B.S. in Biology (1984) from Oakland City College. She has 16 years experience in the environmental laboratory industry and has been with the Chicago laboratory since 1989. Ms. Gibson has been Unit Leader for the Pesticide/PCB group for approximately three years, with six years experience in GC analysis of pesticides and PCBs and has recently been promoted to Section Manager of Chromatography. She has experience utilizing methods from SW846, 40CFR, CLP OLM03.2 and OLC02.1. Her responsibilities included sample analysis, supervision of day to day operations and data review. She served in the metals department for one year performing various duties, including metals analysis on a Flame AA, metals digestions and TCLP extractions. Her initial duties were in log-in, where she worked for one year receiving, tracking

and storing samples, entering log-in data in the computer system and reviewing associated paperwork.

Metals Laboratory Section Manager, Jodi L. Gromala

Ms. Gromala has a B.S. degree in Biology from Bradley University. She has over 19 years of laboratory experience. Ms. Gromala has an extensive background in inorganic metals analyses using SW-846 Methods, U.S. EPA CLP ILM04.0, Standard Methods for the Examination of Waters, EPA 600 Series Methods and ASTM Methods of Analyses. Previously Ms. Gromala was Unit Leader for the Metals Department and her experience includes ICP, GFAA and Hg analysis and Sample Preparation. She has been with the Chicago laboratory since 1988.

Sample Receipt Manager, Jeffrey A. James

Mr. James holds a B.A. in Music Education from Eastern Illinois University. He has 16 years of environmental laboratory experience and has been with the Chicago laboratory since 1989. Mr. James previously served as a project manager for several large industrial programs, including NPDES, municipal and wastewater clients. He now manages the Sample Receipt Department. His expertise includes consultation on the most cost effective approaches for environmental sampling. His experience includes sampling of a variety of matrices, well development, combustible gas monitoring, and field data collection. He has extensive training in environmental health and safety procedures, and hazardous shipping. Mr. James previously served as Field Sampling Unit Leader, Bottle Project Unit Leader, Facility Manager, and Field Technician.

STL Sacramento

Quality Assurance Manager, Pam Schemmer

Ms. Schemmer has a BS degree in Chemistry from the University of Iowa. She brings more than 12 years of experience in the analytical industry to her current role as Quality Assurance Manager. She began her career as an analyst, and quickly advanced into increasingly responsible management positions. Her attention to detail and excellent communication skills, make Ms. Schemmer an excellent contributor to our STL team. As a senior member of management Ms. Schemmer directs and monitors quality assurance activities at the Sacramento facility. She is responsible for reports to management, client concerns, project plan review, lab performance review, and review of procedures that will ensure the production of data of a defined quality.

Manager of Project Management, Robert Hrabak

Mr. Hrabak has a BS in Biological Sciences from the University of California. Over the past 16 years Mr. Hrabak has specialized in the area of the Advanced Technology Group, focusing on low-resolution dioxins and specialty chemicals. His extensive technical knowledge in these areas and excellent organizational skills, make him the ideal choice to manage these projects in the laboratory. His leadership ability was recognized in 1991 when he was included in a team of employees evaluating the application of high performance work teams in our environmental laboratory. These same skills were utilized in 1999 when he was chosen to coordinate the implementation of a new LIMS at the laboratory facility. In 1994 his customer focus and service skills were recognized by awarding him with the Presidential Exceptional Achievement Award, which is presented annually to only one percent of the company's employees.

ATTACHMENT C

Laboratory Certifications

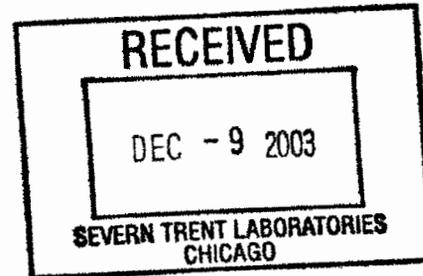


REPLY TO
ATTENTION OF

DEPARTMENT OF THE ARMY
U.S. ARMY CORPS OF ENGINEERS
12565 WEST CENTER ROAD
OMAHA NE 68144-3869

December 1, 2003

Hazardous, Toxic and Radioactive Waste
Center of Expertise



Ms. Donna J. McCarthy
STL Chicago
2417 Bond Street
University Park, IL 60466-3182

Dear Ms. McCarthy:

This correspondence addresses the recent evaluation of STL Chicago of University Park, IL by the U.S. Army Corps of Engineers (USACE) for chemical analysis in support of the USACE Hazardous, Toxic and Radioactive Waste Program.

Your laboratory is now validated for the parameters listed below:

METHOD ⁽¹⁾	PARAMETER	MATRIX ⁽¹⁾
300.0	Anions ⁽⁶⁾	Water ⁽³⁾
300.0	Anions ⁽⁶⁾	Solids ⁽⁴⁾
9010B/9014	Cyanide	Water ⁽³⁾
9010B/9014	Cyanide	Solids ⁽³⁾
8330	Explosives	Water ⁽⁴⁾
8330	Explosives	Solids ⁽³⁾
8151A	Herbicides	Water ⁽³⁾
8151A	Herbicides	Solids ⁽³⁾
7196A	Hexavalent Chromium	Water ⁽³⁾
3060A/7196A	Hexavalent Chromium	Solids ⁽³⁾
1664A	Oil & Grease	Water ⁽³⁾
9071B	Oil & Grease	Solids ⁽⁴⁾
3510C/3520C/8081A	Organochlorine Pesticides	Water ⁽³⁾
3541/3550B/8081A	Organochlorine Pesticides	Solids ⁽³⁾
9065/9066	Phenolics	Water ⁽³⁾
9065/9066	Phenolics	Solids ⁽⁴⁾

3510C/3520C/8082	Polychlorinated Biphenyls	Water ⁽³⁾
3541/3550B/8082	Polychlorinated Biphenyls	Solids ⁽³⁾
3510C/3520C/8310	Polynuclear Aromatic Hydrocarbons	Water ⁽³⁾
3541/3550B/8310	Polynuclear Aromatic Hydrocarbons	Solids ⁽³⁾
3510C/3520C/8270C	Semivolatile Organics	Water ⁽³⁾
3541/3550B/8270C	Semivolatile Organics	Solids ⁽³⁾
3510C/3520C/8270C SIM	Semivolatile Organics and Polychlorinated Naphthalenes	Water ⁽⁷⁾
3541/3550B/8270C SIM	Semivolatile Organics and Polychlorinated Naphthalenes	Solids ⁽⁷⁾
3005A/3010A/6010B/ 3020A/7000A Series	TAL Metals ⁽⁵⁾	Water ⁽³⁾
3050B/6010B/7000A Series	TAL Metals ⁽⁵⁾	Solids ⁽³⁾
9060	Total Organic Carbon	Water ⁽³⁾
9060M	Total Organic Carbon	Solids ⁽⁴⁾
3510C/3520C/Mod 8015B	TPH - DRO	Water ⁽³⁾
3541/3550B/Mod 8015B	TPH - DRO	Solids ⁽³⁾
5030B/5035/Mod 8015B	TPH - GRO	Water ⁽³⁾
5035/Mod 8015B	TPH - GRO	Solids ⁽³⁾
5030B/5035/8260B	Volatile Organics	Water ⁽³⁾
5035/8260B	Volatile Organics	Solids ⁽³⁾

- Remarks:
- 1) Sample preparation methods have been added to reflect program policy change.
 - 2) "Solids" includes soils, sediments, and solid waste.
 - 3) The laboratory has successfully analyzed a Proficiency Testing (PT) sample for this method/matrix.
 - 4) Approval is based on review of SOPs, performance data, and PT results of other matrices.
 - 5) TAL Metals: Aluminum, antimony, arsenic, barium, beryllium, cadmium, calcium, chromium, cobalt, copper, iron, lead, magnesium, manganese, mercury, nickel, potassium, selenium, silver, sodium, thallium, vanadium, and zinc.
 - 6) Anions: Chloride, fluoride, sulfate, nitrate, nitrite, and ortho-phosphate.
 - 7) Approval for polychlorinated naphthalene is based on review of SOPs and performance data only.

Enclosed for your information is a copy of the Laboratory Inspection and Evaluation Report. Please see the enclosed Laboratory Inspection and Evaluation Report for unresolved action items.

Based on the successful analysis of the National Environmental Laboratory Accreditation Conference Proficiency Testing samples for the appropriate fields of testing, the results of the laboratory inspection, and your Corrective Action Report, your laboratory will be validated for sample analysis by the methods listed above. The evaluation, which was conducted for your facility, is based substantially on ISO Guide 25 (General Requirements for the Competence of Testing Laboratories) and USACE Engineering Manual (EM) 200-1-3, Appendix I (Shell for Analytical Chemistry Requirements). The period of validation is 24 months and expires on December 1, 2005.

The USACE reserves the right to conduct additional laboratory inspections or to suspend validation status for any or all of the listed parameters if deemed necessary. It should be noted that your laboratory may not subcontract USACE analytical work to any other laboratory location without the approval of this office. This laboratory validation does not guarantee the delivery of any analytical samples from a USACE Contracting Officer Representative.

Any questions or comments can be directed to Chung-Rei Mao at (402) 697-2570. General questions regarding laboratory validation may be directed to the Laboratory Validation Coordinator at (402) 697-2574.

Sincerely,



Marcia C. Davies, Ph.D.
Director, USACE Hazardous,
Toxic and Radioactive Waste
Center of Expertise

Enclosure



**STATE OF ILLINOIS
ENVIRONMENTAL PROTECTION AGENCY**



ENVIRONMENTAL LABORATORY ACCREDITATION

is hereby granted to

**STL CHICAGO
2417 BOND STREET
UNIVERSITY PARK, IL 60466-3182**

ACCREDITATION NUMBER #100201



According to the Illinois Administrative Code, Title 35, Subtitle A, Chapter II, Part 186, ACCREDITATION OF LABORATORIES FOR DRINKING WATER, WASTEWATER AND HAZARDOUS WASTES ANALYSIS, the State of Illinois formally recognizes that this laboratory is technically competent to perform the environmental analyses listed on the scope of accreditation detailed below.

The laboratory agrees to perform all analyses listed on this scope of accreditation according to the Part 186 requirements and acknowledges that continued accreditation is dependent on successful ongoing compliance with the applicable requirements of Part 186. Please contact the Illinois EPA Environmental Laboratory Accreditation Program (IL ELAP) to verify the laboratory's scope of accreditation and accreditation status. Accreditation by the State of Illinois is not an endorsement or a guarantee of validity of the data generated by the laboratory.

Scott D. Siders

Scott D. Siders
Accreditation Officer
Environmental Laboratory Accreditation Program

Certificate No.: 001027
Expiration Date: 04/30/2005
Issued On: 04/27/2004

**State of Illinois
Environmental Protection Agency**

Certificate No.:

001027

Awards the Certificate of Approval

STL Chicago
2417 Bond Street
University Park, IL 60466-3182

According to the Illinois Administrative Code, Title 35, Subtitle A, Chapter II, Part 186, ACCREDITATION OF LABORATORIES FOR DRINKING WATER, WASTEWATER AND HAZARDOUS WASTES ANALYSIS, the State of Illinois formally recognizes that this laboratory is technically competent to perform the environmental analyses listed on the scope of accreditation detailed below.

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Drinking Water, inorganic

SM2120B, 18Ed

Color

SM2130B, 18Ed

Turbidity

SM2150B, 18Ed

Odor

SM2320B, 18Ed

Alkalinity

SM2330B, 18Ed

Corrosivity (Langlier Index)

SM2340B, 18Ed

Hardness

SM2340C, 18Ed

Hardness

SM2510B, 18Ed

Conductivity

SM2540C, 18Ed

Total Dissolved Solids

SM4500Cl-F, 18Ed

Chlorine

SM4500CN-CE18Ed

Cyanide

SM4500F-C, 18Ed

Fluoride

SM4500H-B, 18Ed

Hydrogen ion (pH)

SM4500NO2B, 18Ed

Nitrite

SM4500NO3F, 18Ed

Nitrate

SM4500P-E, 18Ed

Orthophosphate

SM5310C, 19Ed

Dissolved Organic Carbon

Total Organic Carbon (TOC)

State of Illinois
Environmental Protection Agency
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STL Chicago
 2417 Bond Street
 University Park, IL 60466-3182

Drinking Water, Inorganic

USEPA150.1

Hydrogen ion (pH)

USEPA180.1

Turbidity

USEPA200.7R4.4

Aluminum

Beryllium

Chromium

Iron

Nickel

Sodium

Arsenic

Cadmium

Copper

Magnesium

Silica

Zinc

Barium

Calcium

Hardness (calc.)

Manganese

Silver

USEPA200.9R2.2

Antimony

Chromium

Silver

Arsenic

Lead

Thallium

Cadmium

Selenium

USEPA245.1R3.0

Mercury

USEPA300.0R2.1

Chloride

Nitrite

Fluoride

Orthophosphate

Nitrate

Sulfate

USEPA353.2R2.0

Nitrate

Hazardous and Solid Waste, Inorganic

1010

Ignitability

1311

TCLP (Organic and Inorganic)

1312

Synthetic Precipitation Leaching Procedure

5050

Bomb Preparation

6010B

Aluminum

Barium

Cadmium

Cobalt

Lead

Molybdenum

Selenium

Sodium

Tin

Zinc

Antimony

Beryllium

Calcium

Copper

Magnesium

Nickel

Silica

Strontium

Titanium

Arsenic

Boron

Chromium

Iron

Manganese

Potassium

Silver

Thallium

Vanadium

7041

Antimony

State of Illinois
Environmental Protection Agency
Awards the Certificate of Approval

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001027

STL Chicago
2417 Bond Street
University Park, IL 60466-3182

Hazardous and Solid Waste, Inorganic

7060A

Arsenic

7131A

Cadmium

7191

Chromium

7196A

Chromium VI

7421

Lead

7470A

Mercury

7471A

Mercury

7740

Selenium

7761

Silver

7841

Thallium

9010B

Cyanide

9014

Cyanide

9020B

TOX - Total Organic Halides

9030B

Sulfides

9034

Sulfides

9038

Sulfate

9040B

Hydrogen Ion (pH)

9041A

Hydrogen Ion (pH)

9045C

Hydrogen Ion (pH)

9050A

Specific Conductance

9056

Bromide

Chloride

Fluoride

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STL Chicago
 2417 Bond Street
 University Park, IL 60466-3182

Hazardous and Solid Waste, Inorganic

9056

Nitrate

Nitrite

Phosphate

Sulfate

9060

Total Organic Carbon (TOC)

9066

Phenolics

9071B

Oil and Grease Extractable

9081

Cation-exchange Capacity

9095A

Paint Filter

9251

Chloride

Chapter 7/9014

Reactive Cyanide

Chapter 7/9034

Reactive Sulfide

Hazardous and Solid Waste, Organic

8015B

Diesel range organics (DRO)

Gasoline range organics (GRO)

8081A

4,4'-DDD

4,4'-DDE

4,4'-DDT

Alachlor

Aldrin

alpha-BHC

alpha-Chlordane

Atrazine

beta-BHC

Chlordane - not otherwise specified

delta-BHC

Dieldrin

Endosulfan I

Endosulfan II

Endosulfan sulfate

Endrin

Endrin aldehyde

Endrin ketone

gamma-BHC (Lindane)

gamma-Chlordane

Heptachlor

Heptachlor epoxide

Isodrin

Kepone

Methoxychlor

Simazine

Toxaphene

8082

PCB-1016

PCB-1221

PCB-1232

PCB-1242

PCB-1248

PCB-1254

PCB-1260

8141A

Dimethoate

Disulfoton

Famphur

Parathion ethyl

Parathion methyl

Phorate

Sulfotepp

Thionazine (Zinophos)

8151A

2,4,5-T

2,4,5-TP (Silvex)

2,4-D

2,4-DB

4-Nitrophenol

Dalapon

Dicamba

Dichloroprop

Dinoseb

Pentachlorophenol

Picloram

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STL Chicago
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Hazardous and Solid Waste, Organic

1,1,1,2-Tetrachloroethane
1,1,2-Trichloroethane
1,1-Dichloropropene
1,2,4-Trichlorobenzene
1,2-Dibromoethane (EDB)
1,2-Dichloropropane
1,3-Dichlorobenzene
1-Chlorohexane
2-Butanone (Methyl ethyl ketone, MEK)
2-Chlorotoluene
2-Methylnaphthalene
4-Methyl-2-pentanone (Methyl isobutyl ketone, MIBK)
Acrolein (Propenal)
Benzene
Bromodichloromethane
Carbon disulfide
Chlorodibromomethane (Dibromochloromethane)
Chloromethane
Dibromomethane
Ethyl acetate
Ethylbenzene
Isopropylbenzene
Methyl ethyl ketone
Methyl methacrylate
Naphthalene
o-Toluidine
p-Isopropyltoluene
sec-Butylbenzene
Tetrachloroethene
trans-1,2-Dichloroethene
Trichloroethene
Trichlorotrifluoromethane
Vinylidene chloride

8260B

1,1,1-Trichloroethane
1,1-Dichloroethane
1,2,3-Trichlorobenzene
1,2,4-Trimethylbenzene
1,2-Dichlorobenzene
1,3,5-TCB
1,3-Dichloropropane
1-Chlorohexane
2-Chloro-1,3-butadiene (Chloroprene)
2-Hexanone
2-Nitropropane
Acetone
Acrylonitrile
Bromobenzene
Bromoform
Carbon tetrachloride
Chloroethane
cis-1,2-Dichloroethene
Dichlorodifluoromethane
Ethyl ether
Hexachlorobutadiene
Malononitrile
Methyl iodide (Iodomethane)
Methyl-t-butyl ether
n-Butylbenzene
o-Xylene
Propionitrile (Ethyl cyanide)
Styrene
Tetrahydrofuran
trans-1,3-Dichloropropene
Trichlorofluoromethane
Vinyl acetate
Xylenes (Total)

1,1,2,2-Tetrachloroethane
1,1-Dichloroethene
1,2,3-Trichloropropane
1,2-Dibromo-3-chloropropane (DBCP)
1,2-Dichloroethane
1,3,5-Trimethylbenzene
1,4-Dichlorobenzene
2,2-Dichloropropane
2-Chloroethyl vinyl ether
2-Methyl-1-propanol (Isobutyl alcohol)
4-Chlorotoluene
Acetonitrile
Allyl chloride
Bromochloromethane
Bromomethane
Chlorobenzene
Chloroform
cis-1,3-Dichloropropene
Dichloromethane (Methylene chloride)
Ethyl methacrylate
Isopropyl ether
Methacrylonitrile
Methyl isobutyl ketone
m-Xylene
n-Propylbenzene
Pentachloroethane
p-Xylene
tert-Butylbenzene
Toluene
trans-1,4-Dichloro-2-butene
Trichlorotrifluoroethane
Vinyl chloride

8270C

1,2,4,5-Tetrachlorobenzene
1,2-Diphenylhydrazine
1,3-Dinitrobenzene (1,3-DNB)
1,4-Naphthoquinone
2,3,4,6-Tetrachlorophenol
2,4-Dichlorophenol
2,4-Dinitrotoluene (2,4-DNT)
2-Acetylaminofluorene
2-Methylnaphthalene
2-Nitroaniline
3,3'-Dimethylbenzidine
4,6-Dinitro-2-methylphenol

1,2,4-Trichlorobenzene
1,3,5-Trinitrobenzene (1,3,5-TNB)
1,4-Dichlorobenzene
1,4-Phenylenediamine
2,4,5-Trichlorophenol
2,4-Dimethylphenol
2,6-Dichlorophenol
2-Chloronaphthalene
2-Methylpyridine (2-Picoline)
2-Nitrophenol
3-Methylcholanthrene
4-Aminobiphenyl

1,2-Dichlorobenzene
1,3-Dichlorobenzene
1,4-Dioxane
1-Naphthylamine
2,4,6-Trichlorophenol
2,4-Dinitrophenol
2,6-Dinitrotoluene (2,6-DNT)
2-Chlorophenol
2-Naphthylamine
3,3'-Dichlorobenzidine
3-Nitroaniline
4-Bromophenyl phenyl ether

State of Illinois
Environmental Protection Agency
Awards the Certificate of Approval

Certificate No.: 001027

STL Chicago
 2417 Bond Street
 University Park, IL 60466-3182

Hazardous and Solid Waste, Organic

4-Chloroaniline	8270C	4-Chloro-3-methylphenol
4-Nitrophenol	4-Chlorophenyl phenyl ether	4-Nitroaniline
7,12-Dimethylbenz(a)anthracene	4-Nitroquinoline-1-oxide	5-Nitro-o-toluidine
Acetophenone	Acenaphthene	Acenaphthylene
Anthracene	alpha,alpha-Dimethylphenethylamine	Aniline
Benzo(a)anthracene	Aramite	Benzidine
Benzo(g,h,i)perylene	Benzo(a)pyrene	Benzo(b)fluoranthene
Benzyl alcohol	Benzo(k)fluoranthene	Benzoic acid
Bis(2-chloroisopropyl) ether	Bis(2-chloroethoxy) methane	Bis(2-chloroethyl) ether
Carbazole	Bis(2-ethylhexyl) phthalate	Butyl benzyl phthalate
Diallate	Chlorobenzilate	Chrysene
Diethyl phthalate	Dibenz(a,h)anthracene	Dibenzofuran
Di-n-octyl phthalate	Dimethyl phthalate	Di-n-butyl phthalate
Ethyl methanesulfonate	Dinoseb	Diphenylamine
Hexachlorobenzene	Fluoranthene	Fluorene
Hexachloroethane	Hexachlorobutadiene	Hexachlorocyclopentadiene
Indeno(1,2,3-cd) pyrene	Hexachlorophene	Hexachloropropene
m-Cresol (3-Methylphenol)	Isophorone	Isosafrole
Methyl methanesulfonate	m-Dinitrobenzene	Methapyrilene
N-Nitrosodiethylamine	Naphthalene	Nitrobenzene
N-Nitrosodi-n-propylamine	N-Nitrosodimethylamine	N-Nitrosodi-n-butylamine (N-Nitrosodibutylamin
N-Nitrosomorpholine	N-Nitrosodiphenylamine	N-Nitrosomethylethylamine
o-Cresol (2-Methylphenol)	N-Nitrosopiperidine	N-Nitrosopyrrolidine
p-Cresol (4-Methylphenol)	o-Toluidine	Parathion
Pentachloronitrobenzene	p-Dimethylaminoazobenzene	Pentachlorobenzene
Phenanthrene	Pentachlorophenol	Phenacetin
Pronamide	Phenol	p-Phenylenediamine
Pyridine	Pyrene	Pyridine
	Safrole	
8310		
Acenaphthene	Acenaphthylene	Anthracene
Benzo(a)anthracene	Benzo(a)pyrene	Benzo(b)fluoranthene
Benzo(g,h,i)perylene	Benzo(k)fluoranthene	Chrysene
Dibenz(a,h)anthracene	Fluoranthene	Fluorene
Indeno(1,2,3-cd) pyrene	Naphthalene	Phenanthrene
Pyrene		
8330		
1,3,5-Trinitrobenzene (1,3,5-TBN)	1,3-Dinitrobenzene (1,3-DNB)	2,4,6-Trinitrotoluene (2,4,6-TNT)
2,4-Dinitrotoluene (2,4-DNT)	2,6-Dinitrotoluene (2,6-DNT)	2-Amino-4,6-dinitrotoluene (2-Am-DNT)
4-Amino-2,6-dinitrotoluene (4-Am-DNT)	Hexahydro-1,3,5-trinitro-1,3,5-triazine (RDX)	Methyl-2,4,6-trinitrophenylnitramine (Tetryl)
m-Nitrotoluene (3-Nitrotoluene, 3-NT)	Nitrobenzene	Octahydro-1,3,5,7-tetranitro-1,3,5,7-tetrazocine
o-Nitrotoluene (2-Nitrotoluene, 2-NT)	p-Nitrotoluene (4-Nitrotoluene, 4-NT)	

Wastewater, Inorganic

HACH8000
 Chemical Oxygen Demand (COD)
 SM3500Cr-D, 18Ed

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STL Chicago
 2417 Bond Street
 University Park, IL 60466-3182

Wastewater, Inorganic

SM3500Cr-D, 18Ed

Chromium VI

SM4500P-E, 18Ed

Orthophosphate (as P)

SM5210B, 18Ed

Biochemical Oxygen Demand (BOD)

Carbonaceous Biochemical Oxygen Demand (C

SM5310C, 18Ed

Total organic carbon (TOC)

USEPA110.2

Color

USEPA120.1

Specific Conductance

USEPA130.2

Hardness

USEPA150.1

Hydrogen Ion (pH)

USEPA160.1

Residue (TDS)

USEPA160.2

Residue (TSS)

USEPA160.3

Residue (Total)

USEPA160.4

Residue (Volatile)

USEPA1664RA

Oil and Grease

USEPA180.1

Turbidity

USEPA200.7R4.4

Aluminum

Antimony

Arsenic

Barium

Beryllium

Boron

Cadmium

Calcium

Chromium

Cobalt

Copper

Hardness (calc.)

Iron

Lead

Magnesium

Manganese

Molybdenum

Nickel

Potassium

Selenium

Silica

Silver

Sodium

Thallium

Tin

Titanium

Vanadium

Zinc

USEPA204.2

Antimony

USEPA206.2

Arsenic

USEPA213.2

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STL Chicago
 2417 Bond Street
 University Park, IL 60466-3182

<i>Wastewater, Inorganic</i>	<i>USEPA213.2</i>	<i>Cadmium</i>
<i>USEPA218.2</i>		
Chromium		
<i>USEPA239.2</i>		
Lead		
<i>USEPA245.1</i>		
Mercury		
<i>USEPA270.2</i>		
Selenium		
<i>USEPA272.2</i>		
Silver		
<i>USEPA279.2</i>		
Thallium		
<i>USEPA300.0R2.1</i>		
Bromide	Chloride	Fluoride
Nitrate	Nitrate-Nitrite (sum)	Nitrite
Orthophosphate (as P)	Sulfate	
<i>USEPA305.1</i>		
Acidity		
<i>USEPA310.1</i>		
Alkalinity		
<i>USEPA325.2</i>		
Chloride		
<i>USEPA330.4</i>		
Chlorine		
<i>USEPA335.1</i>		
Cyanide, Amenable		
<i>USEPA335.2</i>		
Cyanide		
<i>USEPA340.2</i>		
Fluoride		
<i>USEPA350.2</i>		
Ammonia		
<i>USEPA351.3</i>		
Total Kjeldahl Nitrogen		
<i>USEPA353.2</i>		
Nitrate (total)	Nitrate-Nitrite (sum)	
<i>USEPA354.1</i>		
Nitrite		
<i>USEPA360.1</i>		
Oxygen		
<i>USEPA365.2</i>		

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STL Chicago
 2417 Bond Street
 University Park, IL 60466-3182

Wastewater, Inorganic	USEPA365.2	Orthophosphate
Phosphorus		
USEPA375.4		
Sulfate		
USEPA376.1		
Sulfide		
USEPA405.1		
Biochemical Oxygen Demand (BOD)		
USEPA415.1		
Total organic carbon (TOC)		
USEPA420.2		
Phenolics		
Wastewater, Organic		
USEPA608		
4,4'-DDD	4,4'-DDE	4,4'-DDT
Aldrin	alpha-BHC	beta-BHC
Chlordane	delta-BHC	Dieldrin
Endosulfan I	Endosulfan II	Endosulfan sulfate
Endrin	Endrin aldehyde	gamma-BHC (Lindane)
Heptachlor	Heptachlor epoxide	Methoxychlor
PCB-1016	PCB-1221	PCB-1232
PCB-1242	PCB-1248	PCB-1254
PCB-1260	Toxaphene	
USEPA610		
Acenaphthene	Acenaphthylene	Anthracene
Benzo(a)anthracene	Benzo(a)pyrene	Benzo(b)fluoranthene
Benzo(g,h,i)perylene	Benzo(k)fluoranthene	Chrysene
Dibenz(a,h)anthracene	Fluoranthene	Fluorene
Indeno(1,2,3-cd) pyrene	Naphthalene	Phenanthrene
Pyrene		
USEPA624		
1,1,1-Trichloroethane	1,1,2,2-Tetrachloroethane	1,1,2-Trichloroethane
1,1-Dichloroethane	1,1-Dichloroethene	1,2-Dichlorobenzene
1,2-Dichloroethane	1,2-Dichloropropane	1,3-Dichlorobenzene
1,4-Dichlorobenzene	2-Chloroethylvinyl ether	Acrolein
Acrylonitrile	Benzene	Bromodichloromethane
Bromoform	Bromomethane	Carbon tetrachloride
Chlorobenzene	Chloroethane	Chloroform
Chloromethane	cis-1,3-Dichloropropene	Dibromochloromethane
Dichloromethane (Methylene chloride)	Ethylbenzene	Tetrachloroethene
Toluene	trans-1,2-Dichloroethene	trans-1,3-Dichloropropene
Trichloroethene	Trichlorofluoromethane	Vinyl chloride
Xylenes (total)		
USEPA625		
1,2,4-Trichlorobenzene	1,2-Dichlorobenzene	1,3-Dichlorobenzene

State of Illinois
Environmental Protection Agency
Awards the Certificate of Approval

Certificate No.:

001027

STL Chicago
2417 Bond Street
University Park, IL 60466-3182

Wastewater, Organic

2,2-Oxybis(1-chloropropane)
2,4-Dichlorophenol
2,4-Dinitrotoluene (2,4-DNT)
2-Chlorophenol
3,3'-Dichlorobenzidine
4-Chlorophenyl phenyl ether
Acenaphthylene
Benzo(a)anthracene
Benzo(g,h,i)perylene
Bis(2-chloroethoxy) methane
Chrysene
Dimethyl phthalate
Fluoranthene
Hexachlorobutadiene
Indeno(1,2,3-cd) pyrene
Nitrobenzene
N-Nitrosodiphenylamine
Phenol

USEPA625

2,4,5-Trichlorophenol
2,4-Dimethylphenol
2,6-Dinitrotoluene (2,6-DNT)
2-Methyl-4,6-dinitrophenol
4-Bromophenyl phenyl ether
4-Nitrophenol
Anthracene
Benzo(a)pyrene
Benzo(k)fluoranthene
Bis(2-chloroethyl) ether
Dibenz(a,h)anthracene
Di-n-butyl phthalate
Fluorene
Hexachlorocyclopentadiene
Isophorone
N-Nitrosodimethylamine
Pentachlorophenol
Pyrene

1,4-Dichlorobenzene
2,4,6-Trichlorophenol
2,4-Dinitrophenol
2-Chloronaphthalene
2-Nitrophenol
4-Chloro-3-methylphenol
Acenaphthene
Benzidine
Benzo(b)fluoranthene
Benzyl butyl phthalate
Bis(2-ethylhexyl) phthalate
Diethyl phthalate
Di-n-octyl phthalate
Hexachlorobenzene
Hexachloroethane
Naphthalene
N-Nitrosodi-n-propylamine
Phenanthrene



REPLY TO
ATTENTION OF

DEPARTMENT OF THE ARMY
U.S. ARMY CORPS OF ENGINEERS
12565 WEST CENTER ROAD
OMAHA NE 68144-3869

September 29, 2004

Hazardous, Toxic and Radioactive Waste
Center of Expertise

Pamela Schemmer
STL Sacramento
880 Riverside Parkway
West Sacramento, CA 95605

Dear Ms. Schemmer:

This correspondence addresses the ongoing validation status of STL Sacramento of West Sacramento, CA by the U.S. Army Corps of Engineers (USACE) for chemical analysis in support of the Hazardous, Toxic and Radioactive Waste Program.

Your laboratory is now validated for the parameters listed below:

METHODS	PARAMETERS	MATRIX ⁽¹⁾
IO-3/6020/TSP ⁽²⁾	TAL Metals ⁽³⁾	Air ⁽⁴⁾
6010B/7000A ⁽⁵⁾	TAL Metals ⁽³⁾	Water ⁽⁶⁾
6010B/7000A ⁽⁵⁾	TAL Metals ⁽³⁾	Solids ⁽⁶⁾
6020/7470A	TAL Metals ⁽³⁾	Water ⁽⁶⁾
6020/7471A	TAL Metals ⁽³⁾	Solids ⁽⁶⁾
8015M	Total Petroleum Hydrocarbons – Gasoline Range Organics	Water ⁽⁶⁾
8015M	Total Petroleum Hydrocarbons – Gasoline Range Organics	Solids ⁽⁶⁾
8015M	Total Petroleum Hydrocarbons – Diesel Range Organics	Water ⁽⁶⁾
8015M	Total Petroleum Hydrocarbons – Diesel Range Organics	Solids ⁽⁶⁾
8021B	Benzene, Toluene, Ethylbenzene, and Xylenes	Water ⁽⁶⁾
8021B	Benzene, Toluene, Ethylbenzene, and Xylenes	Solids ⁽⁴⁾
8081A	Organochlorine Pesticides	Water ⁽⁶⁾
8081A	Organochlorine Pesticides	Solids ⁽⁶⁾
TO-4A/1668A ⁽⁷⁾	Polychlorinated Biphenyls-Congeners	Air ⁽⁴⁾
8082	Polychlorinated Biphenyls	Water ⁽⁶⁾
8082	Polychlorinated Biphenyls	Solids ⁽⁶⁾
8151A	Chlorinated herbicides	Water ⁽⁶⁾
TO-14A/15 ⁽⁸⁾	Volatile Organics	Air ⁽⁴⁾
8260B	Volatile Organics	Water ⁽⁶⁾
8260B	Volatile Organics	Solids ⁽⁶⁾
8270C	Semivolatile Organics	Water ⁽⁶⁾
8270C	Semivolatile Organics	Solids ⁽⁶⁾

TO-13A ⁽⁸⁾	Polynuclear Aromatic Hydrocarbons	Air ⁽⁴⁾
8270C SIM	Polynuclear Aromatic Hydrocarbons	Water ⁽⁴⁾
8270C SIM	Polynuclear Aromatic Hydrocarbons	Solids ⁽⁴⁾
TO-9A ⁽⁸⁾	Dioxins	Air ⁽⁴⁾
8280A	Dioxins	Water ⁽⁴⁾
8280A	Dioxins	Solids ⁽⁶⁾
8290	Dioxins	Water ⁽⁶⁾
8290	Dioxins	Solids ⁽⁶⁾
8310	Polynuclear Aromatic Hydrocarbons	Water ⁽⁶⁾
8310	Polynuclear Aromatic Hydrocarbons	Solids ⁽⁶⁾
8321A	Explosives	Water ⁽⁶⁾
8321A	Explosives	Solids ⁽⁴⁾
8330	Explosives	Water ⁽⁶⁾
8330	Explosives	Solids ⁽⁴⁾
9012A	Cyanide	Water ⁽⁶⁾
9012A	Cyanide	Solids ⁽⁶⁾
314.0	Perchlorate	Water ⁽⁶⁾
314.0	Perchlorate	Solids ⁽⁴⁾
300.0/9056	Anions ⁽⁹⁾	Water ⁽⁶⁾
300.0/9056	Anions ⁽⁹⁾	Solids ⁽⁶⁾

- Remarks: (1) "Solids" includes soils, sediments, and solid waste.
- (2) TAL metals in collected total suspended particulate (TSP) matter from ambient air.
- (3) TAL Metals: aluminum, antimony, arsenic, barium, beryllium, cadmium, calcium, chromium, cobalt, copper, iron, lead, magnesium, manganese, mercury, nickel, potassium, selenium, silver, sodium, thallium, vanadium, and zinc.
- (4) Approval for this parameter is based on review of SOPs and/or evaluation of PT results for other matrices.
- (5) Method 7000A includes only Methods 7470A and 7471A for mercury in water and soil, respectively.
- (6) The laboratory has demonstrated acceptable proficiency for the parameters/matrix/methods by successful analysis of proficiency testing (PT) samples from a NELAC accredited PT provider(s) or other reliable PT providers if a NELAC accredited PT provider is not available.
- (7) Samples collected on PUF/XAD cartridge, followed by solvent extraction.
- (8) Analysis of whole air sample collected in specially treated canister.
- (9) Anions: chloride, fluoride, sulfate, nitrate, and orthophosphate.

Based on acceptable past performance of your laboratory, the validation of your laboratory is hereby extended from September 30, 2004 to March 31, 2005 to provide time to complete the revalidation process.

The USACE reserves the right to conduct additional laboratory inspections or to suspend validation status for any or all of the listed parameters if deemed necessary. It should be noted that your laboratory may not subcontract USACE analytical work to any other laboratory location without the approval of this office. This laboratory validation does not guarantee the delivery of any analytical samples from a USACE Contracting Officer Representative.

Any questions or comments can be directed to Kevin Coats at (402) 697-2563. General questions regarding laboratory validation may be directed to the Laboratory Validation Coordinator at (402) 697-2574.

Sincerely,


Marcia C. Davies, Ph.D.
Director, USACE Hazardous,
Toxic and Radioactive Waste
Center of Expertise



**STATE OF CALIFORNIA
DEPARTMENT OF HEALTH SERVICES**

**ENVIRONMENTAL LABORATORY ACCREDITATION PROGRAM
NELAP - RECOGNIZED**

ACCREDITATION

Is hereby granted to

STL - SACRAMENTO

880 RIVERSIDE PARKWAY

WEST SACRAMENTO, CA 95605

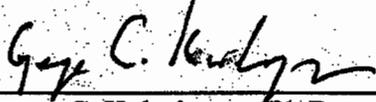
Scope of accreditation is limited to the
"NELAP Fields of Accreditation"
which accompanies this Certificate.

Continued accredited status depends on successful
ongoing participation in the program.

This Certificate is granted in accordance with provisions of
Section 100825, et seq. of the Health and Safety Code.

Certificate No: **01119CA**
Expiration Date: **01/31/2006**
Effective Date: **01/31/2005**

Berkeley, California
subject to forfeiture or revocation.



George C. Kulasingam, Ph.D.
Program Chief
Environmental Laboratory Accreditation Program

CALIFORNIA DEPARTMENT OF HEALTH SERVICES
ENVIRONMENTAL LABORATORY ACCREDITATION PROGRAM - NELAP RECOGNIZED
NELAP Fields of Accreditation

STL - SACRAMENTO

Lab Phone (916) 373-5600

880 RIVERSIDE PARKWAY
WEST SACRAMENTO, CA 95605

Certificate No: 01119CA Renew Date: 1/31/2006

INTERIM

102 - Inorganic Chemistry of Drinking Water

102.045 001 EPA 314.0 Perchlorate

105 - Semi-volatile Organic Chemistry of Drinking Water

105.230 001 EPA 1613 2,3,7,8-Tetrachlorodibenzo-p-dioxin (TCDD)

108 - Inorganic Chemistry of Wastewater

108.020	001	EPA 120.1	Conductivity
108.040	001	EPA 130.2	Hardness
108.050	001	EPA 150.1	pH
108.060	001	EPA 160.1	Residue, Filterable
108.070	001	EPA 160.2	Residue, Non-filterable
108.080	001	EPA 160.3	Residue, Total
108.090	001	EPA 160.4	Residue, Volatile
108.100	001	EPA 160.5	Residue, Settleable
108.110	001	EPA 180.1	Turbidity
108.112	002	EPA 200.7	Calcium
108.112	004	EPA 200.7	Magnesium
108.112	005	EPA 200.7	Potassium
108.112	006	EPA 200.7	Silica
108.112	007	EPA 200.7	Sodium
108.120	001	EPA 300.0	Bromide
108.120	002	EPA 300.0	Chloride
108.120	004	EPA 300.0	Nitrate
108.120	005	EPA 300.0	Nitrite
108.120	006	EPA 300.0	Nitrate-nitrite, Total
108.120	007	EPA 300.0	Phosphate, Ortho
108.120	008	EPA 300.0	Sulfate
108.140	001	EPA 310.1	Alkalinity
108.181	001	EPA 335.2	Cyanide, Total
108.191	001	EPA 340.2	Fluoride
108.200	001	EPA 350.1	Ammonia
108.211	001	EPA 351.2	Kjeldahl Nitrogen
108.231	001	EPA 353.2	Nitrate calc.
108.232	001	EPA 353.2	Nitrate-nitrite, Total
108.233	001	EPA 353.2	Nitrite
108.264	001	EPA 365.3	Phosphate, Ortho
108.265	001	EPA 365.3	Phosphorus, Total
108.291	001	EPA 376.2	Sulfide
108.323	001	EPA 410.4	Chemical Oxygen Demand
108.340	001	EPA 415.1	Total Organic Carbon
108.362	001	EPA 420.4	Phenols, Total

As of 2/1/2005, this list supersedes all previous lists for this certificate number.
Customers: Please verify the current accreditation standing with the State.

STL - SACRAMENTO

Certificate No: 01119CA
Renew Date: 1/31/2006

108.380	001	EPA 1664	Oil and Grease
108.410	001	SM2320B	Alkalinity
108.420	001	SM2340B	Hardness (calc.)
108.421	001	SM2340C	Hardness
108.441	001	SM2540C	Residue, Filterable
108.472	001	SM4500-CN E	Cyanide, Total
108.473	001	SM4500-CN G	Cyanide, amenable
108.611	001	SM5310C	Total Organic Carbon

109 - Toxic Chemical Elements of Wastewater

109.010	001	EPA 200.7	Aluminum
109.010	002	EPA 200.7	Antimony
109.010	003	EPA 200.7	Arsenic
109.010	004	EPA 200.7	Barium
109.010	005	EPA 200.7	Beryllium
109.010	007	EPA 200.7	Cadmium
109.010	009	EPA 200.7	Chromium
109.010	010	EPA 200.7	Cobalt
109.010	011	EPA 200.7	Copper
109.010	012	EPA 200.7	Iron
109.010	013	EPA 200.7	Lead
109.010	015	EPA 200.7	Manganese
109.010	016	EPA 200.7	Molybdenum
109.010	017	EPA 200.7	Nickel
109.010	019	EPA 200.7	Selenium
109.010	021	EPA 200.7	Silver
109.010	023	EPA 200.7	Thallium
109.010	024	EPA 200.7	Tin
109.010	025	EPA 200.7	Titanium
109.010	026	EPA 200.7	Vanadium
109.010	027	EPA 200.7	Zinc
109.020	001	EPA 200.8	Aluminum
109.020	002	EPA 200.8	Antimony
109.020	003	EPA 200.8	Arsenic
109.020	004	EPA 200.8	Barium
109.020	005	EPA 200.8	Beryllium
109.020	006	EPA 200.8	Cadmium
109.020	007	EPA 200.8	Chromium
109.020	008	EPA 200.8	Cobalt
109.020	009	EPA 200.8	Copper
109.020	010	EPA 200.8	Lead
109.020	011	EPA 200.8	Manganese
109.020	012	EPA 200.8	Molybdenum
109.020	013	EPA 200.8	Nickel
109.020	014	EPA 200.8	Selenium
109.020	015	EPA 200.8	Silver
109.020	016	EPA 200.8	Thallium
109.020	017	EPA 200.8	Vanadium
109.020	018	EPA 200.8	Zinc

As of 2/1/2005 , this list supersedes all previous lists for this certificate number.
Customers: Please verify the current accreditation standing with the State.

111 - Semi-volatile Organic Chemistry of Wastewater

111.110 001	EPA 1613	2,3,7,8-Tetrachlorodibenzo-p-dioxin (TCDD)
111.110 002	EPA 1613	1,2,3,7,8-Pentachlorodibenzo-p-dioxin (PeCDD)
111.110 003	EPA 1613	1,2,3,4,7,8-Hexachlorodibenzo-p-dioxin (HxCDD)
111.110 004	EPA 1613	1,2,3,6,7,8-Hexachlorodibenzo-p-dioxin (HxCDD)
111.110 005	EPA 1613	1,2,3,7,8,9-Hexachlorodibenzo-p-dioxin (HxCDD)
111.110 006	EPA 1613	1,2,3,4,6,7,8-Heptachlorodibenzo-p-dioxin (HpCDD)
111.110 007	EPA 1613	1,2,3,4,6,7,8,9-Octachlorodibenzo-p-dioxin (OCDD)
111.110 008	EPA 1613	2,3,7,8-Tetrachlorodibenzofuran (TCDF)
111.110 009	EPA 1613	1,2,3,7,8-Pentachlorodibenzofuran (PeCDF)
111.110 010	EPA 1613	2,3,4,7,8-Pentachlorodibenzofuran (PeCDF)
111.110 011	EPA 1613	1,2,3,4,7,8-Hexachlorodibenzofuran (HxCDF)
111.110 012	EPA 1613	1,2,3,6,7,8-Hexachlorodibenzofuran (HxCDF)
111.110 013	EPA 1613	1,2,3,7,8,9-Hexachlorodibenzofuran (HxCDF)
111.110 014	EPA 1613	2,3,4,6,7,8-Hexachlorodibenzofuran (HxCDF)
111.110 015	EPA 1613	1,2,3,4,6,7,8-Heptachlorodibenzofuran (HpCDF)
111.110 016	EPA 1613	1,2,3,4,7,8,9-Heptachlorodibenzofuran (HpCDF)
111.110 017	EPA 1613	1,2,3,4,6,7,8,9-Octachlorodibenzofuran (OCDF)
111.110 018	EPA 1613	Total TCDD
111.110 019	EPA 1613	Total PeCDD
111.110 020	EPA 1613	Total HxCDD
111.110 021	EPA 1613	Total HpCDD
111.110 022	EPA 1613	Total TCDF
111.110 023	EPA 1613	Total PeCDF
111.110 024	EPA 1613	Total HxCDF
111.110 025	EPA 1613	Total HpCDF

114 - Inorganic Chemistry of Hazardous Waste

114.010 001	EPA 6010B	Antimony	
114.010 002	EPA 6010B	Arsenic	
114.010 003	EPA 6010B	Barium	
114.010 004	EPA 6010B	Beryllium	
114.010 005	EPA 6010B	Cadmium	
114.010 006	EPA 6010B	Chromium	
114.010 007	EPA 6010B	Cobalt	
114.010 008	EPA 6010B	Copper	
114.010 009	EPA 6010B	Lead	
114.010 010	EPA 6010B	Molybdenum	
114.010 011	EPA 6010B	Nickel	
114.010 012	EPA 6010B	Selenium	
114.010 013	EPA 6010B	Silver	
114.010 014	EPA 6010B	Thallium	
114.010 015	EPA 6010B	Vanadium	
114.010 016	EPA 6010B	Zinc	
114.010 026	EPA 6010B	Silica	Aqueous
114.010 027	EPA 6010B	Sodium	
114.020 001	EPA 6020	Antimony	
114.020 002	EPA 6020	Arsenic	
114.020 003	EPA 6020	Barium	

As of 2/1/2005 , this list supersedes all previous lists for this certificate number.
 Customers: Please verify the current accreditation standing with the State.

STL - SACRAMENTO**Certificate No:** 01119CA
Renew Date: 1/31/2006

114.020	004	EPA 6020	Beryllium
114.020	005	EPA 6020	Cadmium
114.020	006	EPA 6020	Chromium
114.020	007	EPA 6020	Cobalt
114.020	008	EPA 6020	Copper
114.020	009	EPA 6020	Lead
114.020	010	EPA 6020	Molybdenum
114.020	011	EPA 6020	Nickel
114.020	012	EPA 6020	Selenium
114.020	013	EPA 6020	Silver
114.020	014	EPA 6020	Thallium
114.020	015	EPA 6020	Vanadium
114.020	016	EPA 6020	Zinc
114.103	001	EPA 7196A	Chromium (VI)
114.140	001	EPA 7470A	Mercury
114.141	001	EPA 7471A	Mercury
114.221	001	EPA 9012A	Cyanide, Total
114.240	001	EPA 9040	pH
114.241	001	EPA 9045	pH
114.250	001	EPA 9056	Fluoride
114.291	001	EPA 340.2	Fluoride

115 - Extraction Test of Hazardous Waste

115.021	001	EPA 1311	TCLP Inorganics
115.022	001	EPA 1311	TCLP Extractables
115.030	001	CCR Chapter11, Article 5, Appendix II	Waste Extraction Test (WET)

116 - Volatile Organic Chemistry of Hazardous Waste

116.030	001	EPA 8015B	Gasoline-range Organics
116.040	002	EPA 8021B	Benzene
116.040	039	EPA 8021B	Ethylbenzene
116.040	041	EPA 8021B	Methyl tert-butyl Ether (MTBE)
116.040	047	EPA 8021B	Toluene
116.040	056	EPA 8021B	Xylenes, Total
116.080	001	EPA 8260B	Acetone
116.080	002	EPA 8260B	Acetonitrile
116.080	003	EPA 8260B	Acrolein
116.080	004	EPA 8260B	Acrylonitrile
116.080	006	EPA 8260B	Allyl Chloride
116.080	007	EPA 8260B	Benzene
116.080	010	EPA 8260B	Bromochloromethane
116.080	011	EPA 8260B	Bromodichloromethane
116.080	012	EPA 8260B	Bromoform
116.080	013	EPA 8260B	Bromomethane
116.080	015	EPA 8260B	Carbon Disulfide
116.080	016	EPA 8260B	Carbon Tetrachloride
116.080	018	EPA 8260B	Chlorobenzene
116.080	019	EPA 8260B	Chloroethane
116.080	020	EPA 8260B	2-Chloroethyl Vinyl Ether
116.080	021	EPA 8260B	Chloroform

As of 2/1/2005, this list supersedes all previous lists for this certificate number.
Customers: Please verify the current accreditation standing with the State.

STL - SACRAMENTO

Certificate No: 01119CA
Renew Date: 1/31/2006

116.080	022	EPA 8260B	Chloromethane
116.080	023	EPA 8260B	Chloroprene
116.080	026	EPA 8260B	Dibromochloromethane
116.080	027	EPA 8260B	Dibromochloropropane
116.080	028	EPA 8260B	1,2-Dibromoethane
116.080	029	EPA 8260B	Dibromofluoromethane
116.080	030	EPA 8260B	Dibromomethane
116.080	031	EPA 8260B	1,2-Dichlorobenzene
116.080	032	EPA 8260B	1,3-Dichlorobenzene
116.080	033	EPA 8260B	1,4-Dichlorobenzene
116.080	035	EPA 8260B	trans-1,4-Dichloro-2-butene
116.080	036	EPA 8260B	Dichlorodifluoromethane
116.080	037	EPA 8260B	1,1-Dichloroethane
116.080	038	EPA 8260B	1,2-Dichloroethane
116.080	039	EPA 8260B	1,1-Dichloroethene
116.080	040	EPA 8260B	trans-1,2-Dichloroethene
116.080	041	EPA 8260B	cis-1,2-Dichloroethene
116.080	042	EPA 8260B	1,2-Dichloropropane
116.080	043	EPA 8260B	1,3-Dichloropropane
116.080	044	EPA 8260B	2,2-Dichloropropane
116.080	045	EPA 8260B	1,1-Dichloropropene
116.080	046	EPA 8260B	cis-1,3-Dichloropropene
116.080	047	EPA 8260B	trans-1,3-Dichloropropene
116.080	050	EPA 8260B	1,4-Dioxane
116.080	053	EPA 8260B	Ethylbenzene
116.080	055	EPA 8260B	Ethyl Methacrylate
116.080	056	EPA 8260B	Hexachlorobutadiene
116.080	058	EPA 8260B	2-Hexanone (MBK)
116.080	059	EPA 8260B	Iodomethane
116.080	060	EPA 8260B	Isobutyl Alcohol
116.080	062	EPA 8260B	Methacrylonitrile
116.080	064	EPA 8260B	Methyl tert-butyl Ether (MTBE)
116.080	065	EPA 8260B	Methylene Chloride
116.080	066	EPA 8260B	Methyl Ethyl Ketone
116.080	067	EPA 8260B	Methyl Methacrylate
116.080	068	EPA 8260B	4-Methyl-2-pentanone (MIBK)
116.080	069	EPA 8260B	Naphthalene
116.080	078	EPA 8260B	Propionitrile
116.080	081	EPA 8260B	1,1,1,2-Tetrachloroethane
116.080	082	EPA 8260B	1,1,2,2-Tetrachloroethane
116.080	083	EPA 8260B	Tetrachloroethene
116.080	084	EPA 8260B	Toluene
116.080	086	EPA 8260B	1,2,3-Trichlorobenzene
116.080	087	EPA 8260B	1,2,4-Trichlorobenzene
116.080	088	EPA 8260B	1,1,1-Trichloroethane
116.080	089	EPA 8260B	1,1,2-Trichloroethane
116.080	090	EPA 8260B	Trichloroethene
116.080	091	EPA 8260B	Trichlorofluoromethane

As of 2/1/2005 , this list supersedes all previous lists for this certificate number.
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STL - SACRAMENTO

Certificate No: 01119CA
Renew Date: 1/31/2006

116.080	092	EPA 8260B	1,2,3-Trichloropropane
116.080	093	EPA 8260B	Vinyl Acetate
116.080	094	EPA 8260B	Vinyl Chloride
116.080	095	EPA 8260B	Xylenes, Total
116.080	096	EPA 8260B	tert-Amyl Methyl Ether (TAME)
116.080	097	EPA 8260B	tert-Butyl Alcohol (TBA)
116.080	098	EPA 8260B	Ethyl tert-butyl Ether (ETBE)
116.080	099	EPA 8260B	Bromobenzene
116.080	100	EPA 8260B	n-Butylbenzene
116.080	101	EPA 8260B	sec-Butylbenzene
116.080	102	EPA 8260B	tert-Butylbenzene
116.080	103	EPA 8260B	2-Chlorotoluene
116.080	104	EPA 8260B	4-Chlorotoluene
116.080	105	EPA 8260B	Isopropylbenzene
116.080	106	EPA 8260B	N-propylbenzene
116.080	107	EPA 8260B	Styrene
116.080	108	EPA 8260B	1,2,4-Trimethylbenzene
116.080	109	EPA 8260B	1,3,5-Trimethylbenzene
116.110	001	LUFT	Total Petroleum Hydrocarbons - Gasoline

117 - Semi-volatile Organic Chemistry of Hazardous Waste

117.010	001	EPA 8015B	Diesel-range Total Petroleum Hydrocarbons
117.016	001	LUFT	Diesel-range Total Petroleum Hydrocarbons
117.110	001	EPA 8270C	Acenaphthene
117.110	002	EPA 8270C	Acenaphthylene
117.110	003	EPA 8270C	Acetophenone
117.110	004	EPA 8270C	2-Acetylaminofluorene
117.110	006	EPA 8270C	4-Aminobiphenyl
117.110	007	EPA 8270C	Aniline
117.110	008	EPA 8270C	Anthracene
117.110	009	EPA 8270C	Aramite
117.110	010	EPA 8270C	Benzidine
117.110	011	EPA 8270C	Benz(a)anthracene
117.110	012	EPA 8270C	Benzo(b)fluoranthene
117.110	013	EPA 8270C	Benzo(k)fluoranthene
117.110	014	EPA 8270C	Benzo(g,h,i)perylene
117.110	015	EPA 8270C	Benzo(a)pyrene
117.110	016	EPA 8270C	Benzoic Acid
117.110	018	EPA 8270C	Benzyl Alcohol
117.110	019	EPA 8270C	Benzyl Butyl Phthalate
117.110	020	EPA 8270C	Bis(2-chloroethoxy)methane
117.110	021	EPA 8270C	Bis(2-chloroethyl) Ether
117.110	022	EPA 8270C	Bis(2-chloroisopropyl) Ether
117.110	023	EPA 8270C	Di(2-ethylhexyl) Phthalate
117.110	024	EPA 8270C	4-Bromophenyl Phenyl Ether
117.110	025	EPA 8270C	Carbazole
117.110	026	EPA 8270C	4-Chloroaniline
117.110	027	EPA 8270C	4-Chloro-3-methylphenol
117.110	028	EPA 8270C	1-Chloronaphthalene

As of 2/1/2005 , this list supersedes all previous lists for this certificate number.
Customers: Please verify the current accreditation standing with the State.

STL - SACRAMENTO

Certificate No: 01119CA
Renew Date: 1/31/2006

117.110	029	EPA 8270C	2-Chloronaphthalene
117.110	030	EPA 8270C	2-Chlorophenol
117.110	031	EPA 8270C	4-Chlorophenyl Phenyl Ether
117.110	032	EPA 8270C	Chrysene
117.110	035	EPA 8270C	Dibenz(a,j)acridine
117.110	036	EPA 8270C	Dibenz(a,h)anthracene
117.110	037	EPA 8270C	Dibenzofuran
117.110	039	EPA 8270C	1,2-Dichlorobenzene
117.110	040	EPA 8270C	1,3-Dichlorobenzene
117.110	041	EPA 8270C	1,4-Dichlorobenzene
117.110	042	EPA 8270C	3,3'-Dichlorobenzidine
117.110	043	EPA 8270C	2,4-Dichlorophenol
117.110	044	EPA 8270C	2,6-Dichlorophenol
117.110	045	EPA 8270C	Diethyl Phthalate
117.110	050	EPA 8270C	p-Dimethylaminoazobenzene
117.110	051	EPA 8270C	7,12-Dimethylbenz(a)anthracene
117.110	052	EPA 8270C	a,a-Dimethylphenethylamine
117.110	053	EPA 8270C	2,4-Dimethylphenol
117.110	054	EPA 8270C	Dimethyl Phthalate
117.110	055	EPA 8270C	Di-n-butyl phthalate
117.110	056	EPA 8270C	Di-n-octyl phthalate
117.110	057	EPA 8270C	1,2-Dinitrobenzene
117.110	058	EPA 8270C	1,3-Dinitrobenzene
117.110	059	EPA 8270C	1,4-Dinitrobenzene
117.110	060	EPA 8270C	2,4-Dinitrophenol
117.110	061	EPA 8270C	2,4-Dinitrotoluene
117.110	062	EPA 8270C	2,6-Dinitrotoluene
117.110	063	EPA 8270C	Diphenylamine
117.110	064	EPA 8270C	1,2-Diphenylhydrazine
117.110	066	EPA 8270C	Ethyl Methanesulfonate
117.110	067	EPA 8270C	Fluoranthene
117.110	068	EPA 8270C	Fluorene
117.110	069	EPA 8270C	Hexachlorobenzene
117.110	070	EPA 8270C	Hexachlorobutadiene
117.110	071	EPA 8270C	Hexachlorocyclopentadiene
117.110	072	EPA 8270C	Hexachloroethane
117.110	074	EPA 8270C	Hexachloropropene
117.110	075	EPA 8270C	Indeno(1,2,3-c,d)pyrene
117.110	076	EPA 8270C	Isophorone
117.110	077	EPA 8270C	Isosafrole
117.110	079	EPA 8270C	3-Methylcholanthrene
117.110	080	EPA 8270C	2-Methyl-4,6-dinitrophenol
117.110	082	EPA 8270C	Methyl Methanesulfonate
117.110	083	EPA 8270C	2-Methylnaphthalene
117.110	084	EPA 8270C	2-Methylphenol
117.110	085	EPA 8270C	3-Methylphenol
117.110	086	EPA 8270C	4-Methylphenol
117.110	087	EPA 8270C	Naphthalene

As of 2/1/2005 , this list supersedes all previous lists for this certificate number.
 Customers: Please verify the current accreditation standing with the State.

STL - SACRAMENTO**Certificate No:** 01119CA
Renew Date: 1/31/2006

117.110	088	EPA 8270C	1,4-Naphthoquinone
117.110	089	EPA 8270C	1-Naphthylamine
117.110	090	EPA 8270C	2-Naphthylamine
117.110	092	EPA 8270C	2-Nitroaniline
117.110	093	EPA 8270C	3-Nitroaniline
117.110	094	EPA 8270C	4-Nitroaniline
117.110	095	EPA 8270C	Nitrobenzene
117.110	096	EPA 8270C	2-Nitrophenol
117.110	097	EPA 8270C	4-Nitrophenol
117.110	098	EPA 8270C	N-nitrosodi-n-butylamine
117.110	099	EPA 8270C	N-nitrosodiethylamine
117.110	100	EPA 8270C	N-nitrosodimethylamine
117.110	101	EPA 8270C	N-nitrosodi-n-propylamine
117.110	102	EPA 8270C	N-nitrosodiphenylamine
117.110	103	EPA 8270C	N-nitrosomethylethylamine
117.110	104	EPA 8270C	N-nitrosomorpholine
117.110	105	EPA 8270C	N-nitrosopiperidine
117.110	106	EPA 8270C	N-nitrosopyrrolidine
117.110	107	EPA 8270C	5-Nitro-o-toluidine
117.110	108	EPA 8270C	Pentachlorobenzene
117.110	109	EPA 8270C	Pentachloronitrobenzene
117.110	110	EPA 8270C	Pentachlorophenol
117.110	111	EPA 8270C	Phenacetin
117.110	112	EPA 8270C	Phenanthrene
117.110	113	EPA 8270C	Phenol
117.110	114	EPA 8270C	1,4-Phenylenediamine
117.110	116	EPA 8270C	2-Picoline
117.110	117	EPA 8270C	Pronamide
117.110	119	EPA 8270C	Pyrene
117.110	120	EPA 8270C	Pyridine
117.110	122	EPA 8270C	Safrole
117.110	124	EPA 8270C	1,2,4,5-Tetrachlorobenzene
117.110	125	EPA 8270C	2,3,4,6-Tetrachlorophenol
117.110	128	EPA 8270C	o-Toluidine
117.110	129	EPA 8270C	1,2,4-Trichlorobenzene
117.110	130	EPA 8270C	2,4,5-Trichlorophenol
117.110	131	EPA 8270C	2,4,6-Trichlorophenol
117.110	132	EPA 8270C	1,3,5-Trinitrobenzene
117.111	015	EPA 8270C	Chlorobenzilate
117.111	021	EPA 8270C	Diallate
117.111	025	EPA 8270C	Dimethoate
117.111	039	EPA 8270C	Isodrin
117.111	054	EPA 8270C	Parathion Ethyl
117.111	055	EPA 8270C	Parathion Methyl
117.111	056	EPA 8270C	Phorate
117.111	058	EPA 8270C	Sulfotepp
117.111	061	EPA 8270C	O,O,O-triethyl Phosphorothioate
117.111	062	EPA 8270C	Trifluralin

As of 2/1/2005, this list supersedes all previous lists for this certificate number.
Customers: Please verify the current accreditation standing with the State.

STL - SACRAMENTO

Certificate No: 01119CA
Renew Date: 1/31/2006

117.120 001	EPA 8280A	2,3,7,8-Tetrachlorodibenzo-p-dioxin (TCDD)
117.120 002	EPA 8280A	1,2,3,7,8-Pentachlorodibenzo-p-dioxin (PeCDD)
117.120 003	EPA 8280A	1,2,3,4,7,8-Hexachlorodibenzo-p-dioxin (HxCDD)
117.120 004	EPA 8280A	1,2,3,6,7,8-Hexachlorodibenzo-p-dioxin (HxCDD)
117.120 005	EPA 8280A	1,2,3,7,8,9-Hexachlorodibenzo-p-dioxin (HxCDD)
117.120 006	EPA 8280A	2,3,7,8-Tetrachlorodibenzofuran (TCDF)
117.120 007	EPA 8280A	1,2,3,7,8-Pentachlorodibenzofuran (PeCDF)
117.120 008	EPA 8280A	2,3,4,7,8-Pentachlorodibenzofuran (PeCDF)
117.120 009	EPA 8280A	1,2,3,4,7,8-Hexachlorodibenzofuran (HxCDF)
117.120 010	EPA 8280A	1,2,3,6,7,8-Hexachlorodibenzofuran (HxCDF)
117.120 011	EPA 8280A	1,2,3,7,8,9-Hexachlorodibenzofuran (HxCDF)
117.120 012	EPA 8280A	2,3,4,6,7,8-Hexachlorodibenzofuran (HxCDF)
117.120 013	EPA 8280A	Total TCDD
117.120 014	EPA 8280A	Total PeCDD
117.120 015	EPA 8280A	Total HxCDD
117.120 016	EPA 8280A	Total TCDF
117.120 017	EPA 8280A	Total PeCDF
117.120 018	EPA 8280A	Total HxCDF
117.120 019	EPA 8280A	1,2,3,4,6,7,8-Heptachlorodibenzo-p-dioxin (HpCDD)
117.120 020	EPA 8280A	1,2,3,4,6,7,8,9-Octachlorodibenzo-p-dioxin (OCDD)
117.120 021	EPA 8280A	1,2,3,4,6,7,8-Heptachlorodibenzofuran (HpCDF)
117.120 022	EPA 8280A	1,2,3,4,7,8,9-Heptachlorodibenzofuran (HpCDF)
117.120 023	EPA 8280A	1,2,3,4,6,7,8,9-Octachlorodibenzofuran (OCDF)
117.120 024	EPA 8280A	Total HpCDD
117.120 025	EPA 8280A	Total HpCDF
117.130 001	EPA 8290	2,3,7,8-Tetrachlorodibenzo-p-dioxin (TCDD)
117.130 002	EPA 8290	1,2,3,7,8-Pentachlorodibenzo-p-dioxin (PeCDD)
117.130 003	EPA 8290	1,2,3,4,7,8-Hexachlorodibenzo-p-dioxin (HxCDD)
117.130 004	EPA 8290	1,2,3,6,7,8-Hexachlorodibenzo-p-dioxin (HxCDD)
117.130 005	EPA 8290	1,2,3,7,8,9-Hexachlorodibenzo-p-dioxin (HxCDD)
117.130 006	EPA 8290	2,3,7,8-Tetrachlorodibenzofuran (TCDF)
117.130 007	EPA 8290	1,2,3,7,8-Pentachlorodibenzofuran (PeCDF)
117.130 008	EPA 8290	2,3,4,7,8-Pentachlorodibenzofuran (PeCDF)
117.130 009	EPA 8290	1,2,3,4,7,8-Hexachlorodibenzofuran (HxCDF)
117.130 010	EPA 8290	1,2,3,6,7,8-Hexachlorodibenzofuran (HxCDF)
117.130 011	EPA 8290	1,2,3,7,8,9-Hexachlorodibenzofuran (HxCDF)
117.130 012	EPA 8290	2,3,4,6,7,8-Hexachlorodibenzofuran (HxCDF)
117.130 013	EPA 8290	1,2,3,4,6,7,8-Heptachlorodibenzo-p-dioxin (HpCDD)
117.130 014	EPA 8290	1,2,3,4,6,7,8-Heptachlorodibenzofuran (HpCDF)
117.130 015	EPA 8290	1,2,3,4,7,8,9-Heptachlorodibenzofuran (HpCDF)
117.130 016	EPA 8290	1,2,3,4,6,7,8,9-Octachlorodibenzo-p-dioxin (OCDD)
117.130 017	EPA 8290	1,2,3,4,6,7,8,9-Octachlorodibenzofuran (OCDF)
117.140 001	EPA 8310	Acenaphthene
117.140 002	EPA 8310	Acenaphthylene
117.140 003	EPA 8310	Anthracene
117.140 004	EPA 8310	Benz(a)anthracene
117.140 005	EPA 8310	Benzo(a)pyrene
117.140 006	EPA 8310	Benzo(b)fluoranthene

As of 2/1/2005 , this list supersedes all previous lists for this certificate number.
 Customers: Please verify the current accreditation standing with the State.

STL - SACRAMENTO

Certificate No: 01119CA
Renew Date: 1/31/2006

117.140	007	EPA 8310	Benzo(k)fluoranthene
117.140	008	EPA 8310	Benzo(g,h,i)perylene
117.140	009	EPA 8310	Chrysene
117.140	010	EPA 8310	Dibenz(a,h)anthracene
117.140	011	EPA 8310	Fluoranthene
117.140	012	EPA 8310	Fluorene
117.140	013	EPA 8310	Indeno(1,2,3-c,d)pyrene
117.140	014	EPA 8310	Naphthalene
117.140	015	EPA 8310	Phenanthrene
117.140	016	EPA 8310	Pyrene
117.170	001	EPA 8330	4-Amino-2,6-dinitrotoluene
117.170	002	EPA 8330	2-Amino-4,6-dinitrotoluene
117.170	003	EPA 8330	1,3-Dinitrobenzene
117.170	004	EPA 8330	2,4-Dinitrotoluene
117.170	005	EPA 8330	2,6-Dinitrotoluene
117.170	006	EPA 8330	Hexahydro-1,3,5-trinitro-1,3,5-triazine (RDX)
117.170	007	EPA 8330	Methyl-2,4,6-trinitrophenylnitramine
117.170	008	EPA 8330	Nitrobenzene
117.170	009	EPA 8330	2-Nitrotoluene
117.170	010	EPA 8330	3-Nitrotoluene
117.170	011	EPA 8330	4-Nitrotoluene
117.170	012	EPA 8330	Octahydro-1,3,5,7-tetranitro-1,3,5,7-tetrazocine
117.170	013	EPA 8330	1,3,5-Trinitrobenzene
117.170	014	EPA 8330	2,4,6-Trinitrotoluene
117.210	001	EPA 8081A	Aldrin
117.210	002	EPA 8081A	a-BHC
117.210	003	EPA 8081A	b-BHC
117.210	004	EPA 8081A	d-BHC
117.210	005	EPA 8081A	g-BHC (Lindane)
117.210	007	EPA 8081A	a-Chlordane
117.210	008	EPA 8081A	g-Chlordane
117.210	009	EPA 8081A	Chlordane (tech.)
117.210	010	EPA 8081A	Chlorobenzilate
117.210	013	EPA 8081A	4,4'-DDD
117.210	014	EPA 8081A	4,4'-DDE
117.210	015	EPA 8081A	4,4'-DDT
117.210	016	EPA 8081A	Diallate
117.210	020	EPA 8081A	Dieldrin
117.210	021	EPA 8081A	Endosulfan I
117.210	022	EPA 8081A	Endosulfan II
117.210	023	EPA 8081A	Endosulfan Sulfate
117.210	024	EPA 8081A	Endrin
117.210	025	EPA 8081A	Endrin Aldehyde
117.210	026	EPA 8081A	Endrin Ketone
117.210	027	EPA 8081A	Heptachlor
117.210	028	EPA 8081A	Heptachlor Epoxide
117.210	031	EPA 8081A	Isodrin
117.210	033	EPA 8081A	Methoxychlor

As of 2/1/2005 , this list supersedes all previous lists for this certificate number.
 Customers: Please verify the current accreditation standing with the State.

STL - SACRAMENTO

Certificate No: 01119CA
Renew Date: 1/31/2006

117.210	039	EPA 8081A	Toxaphene
117.220	001	EPA 8082	PCB-1016
117.220	002	EPA 8082	PCB-1221
117.220	003	EPA 8082	PCB-1232
117.220	004	EPA 8082	PCB-1242
117.220	005	EPA 8082	PCB-1248
117.220	006	EPA 8082	PCB-1254
117.220	007	EPA 8082	PCB-1260

As of 2/1/2005 , this list supersedes all previous lists for this certificate number.
Customers: Please verify the current accreditation standing with the State.



Jeb Bush
Governor

John O. Agwunobi, M.D., M.B.A.
Secretary

March 7, 2005
I. D. # E87570

CERTIFIED MAIL NUMBER 7001 2510 0002 7549 1934

Eric Redman
STL Sacramento
880 Riverside Parkway
West Sacramento, CA 96505

Dear Laboratory Director:

In the e-mail dated March 5, 2005, authorized personnel at your laboratory indicated that certification for the following was to be voluntarily relinquished:

Solid and Chemical Materials

- EPA 1310 EP-TOX Extraction

- EPA 8021 1,1,2,2-Tetrachloroethane
- EPA 8021 1,1,2-Trichloroethane
- EPA 8021 1,1-Dichloroethane
- EPA 8021 1,1-Dichloroethylene
- EPA 8021 1,2-Dichlorobenzene
- EPA 8021 1,2-Dichloroethane
- EPA 8021 1,2-Dichloropropane
- EPA 8021 1,3-Dichlorobenzene
- EPA 8021 1,4-Dichlorobenzene
- EPA 8021 2-Chloroethyl vinyl ether
- EPA 8021 Bromochloromethane
- EPA 8021 Bromodichloromethane
- EPA 8021 Bromoform
- EPA 8021 Carbon Tetrachloride
- EPA 8021 Chlorobenzene
- EPA 8021 Chloroethane
- EPA 8021 Chloroform
- EPA 8021 cis-1,2-Dichloroethylene
- EPA 8021 cis-1,3-Dichloropropene
- EPA 8021 Dibromochloromethane
- EPA 8021 Dichlorodifluoromethane
- EPA 8021 Methyl bromide
- EPA 8021 Methyl chloride
- EPA 8021 Methylene chloride
- EPA 8021 Tetrachloroethylene
- EPA 8021 trans-1,2-Dichloroethylene
- EPA 8021 trans-1,3-Dichloropropylene
- EPA 8021 Trichloroethene
- EPA 8021 Trichlorofluoromethane
- EPA 8021 Vinyl chloride

- EPA 8151 2,4-DB
- EPA 8151 Dalapon
- EPA 8151 Dichloroprop
- EPA 8151 Dinoseb
- EPA 8151 MCPA



EPA 8151 MCPP

EPA 8270 5,5-Diphenylhydantoin

EPA 8270 Benzo(j)fluoranthene

EPA 8270 Dinoseb

Non-Potable Water

EPA 300.0 Fluoride

EPA 335.4 Cyanide

Please be advised that the Department of Health has made the necessary changes to the scope of your laboratory's certification final as of March 5, 2005. The attached Laboratory Scope of Accreditation reflects the Fields of Accreditation for which the laboratory is now certified.

If there are any questions regarding this action, please contact the Environmental Laboratory Certification Program immediately at 904-791-1599 or e-mail me at steve_arms@doh.state.fl.us.

Sincerely,



Stephen A. Arms

Administrator

Environmental Laboratory Certification Program

SAA\nr

Enclosure: Laboratory Scope of Accreditation

CERTIFICATE OF SERVICE

I HEREBY CERTIFY that the foregoing notice was sent to STL Sacramento via United States Certified Mail this 8th day of March 2005.


Signature

Jeb Bush
Governor



John O. Agwunobi, M.D., M.B.A.
Secretary

Laboratory Scope of Accreditation

Page 1 of 34

**THIS LISTING OF ACCREDITED ANALYTES SHOULD BE USED ONLY WHEN
ASSOCIATED WITH A VALID CERTIFICATE**

State Laboratory ID: E87570

EPA Lab Code: CA00044

(916) 373-5600

E87570

STL Sacramento

880 Riverside Parkway

West Sacramento, CA 95605

Matrix: Drinking Water

Analyte	Method/Tech	Category	Certification Type	Effective Date
2,3,7,8-Tetrachlorodibenzo-p-dioxin	EPA 1613	Dioxin	NELAP	9/24/2001

"STATE" indicates certification for the analyte by the method specified. "NELAP" further indicates certification compliant with the NELAC Standards.

NON-TRANSFERABLE 03/07/2005-E87570

Jeb Bush
Governor



John O. Agwunobi, M.D., M.B.A.
Secretary

Laboratory Scope of Accreditation

Page 2 of 34

THIS LISTING OF ACCREDITED ANALYTES SHOULD BE USED ONLY WHEN
ASSOCIATED WITH A VALID CERTIFICATE

State Laboratory ID: E87570

EPA Lab Code: CA00044

(916) 373-5600

E87570

STL Sacramento
880 Riverside Parkway
West Sacramento, CA 95605

Matrix: Non-Potable Water

Analyte	Method/Tech	Category	Certification Type	Effective Date
1,2,3,4,6,7,8,9-Octachlorodibenzofuran (OCDF)	EPA 1613	Extractable Organics	NELAP	9/24/2001
1,2,3,4,6,7,8,9-Octachlorodibenzo-p-dioxin (OCDD)	EPA 1613	Extractable Organics	NELAP	9/24/2001
1,2,3,4,6,7,8-Heptachlorodibenzofuran (1,2,3,4,6,7,8-hpcdf)	EPA 1613	Extractable Organics	NELAP	9/24/2001
1,2,3,4,6,7,8-Heptachlorodibenzo-p-dioxin (1,2,3,4,6,7,8-hpcdd)	EPA 1613	Extractable Organics	NELAP	9/24/2001
1,2,3,4,7,8,9-Heptachlorodibenzofuran (1,2,3,4,7,8,9-hpcdf)	EPA 1613	Extractable Organics	NELAP	9/24/2001
1,2,3,4,7,8-Hxcdd	EPA 1613	Extractable Organics	NELAP	9/24/2001
1,2,3,4,7,8-Hxcdf	EPA 1613	Extractable Organics	NELAP	9/24/2001
1,2,3,6,7,8-Hxcdd	EPA 1613	Extractable Organics	NELAP	9/24/2001
1,2,3,6,7,8-Hxcdf	EPA 1613	Extractable Organics	NELAP	9/24/2001
1,2,3,7,8,9-Hxcdd	EPA 1613	Extractable Organics	NELAP	9/24/2001
1,2,3,7,8,9-Hxcdf	EPA 1613	Extractable Organics	NELAP	9/24/2001
1,2,3,7,8-Pecdd	EPA 1613	Extractable Organics	NELAP	9/24/2001
1,2,3,7,8-Pecdf	EPA 1613	Extractable Organics	NELAP	9/24/2001
1,3,5-Trinitrobenzene (1,3,5-TNB)	SAC-LC-0001	Extractable Organics	NELAP	9/24/2001
1,3-Dinitrobenzene (1,3-DNB)	SAC-LC-0001	Extractable Organics	NELAP	9/24/2001
1-Methylnaphthalene	SAC-ID-0015	Extractable Organics	NELAP	9/24/2001
1-Methylphenanthrene	SAC-ID-0015	Extractable Organics	NELAP	9/24/2001
2,2',3,3',4,4',5,5',6-Nonachlorobiphenyl (BZ 206)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	6/10/2003
2,2',3,3',4,4',5,5'-Octachlorobiphenyl (BZ 194)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	6/10/2003
2,2',3,3',4,4',5,6,6'-Nonachlorobiphenyl (BZ 207)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	6/10/2003
2,2',3,3',4,4',5,6-Octachlorobiphenyl (BZ 195)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	6/10/2003
2,2',3,3',4,4',5-Heptachlorobiphenyl (BZ 170)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	6/10/2003
2,2',3,3',4,4',6,6'-Octachlorobiphenyl (BZ 197)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	6/10/2003
2,2',3,3',4,4',6-Heptachlorobiphenyl (BZ 171)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	6/10/2003
2,2',3,3',4,4'-Hexachlorobiphenyl (BZ 128)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	6/10/2003
2,2',3,3',4,5,5',6,6'-Nonachlorobiphenyl (BZ 208)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	6/10/2003
2,2',3,3',4,5,5',6-Octachlorobiphenyl (BZ 198)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	6/10/2003
2,2',3,3',4,5,5',6'-Octachlorobiphenyl (BZ 199)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	6/10/2003
2,2',3,3',4,5,5'-Heptachlorobiphenyl (BZ 172)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	6/10/2003
2,2',3,3',4,5,6,6'-Octachlorobiphenyl (BZ 200)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	6/10/2003
2,2',3,3',4,5',6,6'-Octachlorobiphenyl (BZ 201)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	6/10/2003
2,2',3,3',4,5,6-Heptachlorobiphenyl (BZ 173)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	6/10/2003
2,2',3,3',4,5,6'-Heptachlorobiphenyl (BZ 174)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	6/10/2003
2,2',3,3',4,5',6-Heptachlorobiphenyl (BZ 175)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	6/10/2003
2,2',3,3',4,5',6'-Heptachlorobiphenyl (BZ 177)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	6/10/2003

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NON-TRANSFERABLE 03/07/2005-E87570

Laboratory Scope of Accreditation

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State Laboratory ID: E87570

EPA Lab Code: CA00044

(916) 373-5600

E87570

STL Sacramento
880 Riverside Parkway
West Sacramento, CA 95605

Matrix: Non-Potable Water

Analyte	Method/Tech	Category	Certification Type	Effective Date
2,2',3,3',4,5-Hexachlorobiphenyl (BZ 129)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	6/10/2003
2,2',3,3',4,5'-Hexachlorobiphenyl (BZ 130)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	6/10/2003
2,2',3,3',4,6,6'-Heptachlorobiphenyl (BZ 176)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	6/10/2003
2,2',3,3',4,6-Hexachlorobiphenyl (BZ 131)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	6/10/2003
2,2',3,3',4-Pentachlorobiphenyl (BZ 82)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	6/10/2003
2,2',3,3',5,5',6,6'-Octachlorobiphenyl (BZ 202)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	6/10/2003
2,2',3,3',5,5',6-Heptachlorobiphenyl (BZ 178)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	6/10/2003
2,2',3,3',5,5'-Hexachlorobiphenyl (BZ 133)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	6/10/2003
2,2',3,3',5,6,6'-Heptachlorobiphenyl (BZ 179)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	6/10/2003
2,2',3,3',5,6-Hexachlorobiphenyl (BZ 134)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	6/10/2003
2,2',3,3',5,6'-Hexachlorobiphenyl (BZ 135)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	6/10/2003
2,2',3,3',5-Pentachlorobiphenyl (BZ 83)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	6/10/2003
2,2',3,3',6,6'-Hexachlorobiphenyl (BZ 136)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	6/10/2003
2,2',3,3',6-Pentachlorobiphenyl (BZ 84)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	6/10/2003
2,2',3,3'-Tetrachlorobiphenyl (BZ 40)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	6/10/2003
2,2',3,4,4',5,5',6-Octachlorobiphenyl (BZ 203)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	6/10/2003
2,2',3,4,4',5,5'-Heptachlorobiphenyl (BZ 180)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	6/10/2003
2,2',3,4,4',5,6,6'-Octachlorobiphenyl (BZ 204)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	6/10/2003
2,2',3,4,4',5,6-Heptachlorobiphenyl (BZ 181)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	6/10/2003
2,2',3,4,4',5'-Heptachlorobiphenyl (BZ 183)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	6/10/2003
2,2',3,4,4',5-Hexachlorobiphenyl (BZ 137)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	6/10/2003
2,2',3,4,4',5'-Hexachlorobiphenyl (BZ 138)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	6/10/2003
2,2',3,4,4',6-Heptachlorobiphenyl (BZ 184)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	6/10/2003
2,2',3,4,4',6'-Hexachlorobiphenyl (BZ 140)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	6/10/2003
2,2',3,4,5,5',6-Heptachlorobiphenyl (BZ 185)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	6/10/2003
2,2',3,4',5,5',6-Heptachlorobiphenyl (BZ 187)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	6/10/2003
2,2',3,4,5,5'-Hexachlorobiphenyl (BZ 141)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	6/10/2003
2,2',3,4',5,5'-Hexachlorobiphenyl (BZ 146)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	6/10/2003
2,2',3,4,5,6,6'-Heptachlorobiphenyl (BZ 186)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	6/10/2003
2,2',3,4',5,6,6'-Heptachlorobiphenyl (BZ 188)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	6/10/2003
2,2',3,4,5,6'-Hexachlorobiphenyl (BZ 143)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	6/10/2003
2,2',3,4',5,6-Hexachlorobiphenyl (BZ 147)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	6/10/2003
2,2',3,4',5,6'-Hexachlorobiphenyl (BZ 148)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	6/10/2003
2,2',3,4',5,6-Hexachlorobiphenyl (BZ 149)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	6/10/2003
2,2',3,4,5'-Pentachlorobiphenyl (BZ 87)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	6/10/2003
2,2',3,4,6,6'-Hexachlorobiphenyl (BZ 145)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	6/10/2003

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NON-TRANSFERABLE 03/07/2005-E87570



Laboratory Scope of Accreditation

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State Laboratory ID: E87570

EPA Lab Code: CA00044

(916) 373-5600

E87570

STL Sacramento
880 Riverside Parkway
West Sacramento, CA 95605

Matrix: Non-Potable Water

Analyte	Method/Tech	Category	Certification Type	Effective Date
2,2',3,4',6,6'-Hexachlorobiphenyl (BZ 150)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	6/10/2003
2,2',3,4,6-Pentachlorobiphenyl (BZ 88)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	6/10/2003
2,2',3,4',6-Pentachlorobiphenyl (BZ 91)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	6/10/2003
2,2',3,4'-Tetrachlorobiphenyl (BZ 42)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	6/10/2003
2,2',3,5,5',6-Hexachlorobiphenyl (BZ 151)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	6/10/2003
2,2',3,5,5'-Pentachlorobiphenyl (BZ 92)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	6/10/2003
2,2',3,5,6,6'-Hexachlorobiphenyl (BZ 152)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	6/10/2003
2,2',3,5,6'-Pentachlorobiphenyl (BZ 94)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	6/10/2003
2,2',3,5',6-Pentachlorobiphenyl (BZ 95)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	6/10/2003
2,2',3,5'-Tetrachlorobiphenyl (BZ 44)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	6/10/2003
2,2',3,6,6'-Pentachlorobiphenyl (BZ 96)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	6/10/2003
2,2',3,6-Tetrachlorobiphenyl (BZ 45)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	6/10/2003
2,2',3,6'-Tetrachlorobiphenyl (BZ 46)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	6/10/2003
2,2',3-Trichlorobiphenyl (BZ 16)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	6/10/2003
2,2',4,4',5,5'-Hexachlorobiphenyl (BZ 153)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	6/10/2003
2,2',4,4',5,6'-Hexachlorobiphenyl (BZ 154)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	6/10/2003
2,2',4,4',5-Pentachlorobiphenyl (BZ 99)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	6/10/2003
2,2',4,4',6,6'-Hexachlorobiphenyl (BZ 155)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	6/10/2003
2,2',4,4',6-Pentachlorobiphenyl (BZ 100)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	6/10/2003
2,2',4,4'-Tetrachlorobiphenyl (BZ 47)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	6/10/2003
2,2',4,5,5'-Pentachlorobiphenyl (BZ 101)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	6/10/2003
2,2',4,5,6'-Pentachlorobiphenyl (BZ 102)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	6/10/2003
2,2',4,5,6-Pentachlorobiphenyl (BZ 103)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	6/10/2003
2,2',4,5'-Tetrachlorobiphenyl (BZ 49)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	6/10/2003
2,2',4,6,6'-Pentachlorobiphenyl (BZ 104)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	6/10/2003
2,2',4,6-Tetrachlorobiphenyl (BZ 50)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	6/10/2003
2,2',4,6'-Tetrachlorobiphenyl (BZ 51)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	6/10/2003
2,2',4-Trichlorobiphenyl (BZ 17)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	6/10/2003
2,2',5,5'-Tetrachlorobiphenyl (BZ 52)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	6/10/2003
2,2',5,6'-Tetrachlorobiphenyl (BZ 53)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	6/10/2003
2,2',5-Trichlorobiphenyl (BZ 18)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	6/10/2003
2,2',6,6'-Tetrachlorobiphenyl (BZ 54)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	6/10/2003
2,2',6-Trichlorobiphenyl (BZ 19)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	6/10/2003
2,3,3',4,4',5,5',6-Octachlorobiphenyl (BZ 205)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	6/10/2003
2,3,3',4,4',5,5'-Heptachlorobiphenyl (BZ 189)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	6/10/2003
2,3,3',4,4',5,6-Heptachlorobiphenyl (BZ 190)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	6/10/2003

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NON-TRANSFERABLE 03/07/2005-E87570

Laboratory Scope of Accreditation

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EPA Lab Code: CA00044

(916) 373-5600

E87570

STL Sacramento
880 Riverside Parkway
West Sacramento, CA 95605

Matrix: Non-Potable Water

Analyte	Method/Tech	Category	Certification Type	Effective Date
2,3,3',4,4',5',6-Heptachlorobiphenyl (BZ 191)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	6/10/2003
2,3,3',4,4',5-Hexachlorobiphenyl (BZ 156)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	6/10/2003
2,3,3',4,4',5'-Hexachlorobiphenyl (BZ 157)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	6/10/2003
2,3,3',4,4',6-Hexachlorobiphenyl (BZ 158)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	6/10/2003
2,3,3',4,4'-Pentachlorobiphenyl (BZ 105)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	6/10/2003
2,3,3',4',5,5',6-Heptachlorobiphenyl (BZ 193)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	6/10/2003
2,3,3',4,5,5'-Hexachlorobiphenyl (BZ 159)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	6/10/2003
2,3,3',4',5,5'-Hexachlorobiphenyl (BZ 162)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	6/10/2003
2,3,3',4,5,6-Hexachlorobiphenyl (BZ 161)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	6/10/2003
2,3,3',4',5'-Pentachlorobiphenyl (BZ 122)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	6/10/2003
2,3,3',4,6-Pentachlorobiphenyl (BZ 109)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	6/10/2003
2,3,3',4',6-Pentachlorobiphenyl (BZ 110)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	6/10/2003
2,3,3',4-Tetrachlorobiphenyl (BZ 55)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	6/10/2003
2,3,3',5,6-Pentachlorobiphenyl (BZ 112)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	6/10/2003
2,3,3',5',6-Pentachlorobiphenyl (BZ 113)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	6/10/2003
2,3,3',5-Tetrachlorobiphenyl (BZ 57)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	6/10/2003
2,3,3',5'-Tetrachlorobiphenyl (BZ 58)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	6/10/2003
2,3,3',6-Tetrachlorobiphenyl (BZ 59)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	6/10/2003
2,3',4,4',5,5'-Hexachlorobiphenyl (BZ 167)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	6/10/2003
2,3,4,4',5,6-Hexachlorobiphenyl (BZ 166)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	6/10/2003
2,3',4,4',5',6-Hexachlorobiphenyl (BZ 168)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	6/10/2003
2',3,4,4',5-Pentachlorobiphenyl	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	6/10/2003
2,3,4,4',5-Pentachlorobiphenyl (BZ 114)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	6/10/2003
2,3',4,4',5-Pentachlorobiphenyl (BZ 118)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	6/10/2003
2,3',4,4',5'-Pentachlorobiphenyl (BZ 123)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	6/10/2003
2,3,4,4',6-Pentachlorobiphenyl (BZ 115)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	6/10/2003
2,3',4,4',6-Pentachlorobiphenyl (BZ 119)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	6/10/2003
2,3,4,4'-Tetrachlorobiphenyl (BZ 60)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	6/10/2003
2,3',4,4'-Tetrachlorobiphenyl (BZ 66)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	6/10/2003
2,3',4,5,5'-Pentachlorobiphenyl (BZ 120)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	6/10/2003
2,3',4',5,5'-Pentachlorobiphenyl (BZ 124)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	6/10/2003
2,3,4',5-Tetrachlorobiphenyl (BZ 63)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	6/10/2003
2,3',4,5-Tetrachlorobiphenyl (BZ 67)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	6/10/2003
2,3',4',5-Tetrachlorobiphenyl (BZ 70)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	6/10/2003
2,3,4,6,7,8-Hxcdf	EPA 1613	Extractable Organics	NELAP	9/24/2001
2,3,4',6-Tetrachlorobiphenyl (BZ 64)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	6/10/2003

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NON-TRANSFERABLE 03/07/2005-E87570

Laboratory Scope of Accreditation

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State Laboratory ID: **E87570**

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(916) 373-5600

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**STL Sacramento
880 Riverside Parkway
West Sacramento, CA 95605**

Matrix: Non-Potable Water

Analyte	Method/Tech	Category	Certification Type	Effective Date
2,3',4,6-Tetrachlorobiphenyl (BZ 69)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	6/10/2003
2,3',4',6-Tetrachlorobiphenyl (BZ 71)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	6/10/2003
2,3,4,7,8-Peocdf	EPA 1613	Extractable Organics	NELAP	9/24/2001
2,3,4'-Trichlorobiphenyl (BZ 22)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	6/10/2003
2,3',4-Trichlorobiphenyl (BZ 25)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	6/10/2003
2,3',4'-Trichlorobiphenyl (BZ 33)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	6/10/2003
2,3',5,5'-Tetrachlorobiphenyl (BZ 72)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	6/10/2003
2,3,5,6-Tetrachlorobiphenyl (BZ 65)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	6/10/2003
2,3',5',6-Tetrachlorobiphenyl (BZ 73)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	6/10/2003
2,3,5-Trichlorobiphenyl (BZ 23)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	6/10/2003
2,3',5-Trichlorobiphenyl (BZ 26)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	6/10/2003
2,3',5'-Trichlorobiphenyl (BZ 34)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	6/10/2003
2,3,5-Trimethylnaphthalene	SAC-ID-0015	Extractable Organics	NELAP	9/24/2001
2,3',6-Trichlorobiphenyl (BZ 27)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	6/10/2003
2,3,7,8-TCDF	EPA 1613	Extractable Organics	NELAP	9/24/2001
2,3,7,8-Tetrachlorodibenzo-p-dioxin	EPA 1613	Extractable Organics	NELAP	9/24/2001
2,3-Dichlorobiphenyl (BZ 5)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	6/10/2003
2,3'-Dichlorobiphenyl (BZ 6)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	6/10/2003
2,4,4',5-Tetrachlorobiphenyl (BZ 74)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	6/10/2003
2,4,4',6-Tetrachlorobiphenyl (BZ 75)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	6/10/2003
2,4,4'-Trichlorobiphenyl (BZ 28)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	6/10/2003
2,4,5-Trichlorobiphenyl (BZ 29)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	6/10/2003
2,4',5-Trichlorobiphenyl (BZ 31)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	6/10/2003
2,4,6-Trichlorobiphenyl (BZ 30)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	6/10/2003
2,4,6-Trinitrotoluene (2,4,6-TNT)	SAC-LC-0001	Extractable Organics	NELAP	9/24/2001
2,4'-DDD	SAC-ID-0014	Pesticides-Herbicides-PCB's	NELAP	9/24/2001
2,4'-DDE	SAC-ID-0014	Pesticides-Herbicides-PCB's	NELAP	9/24/2001
2,4'-DDT	SAC-ID-0014	Pesticides-Herbicides-PCB's	NELAP	9/24/2001
2,4'-Dichlorobiphenyl (BZ 8)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	6/10/2003
2,4-Dinitrotoluene (2,4-DNT)	SAC-LC-0001	Extractable Organics	NELAP	9/24/2001
2,5-Dichlorobiphenyl (BZ 9)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	6/10/2003
2,6-Dichlorobiphenyl (BZ 10)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	6/10/2003
2,6-Dimethylnaphthalene	SAC-ID-0015	Extractable Organics	NELAP	9/24/2001
2,6-Dinitrotoluene (2,6-DNT)	SAC-LC-0001	Extractable Organics	NELAP	9/24/2001
2-Amino-4,6-dinitrotoluene (2-am-dnt)	SAC-LC-0001	Extractable Organics	NELAP	9/24/2001
2-Chlorobiphenyl (BZ 1)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	6/10/2003

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Laboratory Scope of Accreditation

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State Laboratory ID: E87570

EPA Lab Code: CA00044

(916) 373-5600

E87570
STL Sacramento
880 Riverside Parkway
West Sacramento, CA 95605

Matrix: Non-Potable Water

Analyte	Method/Tech	Category	Certification Type	Effective Date
2-Methylnaphthalene	SAC-ID-0015	Extractable Organics	NELAP	9/24/2001
2-Nitrotoluene	SAC-LC-0001	Extractable Organics	NELAP	9/24/2001
3,3',4,4',5,5'-Hexachlorobiphenyl (BZ 169)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	6/10/2003
3,3',4,4',5-Pentachlorobiphenyl (BZ 126)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	6/10/2003
3,3',4,4'-Tetrachlorobiphenyl (BZ 77)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	6/10/2003
3,3',4,5-Tetrachlorobiphenyl (BZ 78)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	6/10/2003
3,3',4,5'-Tetrachlorobiphenyl (BZ 79)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	6/10/2003
3,3',4-Trichlorobiphenyl (BZ 35)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	6/10/2003
3,3',5-Trichlorobiphenyl (BZ 36)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	6/10/2003
3,3'-Dichlorobiphenyl (BZ 11)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	6/10/2003
3,4,4',5-Tetrachlorobiphenyl (BZ 81)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	6/10/2003
3,4,4'-Trichlorobiphenyl (BZ 37)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	6/10/2003
3,4,5-Trichlorobiphenyl (BZ 38)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	6/10/2003
3,4',5-Trichlorobiphenyl (BZ 39)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	6/10/2003
3,4'-Dichlorobiphenyl (BZ 13)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	6/10/2003
3,5-Dichlorobiphenyl (BZ 14)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	6/10/2003
3-Chlorobiphenyl (BZ 2)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	6/10/2003
3-Nitrotoluene	SAC-LC-0001	Extractable Organics	NELAP	9/24/2001
4,4'-DDD	SAC-ID-0014	Pesticides-Herbicides-PCB's	NELAP	9/24/2001
4,4'-DDE	SAC-ID-0014	Pesticides-Herbicides-PCB's	NELAP	9/24/2001
4,4'-DDT	SAC-ID-0014	Pesticides-Herbicides-PCB's	NELAP	9/24/2001
4,4'-Dichlorobiphenyl (BZ 15)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	6/10/2003
4-Amino-2,6-dinitrotoluene (4-am-dnt)	SAC-LC-0001	Extractable Organics	NELAP	9/24/2001
4-Chlorobiphenyl (BZ 3)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	6/10/2003
4-Nitrotoluene	SAC-LC-0001	Extractable Organics	NELAP	9/24/2001
Acenaphthene	SAC-ID-0015	Extractable Organics	NELAP	9/24/2001
Acenaphthylene	SAC-ID-0015	Extractable Organics	NELAP	9/24/2001
Acetophenone	SAC-MS-0003	Extractable Organics	NELAP	11/7/2003
Aldrin	SAC-ID-0014	Pesticides-Herbicides-PCB's	NELAP	9/24/2001
Alkalinity as CaCO ₃	EPA 310.1	General Chemistry	NELAP	9/24/2001
Alkalinity as CaCO ₃	SM 2320 B	General Chemistry	NELAP	9/24/2001
alpha-BHC (alpha-Hexachlorocyclohexane)	SAC-ID-0014	Pesticides-Herbicides-PCB's	NELAP	9/24/2001
alpha-Chlordane	SAC-ID-0014	Pesticides-Herbicides-PCB's	NELAP	9/24/2001
Aluminum	EPA 6010	Metals	NELAP	7/1/2003
Amenable cyanide	SM 4500-CN G	General Chemistry	NELAP	9/24/2001
Ammonia as N	EPA 350.1	General Chemistry	NELAP	9/24/2001

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Laboratory Scope of Accreditation

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State Laboratory ID: E87570

EPA Lab Code: CA00044

(916) 373-5600

E87570
STL Sacramento
880 Riverside Parkway
West Sacramento, CA 95605

Matrix: Non-Potable Water

Analyte	Method/Tech	Category	Certification Type	Effective Date
Anthracene	SAC-ID-0015	Extractable Organics	NELAP	9/24/2001
Benzo(a)anthracene	SAC-ID-0015	Extractable Organics	NELAP	9/24/2001
Benzo(a)pyrene	SAC-ID-0015	Extractable Organics	NELAP	9/24/2001
Benzo(b)fluoranthene	SAC-ID-0015	Extractable Organics	NELAP	9/24/2001
Benzo(e)pyrene	SAC-ID-0015	Extractable Organics	NELAP	9/24/2001
Benzo(g,h,i)perylene	SAC-ID-0015	Extractable Organics	NELAP	9/24/2001
Benzo(k)fluoranthene	SAC-ID-0015	Extractable Organics	NELAP	9/24/2001
Benzothiazole	SAC-MS-0003	Extractable Organics	NELAP	11/7/2003
beta-BHC (beta-Hexachlorocyclohexane)	SAC-ID-0014	Pesticides-Herbicides-PCB's	NELAP	9/24/2001
Biphenyl	SAC-ID-0015	Extractable Organics	NELAP	9/24/2001
Bismuth	SAC-MT-0001	Metals	NELAP	7/1/2003
Boron	EPA 6010	Metals	NELAP	7/1/2003
Boron	SAC-MT-0001	Metals	NELAP	7/1/2003
Calcium	EPA 6010	Metals	NELAP	7/1/2003
Cerium	SAC-MT-0001	Metals	NELAP	7/1/2003
Chemical oxygen demand	EPA 410.4	General Chemistry	NELAP	9/24/2001
Chloride	EPA 300.0	General Chemistry	NELAP	9/24/2001
Chloropicrin	SAC-MS-0003	Extractable Organics	NELAP	11/7/2003
Chromium VI	SM3500Cr E	General Chemistry	NELAP	9/24/2001
Chrysene	SAC-ID-0015	Extractable Organics	NELAP	9/24/2001
cis-Nonachlor	SAC-ID-0014	Pesticides-Herbicides-PCB's	NELAP	9/24/2001
Conductivity	EPA 120.1	General Chemistry	NELAP	9/24/2001
Cyanide	SM 4500CN-E	General Chemistry	NELAP	9/24/2001
Decachlorobiphenyl (BZ 209)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	6/10/2003
delta-BHC	SAC-ID-0014	Pesticides-Herbicides-PCB's	NELAP	9/24/2001
Dibenz(a,h) anthracene	SAC-ID-0015	Extractable Organics	NELAP	9/24/2001
Dieldrin	SAC-ID-0014	Pesticides-Herbicides-PCB's	NELAP	9/24/2001
Diisopropyl methyl phosphonate	SAC-LC-0004	Extractable Organics	NELAP	9/24/2001
Dimethyl disulfide	SAC-MS-0003	Extractable Organics	NELAP	11/7/2003
Dimethyl methyl phosphonate	SAC-LC-0004	Extractable Organics	NELAP	9/24/2001
Endosulfan I	SAC-ID-0014	Pesticides-Herbicides-PCB's	NELAP	9/24/2001
Endosulfan II	SAC-ID-0014	Pesticides-Herbicides-PCB's	NELAP	9/24/2001
Endosulfan sulfate	SAC-ID-0014	Pesticides-Herbicides-PCB's	NELAP	9/24/2001
Endrin	SAC-ID-0014	Pesticides-Herbicides-PCB's	NELAP	9/24/2001
Endrin aldehyde	SAC-ID-0014	Pesticides-Herbicides-PCB's	NELAP	9/24/2001
Endrin ketone	SAC-ID-0014	Pesticides-Herbicides-PCB's	NELAP	9/24/2001

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State Laboratory ID: E87570

EPA Lab Code: CA00044

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STL Sacramento
880 Riverside Parkway
West Sacramento, CA 95605

Matrix: Non-Potable Water

Analyte	Method/Tech	Category	Certification Type	Effective Date
Ethylmethylphosphonic acid	SAC-LC-0004	Extractable Organics	NELAP	9/24/2001
Fluoranthene	SAC-ID-0015	Extractable Organics	NELAP	9/24/2001
Fluorene	SAC-ID-0015	Extractable Organics	NELAP	9/24/2001
Fluoride	EPA 340.2	General Chemistry	NELAP	9/24/2001
gamma-BHC (Lindane, gamma-Hexachlorocyclohexane)	SAC-ID-0014	Pesticides-Herbicides-PCB's	NELAP	9/24/2001
gamma-Chlordane	SAC-ID-0014	Pesticides-Herbicides-PCB's	NELAP	9/24/2001
Hardness	EPA 130.2	General Chemistry	NELAP	9/24/2001
Heptachlor	SAC-ID-0014	Pesticides-Herbicides-PCB's	NELAP	9/24/2001
Heptachlor epoxide	SAC-ID-0014	Pesticides-Herbicides-PCB's	NELAP	9/24/2001
Hexachlorobenzene	SAC-ID-0014	Pesticides-Herbicides-PCB's	NELAP	9/24/2001
Indeno(1,2,3-cd)pyrene	SAC-ID-0015	Extractable Organics	NELAP	9/24/2001
Iron	EPA 6010	Metals	NELAP	7/1/2003
Iron	SAC-MT-0001	Metals	NELAP	7/1/2003
Isopropylmethylphosphonic acid	SAC-LC-0004	Extractable Organics	NELAP	9/24/2001
Kjeldahl nitrogen - total	EPA 351.2	General Chemistry	NELAP	9/24/2001
Lithium	EPA 6010	Metals	NELAP	7/1/2003
Lithium	SAC-MT-0001	Metals	NELAP	7/1/2003
Magnesium	EPA 6010	Metals	NELAP	7/1/2003
Magnesium	SAC-MT-0001	Metals	NELAP	7/1/2003
Manganese	EPA 6010	Metals	NELAP	7/1/2003
Methoxychlor	SAC-ID-0014	Pesticides-Herbicides-PCB's	NELAP	9/24/2001
Methylphosphonic acid	SAC-LC-0004	Extractable Organics	NELAP	9/24/2001
Mirex	SAC-ID-0014	Pesticides-Herbicides-PCB's	NELAP	9/24/2001
Naphthalene	SAC-ID-0015	Extractable Organics	NELAP	9/24/2001
Nitrate as N	EPA 300.0	General Chemistry	NELAP	9/24/2001
Nitrate as N	EPA 353.2	General Chemistry	NELAP	9/24/2001
Nitrate-nitrite	EPA 300.0	General Chemistry	NELAP	9/24/2001
Nitrate-nitrite	EPA 353.2	General Chemistry	NELAP	9/24/2001
Nitrite as N	EPA 300.0	General Chemistry	NELAP	9/24/2001
Nitrite as N	EPA 353.2	General Chemistry	NELAP	9/24/2001
Nitrobenzene	SAC-LC-0001	Extractable Organics	NELAP	9/24/2001
Nitrocellulose	SAC-WC-0050	General Chemistry	NELAP	9/24/2001
Nitroglycerin	SAC-LC-0009	Extractable Organics	NELAP	11/7/2003
Nitroguanidine	SAC-LC-0010	Extractable Organics	NELAP	11/7/2003
o-Chloroacetophenone	SAC-MS-0003	Extractable Organics	NELAP	11/7/2003
Octahydro-1,3,5,7-tetranitro-1,3,5,7-tetrazocine (HMX)	SAC-LC-0001	Extractable Organics	NELAP	9/24/2001

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NON-TRANSFERABLE 03/07/2005-E87570

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Laboratory Scope of Accreditation

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State Laboratory ID: E87570

EPA Lab Code: CA00044

(916) 373-5600

E87570

STL Sacramento

880 Riverside Parkway

West Sacramento, CA 95605

Matrix: Non-Potable Water

Analyte	Method/Tech	Category	Certification Type	Effective Date
Oil & Grease	EPA 1664	General Chemistry	NELAP	9/24/2001
Orthophosphate as P	EPA 300.0	General Chemistry	NELAP	9/24/2001
Orthophosphate as P	EPA 365.3	General Chemistry	NELAP	9/24/2001
Oxychlorodane	SAC-ID-0014	Pesticides-Herbicides-PCB's	NELAP	9/24/2001
p-Chlorophenyl methyl sulfide	SAC-MS-0003	Extractable Organics	NELAP	11/7/2003
p-Chlorophenyl methyl sulfone	SAC-MS-0003	Extractable Organics	NELAP	11/7/2003
p-Chlorophenyl methyl sulfoxide	SAC-MS-0003	Extractable Organics	NELAP	11/7/2003
p-Dithiane	SAC-MS-0003	Extractable Organics	NELAP	11/7/2003
Pentaerythritoltetranitrate	SAC-LC-0009	Extractable Organics	NELAP	11/7/2003
Perchlorate	SAC-LC-0012	General Chemistry	NELAP	11/7/2003
Perylene	SAC-ID-0015	Extractable Organics	NELAP	9/24/2001
pH	EPA 150.1	General Chemistry	NELAP	9/24/2001
Phenanthrene	SAC-ID-0015	Extractable Organics	NELAP	9/24/2001
Phosphorus, total	EPA 365.3	General Chemistry	NELAP	9/24/2001
Phosphorus, total	EPA 6010	Metals	NELAP	7/1/2003
Phosphorus, total	SAC-MT-0001	Metals	NELAP	7/1/2003
Potassium	SAC-MT-0001	Metals	NELAP	7/1/2003
p-Oxathiane	SAC-MS-0003	Extractable Organics	NELAP	11/7/2003
Pyrene	SAC-ID-0015	Extractable Organics	NELAP	9/24/2001
RDX (hexahydro-1,3,5-trinitro-1,3,5-triazine)	SAC-LC-0001	Extractable Organics	NELAP	9/24/2001
Residue-filterable (TDS)	EPA 160.1	General Chemistry	NELAP	9/24/2001
Residue-filterable (TDS)	SM 2540 C	General Chemistry	NELAP	9/24/2001
Residue-nonfilterable (TSS)	EPA 160.2	General Chemistry	NELAP	9/24/2001
Residue-settleable	EPA 160.5	General Chemistry	NELAP	9/24/2001
Residue-total	EPA 160.3	General Chemistry	NELAP	9/24/2001
Residue-volatile	EPA 160.4	General Chemistry	NELAP	9/24/2001
Sodium	SAC-MT-0001	Metals	NELAP	7/1/2003
Strontium	SAC-MT-0001	Metals	NELAP	7/1/2003
Sulfate	EPA 300.0	General Chemistry	NELAP	9/24/2001
Sulfide	EPA 376.2	General Chemistry	NELAP	9/24/2001
Tetryl (methyl-2,4,6-trinitrophenylnitramine)	SAC-LC-0001	Extractable Organics	NELAP	9/24/2001
Thiodiglycol	SAC-LC-0004	Extractable Organics	NELAP	9/24/2001
Thulium	SAC-MT-0001	Metals	NELAP	7/1/2003
Tin	SAC-MT-0001	Metals	NELAP	7/1/2003
Titanium	SAC-MT-0001	Metals	NELAP	7/1/2003
Total organic carbon	EPA 415.1	General Chemistry	NELAP	9/24/2001

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Laboratory Scope of Accreditation

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STL Sacramento
880 Riverside Parkway
West Sacramento, CA 95605

Matrix: Non-Potable Water

Analyte	Method/Tech	Category	Certification Type	Effective Date
Total organic carbon	SM 5310C	General Chemistry	NELAP	9/24/2001
Total phenolics	EPA 420.4	General Chemistry	NELAP	9/24/2001
trans Nanochlor	SAC-ID-0014	Pesticides-Herbicides-PCB's	NELAP	9/24/2001
Turbidity	EPA 180.1	General Chemistry	NELAP	9/24/2001
Uranium	SAC-MT-0001	Metals	NELAP	7/1/2003
Zirconium	SAC-MT-0001	Metals	NELAP	7/1/2003

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STL Sacramento
880 Riverside Parkway
West Sacramento, CA 95605

Matrix: Solid and Chemical Materials

Analyte	Method/Tech	Category	Certification Type	Effective Date
1,1,1,2-Tetrachloroethane	EPA 8260	Volatile Organics	NELAP	9/24/2001
1,1,1-Trichloroethane	EPA 8260	Volatile Organics	NELAP	9/24/2001
1,1,2,2-Tetrachloroethane	EPA 8260	Volatile Organics	NELAP	9/24/2001
1,1,2-Trichloroethane	EPA 8260	Volatile Organics	NELAP	9/24/2001
1,1-Dichloroethane	EPA 8260	Volatile Organics	NELAP	9/24/2001
1,1-Dichloroethylene	EPA 8260	Volatile Organics	NELAP	9/24/2001
1,1-Dichloropropene	EPA 8260	Volatile Organics	NELAP	9/24/2001
1,2,3,4,6,7,8,9-Octachlorodibenzofuran (OCDF)	EPA 8290	Extractable Organics	NELAP	9/24/2001
1,2,3,4,6,7,8,9-Octachlorodibenzo-p-dioxin (OCDD)	EPA 8290	Extractable Organics	NELAP	9/24/2001
1,2,3,4,6,7,8-Heptachlorodibenzofuran (1,2,3,4,6,7,8-hpcdf)	EPA 8290	Extractable Organics	NELAP	9/24/2001
1,2,3,4,6,7,8-Heptachlorodibenzo-p-dioxin (1,2,3,4,6,7,8-hpcdd)	EPA 8290	Extractable Organics	NELAP	9/24/2001
1,2,3,4,7,8,9-Heptachlorodibenzofuran (1,2,3,4,7,8,9-hpcdf)	EPA 8290	Extractable Organics	NELAP	9/24/2001
1,2,3,4,7,8-Hxcdd	EPA 8280	Extractable Organics	NELAP	9/24/2001
1,2,3,4,7,8-Hxcdd	EPA 8290	Extractable Organics	NELAP	9/24/2001
1,2,3,4,7,8-Hxcdf	EPA 8280	Extractable Organics	NELAP	9/24/2001
1,2,3,4,7,8-Hxcdf	EPA 8290	Extractable Organics	NELAP	9/24/2001
1,2,3,6,7,8-Hxcdd	EPA 8280	Extractable Organics	NELAP	9/24/2001
1,2,3,6,7,8-Hxcdd	EPA 8290	Extractable Organics	NELAP	9/24/2001
1,2,3,6,7,8-Hxcdf	EPA 8280	Extractable Organics	NELAP	9/24/2001
1,2,3,6,7,8-Hxcdf	EPA 8290	Extractable Organics	NELAP	9/24/2001
1,2,3,7,8,9-Hxcdd	EPA 8280	Extractable Organics	NELAP	9/24/2001
1,2,3,7,8,9-Hxcdd	EPA 8290	Extractable Organics	NELAP	9/24/2001
1,2,3,7,8,9-Hxcdf	EPA 8280	Extractable Organics	NELAP	9/24/2001
1,2,3,7,8,9-Hxcdf	EPA 8290	Extractable Organics	NELAP	9/24/2001
1,2,3,7,8-Pecdd	EPA 8280	Extractable Organics	NELAP	9/24/2001
1,2,3,7,8-Pecdd	EPA 8290	Extractable Organics	NELAP	9/24/2001
1,2,3,7,8-Pecdf	EPA 8280	Extractable Organics	NELAP	9/24/2001
1,2,3,7,8-Pecdf	EPA 8290	Extractable Organics	NELAP	9/24/2001
1,2,3-Trichlorobenzene	EPA 8260	Volatile Organics	NELAP	9/24/2001
1,2,3-Trichloropropane	EPA 8260	Volatile Organics	NELAP	9/24/2001
1,2,4,5-Tetrachlorobenzene	EPA 8270	Extractable Organics	NELAP	9/24/2001
1,2,4-Trichlorobenzene	EPA 8260	Volatile Organics	NELAP	9/24/2001
1,2,4-Trichlorobenzene	EPA 8270	Extractable Organics	NELAP	9/24/2001
1,2-Dibromo-3-chloropropane (DBCP)	EPA 8260	Volatile Organics	NELAP	9/24/2001
1,2-Dibromoethane (EDB, Ethylene dibromide)	EPA 8260	Volatile Organics	NELAP	9/24/2001

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STL Sacramento
880 Riverside Parkway
West Sacramento, CA 95605

Matrix: Solid and Chemical Materials

Analyte	Method/Tech	Category	Certification Type	Effective Date
1,2-Dichlorobenzene	EPA 8260	Volatile Organics	NELAP	9/24/2001
1,2-Dichlorobenzene	EPA 8270	Extractable Organics	NELAP	9/24/2001
1,2-Dichloroethane	EPA 8260	Volatile Organics	NELAP	9/24/2001
1,2-Dichloropropane	EPA 8260	Volatile Organics	NELAP	9/24/2001
1,2-Dinitrobenzene	EPA 8270	Extractable Organics	NELAP	9/24/2001
1,2-Diphenylhydrazine	EPA 8270	Extractable Organics	NELAP	9/24/2001
1,3,5-Trinitrobenzene (1,3,5-TNB)	EPA 8095	Extractable Organics	NELAP	11/7/2003
1,3,5-Trinitrobenzene (1,3,5-TNB)	EPA 8270	Extractable Organics	NELAP	9/24/2001
1,3,5-Trinitrobenzene (1,3,5-TNB)	EPA 8330	Extractable Organics	NELAP	9/24/2001
1,3,5-Trinitrobenzene (1,3,5-TNB)	SAC-LC-0001	Extractable Organics	NELAP	9/24/2001
1,3-Dichlorobenzene	EPA 8260	Volatile Organics	NELAP	9/24/2001
1,3-Dichlorobenzene	EPA 8270	Extractable Organics	NELAP	9/24/2001
1,3-Dichloropropane	EPA 8260	Volatile Organics	NELAP	9/24/2001
1,3-Dinitrobenzene (1,3-DNB)	EPA 8095	Extractable Organics	NELAP	11/7/2003
1,3-Dinitrobenzene (1,3-DNB)	EPA 8270	Extractable Organics	NELAP	9/24/2001
1,3-Dinitrobenzene (1,3-DNB)	EPA 8330	Extractable Organics	NELAP	9/24/2001
1,3-Dinitrobenzene (1,3-DNB)	SAC-LC-0001	Extractable Organics	NELAP	9/24/2001
1,4-Dichlorobenzene	EPA 8260	Volatile Organics	NELAP	9/24/2001
1,4-Dichlorobenzene	EPA 8270	Extractable Organics	NELAP	9/24/2001
1,4-Dinitrobenzene	EPA 8270	Extractable Organics	NELAP	9/24/2001
1,4-Naphthoquinone	EPA 8270	Extractable Organics	NELAP	9/24/2001
1,4-Phenylenediamine	EPA 8270	Extractable Organics	NELAP	9/24/2001
1-Chloronaphthalene	EPA 8270	Extractable Organics	NELAP	9/24/2001
1-Methylnaphthalene	SAC-ID-0015	Extractable Organics	NELAP	9/24/2001
1-Methylphenanthrene	SAC-ID-0015	Extractable Organics	NELAP	9/24/2001
1-Naphthylamine	EPA 8270	Extractable Organics	NELAP	9/24/2001
2,2',3,3',4,4',5,5',6'-Nonachlorobiphenyl (BZ 206)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	6/10/2003
2,2',3,3',4,4',5,5',6'-Octachlorobiphenyl (BZ 194)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	6/10/2003
2,2',3,3',4,4',5,5',6'-Nonachlorobiphenyl (BZ 207)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	6/10/2003
2,2',3,3',4,4',5,6'-Octachlorobiphenyl (BZ 195)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	6/10/2003
2,2',3,3',4,4',5-Heptachlorobiphenyl (BZ 170)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	6/10/2003
2,2',3,3',4,4',6'-Octachlorobiphenyl (BZ 197)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	6/10/2003
2,2',3,3',4,4',6-Heptachlorobiphenyl (BZ 171)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	6/10/2003
2,2',3,3',4,4'-Hexachlorobiphenyl (BZ 128)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	6/10/2003
2,2',3,3',4,5,5',6'-Nonachlorobiphenyl (BZ 208)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	6/10/2003
2,2',3,3',4,5,5',6-Octachlorobiphenyl (BZ 198)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	6/10/2003

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NON-TRANSFERABLE 03/07/2005-E87570

Jeb Bush
Governor



John O. Agwunobi, M.D., M.B.A.
Secretary

Laboratory Scope of Accreditation

Page 14 of 34

THIS LISTING OF ACCREDITED ANALYTES SHOULD BE USED ONLY WHEN
ASSOCIATED WITH A VALID CERTIFICATE

State Laboratory ID: E87570

EPA Lab Code: CA00044

(916) 373-5600

E87570

STL Sacramento

880 Riverside Parkway

West Sacramento, CA 95605

Matrix: Solid and Chemical Materials

Analyte	Method/Tech	Category	Certification Type	Effective Date
2,2',3,3',4,5,5',6'-Octachlorobiphenyl (BZ 199)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	6/10/2003
2,2',3,3',4,5,5'-Heptachlorobiphenyl (BZ 172)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	6/10/2003
2,2',3,3',4,5,6,6'-Octachlorobiphenyl (BZ 200)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	6/10/2003
2,2',3,3',4,5',6,6'-Octachlorobiphenyl (BZ 201)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	6/10/2003
2,2',3,3',4,5,6-Heptachlorobiphenyl (BZ 173)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	6/10/2003
2,2',3,3',4,5,6-Heptachlorobiphenyl (BZ 174)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	6/10/2003
2,2',3,3',4,5,6-Heptachlorobiphenyl (BZ 175)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	6/10/2003
2,2',3,3',4,5,6'-Heptachlorobiphenyl (BZ 177)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	6/10/2003
2,2',3,3',4,5-Hexachlorobiphenyl (BZ 129)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	6/10/2003
2,2',3,3',4,5'-Hexachlorobiphenyl (BZ 130)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	6/10/2003
2,2',3,3',4,6'-Heptachlorobiphenyl (BZ 176)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	6/10/2003
2,2',3,3',4,6-Hexachlorobiphenyl (BZ 131)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	6/10/2003
2,2',3,3',4-Pentachlorobiphenyl (BZ 82)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	6/10/2003
2,2',3,3',5,5',6,6'-Octachlorobiphenyl (BZ 202)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	6/10/2003
2,2',3,3',5,5',6-Heptachlorobiphenyl (BZ 178)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	6/10/2003
2,2',3,3',5,5'-Hexachlorobiphenyl (BZ 133)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	6/10/2003
2,2',3,3',5,6,6'-Heptachlorobiphenyl (BZ 179)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	6/10/2003
2,2',3,3',5,6-Hexachlorobiphenyl (BZ 134)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	6/10/2003
2,2',3,3',5,6'-Hexachlorobiphenyl (BZ 135)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	6/10/2003
2,2',3,3',5-Pentachlorobiphenyl (BZ 83)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	6/10/2003
2,2',3,3',6,6'-Hexachlorobiphenyl (BZ 136)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	6/10/2003
2,2',3,3',6-Pentachlorobiphenyl (BZ 84)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	6/10/2003
2,2',3,3'-Tetrachlorobiphenyl (BZ 40)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	6/10/2003
2,2',3,4,4',5,5',6-Octachlorobiphenyl (BZ 203)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	6/10/2003
2,2',3,4,4',5,5'-Heptachlorobiphenyl (BZ 180)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	6/10/2003
2,2',3,4,4',5,6,6'-Octachlorobiphenyl (BZ 204)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	6/10/2003
2,2',3,4,4',5,6-Heptachlorobiphenyl (BZ 181)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	6/10/2003
2,2',3,4,4',5,6-Heptachlorobiphenyl (BZ 183)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	6/10/2003
2,2',3,4,4',5-Hexachlorobiphenyl (BZ 137)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	6/10/2003
2,2',3,4,4',5'-Hexachlorobiphenyl (BZ 138)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	6/10/2003
2,2',3,4,4',6,6'-Heptachlorobiphenyl (BZ 184)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	6/10/2003
2,2',3,4,4',6-Hexachlorobiphenyl (BZ 140)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	6/10/2003
2,2',3,4,5,5',6-Heptachlorobiphenyl (BZ 185)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	6/10/2003
2,2',3,4,5,5',6-Heptachlorobiphenyl (BZ 187)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	6/10/2003
2,2',3,4,5,5'-Hexachlorobiphenyl (BZ 141)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	6/10/2003
2,2',3,4,5,5'-Hexachlorobiphenyl (BZ 146)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	6/10/2003

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NON-TRANSFERABLE 03/07/2005-E87570

Laboratory Scope of Accreditation

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State Laboratory ID: E87570

EPA Lab Code:

CA00044

(916) 373-5600

E87570

STL Sacramento
880 Riverside Parkway
West Sacramento, CA 95605

Matrix: Solid and Chemical Materials

Analyte	Method/Tech	Category	Certification Type	Effective Date
2,2',3,4,5,6,6'-Heptachlorobiphenyl (BZ 186)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	6/10/2003
2,2',3,4',5,6,6'-Heptachlorobiphenyl (BZ 188)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	6/10/2003
2,2',3,4,5,6'-Hexachlorobiphenyl (BZ 143)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	6/10/2003
2,2',3,4',5,6'-Hexachlorobiphenyl (BZ 147)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	6/10/2003
2,2',3,4',5,6'-Hexachlorobiphenyl (BZ 148)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	6/10/2003
2,2',3,4',5',6'-Hexachlorobiphenyl (BZ 149)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	6/10/2003
2,2',3,4,5'-Pentachlorobiphenyl (BZ 87)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	6/10/2003
2,2',3,4,6,6'-Hexachlorobiphenyl (BZ 145)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	6/10/2003
2,2',3,4',6,6'-Hexachlorobiphenyl (BZ 150)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	6/10/2003
2,2',3,4,6-Pentachlorobiphenyl (BZ 88)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	6/10/2003
2,2',3,4',6-Pentachlorobiphenyl (BZ 91)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	6/10/2003
2,2',3,4'-Tetrachlorobiphenyl (BZ 42)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	6/10/2003
2,2',3,5,5',6-Hexachlorobiphenyl (BZ 151)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	6/10/2003
2,2',3,5,5'-Pentachlorobiphenyl (BZ 92)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	6/10/2003
2,2',3,5,6,6'-Hexachlorobiphenyl (BZ 152)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	6/10/2003
2,2',3,5,6'-Pentachlorobiphenyl (BZ 94)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	6/10/2003
2,2',3,5',6-Pentachlorobiphenyl (BZ 95)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	6/10/2003
2,2',3,5'-Tetrachlorobiphenyl (BZ 44)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	6/10/2003
2,2',3,6,6'-Pentachlorobiphenyl (BZ 96)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	6/10/2003
2,2',3,6-Tetrachlorobiphenyl (BZ 45)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	6/10/2003
2,2',3,6'-Tetrachlorobiphenyl (BZ 46)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	6/10/2003
2,2',3-Trichlorobiphenyl (BZ 16)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	6/10/2003
2,2',4,4',5,5'-Hexachlorobiphenyl (BZ 153)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	6/10/2003
2,2',4,4',5,6'-Hexachlorobiphenyl (BZ 154)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	6/10/2003
2,2',4,4',5-Pentachlorobiphenyl (BZ 99)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	6/10/2003
2,2',4,4',6,6'-Hexachlorobiphenyl (BZ 155)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	6/10/2003
2,2',4,4',6-Pentachlorobiphenyl (BZ 100)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	6/10/2003
2,2',4,4'-Tetrachlorobiphenyl (BZ 47)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	6/10/2003
2,2',4,5,5'-Pentachlorobiphenyl (BZ 101)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	6/10/2003
2,2',4,5,6'-Pentachlorobiphenyl (BZ 102)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	6/10/2003
2,2',4,5',6-Pentachlorobiphenyl (BZ 103)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	6/10/2003
2,2',4,5'-Tetrachlorobiphenyl (BZ 49)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	6/10/2003
2,2',4,6,6'-Pentachlorobiphenyl (BZ 104)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	6/10/2003
2,2',4,6-Tetrachlorobiphenyl (BZ 50)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	6/10/2003
2,2',4,6'-Tetrachlorobiphenyl (BZ 51)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	6/10/2003
2,2',4-Trichlorobiphenyl (BZ 17)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	6/10/2003

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Laboratory Scope of Accreditation

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State Laboratory ID: E87570

EPA Lab Code: CA00044

(916) 373-5600

E87570

STL Sacramento
880 Riverside Parkway
West Sacramento, CA 95605

Matrix: Solid and Chemical Materials

Analyte	Method/Tech	Category	Certification Type	Effective Date
2,2',5,5'-Tetrachlorobiphenyl (BZ 52)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	6/10/2003
2,2',5,6'-Tetrachlorobiphenyl (BZ 53)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	6/10/2003
2,2',5-Trichlorobiphenyl (BZ 18)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	6/10/2003
2,2',6,6'-Tetrachlorobiphenyl (BZ 54)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	6/10/2003
2,2',6-Trichlorobiphenyl (BZ 19)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	6/10/2003
2,2-Dichloropropane	EPA 8260	Volatile Organics	NELAP	9/24/2001
2,3,3',4,4',5,5',6-Octachlorobiphenyl (BZ 205)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	6/10/2003
2,3,3',4,4',5,5'-Heptachlorobiphenyl (BZ 189)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	6/10/2003
2,3,3',4,4',5,6-Heptachlorobiphenyl (BZ 190)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	6/10/2003
2,3,3',4,4',5,6-Heptachlorobiphenyl (BZ 191)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	6/10/2003
2,3,3',4,4',5-Hexachlorobiphenyl (BZ 156)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	6/10/2003
2,3,3',4,4',5-Hexachlorobiphenyl (BZ 157)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	6/10/2003
2,3,3',4,4',6-Hexachlorobiphenyl (BZ 158)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	6/10/2003
2,3,3',4,4'-Pentachlorobiphenyl (BZ 105)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	6/10/2003
2,3,3',4',5,5',6-Heptachlorobiphenyl (BZ 193)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	6/10/2003
2,3,3',4,5,5'-Hexachlorobiphenyl (BZ 159)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	6/10/2003
2,3,3',4,5,5'-Hexachlorobiphenyl (BZ 162)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	6/10/2003
2,3,3',4,5,6-Hexachlorobiphenyl (BZ 161)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	6/10/2003
2,3,3',4',5'-Pentachlorobiphenyl (BZ 122)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	6/10/2003
2,3,3',4,6-Pentachlorobiphenyl (BZ 109)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	6/10/2003
2,3,3',4',6-Pentachlorobiphenyl (BZ 110)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	6/10/2003
2,3,3',4-Tetrachlorobiphenyl (BZ 55)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	6/10/2003
2,3,3',5,6-Pentachlorobiphenyl (BZ 112)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	6/10/2003
2,3,3',5,6-Pentachlorobiphenyl (BZ 113)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	6/10/2003
2,3,3',5-Tetrachlorobiphenyl (BZ 57)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	6/10/2003
2,3,3',5'-Tetrachlorobiphenyl (BZ 58)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	6/10/2003
2,3,3',6-Tetrachlorobiphenyl (BZ 59)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	6/10/2003
2,3',4,4',5,5'-Hexachlorobiphenyl (BZ 167)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	6/10/2003
2,3,4,4',5,6-Hexachlorobiphenyl (BZ 166)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	6/10/2003
2,3',4,4',5',6-Hexachlorobiphenyl (BZ 168)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	6/10/2003
2',3,4,4',5-Pentachlorobiphenyl	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	6/10/2003
2,3,4,4',5-Pentachlorobiphenyl (BZ 114)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	6/10/2003
2,3',4,4',5-Pentachlorobiphenyl (BZ 118)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	6/10/2003
2,3',4,4',5'-Pentachlorobiphenyl (BZ 123)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	6/10/2003
2,3,4,4',6-Pentachlorobiphenyl (BZ 115)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	6/10/2003
2,3',4,4',6-Pentachlorobiphenyl (BZ 119)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	6/10/2003

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Jeb Bush
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John O. Agwunobi, M.D., M.B.A.
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Laboratory Scope of Accreditation

Page 17 of 34

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STL Sacramento

880 Riverside Parkway

West Sacramento, CA 95605

Matrix: Solid and Chemical Materials

Analyte	Method/Tech	Category	Certification Type	Effective Date
2,3,4,4'-Tetrachlorobiphenyl (BZ 60)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	6/10/2003
2,3',4,4'-Tetrachlorobiphenyl (BZ 66)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	6/10/2003
2,3',4,5,5'-Pentachlorobiphenyl (BZ 120)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	6/10/2003
2,3',4',5,5'-Pentachlorobiphenyl (BZ 124)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	6/10/2003
2,3,4',5-Tetrachlorobiphenyl (BZ 63)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	6/10/2003
2,3',4,5-Tetrachlorobiphenyl (BZ 67)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	6/10/2003
2,3',4',5-Tetrachlorobiphenyl (BZ 70)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	6/10/2003
2,3,4,6,7,8-Hxcdf	EPA 8280	Extractable Organics	NELAP	9/24/2001
2,3,4,6,7,8-Hxcdf	EPA 8290	Extractable Organics	NELAP	9/24/2001
2,3,4',6-Tetrachlorobiphenyl (BZ 64)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	6/10/2003
2,3',4,6-Tetrachlorobiphenyl (BZ 69)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	6/10/2003
2,3',4',6-Tetrachlorobiphenyl (BZ 71)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	6/10/2003
2,3,4,6-Tetrachlorophenol	EPA 8270	Extractable Organics	NELAP	9/24/2001
2,3,4,7,8-Pecdf	EPA 8280	Extractable Organics	NELAP	9/24/2001
2,3,4,7,8-Pecdf	EPA 8290	Extractable Organics	NELAP	9/24/2001
2,3,4'-Trichlorobiphenyl (BZ 22)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	6/10/2003
2,3',4-Trichlorobiphenyl (BZ 25)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	6/10/2003
2,3',4'-Trichlorobiphenyl (BZ 33)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	6/10/2003
2,3',5,5'-Tetrachlorobiphenyl (BZ 72)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	6/10/2003
2,3,5,6-Tetrachlorobiphenyl (BZ 65)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	6/10/2003
2,3',5',6-Tetrachlorobiphenyl (BZ 73)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	6/10/2003
2,3,5-Trichlorobiphenyl (BZ 23)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	6/10/2003
2,3',5-Trichlorobiphenyl (BZ 26)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	6/10/2003
2,3',5'-Trichlorobiphenyl (BZ 34)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	6/10/2003
2,3,5-Trimethylnaphthalene	SAC-ID-0015	Extractable Organics	NELAP	9/24/2001
2,3',6-Trichlorobiphenyl (BZ 27)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	6/10/2003
2,3,7,8-TCDF	EPA 8280	Extractable Organics	NELAP	9/24/2001
2,3,7,8-TCDF	EPA 8290	Extractable Organics	NELAP	9/24/2001
2,3,7,8-Tetrachlorodibenzo-p-dioxin	EPA 8280	Extractable Organics	NELAP	9/24/2001
2,3,7,8-Tetrachlorodibenzo-p-dioxin	EPA 8290	Extractable Organics	NELAP	9/24/2001
2,3-Dichlorobiphenyl (BZ 5)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	6/10/2003
2,3'-Dichlorobiphenyl (BZ 6)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	6/10/2003
2,4,4',5-Tetrachlorobiphenyl (BZ 74)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	6/10/2003
2,4,4',6-Tetrachlorobiphenyl (BZ 75)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	6/10/2003
2,4,4'-Trichlorobiphenyl (BZ 28)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	6/10/2003
2,4,5-Trichlorobiphenyl (BZ 29)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	6/10/2003

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Laboratory Scope of Accreditation

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EPA Lab Code:

CA00044

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STL Sacramento
880 Riverside Parkway
West Sacramento, CA 95605

Matrix: Solid and Chemical Materials

Analyte	Method/Tech	Category	Certification Type	Effective Date
2,4',5-Trichlorobiphenyl (BZ 31)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	6/10/2003
2,4,5-Trichlorophenol	EPA 8270	Extractable Organics	NELAP	9/24/2001
2,4,6-Trichlorobiphenyl (BZ 30)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	6/10/2003
2,4,6-Trichlorophenol	EPA 8270	Extractable Organics	NELAP	9/24/2001
2,4,6-Trinitrophenylmethyl nitramine	EPA 8095	Extractable Organics	NELAP	11/7/2003
2,4,6-Trinitrotoluene (2,4,6-TNT)	EPA 8095	Extractable Organics	NELAP	11/7/2003
2,4,6-Trinitrotoluene (2,4,6-TNT)	EPA 8330	Extractable Organics	NELAP	9/24/2001
2,4,6-Trinitrotoluene (2,4,6-TNT)	SAC-LC-0001	Extractable Organics	NELAP	9/24/2001
2,4'-DDD	SAC-ID-0014	Pesticides-Herbicides-PCB's	NELAP	9/24/2001
2,4'-DDE	SAC-ID-0014	Pesticides-Herbicides-PCB's	NELAP	9/24/2001
2,4'-DDT	SAC-ID-0014	Pesticides-Herbicides-PCB's	NELAP	9/24/2001
2,4'-Dichlorobiphenyl (BZ 8)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	6/10/2003
2,4-Dichlorophenol	EPA 8270	Extractable Organics	NELAP	9/24/2001
2,4-Dimethylphenol	EPA 8270	Extractable Organics	NELAP	9/24/2001
2,4-Dinitrophenol	EPA 8270	Extractable Organics	NELAP	9/24/2001
2,4-Dinitrotoluene (2,4-DNT)	EPA 8095	Extractable Organics	NELAP	11/7/2003
2,4-Dinitrotoluene (2,4-DNT)	EPA 8270	Extractable Organics	NELAP	9/24/2001
2,4-Dinitrotoluene (2,4-DNT)	EPA 8330	Extractable Organics	NELAP	9/24/2001
2,4-Dinitrotoluene (2,4-DNT)	SAC-LC-0001	Extractable Organics	NELAP	9/24/2001
2,5-Dichlorobiphenyl (BZ 9)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	6/10/2003
2,6-Dichlorobiphenyl (BZ 10)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	6/10/2003
2,6-Dichlorophenol	EPA 8270	Extractable Organics	NELAP	9/24/2001
2,6-Dimethylnaphthalene	SAC-ID-0015	Extractable Organics	NELAP	9/24/2001
2,6-Dinitrotoluene (2,6-DNT)	EPA 8095	Extractable Organics	NELAP	11/7/2003
2,6-Dinitrotoluene (2,6-DNT)	EPA 8270	Extractable Organics	NELAP	9/24/2001
2,6-Dinitrotoluene (2,6-DNT)	EPA 8330	Extractable Organics	NELAP	9/24/2001
2,6-Dinitrotoluene (2,6-DNT)	SAC-LC-0001	Extractable Organics	NELAP	9/24/2001
2-Acetylaminofluorene	EPA 8270	Extractable Organics	NELAP	9/24/2001
2-Amino-4,6-dinitrotoluene (2-am-dnt)	EPA 8095	Extractable Organics	NELAP	11/7/2003
2-Amino-4,6-dinitrotoluene (2-am-dnt)	EPA 8330	Extractable Organics	NELAP	9/24/2001
2-Amino-4,6-dinitrotoluene (2-am-dnt)	SAC-LC-0001	Extractable Organics	NELAP	9/24/2001
2-Butanone (Methyl ethyl ketone, MEK)	EPA 8260	Volatile Organics	NELAP	9/24/2001
2-Chlorobiphenyl (BZ 1)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	6/10/2003
2-Chloroethyl vinyl ether	EPA 8260	Volatile Organics	NELAP	9/24/2001
2-Chloronaphthalene	EPA 8270	Extractable Organics	NELAP	9/24/2001
2-Chlorophenol	EPA 8270	Extractable Organics	NELAP	9/24/2001

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880 Riverside Parkway
West Sacramento, CA 95605

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Analyte	Method/Tech	Category	Certification Type	Effective Date
2-Hexanone	EPA 8260	Volatile Organics	NELAP	9/24/2001
2-Methyl-4,6-dinitrophenol	EPA 8270	Extractable Organics	NELAP	9/24/2001
2-Methylnaphthalene	EPA 8270	Extractable Organics	NELAP	9/24/2001
2-Methylnaphthalene	SAC-ID-0015	Extractable Organics	NELAP	9/24/2001
2-Methylphenol (o-Cresol)	EPA 8270	Extractable Organics	NELAP	9/24/2001
2-Nitroaniline	EPA 8270	Extractable Organics	NELAP	9/24/2001
2-Nitrophenol	EPA 8270	Extractable Organics	NELAP	9/24/2001
2-Nitrotoluene	EPA 8095	Extractable Organics	NELAP	11/7/2003
2-Nitrotoluene	EPA 8330	Extractable Organics	NELAP	9/24/2001
2-Nitrotoluene	SAC-LC-0001	Extractable Organics	NELAP	9/24/2001
2-Picoline (2-Methylpyridine)	EPA 8270	Extractable Organics	NELAP	9/24/2001
3,3',4,4',5,5'-Hexachlorobiphenyl (BZ 169)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	6/10/2003
3,3',4,4',5-Pentachlorobiphenyl (BZ 126)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	6/10/2003
3,3',4,4'-Tetrachlorobiphenyl (BZ 77)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	6/10/2003
3,3',4,5-Tetrachlorobiphenyl (BZ 78)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	6/10/2003
3,3',4,5'-Tetrachlorobiphenyl (BZ 79)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	6/10/2003
3,3',4-Trichlorobiphenyl (BZ 35)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	6/10/2003
3,3',5-Trichlorobiphenyl (BZ 36)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	6/10/2003
3,3'-Dichlorobenzidine	EPA 8270	Extractable Organics	NELAP	9/24/2001
3,3'-Dichlorobiphenyl (BZ 11)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	6/10/2003
3,4,4',5-Tetrachlorobiphenyl (BZ 81)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	6/10/2003
3,4,4'-Trichlorobiphenyl (BZ 37)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	6/10/2003
3,4,5-Trichlorobiphenyl (BZ 38)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	6/10/2003
3,4',5-Trichlorobiphenyl (BZ 39)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	6/10/2003
3,4'-Dichlorobiphenyl (BZ 13)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	6/10/2003
3,5-Dichlorobiphenyl (BZ 14)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	6/10/2003
3-Chlorobiphenyl (BZ 2)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	6/10/2003
3-Methylcholanthrene	EPA 8270	Extractable Organics	NELAP	9/24/2001
3-Methylphenol (m-Cresol)	EPA 8270	Extractable Organics	NELAP	9/24/2001
3-Nitroaniline	EPA 8270	Extractable Organics	NELAP	9/24/2001
3-Nitrotoluene	EPA 8095	Extractable Organics	NELAP	11/7/2003
3-Nitrotoluene	EPA 8330	Extractable Organics	NELAP	9/24/2001
3-Nitrotoluene	SAC-LC-0001	Extractable Organics	NELAP	9/24/2001
4,4'-DDD	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	9/24/2001
4,4'-DDD	SAC-ID-0014	Pesticides-Herbicides-PCB's	NELAP	9/24/2001
4,4'-DDE	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	9/24/2001

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4,4'-DDE	SAC-ID-0014	Pesticides-Herbicides-PCB's	NELAP	9/24/2001
4,4'-DDT	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	9/24/2001
4,4'-DDT	SAC-ID-0014	Pesticides-Herbicides-PCB's	NELAP	9/24/2001
4,4'-Dichlorobiphenyl (BZ 15)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	6/10/2003
4-Amino-2,6-dinitrotoluene (4-am-dnt)	EPA 8095	Extractable Organics	NELAP	11/7/2003
4-Amino-2,6-dinitrotoluene (4-am-dnt)	EPA 8330	Extractable Organics	NELAP	9/24/2001
4-Amino-2,6-dinitrotoluene (4-am-dnt)	SAC-LC-0001	Extractable Organics	NELAP	9/24/2001
4-Aminobiphenyl	EPA 8270	Extractable Organics	NELAP	9/24/2001
4-Bromophenyl phenyl ether	EPA 8270	Extractable Organics	NELAP	9/24/2001
4-Chloro-3-methylphenol	EPA 8270	Extractable Organics	NELAP	9/24/2001
4-Chloroaniline	EPA 8270	Extractable Organics	NELAP	9/24/2001
4-Chlorobiphenyl (BZ 3)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	6/10/2003
4-Chlorophenyl phenylether	EPA 8270	Extractable Organics	NELAP	9/24/2001
4-Dimethyl aminoazobenzene	EPA 8270	Extractable Organics	NELAP	9/24/2001
4-Methyl-2-pentanone (MIBK)	EPA 8260	Volatile Organics	NELAP	9/24/2001
4-Methylphenol (p-Cresol)	EPA 8270	Extractable Organics	NELAP	9/24/2001
4-Nitroaniline	EPA 8270	Extractable Organics	NELAP	9/24/2001
4-Nitrophenol	EPA 8270	Extractable Organics	NELAP	9/24/2001
4-Nitrotoluene	EPA 8095	Extractable Organics	NELAP	11/7/2003
4-Nitrotoluene	EPA 8330	Extractable Organics	NELAP	9/24/2001
4-Nitrotoluene	SAC-LC-0001	Extractable Organics	NELAP	9/24/2001
5-Nitro-o-toluidine	EPA 8270	Extractable Organics	NELAP	9/24/2001
7,12-Dimethylbenz(a) anthracene	EPA 8270	Extractable Organics	NELAP	9/24/2001
a-a-Dimethylphenethylamine	EPA 8270	Extractable Organics	NELAP	9/24/2001
Acenaphthene	EPA 8270	Extractable Organics	NELAP	9/24/2001
Acenaphthene	EPA 8310	Extractable Organics	NELAP	9/24/2001
Acenaphthene	SAC-ID-0015	Extractable Organics	NELAP	9/24/2001
Acenaphthylene	EPA 8270	Extractable Organics	NELAP	9/24/2001
Acenaphthylene	EPA 8310	Extractable Organics	NELAP	9/24/2001
Acenaphthylene	SAC-ID-0015	Extractable Organics	NELAP	9/24/2001
Acetone	EPA 8260	Volatile Organics	NELAP	9/24/2001
Acetonitrile	EPA 8260	Volatile Organics	NELAP	9/24/2001
Acetophenone	EPA 8270	Extractable Organics	NELAP	9/24/2001
Acetophenone	SAC-MS-0003	Extractable Organics	NELAP	11/7/2003
Acrolein (Propenal)	EPA 8260	Volatile Organics	NELAP	9/24/2001
Acrylonitrile	EPA 8260	Volatile Organics	NELAP	9/24/2001

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Analyte	Method/Tech	Category	Certification Type	Effective Date
Aldrin	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	9/24/2001
Aldrin	SAC-ID-0014	Pesticides-Herbicides-PCB's	NELAP	9/24/2001
Allyl chloride (3-Chloropropene)	EPA 8260	Volatile Organics	NELAP	9/24/2001
alpha-BHC (alpha-Hexachlorocyclohexane)	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	9/24/2001
alpha-BHC (alpha-Hexachlorocyclohexane)	SAC-ID-0014	Pesticides-Herbicides-PCB's	NELAP	9/24/2001
alpha-Chlordane	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	9/24/2001
alpha-Chlordane	SAC-ID-0014	Pesticides-Herbicides-PCB's	NELAP	9/24/2001
Aluminum	EPA 6010	Metals	NELAP	6/10/2003
Aniline	EPA 8270	Extractable Organics	NELAP	9/24/2001
Anthracene	EPA 8270	Extractable Organics	NELAP	9/24/2001
Anthracene	EPA 8310	Extractable Organics	NELAP	9/24/2001
Anthracene	SAC-ID-0015	Extractable Organics	NELAP	9/24/2001
Antimony	EPA 6010	Metals	NELAP	9/24/2001
Antimony	EPA 6020	Metals	NELAP	9/24/2001
Aramite	EPA 8270	Extractable Organics	NELAP	9/24/2001
Aroclor-1016 (PCB-1016)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	9/24/2001
Aroclor-1221 (PCB-1221)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	9/24/2001
Aroclor-1232 (PCB-1232)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	9/24/2001
Aroclor-1242 (PCB-1242)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	9/24/2001
Aroclor-1248 (PCB-1248)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	9/24/2001
Aroclor-1254 (PCB-1254)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	9/24/2001
Aroclor-1260 (PCB-1260)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	9/24/2001
Arsenic	EPA 6010	Metals	NELAP	9/24/2001
Arsenic	EPA 6020	Metals	NELAP	9/24/2001
Barium	EPA 6010	Metals	NELAP	9/24/2001
Barium	EPA 6020	Metals	NELAP	9/24/2001
Benzene	EPA 8260	Volatile Organics	NELAP	9/24/2001
Benzidine	EPA 8270	Extractable Organics	NELAP	9/24/2001
Benzo(a)anthracene	EPA 8270	Extractable Organics	NELAP	9/24/2001
Benzo(a)anthracene	EPA 8310	Extractable Organics	NELAP	9/24/2001
Benzo(a)anthracene	SAC-ID-0015	Extractable Organics	NELAP	9/24/2001
Benzo(a)pyrene	EPA 8270	Extractable Organics	NELAP	9/24/2001
Benzo(a)pyrene	EPA 8310	Extractable Organics	NELAP	9/24/2001
Benzo(a)pyrene	SAC-ID-0015	Extractable Organics	NELAP	9/24/2001
Benzo(b)fluoranthene	EPA 8270	Extractable Organics	NELAP	9/24/2001
Benzo(b)fluoranthene	EPA 8310	Extractable Organics	NELAP	9/24/2001

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Benzo(b)fluoranthene	SAC-ID-0015	Extractable Organics	NELAP	9/24/2001
Benzo(e)pyrene	SAC-ID-0015	Extractable Organics	NELAP	9/24/2001
Benzo(g,h,i)perylene	EPA 8270	Extractable Organics	NELAP	9/24/2001
Benzo(g,h,i)perylene	EPA 8310	Extractable Organics	NELAP	9/24/2001
Benzo(g,h,i)perylene	SAC-ID-0015	Extractable Organics	NELAP	9/24/2001
Benzo(k)fluoranthene	EPA 8270	Extractable Organics	NELAP	9/24/2001
Benzo(k)fluoranthene	EPA 8310	Extractable Organics	NELAP	9/24/2001
Benzo(k)fluoranthene	SAC-ID-0015	Extractable Organics	NELAP	9/24/2001
Benzoic acid	EPA 8270	Extractable Organics	NELAP	9/24/2001
Benzothiazole	SAC-MS-0003	Extractable Organics	NELAP	11/7/2003
Benzyl alcohol	EPA 8270	Extractable Organics	NELAP	9/24/2001
Beryllium	EPA 6010	Metals	NELAP	9/24/2001
Beryllium	EPA 6020	Metals	NELAP	9/24/2001
beta-BHC (beta-Hexachlorocyclohexane)	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	9/24/2001
beta-BHC (beta-Hexachlorocyclohexane)	SAC-ID-0014	Pesticides-Herbicides-PCB's	NELAP	9/24/2001
beta-Naphthylamine	EPA 8270	Extractable Organics	NELAP	9/24/2001
Biphenyl	SAC-ID-0015	Extractable Organics	NELAP	9/24/2001
bis(2-Chloroethoxy)methane	EPA 8270	Extractable Organics	NELAP	9/24/2001
bis(2-Chloroethyl) ether	EPA 8270	Extractable Organics	NELAP	9/24/2001
bis(2-Chloroisopropyl) ether	EPA 8270	Extractable Organics	NELAP	9/24/2001
bis(2-Ethylhexyl) phthalate (DEHP)	EPA 8270	Extractable Organics	NELAP	9/24/2001
Bismuth	SAC-MT-0001	Metals	NELAP	9/24/2001
Boron	EPA 6010	Metals	NELAP	6/10/2003
Boron	SAC-MT-0001	Metals	NELAP	9/24/2001
Bromochloromethane	EPA 8260	Volatile Organics	NELAP	9/24/2001
Bromodichloromethane	EPA 8260	Volatile Organics	NELAP	9/24/2001
Bromoform	EPA 8260	Volatile Organics	NELAP	9/24/2001
Butyl benzyl phthalate	EPA 8270	Extractable Organics	NELAP	9/24/2001
Cadmium	EPA 6010	Metals	NELAP	9/24/2001
Cadmium	EPA 6020	Metals	NELAP	9/24/2001
Calcium	EPA 6010	Metals	NELAP	6/10/2003
Carbazole	EPA 8270	Extractable Organics	NELAP	9/24/2001
Carbon disulfide	EPA 8260	Volatile Organics	NELAP	9/24/2001
Carbon tetrachloride	EPA 8260	Volatile Organics	NELAP	9/24/2001
Cerium	SAC-MT-0001	Metals	NELAP	9/24/2001
Chlordane (tech.)	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	9/24/2001

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State Laboratory ID: E87570

EPA Lab Code: CA00044

(916) 373-5600

E87570
STL Sacramento
880 Riverside Parkway
West Sacramento, CA 95605

Matrix: Solid and Chemical Materials

Analyte	Method/Tech	Category	Certification Type	Effective Date
Chlorobenzene	EPA 8260	Volatile Organics	NELAP	9/24/2001
Chlorobenzilate	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	9/24/2001
Chloroethane	EPA 8260	Volatile Organics	NELAP	9/24/2001
Chloroform	EPA 8260	Volatile Organics	NELAP	9/24/2001
Chloropicrin	SAC-MS-0003	Extractable Organics	NELAP	11/7/2003
Chloroprene	EPA 8260	Volatile Organics	NELAP	9/24/2001
Chromium	EPA 6010	Metals	NELAP	9/24/2001
Chromium	EPA 6020	Metals	NELAP	9/24/2001
Chrysene	EPA 8270	Extractable Organics	NELAP	9/24/2001
Chrysene	EPA 8310	Extractable Organics	NELAP	9/24/2001
Chrysene	SAC-ID-0015	Extractable Organics	NELAP	9/24/2001
cis-1,2-Dichloroethylene	EPA 8260	Volatile Organics	NELAP	9/24/2001
cis-1,3-Dichloropropene	EPA 8260	Volatile Organics	NELAP	9/24/2001
cis-Nonachlor	SAC-ID-0014	Pesticides-Herbicides-PCB's	NELAP	9/24/2001
Cobalt	EPA 6010	Metals	NELAP	9/24/2001
Cobalt	EPA 6020	Metals	NELAP	9/24/2001
Copper	EPA 6010	Metals	NELAP	9/24/2001
Copper	EPA 6020	Metals	NELAP	9/24/2001
Decachlorobiphenyl (BZ 209)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	6/10/2003
delta-BHC	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	9/24/2001
delta-BHC	SAC-ID-0014	Pesticides-Herbicides-PCB's	NELAP	9/24/2001
Diallate	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	9/24/2001
Dibenz(a, j) acridine	EPA 8270	Extractable Organics	NELAP	9/24/2001
Dibenz(a,h) anthracene	EPA 8270	Extractable Organics	NELAP	9/24/2001
Dibenz(a,h) anthracene	EPA 8310	Extractable Organics	NELAP	9/24/2001
Dibenz(a,h) anthracene	SAC-ID-0015	Extractable Organics	NELAP	9/24/2001
Dibenzofuran	EPA 8270	Extractable Organics	NELAP	9/24/2001
Dibromochloromethane	EPA 8260	Volatile Organics	NELAP	9/24/2001
Dibromofluoromethane	EPA 8260	Volatile Organics	NELAP	9/24/2001
Dibromomethane	EPA 8260	Volatile Organics	NELAP	9/24/2001
Dichlorodifluoromethane	EPA 8260	Volatile Organics	NELAP	9/24/2001
Dieldrin	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	9/24/2001
Dieldrin	SAC-ID-0014	Pesticides-Herbicides-PCB's	NELAP	9/24/2001
Diesel range organics (DRO)	EPA 8015	Extractable Organics	NELAP	9/24/2001
Diethyl phthalate	EPA 8270	Extractable Organics	NELAP	9/24/2001
Diisopropyl methyl phosphonate	SAC-LC-0004	Extractable Organics	NELAP	9/24/2001

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West Sacramento, CA 95605

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Analyte	Method/Tech	Category	Certification Type	Effective Date
Dimethoate	EPA 8270	Pesticides-Herbicides-PCB's	NELAP	9/24/2001
Dimethyl disulfide	SAC-MS-0003	Extractable Organics	NELAP	11/7/2003
Dimethyl methyl phosphonate	SAC-LC-0004	Extractable Organics	NELAP	9/24/2001
Dimethyl phthalate	EPA 8270	Extractable Organics	NELAP	9/24/2001
Di-n-butyl phthalate	EPA 8270	Extractable Organics	NELAP	9/24/2001
Di-n-octyl phthalate	EPA 8270	Extractable Organics	NELAP	9/24/2001
Diphenylamine	EPA 8270	Extractable Organics	NELAP	9/24/2001
Endosulfan I	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	9/24/2001
Endosulfan I	SAC-ID-0014	Pesticides-Herbicides-PCB's	NELAP	9/24/2001
Endosulfan II	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	9/24/2001
Endosulfan II	SAC-ID-0014	Pesticides-Herbicides-PCB's	NELAP	9/24/2001
Endosulfan sulfate	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	9/24/2001
Endosulfan sulfate	SAC-ID-0014	Pesticides-Herbicides-PCB's	NELAP	9/24/2001
Endrin	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	9/24/2001
Endrin	SAC-ID-0014	Pesticides-Herbicides-PCB's	NELAP	9/24/2001
Endrin aldehyde	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	9/24/2001
Endrin aldehyde	SAC-ID-0014	Pesticides-Herbicides-PCB's	NELAP	9/24/2001
Endrin ketone	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	9/24/2001
Endrin ketone	SAC-ID-0014	Pesticides-Herbicides-PCB's	NELAP	9/24/2001
Ethyl methacrylate	EPA 8260	Volatile Organics	NELAP	9/24/2001
Ethyl methanesulfonate	EPA 8270	Extractable Organics	NELAP	9/24/2001
Ethylbenzene	EPA 8021	Volatile Organics	NELAP	9/24/2001
Ethylbenzene	EPA 8260	Volatile Organics	NELAP	9/24/2001
Ethylmethylphosphonic acid	SAC-LC-0004	Extractable Organics	NELAP	9/24/2001
Fluoranthene	EPA 8270	Extractable Organics	NELAP	9/24/2001
Fluoranthene	EPA 8310	Extractable Organics	NELAP	9/24/2001
Fluoranthene	SAC-ID-0015	Extractable Organics	NELAP	9/24/2001
Fluorene	EPA 8270	Extractable Organics	NELAP	9/24/2001
Fluorene	EPA 8310	Extractable Organics	NELAP	9/24/2001
Fluorene	SAC-ID-0015	Extractable Organics	NELAP	9/24/2001
Fluoride	EPA 9056	General Chemistry	NELAP	9/24/2001
gamma-BHC (Lindane, gamma-Hexachlorocyclohexane)	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	9/24/2001
gamma-BHC (Lindane, gamma-Hexachlorocyclohexane)	SAC-ID-0014	Pesticides-Herbicides-PCB's	NELAP	9/24/2001
gamma-Chlordane	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	9/24/2001
gamma-Chlordane	SAC-ID-0014	Pesticides-Herbicides-PCB's	NELAP	9/24/2001
Gasoline range organics (GRO)	EPA 8015	Extractable Organics	NELAP	9/24/2001

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EPA Lab Code: CA00044

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STL Sacramento
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West Sacramento, CA 95605

Matrix: Solid and Chemical Materials

Analyte	Method/Tech	Category	Certification Type	Effective Date
Heptachlor	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	9/24/2001
Heptachlor	SAC-ID-0014	Pesticides-Herbicides-PCB's	NELAP	9/24/2001
Heptachlor epoxide	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	9/24/2001
Heptachlor epoxide	SAC-ID-0014	Pesticides-Herbicides-PCB's	NELAP	9/24/2001
Hexachlorobenzene	EPA 8270	Extractable Organics	NELAP	9/24/2001
Hexachlorobenzene	SAC-ID-0014	Pesticides-Herbicides-PCB's	NELAP	9/24/2001
Hexachlorobutadiene	EPA 8260	Volatile Organics	NELAP	9/24/2001
Hexachlorobutadiene	EPA 8270	Extractable Organics	NELAP	9/24/2001
Hexachlorocyclopentadiene	EPA 8270	Extractable Organics	NELAP	9/24/2001
Hexachloroethane	EPA 8270	Extractable Organics	NELAP	9/24/2001
Hexachloropropene	EPA 8270	Extractable Organics	NELAP	9/24/2001
Indeno(1,2,3-cd)pyrene	EPA 8270	Extractable Organics	NELAP	9/24/2001
Indeno(1,2,3-cd)pyrene	EPA 8310	Extractable Organics	NELAP	9/24/2001
Indeno(1,2,3-cd)pyrene	SAC-ID-0015	Extractable Organics	NELAP	9/24/2001
Iodomethane (Methyl iodide)	EPA 8260	Volatile Organics	NELAP	9/24/2001
Iron	EPA 6010	Metals	NELAP	6/10/2003
Iron	SAC-MT-0001	Metals	NELAP	9/24/2001
Isobutyl alcohol (2-Methyl-1-propanol)	EPA 8260	Volatile Organics	NELAP	9/24/2001
Isodrin	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	9/24/2001
Isodrin	EPA 8270	Pesticides-Herbicides-PCB's	NELAP	9/24/2001
Isophorone	EPA 8270	Extractable Organics	NELAP	9/24/2001
Isopropylmethylphosphonic acid	SAC-LC-0004	Extractable Organics	NELAP	9/24/2001
Isosafrole	EPA 8270	Extractable Organics	NELAP	9/24/2001
Lead	EPA 6010	Metals	NELAP	9/24/2001
Lead	EPA 6020	Metals	NELAP	9/24/2001
Lithium	EPA 6010	Metals	NELAP	6/10/2003
Lithium	SAC-MT-0001	Metals	NELAP	9/24/2001
Magnesium	EPA 6010	Metals	NELAP	6/10/2003
Magnesium	SAC-MT-0001	Metals	NELAP	9/24/2001
Manganese	EPA 6010	Metals	NELAP	6/10/2003
Mercury	EPA 7470	Metals	NELAP	9/24/2001
Mercury	EPA 7471	Metals	NELAP	9/24/2001
Methacrylonitrile	EPA 8260	Volatile Organics	NELAP	9/24/2001
Methoxychlor	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	9/24/2001
Methoxychlor	SAC-ID-0014	Pesticides-Herbicides-PCB's	NELAP	9/24/2001
Methyl bromide (Bromomethane)	EPA 8260	Volatile Organics	NELAP	9/24/2001

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Analyte	Method/Tech	Category	Certification Type	Effective Date
Methyl chloride (Chloromethane)	EPA 8260	Volatile Organics	NELAP	9/24/2001
Methyl methacrylate	EPA 8260	Volatile Organics	NELAP	9/24/2001
Methyl methanesulfonate	EPA 8270	Extractable Organics	NELAP	9/24/2001
Methyl parathion (Parathion, methyl)	EPA 8270	Pesticides-Herbicides-PCB's	NELAP	9/24/2001
Methyl tert-butyl ether (MTBE)	EPA 8021	Volatile Organics	NELAP	9/24/2001
Methyl tert-butyl ether (MTBE)	EPA 8260	Volatile Organics	NELAP	9/24/2001
Methylene chloride	EPA 8260	Volatile Organics	NELAP	9/24/2001
Methylphosphonic acid	SAC-LC-0004	Extractable Organics	NELAP	9/24/2001
Mirex	SAC-ID-0014	Pesticides-Herbicides-PCB's	NELAP	9/24/2001
Molybdenum	EPA 6010	Metals	NELAP	9/24/2001
Molybdenum	EPA 6020	Metals	NELAP	9/24/2001
Naphthalene	EPA 8260	Volatile Organics	NELAP	9/24/2001
Naphthalene	EPA 8270	Extractable Organics	NELAP	9/24/2001
Naphthalene	EPA 8310	Extractable Organics	NELAP	9/24/2001
Naphthalene	SAC-ID-0015	Extractable Organics	NELAP	9/24/2001
Nickel	EPA 6010	Metals	NELAP	9/24/2001
Nickel	EPA 6020	Metals	NELAP	9/24/2001
Nitrobenzene	EPA 8095	Extractable Organics	NELAP	11/7/2003
Nitrobenzene	EPA 8270	Extractable Organics	NELAP	9/24/2001
Nitrobenzene	EPA 8330	Extractable Organics	NELAP	9/24/2001
Nitrobenzene	SAC-LC-0001	Extractable Organics	NELAP	9/24/2001
Nitrocellulose	SAC-WC-0050	General Chemistry	NELAP	9/24/2001
Nitroglycerin	SAC-LC-0009	Extractable Organics	NELAP	11/7/2003
Nitroguanidine	SAC-LC-0010	Extractable Organics	NELAP	11/7/2003
n-Nitrosodiethylamine	EPA 8270	Extractable Organics	NELAP	9/24/2001
n-Nitrosodimethylamine	EPA 8270	Extractable Organics	NELAP	9/24/2001
n-Nitroso-di-n-butylamine	EPA 8270	Extractable Organics	NELAP	9/24/2001
n-Nitrosodi-n-propylamine	EPA 8270	Extractable Organics	NELAP	9/24/2001
n-Nitrosodiphenylamine	EPA 8270	Extractable Organics	NELAP	9/24/2001
n-Nitrosomethylethylamine	EPA 8270	Extractable Organics	NELAP	9/24/2001
n-Nitrosomorpholine	EPA 8270	Extractable Organics	NELAP	9/24/2001
n-Nitrosopiperidine	EPA 8270	Extractable Organics	NELAP	9/24/2001
n-Nitrosopyrrolidine	EPA 8270	Extractable Organics	NELAP	9/24/2001
o-Chloroacetophenone	SAC-MS-0003	Extractable Organics	NELAP	11/7/2003
Octahydro-1,3,5,7-tetranitro-1,3,5,7-tetrazocine (HMX)	EPA 8095	Extractable Organics	NELAP	11/7/2003
Octahydro-1,3,5,7-tetranitro-1,3,5,7-tetrazocine (HMX)	EPA 8330	Extractable Organics	NELAP	9/24/2001

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West Sacramento, CA 95605

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Analyte	Method/Tech	Category	Certification Type	Effective Date
Octahydro-1,3,5,7-tetranitro-1,3,5,7-tetrazocine (HMX)	SAC-LC-0001	Extractable Organics	NELAP	9/24/2001
o-Toluidine	EPA 8270	Extractable Organics	NELAP	9/24/2001
Oxychlorane	SAC-ID-0014	Pesticides-Herbicides-PCB's	NELAP	9/24/2001
Parathion, ethyl	EPA 8270	Pesticides-Herbicides-PCB's	NELAP	9/24/2001
p-Chlorophenyl methyl sulfide	SAC-MS-0003	Extractable Organics	NELAP	11/7/2003
p-Chlorophenyl methyl sulfone	SAC-MS-0003	Extractable Organics	NELAP	11/7/2003
p-Chlorophenyl methyl sulfoxide	SAC-MS-0003	Extractable Organics	NELAP	11/7/2003
p-Dioxane	EPA 8260	Volatile Organics	NELAP	9/24/2001
p-Dithiane	SAC-MS-0003	Extractable Organics	NELAP	11/7/2003
Pentachlorobenzene	EPA 8270	Extractable Organics	NELAP	9/24/2001
Pentachloronitrobenzene	EPA 8270	Extractable Organics	NELAP	9/24/2001
Pentachlorophenol	EPA 8270	Extractable Organics	NELAP	9/24/2001
Pentaerythritol tetranitrate	SAC-LC-0009	Extractable Organics	NELAP	11/7/2003
Perchlorate	SAC-LC-0012	General Chemistry	NELAP	11/7/2003
Perylene	SAC-ID-0015	Extractable Organics	NELAP	9/24/2001
pH	EPA 9045	General Chemistry	NELAP	9/24/2001
Phenacetin	EPA 8270	Extractable Organics	NELAP	9/24/2001
Phenanthrene	EPA 8270	Extractable Organics	NELAP	9/24/2001
Phenanthrene	EPA 8310	Extractable Organics	NELAP	9/24/2001
Phenanthrene	SAC-ID-0015	Extractable Organics	NELAP	9/24/2001
Phenol	EPA 8270	Extractable Organics	NELAP	9/24/2001
Phorate	EPA 8270	Pesticides-Herbicides-PCB's	NELAP	9/24/2001
Phosphorus, total	EPA 6010	Metals	NELAP	6/10/2003
Phosphorus, total	SAC-MT-0001	Metals	NELAP	9/24/2001
Potassium	EPA 6010	Metals	NELAP	6/10/2003
Potassium	SAC-MT-0001	Metals	NELAP	9/24/2001
p-Oxathiane	SAC-MS-0003	Extractable Organics	NELAP	11/7/2003
Pronamide (Kerb)	EPA 8270	Extractable Organics	NELAP	9/24/2001
Propionitrile (Ethyl cyanide)	EPA 8260	Volatile Organics	NELAP	9/24/2001
Pyrene	EPA 8270	Extractable Organics	NELAP	9/24/2001
Pyrene	EPA 8310	Extractable Organics	NELAP	9/24/2001
Pyrene	SAC-ID-0015	Extractable Organics	NELAP	9/24/2001
Pyridine	EPA 8270	Extractable Organics	NELAP	9/24/2001
RDX (hexahydro-1,3,5-trinitro-1,3,5-triazine)	EPA 8095	Extractable Organics	NELAP	11/7/2003
RDX (hexahydro-1,3,5-trinitro-1,3,5-triazine)	EPA 8330	Extractable Organics	NELAP	9/24/2001
RDX (hexahydro-1,3,5-trinitro-1,3,5-triazine)	SAC-LC-0001	Extractable Organics	NELAP	9/24/2001

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Analyte	Method/Tech	Category	Certification Type	Effective Date
Safrole	EPA 8270	Extractable Organics	NELAP	9/24/2001
Selenium	EPA 6010	Metals	NELAP	9/24/2001
Selenium	EPA 6020	Metals	NELAP	9/24/2001
Silica as SiO ₂	EPA 6010	Metals	NELAP	6/10/2003
Silver	EPA 6010	Metals	NELAP	9/24/2001
Silver	EPA 6020	Metals	NELAP	9/24/2001
Sodium	EPA 6010	Metals	NELAP	9/24/2001
Sodium	SAC-MT-0001	Metals	NELAP	9/24/2001
Strontium	EPA 6010	Metals	NELAP	6/10/2003
Strontium	SAC-MT-0001	Metals	NELAP	9/24/2001
Sulfotepp	EPA 8270	Pesticides-Herbicides-PCB's	NELAP	9/24/2001
Tetrachloroethylene (Perchloroethylene)	EPA 8260	Volatile Organics	NELAP	9/24/2001
Tetryl (methyl-2,4,6-trinitrophenylnitramine)	EPA 8330	Extractable Organics	NELAP	9/24/2001
Tetryl (methyl-2,4,6-trinitrophenylnitramine)	SAC-LC-0001	Extractable Organics	NELAP	9/24/2001
Thallium	EPA 6010	Metals	NELAP	9/24/2001
Thallium	EPA 6020	Metals	NELAP	9/24/2001
Thiodiglycol	SAC-LC-0004	Extractable Organics	NELAP	9/24/2001
Thulium	SAC-MT-0001	Metals	NELAP	9/24/2001
Tin	EPA 6010	Metals	NELAP	6/10/2003
Tin	SAC-MT-0001	Metals	NELAP	9/24/2001
Titanium	SAC-MT-0001	Metals	NELAP	9/24/2001
Toluene	EPA 8021	Volatile Organics	NELAP	9/24/2001
Toluene	EPA 8260	Volatile Organics	NELAP	9/24/2001
Total cyanide	EPA 9012	General Chemistry	NELAP	9/24/2001
Total Heptachlorodibenzofuran	EPA 8280	Extractable Organics	NELAP	9/24/2001
Total Heptachlorodibenzo-p-dioxin	EPA 8280	Extractable Organics	NELAP	9/24/2001
Total Hexachlorodibenzofuran	EPA 8280	Extractable Organics	NELAP	9/24/2001
Total Hexachlorodibenzo-p-dioxin	EPA 8280	Extractable Organics	NELAP	9/24/2001
Total Pentachlorodibenzofuran	EPA 8280	Extractable Organics	NELAP	9/24/2001
Total Pentachlorodibenzo-p-dioxin	EPA 8280	Extractable Organics	NELAP	9/24/2001
Total Tetrachlorodibenzofuran	EPA 8280	Extractable Organics	NELAP	9/24/2001
Total Tetrachlorodibenzo-p-dioxin	EPA 8280	Extractable Organics	NELAP	9/24/2001
Toxaphene (Chlorinated camphene)	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	9/24/2001
Toxicity Characteristic Leaching Procedure	EPA 1311	General Chemistry	NELAP	9/24/2001
trans Nanochlor	SAC-ID-0014	Pesticides-Herbicides-PCB's	NELAP	9/24/2001
trans-1,2-Dichloroethylene	EPA 8260	Volatile Organics	NELAP	9/24/2001

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trans-1,3-Dichloropropylene	EPA 8260	Volatile Organics	NELAP	9/24/2001
trans-1,4-Dichloro-2-butene	EPA 8260	Volatile Organics	NELAP	9/24/2001
Trichloroethene (Trichloroethylene)	EPA 8260	Volatile Organics	NELAP	9/24/2001
Trichlorofluoromethane	EPA 8260	Volatile Organics	NELAP	9/24/2001
Uranium	SAC-MT-0001	Metals	NELAP	9/24/2001
Vanadium	EPA 6010	Metals	NELAP	9/24/2001
Vanadium	EPA 6020	Metals	NELAP	9/24/2001
Vinyl acetate	EPA 8260	Volatile Organics	NELAP	9/24/2001
Vinyl chloride	EPA 8260	Volatile Organics	NELAP	9/24/2001
Xylene (total)	EPA 8021	Volatile Organics	NELAP	9/24/2001
Xylene (total)	EPA 8260	Volatile Organics	NELAP	9/24/2001
Zinc	EPA 6010	Metals	NELAP	9/24/2001
Zinc	EPA 6020	Metals	NELAP	9/24/2001
Zirconium	SAC-MT-0001	Metals	NELAP	9/24/2001

"STATE" indicates certification for the analyte by the method specified. "NELAP" further indicates certification compliant with the NELAC Standards.

NON-TRANSFERABLE 03/07/2005-E87570

Jeb Bush
Governor



John O. Agwunobi, M.D., M.B.A.
Secretary

Laboratory Scope of Accreditation

Page 30 of 34

THIS LISTING OF ACCREDITED ANALYTES SHOULD BE USED ONLY WHEN
ASSOCIATED WITH A VALID CERTIFICATE

State Laboratory ID: E87570

EPA Lab Code: CA00044

(916) 373-5600

E87570

STL Sacramento
880 Riverside Parkway
West Sacramento, CA 95605

Matrix: Biological Tissue

Analyte	Method/Tech	Category	Certification Type	Effective Date
2,2',3,3',4,4',5,5',6-Nonachlorobiphenyl (BZ 206)	SAC-ID-0013	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
2,2',3,3',4,4',5,5'-Octachlorobiphenyl (BZ 194)	SAC-ID-0013	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
2,2',3,3',4,4',5-Heptachlorobiphenyl (BZ 170)	SAC-ID-0013	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
2,2',3,3',4,4',6-Heptachlorobiphenyl (BZ 171)	SAC-ID-0013	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
2,2',3,3',4,4'-Hexachlorobiphenyl (BZ 128)	SAC-ID-0013	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
2,2',3,3',4,5,5',6-Octachlorobiphenyl (BZ 198)	SAC-ID-0013	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
2,2',3,3',4,5,6,6'-Octachlorobiphenyl (BZ 200)	SAC-ID-0013	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
2,2',3,4,4',5,5'-Heptachlorobiphenyl (BZ 180)	SAC-ID-0013	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
2,2',3,4,4',5',6-Heptachlorobiphenyl (BZ 183)	SAC-ID-0013	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
2,2',3,4,4',5-Hexachlorobiphenyl (BZ 137)	SAC-ID-0013	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
2,2',3,4,4',5'-Hexachlorobiphenyl (BZ 138)	SAC-ID-0013	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
2,2',3,4,4',6'-Hexachlorobiphenyl (BZ 140)	SAC-ID-0013	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
2,2',3,4,5,6'-Hexachlorobiphenyl (BZ 143)	SAC-ID-0013	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
2,2',3,4',5,6-Hexachlorobiphenyl (BZ 149)	SAC-ID-0013	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
2,2',3,4,5'-Pentachlorobiphenyl (BZ 87)	SAC-ID-0013	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
2,2',3,4',6-Pentachlorobiphenyl (BZ 91)	SAC-ID-0013	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
2,2',3,4-Tetrachlorobiphenyl (BZ 41)	SAC-ID-0013	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
2,2',3,5,5',6-Hexachlorobiphenyl (BZ 151)	SAC-ID-0013	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
2,2',3,5'-Tetrachlorobiphenyl (BZ 44)	SAC-ID-0013	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
2,2',4,4',5,5'-Hexachlorobiphenyl (BZ 153)	SAC-ID-0013	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
2,2',4,4',5-Pentachlorobiphenyl (BZ 99)	SAC-ID-0013	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
2,2',4,4',6-Pentachlorobiphenyl (BZ 100)	SAC-ID-0013	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
2,2',4,5,5'-Pentachlorobiphenyl (BZ 101)	SAC-ID-0013	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
2,2',4,5,6'-Pentachlorobiphenyl (BZ 102)	SAC-ID-0013	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
2,2',4,6'-Tetrachlorobiphenyl (BZ 51)	SAC-ID-0013	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
2,2',5,6'-Tetrachlorobiphenyl (BZ 53)	SAC-ID-0013	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
2,2',5-Trichlorobiphenyl (BZ 18)	SAC-ID-0013	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
2,3,3',4,4',5,5'-Heptachlorobiphenyl (BZ 189)	SAC-ID-0013	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
2,3,3',4,4',5-Hexachlorobiphenyl (BZ 156)	SAC-ID-0013	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
2,3,3',4,4',5'-Hexachlorobiphenyl (BZ 157)	SAC-ID-0013	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
2,3,3',4,4'-Pentachlorobiphenyl (BZ 105)	SAC-ID-0013	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
2,3,3',4',6-Pentachlorobiphenyl (BZ 110)	SAC-ID-0013	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
2,3',4,4',5,5'-Hexachlorobiphenyl (BZ 167)	SAC-ID-0013	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
2',3,4,4',5-Pentachlorobiphenyl	SAC-ID-0013	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
2,3,4,4',5-Pentachlorobiphenyl (BZ 114)	SAC-ID-0013	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
2,3',4,4',5-Pentachlorobiphenyl (BZ 118)	SAC-ID-0013	Pesticides-Herbicides-PCB's	NELAP	7/1/2003

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NON-TRANSFERABLE 03/07/2005-E87570

Jeb Bush
Governor



John O. Agwunobi, M.D., M.B.A.
Secretary

Laboratory Scope of Accreditation

Page 31 of 34

THIS LISTING OF ACCREDITED ANALYTES SHOULD BE USED ONLY WHEN
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State Laboratory ID: E87570

EPA Lab Code: CA00044

(916) 373-5600

E87570

STL Sacramento
880 Riverside Parkway
West Sacramento, CA 95605

Matrix: Biological Tissue

Analyte	Method/Tech	Category	Certification Type	Effective Date
2,3',4',5,5'-Pentachlorobiphenyl (BZ 124)	SAC-ID-0013	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
2,3',4',5-Tetrachlorobiphenyl (BZ 70)	SAC-ID-0013	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
2,3',4,6-Tetrachlorobiphenyl (BZ 69)	SAC-ID-0013	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
2,3',4'-Trichlorobiphenyl (BZ 33)	SAC-ID-0013	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
2,3',5,6-Tetrachlorobiphenyl (BZ 73)	SAC-ID-0013	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
2,3,6-Trichlorobiphenyl (BZ 24)	SAC-ID-0013	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
2,4,4'-Trichlorobiphenyl (BZ 28)	SAC-ID-0013	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
2,4,5-Trichlorobiphenyl (BZ 29)	SAC-ID-0013	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
2,4'-Dichlorobiphenyl (BZ 8)	SAC-ID-0013	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
3,3',4,4',5,5'-Hexachlorobiphenyl (BZ 169)	SAC-ID-0013	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
3,3',4,4',5-Pentachlorobiphenyl (BZ 126)	SAC-ID-0013	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
3,3',4,4'-Tetrachlorobiphenyl (BZ 77)	SAC-ID-0013	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
3,4,4',5-Tetrachlorobiphenyl (BZ 81)	SAC-ID-0013	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
3,4'-Dichlorobiphenyl (BZ 13)	SAC-ID-0013	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
4,4'-Dichlorobiphenyl (BZ 15)	SAC-ID-0013	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
4-Chlorobiphenyl (BZ 3)	SAC-ID-0013	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
Decachlorobiphenyl (BZ 209)	SAC-ID-0013	Pesticides-Herbicides-PCB's	NELAP	7/1/2003

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NON-TRANSFERABLE 03/07/2005-E87570

Jeb Bush
Governor



John O. Agwunobi, M.D., M.B.A.
Secretary

Laboratory Scope of Accreditation

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State Laboratory ID: E87570

EPA Lab Code: CA00044

(916) 373-5600

E87570

STL Sacramento
880 Riverside Parkway
West Sacramento, CA 95605

Matrix: Air and Emissions

Analyte	Method/Tech	Category	Certification Type	Effective Date
1,2,3,4,6,7,8,9-Octachlorodibenzofuran (OCDF)	EPA TO-9A	Extractable Organics	NELAP	9/24/2001
1,2,3,4,6,7,8,9-Octachlorodibenzo-p-dioxin (OCDD)	EPA TO-9A	Extractable Organics	NELAP	9/24/2001
1,2,3,4,6,7,8-Heptachlorodibenzofuran (1,2,3,4,6,7,8-hpddf)	EPA TO-9A	Extractable Organics	NELAP	9/24/2001
1,2,3,4,6,7,8-Heptachlorodibenzo-p-dioxin (1,2,3,4,6,7,8-hpcdd)	EPA TO-9A	Extractable Organics	NELAP	9/24/2001
1,2,3,4,7,8,9-Heptachlorodibenzofuran (1,2,3,4,7,8,9-hpcdf)	EPA TO-9A	Extractable Organics	NELAP	9/24/2001
1,2,3,4,7,8-Hxodd	EPA TO-9A	Extractable Organics	NELAP	9/24/2001
1,2,3,4,7,8-Hxcdf	EPA TO-9A	Extractable Organics	NELAP	9/24/2001
1,2,3,6,7,8-Hxcd	EPA TO-9A	Extractable Organics	NELAP	9/24/2001
1,2,3,6,7,8-Hxcdf	EPA TO-9A	Extractable Organics	NELAP	9/24/2001
1,2,3,7,8,9-Hxodd	EPA TO-9A	Extractable Organics	NELAP	9/24/2001
1,2,3,7,8,9-Hxcdf	EPA TO-9A	Extractable Organics	NELAP	9/24/2001
1,2,3,7,8-Pecdd	EPA TO-9A	Extractable Organics	NELAP	9/24/2001
1,2,3,7,8-Pecdf	EPA TO-9A	Extractable Organics	NELAP	9/24/2001
2,3,4,6,7,8-Hxcd	EPA TO-9A	Extractable Organics	NELAP	9/24/2001
2,3,4,7,8-Pecdf	EPA TO-9A	Extractable Organics	NELAP	9/24/2001
2,3,7,8-TCDF	EPA TO-9A	Extractable Organics	NELAP	9/24/2001
2,3,7,8-Tetrachlorodibenzo-p-dioxin	EPA TO-9A	Extractable Organics	NELAP	9/24/2001
4,4'-DDE	EPA TO-10A	Pesticides-Herbicides-PCB's	NELAP	9/24/2001
4,4'-DDE	EPA TO-4A	Pesticides-Herbicides-PCB's	NELAP	9/24/2001
4,4'-DDE	IP-8	Pesticides-Herbicides-PCB's	NELAP	9/24/2001
4,4'-DDT	EPA TO-10A	Pesticides-Herbicides-PCB's	NELAP	9/24/2001
4,4'-DDT	EPA TO-4A	Pesticides-Herbicides-PCB's	NELAP	9/24/2001
4,4'-DDT	IP-8	Pesticides-Herbicides-PCB's	NELAP	9/24/2001
Acenaphthene	EPA TO-13A	Extractable Organics	NELAP	9/24/2001
Acenaphthene	IP-7	Extractable Organics	NELAP	9/24/2001
Acenaphthylene	EPA TO-13A	Extractable Organics	NELAP	9/24/2001
Acenaphthylene	IP-7	Extractable Organics	NELAP	9/24/2001
Aldrin	EPA TO-10A	Pesticides-Herbicides-PCB's	NELAP	9/24/2001
Aldrin	EPA TO-4A	Pesticides-Herbicides-PCB's	NELAP	9/24/2001
Aldrin	IP-8	Pesticides-Herbicides-PCB's	NELAP	9/24/2001
alpha-BHC (alpha-Hexachlorocyclohexane)	EPA TO-10A	Pesticides-Herbicides-PCB's	NELAP	9/24/2001
alpha-BHC (alpha-Hexachlorocyclohexane)	IP-8	Pesticides-Herbicides-PCB's	NELAP	9/24/2001
Anthracene	EPA TO-13A	Extractable Organics	NELAP	9/24/2001
Anthracene	IP-7	Extractable Organics	NELAP	9/24/2001
Aroclor-1016 (PCB-1016)	EPA TO-10A	Pesticides-Herbicides-PCB's	NELAP	9/24/2001

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NON-TRANSFERABLE 03/07/2005-E87570

Jeb Bush
 Governor



John O. Agwunobi, M.D., M.B.A.
 Secretary

Laboratory Scope of Accreditation

Page 33 of 34

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State Laboratory ID: E87570

EPA Lab Code: CA00044

(916) 373-5600

E87570

STL Sacramento
 880 Riverside Parkway
 West Sacramento, CA 95605

Matrix: Air and Emissions

Analyte	Method/Tech	Category	Certification Type	Effective Date
Aroclor-1016 (PCB-1016)	EPA TO-4A	Pesticides-Herbicides-PCB's	NELAP	9/24/2001
Aroclor-1221 (PCB-1221)	EPA TO-10A	Pesticides-Herbicides-PCB's	NELAP	9/24/2001
Aroclor-1221 (PCB-1221)	EPA TO-4A	Pesticides-Herbicides-PCB's	NELAP	9/24/2001
Aroclor-1232 (PCB-1232)	EPA TO-10A	Pesticides-Herbicides-PCB's	NELAP	9/24/2001
Aroclor-1232 (PCB-1232)	EPA TO-4A	Pesticides-Herbicides-PCB's	NELAP	9/24/2001
Aroclor-1242 (PCB-1242)	EPA TO-10A	Pesticides-Herbicides-PCB's	NELAP	9/24/2001
Aroclor-1242 (PCB-1242)	EPA TO-4A	Pesticides-Herbicides-PCB's	NELAP	9/24/2001
Aroclor-1248 (PCB-1248)	EPA TO-10A	Pesticides-Herbicides-PCB's	NELAP	9/24/2001
Aroclor-1248 (PCB-1248)	EPA TO-4A	Pesticides-Herbicides-PCB's	NELAP	9/24/2001
Aroclor-1254 (PCB-1254)	EPA TO-10A	Pesticides-Herbicides-PCB's	NELAP	9/24/2001
Aroclor-1254 (PCB-1254)	EPA TO-4A	Pesticides-Herbicides-PCB's	NELAP	9/24/2001
Aroclor-1260 (PCB-1260)	EPA TO-10A	Pesticides-Herbicides-PCB's	NELAP	9/24/2001
Aroclor-1260 (PCB-1260)	EPA TO-4A	Pesticides-Herbicides-PCB's	NELAP	9/24/2001
Benzo(a)anthracene	EPA TO-13A	Extractable Organics	NELAP	9/24/2001
Benzo(a)anthracene	IP-7	Extractable Organics	NELAP	9/24/2001
Benzo(a)pyrene	EPA TO-13A	Extractable Organics	NELAP	9/24/2001
Benzo(a)pyrene	IP-7	Extractable Organics	NELAP	9/24/2001
Benzo(b)fluoranthene	EPA TO-13A	Extractable Organics	NELAP	9/24/2001
Benzo(b)fluoranthene	IP-7	Extractable Organics	NELAP	9/24/2001
Benzo(e)pyrene	EPA TO-13A	Extractable Organics	NELAP	9/24/2001
Benzo(e)pyrene	IP-7	Extractable Organics	NELAP	9/24/2001
Benzo(g,h,i)perylene	EPA TO-13A	Extractable Organics	NELAP	9/24/2001
Benzo(g,h,i)perylene	IP-7	Extractable Organics	NELAP	9/24/2001
Benzo(k)fluoranthene	EPA TO-13A	Extractable Organics	NELAP	9/24/2001
Benzo(k)fluoranthene	IP-7	Extractable Organics	NELAP	9/24/2001
Chlordane (tech.)	EPA TO-10A	Pesticides-Herbicides-PCB's	NELAP	9/24/2001
Chlordane (tech.)	EPA TO-4A	Pesticides-Herbicides-PCB's	NELAP	9/24/2001
Chlordane (tech.)	IP-8	Pesticides-Herbicides-PCB's	NELAP	9/24/2001
Chloride	EPA 9057	General Chemistry	NELAP	4/5/2004
Chrysene	EPA TO-13A	Extractable Organics	NELAP	9/24/2001
Chrysene	IP-7	Extractable Organics	NELAP	9/24/2001
Dibenz(a,h) anthracene	EPA TO-13A	Extractable Organics	NELAP	9/24/2001
Dibenz(a,h) anthracene	IP-7	Extractable Organics	NELAP	9/24/2001
Dieldrin	EPA TO-10A	Pesticides-Herbicides-PCB's	NELAP	9/24/2001
Dieldrin	IP-8	Pesticides-Herbicides-PCB's	NELAP	9/24/2001
Endrin	EPA TO-10A	Pesticides-Herbicides-PCB's	NELAP	9/24/2001

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NON-TRANSFERABLE 03/07/2005-E87570



Laboratory Scope of Accreditation

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State Laboratory ID: E87570

EPA Lab Code: CA00044

(916) 373-5600

E87570
STL Sacramento
880 Riverside Parkway
West Sacramento, CA 95605

Matrix: Air and Emissions

Analyte	Method/Tech	Category	Certification Type	Effective Date
Endrin aldehyde	EPA TO-10A	Pesticides-Herbicides-PCB's	NELAP	9/24/2001
Fluoranthene	EPA TO-13A	Extractable Organics	NELAP	9/24/2001
Fluoranthene	IP-7	Extractable Organics	NELAP	9/24/2001
Fluorene	EPA TO-13A	Extractable Organics	NELAP	9/24/2001
Fluorene	IP-7	Extractable Organics	NELAP	9/24/2001
gamma-BHC (Lindane, gamma-Hexachlorocyclohexane)	EPA TO-10A	Pesticides-Herbicides-PCB's	NELAP	9/24/2001
gamma-BHC (Lindane, gamma-Hexachlorocyclohexane)	IP-8	Pesticides-Herbicides-PCB's	NELAP	9/24/2001
Heptachlor	EPA TO-10A	Pesticides-Herbicides-PCB's	NELAP	9/24/2001
Heptachlor	IP-8	Pesticides-Herbicides-PCB's	NELAP	9/24/2001
Heptachlor epoxide	EPA TO-10A	Pesticides-Herbicides-PCB's	NELAP	9/24/2001
Heptachlor epoxide	IP-8	Pesticides-Herbicides-PCB's	NELAP	9/24/2001
Indeno(1,2,3-cd)pyrene	EPA TO-13A	Extractable Organics	NELAP	9/24/2001
Indeno(1,2,3-cd)pyrene	IP-7	Extractable Organics	NELAP	9/24/2001
Methoxychlor	EPA TO-10A	Pesticides-Herbicides-PCB's	NELAP	9/24/2001
Methoxychlor	IP-8	Pesticides-Herbicides-PCB's	NELAP	9/24/2001
Naphthalene	EPA TO-13A	Extractable Organics	NELAP	9/24/2001
Naphthalene	IP-7	Extractable Organics	NELAP	9/24/2001
Phenanthrene	EPA TO-13A	Extractable Organics	NELAP	9/24/2001
Phenanthrene	IP-7	Extractable Organics	NELAP	9/24/2001
Pyrene	EPA TO-13A	Extractable Organics	NELAP	9/24/2001
Pyrene	IP-7	Extractable Organics	NELAP	9/24/2001

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ATTACHMENT D

Soil Permit



**UNITED STATES
DEPARTMENT OF
AGRICULTURE**

**Animal and Plant
Health Inspection
Service**

**Plant Protection and
Quarantine**

Soil Permit

Permit
Number: S-44703 Revised

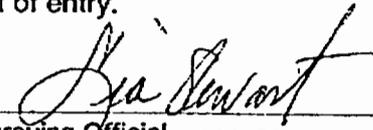
Issued To: STL, Chicago
(Michael J. Healy)
2417 Bond Street
University Park, Illinois 60466

TELEPHONE: (708) 534-5200

Under the authority of the Federal Plant Pest Act of May 23, 1957, permission is hereby granted to the facility/individual named above subject to the following conditions:

1. Valid for shipments of soil not heat treated at the port of entry, only if a Compliance Agreement (PPQ Form 519) has been completed and signed. Compliance Agreements and Soil Permits are non-transferable. If you hold a Soil Permit and you leave your present employer or Company, you must notify your local USDA office promptly. A copy of this permit must accompany all shipments.
2. To be shipped in sturdy, leakproof, containers.
3. To be released without treatment at the port of entry to permittee or authorized user.
4. To be used only for analysis and only in the facility of the permittee at STL, Chicago, located in University Park, Illinois.
5. No use of soil for growing purposes is authorized, including the isolation or culture of organisms imported in soil.
6. All unconsumed soil, containers, and effluent is to be autoclaved, incinerated, or heat treated by the permittee at the conclusion of the project as approved and prescribed by PPQ.
7. This permit authorizes shipments from all foreign sources, including Guam, Hawaii, Puerto Rico, and the U.S. Virgin Islands through any U.S. port of entry.

MARCH 31, 2010
Expiration Date


Approving Official **LIA STEWART**

WARNING: Any alteration, forgery, or unauthorized use of this Federal form is subject to civil penalties of up to \$250,000 (7 U.S.C. s 7734(b)) or punishable by a fine of not more than \$10,000, or imprisonment of not more than 5 years, or both (18 U.S.C. s 1001).



**UNITED STATES
DEPARTMENT OF
AGRICULTURE**

**Animal and Plant
Health Inspection
Service**

**Plant Protection and
Quarantine**

Soil Permit

Permit
Number: S-46613

Issued To: STL Sacramento
(Eric Redman)
880 Riverside Parkway
West Sacramento, California 95605

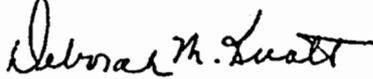
TELEPHONE: (916) 373-5600

Under the authority of the Federal Plant Pest Act of May 23, 1957, permission is hereby granted to the facility/individual named above subject to the following conditions:

1. Valid for shipments of soil not heat treated at the port of entry, only if a compliance agreement (PPQ Form 519) has been completed and signed. Compliance Agreements and Soil permits are non-transferable. If you hold a Soil Permit and you leave your present employer or company, you must notify your local USDA office promptly.
2. To be shipped in sturdy, leakproof, containers and released without treatment at the port of entry.
3. To be used only for analysis and only in the facility of the permittee at STL Sacramento, located in West Sacramento, California.
4. No use of soil for growing purposes is authorized, including the isolation or culture of organisms imported in soil.
5. All unconsumed soil, containers, and effluent is to be autoclaved, incinerated, or heat treated by the permittee at the conclusion of the project as approved and prescribed by Plant Protection and Quarantine.
6. This permit authorizes shipments from all foreign sources, including Guam, Hawaii, Puerto Rico, and the U.S. Virgin Islands through any U.S. port of entry.
7. Permittee shall notify the office at the Yolo County Agricultural Commissioner upon arrival of shipment(s) at Area Cod (530) 666-8140.

JUNE 30, 2005

Expiration Date


Approving Official **DEBORAH M. KNOTT**

COMPLIANCE AGREEMENT

1. NAME AND MAILING ADDRESS OF PERSON OR FIRM STL 880 Riverside Parkway West Sacramento, CA95661	2. LOCATION same
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3. REGULATED ARTICLE(S)
 SOIL SAMPLES - from foreign sources or regulated areas within the United States.

4. APPLICABLE FEDERAL QUARANTINE(S) OR REGULATIONS
 7CFR330.300 and 7CFR330.302 are regulations which restrict the movement of soil into or through the United States as well as from state to state. The State of California also restricts the movement of soil from other

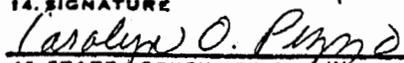
5. I/We agree to the following: / states into California.

Attached Stipulation, Attachment I - Stipulations for Handling Soil Samples.

Compliance Agreements and Departmental (Soil) permits are non-transferable. If you hold a Departmental (Soil) Permit and you leave your present employer or company, you must notify your local USDA office promptly. If permit material is to be used by other persons within the same company, those persons must be under the permittee's supervision or they must apply for their own permit. Notification to this office may help facilitate such circumstances.

7. SIGNATURE 	8. TITLE Laboratory Manager	9. DATE SIGNED 3/1/2000
The affixing of the signatures below will validate this agreement which shall remain in effect until cancelled, but may be revised as necessary or revoked for noncompliance.		10. AGREEMENT NO. 11. DATE OF AGREEMENT

12. PPQ OFFICIAL (Name and Title) Carolyn O. Pizzo PPQ Officer	13. ADDRESS 9550 Micron Avenue, Suite F Sacramento, CA 95827
--	--

14. SIGNATURE 	15. ADDRESS Yolo County Dept. of Agriculture 70 Cottonwood St. Woodland, CA 95695
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15. STATE AGENCY OFFICIAL (Name and Title)	16. ADDRESS Yolo County Dept. of Agriculture 70 Cottonwood St. Woodland, CA 95695
17. SIGNATURE	18. ADDRESS

ATTACHMENT E

**Chain of Custody
and
Job Sample Receipt Checklist Report**

rpjsckl

Job Sample Receipt Checklist Report

V2

Job Number.: 207213	Location.: 57222	Check List Number.: 1	Description.:
Customer Job ID.....:		Job Check List Date.: 12/14/2001	Date of the Report...: 04/14/2005
Project Number.: 20001703	Project Description.: USACE - Culebra Project	Contact.:	Project Manager.....: nsm
Customer.....:			

Questions ?	(Y/N)	Comments
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Chain-of-Custody Present?..... Y

...If "yes", completed properly?..... Y

Custody seal on shipping container?..... Y

...If "yes", custody seal intact?..... Y

Custody seals on sample containers?..... N

...If "yes", custody seal intact?.....

Samples iced?..... Y

Temperature of cooler acceptable? (4 deg C +/- 2). Y 4.8,3.6

Samples received intact (good condition)?..... Y

Volatile samples acceptable? (no headspace).....

Correct containers used?..... Y

Adequate sample volume provided?..... Y

Samples preserved correctly?..... Y

Samples received within holding-time?..... Y

Agreement between COC and sample labels?..... Y

Radioactivity at or below background levels?..... Y

A Sample Discrepancy Report (SDR) was needed?..... N

Comments..... N

If samples were shipped was there an air bill #?.. Y

Sample Custodian Signature/Date..... Y

ATTACHMENT F

Standard Operating Procedures



Controlled Copy
 Controlled Copy No: _____
 Implementation Date: _____

SOP No. SAC-WC-0010
 Revision No. 2.0
 Revision Date 2/18/03
 Page 1 of 20

OPERATION-SPECIFIC STANDARD OPERATING PROCEDURE

TITLE: DETERMINATION OF PERCHLORATE BY

ION CHROMATOGRAPHY BASED ON EPA METHOD 314.0

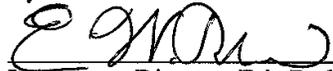
(SUPERSEDES: SAC-WC-0010, REVISION 1.0)

Prepared by: Kristina Hopper

Reviewed by: 
 Technical Specialist, Barry Votaw

Approved by: 
 Quality Assurance Manager, Pamela Schemmer

Approved by: 
 Environmental Health and Safety Coordinator Joe Schairer

Approved by: 
 Laboratory Director, Eric Redman

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1. SCOPE AND APPLICATION

- 1.1. This procedure is based on EPA Method 314.0, Revision 1.0, November 1999, Dionex Application Note 134 and Dionex IonPac AS16 Anion-Exchange Column Literature.
- 1.2. This method covers the determination of perchlorate in drinking, ground, and surface waters using ion chromatography. Soils and wastes may also be analyzed using this procedure, following a DI Leach preparation according to SOP number SAC-WC-0049.
- 1.3. This method is only for use by or under the supervision of analysts experienced in the use of ion chromatography and in the interpretation of the resulting ion chromatograms.
- 1.4. This SOP specifies the use of a Dionex AG16, 4mm Guard column and an AS16, 4-mm Analytical column, and analytical conditions to meet method specifications. Equivalent columns or conditions may be used if method requirements are still met.
- 1.5. The reporting limit is 4.0 ug/L for aqueous samples and 40 ug/kg for solid samples. Lower reporting limits are achievable and may be implemented on a client or project specific basis.

2. SUMMARY OF METHOD

- 2.1. A 1.0 mL volume of sample is introduced into an ion chromatograph (IC). Perchlorate is separated and measured, using a system comprised of an ion chromatographic pump, sample injection valve, guard column, analytical column, suppressor device, and conductivity detector.

3. DEFINITIONS

- 3.1. Definitions of terms used in this SOP may be found in the glossary of the Laboratory Quality Manual (LQM).

4. INTERFERENCES

- 4.1. Method interferences may be caused by contaminants in the reagent water, reagents, glassware, and other sample processing apparatus that lead to discrete artifacts or elevated baselines in an ion chromatogram. These interferences can lead to false

positive results for the target analyte as well as reduced detection limits as a consequence of elevated baseline noise.

- 4.2. Samples and reagent solutions that contain particulates larger than 0.45 microns require filtration to prevent damage to instrument columns and flow systems. Particulates can be separated by filtering the samples, standards, or reagents through a filter syringe with a 0.45-micron filter cartridge. All samples and standards pass through filter caps prior to injection. This filtering is sufficient when small amounts of particulate are present in a sample.
- 4.3. Sample matrices with high concentrations of common anions such as chloride, sulfate, and carbonate can destabilize the baseline in the perchlorate retention time window. This is evidenced by observing a protracted trailing following these anions, extending into the perchlorate window. These anions can be detected by conductivity testing, and dilutions should be performed accordingly.
- 4.4. A noisy baseline will also interfere with accurate recovery. Baseline noise is considered unacceptable if the peak to peak noise is greater than 0.015. If the instrument sits idle for more than a week or runs out of eluent or external water, the suppressor can become dry or overheated and will be unable to produce a clean baseline. Air bubbles trapped in the system, particularly the pump or conductivity cell, can also cause a noisy baseline.
 - 4.4.1. For contaminated NaOH, remake the 32.7mM with a different source of 50% (w/w).
 - 4.4.2. See the instrument manual for specific instructions on priming the pump, regenerating the suppressor, and flushing the conductivity cell.
- 4.5. Over time, some matrices will effect suppressor performance. This is evidenced by reduced peak response or asymmetrical perchlorate peaks, and should be corrected by cleaning the suppressor membranes according to manufacturer's instructions.

5. SAFETY

- 5.1. Procedures shall be carried out in a manner that protects the health and safety of all STL Sacramento associates.
- 5.2. Eye protection that satisfies ANSI Z87.1 (as per the Corporate Safety Manual), a laboratory coat, and appropriate chemically resistant gloves must be worn while samples, standards, solvents, and reagents are being handled. Disposable gloves that

have been contaminated will be removed and discarded as hazardous waste; other gloves will be cleaned immediately.

- 5.3. The health and safety hazards of many of the chemicals used in this procedure have not been fully defined. Each chemical should be regarded as a potential health hazard and exposure should be as low as reasonably achievable. Additional health and safety information can be obtained from the Material Safety Data Sheets (MSDS) maintained in the laboratory. The following specific hazards are known:
 - 5.3.1. The following materials are known to be **corrosive**: Sodium hydroxide.
 - 5.3.2. The following materials are known to be oxidizers: Sodium perchlorate (powder).
- 5.4. All work must be stopped in the event of a known or potential compromise to the health and safety of a STL Sacramento associate. The situation must be reported **immediately** to a laboratory supervisor.
- 5.5. Exposure to chemicals must be maintained **as low as reasonably achievable**; therefore, all samples must be opened, transferred, and prepared in a fume hood, or under other means of mechanical ventilation. Solvent and waste containers will be kept closed unless transfers are being made.
- 5.6. The preparation of standards and reagents will be conducted in a fume hood with the sash closed as far as the operation will permit.
- 5.7. Exercise caution when using syringes with attached filter assemblies. Application of excessive force has on occasion caused a filter disc to burst.

6. EQUIPMENT AND SUPPLIES

- 6.1. Ion Chromatograph (IC) – This method uses IC instrumentation manufactured by Dionex, Model DX500. Equipped with an autosampler, injection valve, pump with 1.5 mL/min flow rate, integrator, 1 mL sample loop, data acquisition system, and set up with the following components:
 - 6.1.1. Columns: Dionex AG16, 4 mm (P/N 055377) and Dionex AS16, 4 mm (P/N 055376).
 - 6.1.2. Suppressor: Dionex ASRS ULTRA (P/N 53946), external water mode, 300 mA current.

- 6.1.3. Detector: Dionex suppressed conductivity detector, Dionex CD20, cell temperature setting at 30 °C
- 6.2. Balance – Analytical balance, capable of accurately weighing to the nearest 0.0001 g.
- 6.3. Syringe, disposable, 2-10 mL capacity and equipped with male pressure fitting.
- 6.4. 0.45 micron acrodisk filters.
- 6.5. Dionex IC sample vials and filter caps –at least 5 mL capacity (P/N 38141).
- 6.6. Various class A analytical glassware of different sizes – graduated cylinder, volumetric flask, pipettes, etc.
- 6.7. Plastic bottles – 2-4L bottles are ideal for water and eluent reservoirs.
- 6.8. Conductivity meter

Note: It is permissible to change columns types, injection volumes, and/or eluents to improve separation or to lower costs, provided that the initial demonstration of capability is repeated and that the specifications as detailed in the reference method 314.0 are met.

7. REAGENTS AND STANDARDS

- 7.1. Reagent grade chemicals shall be used in all tests. Unless otherwise indicated, it is intended that all reagents shall conform to the specifications of the Committee on 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: Distilled or deionized water, free of anions of interest. Water should contain particles no larger than 0.20 micron and have a resistance of at least 18 mega-ohms. For best results, use reagent water that is taken directly from the Nanopure water system.
- 7.3. Eluent solution:
 - 7.3.1. 50% (w/w) NaOH, stock solution: Must be of highest purity (with low carbonate content). Commercially available, preferably in a 500 mL volume.

7.3.2. Eluent working solution: 32.7mM NaOH. Good for 5 days. A 2-liter volume will usually last for approximately 24 hours of non-stop use. A system that automatically generates eluent is an acceptable alternative.

NOTE: Avoid the introduction of carbon dioxide from the air into the 50% (w/w) NaOH. DO NOT shake the 50% (w/w) NaOH bottle or pipette the required aliquot from the top of the solution where sodium carbonate may have formed. IT IS BEST to pipette the aliquot from the middle of the bottle and to minimize the time the solution is exposed to air.

7.3.2.1. Fill a 2000-mL volumetric flask to the mark with Nanopure water. Pipette out 5.23 mL of the reagent water.

7.3.2.2. Transfer the remaining water from the flask to an eluent bottle.

7.3.2.3. De-gas the reagent water with He for at least 10 minutes.

7.3.2.4. Using a glass disposable 5-mL pipette, insert the pipette into the middle of the 50% (w/w) NaOH stock solution and pipette 5.23 mL, making sure that there is minimal solution adhering to the outside of the pipette.

7.3.2.5. Immediately transfer the solution to the reagent bottle. Cover and seal the bottle with parafilm. Gently invert the reagent bottle at least 10 times to properly mix the solution.

7.3.2.6. Remove the parafilm and connect the bottle to the instrument.

7.3.2.7. Dispose of expired eluent waste to the basic waste collection carboy.

7.4. Perchlorate stock solution, 1000 mg/L (or 1,000,000 ug/L): Obtain commercially. Alternatively, use a 1000-mL volumetric flask filled with approximately 600 mL of reagent water. Dissolve 1.2314 grams of sodium perchlorate (99% purity). (Note: sodium perchlorate represents a molar weight fraction of 81.2% perchlorate anion). Dilute to the mark with reagent water. Good for one year.

7.4.1. Intermediate standard solution, 10 mg/L (or 10,000 ug/L): Using a 100 mL volumetric flask containing at least 50 mL of reagent water, pipette 1 mL of the 1000 mg/L stock solution and swirl gently. Dilute to the mark with reagent water. Good for one month.

7.4.2. Working standards: Linear range 2 ug/L to 100 ug/L, good for one month. Dilute the intermediate standard (10,000 ug/L) with reagent water into 200 mL volumetric flasks as follows:

Standard #	Aliquot (mL)	Final Volume (mL)	Final concentration (ug/L)
1	0.04	200	2.00
2	0.08	200	4.00-ICCS
3	0.2	200	10.0
4	0.5	200	25.0
5	1.0	200	50.0-CCV
6	2.0	200	100-CCV

- 7.5. Second-Source Stock Standard, 100 mg/L (or 100,000ug/L): Obtain commercially. Alternatively, the second-source standard can be prepared from a different lot or different manufacturer other than the source of the Calibration Stock Standard. Good for 1 year.
- 7.6. Second-Source Working Standard, 50ug/L: Dilute 0.05 mL of the second-source stock standard to 100-mL in a volumetric flask for a final concentration of 50 ug/L. Good for one month.
- 7.7. Mixed Anion Stock Solution: Dissolve the following salts in reagent water for a final volume of 100 mL: 4.0 grams NaCl, 3.7 grams Na₂SO₄, and 4.4 grams Na₂CO₃. Final concentration: 25,000 mg/L chloride, sulfate, and carbonate anions. Good for one year.
- 7.8. Maximum Conductivity Threshold Standard (MCT) or Initial Performance Check standard (IPC), 25 ug/L perchlorate and 200 ug/L mixed anion standard: Mix 0.25 mL of the 10,000 ug/L perchlorate stock solution with 4 mL mixed anion stock solution (25000 ug/L) to a final volume of 100 mL. Good for one month.

Note: The MCT level can be adjusted, provided that the procedure in reference method 314.0 is followed.

8. SAMPLE COLLECTION, PRESERVATION AND STORAGE

- 8.1. Samples should to be collected in pre-cleaned plastic or glass containers. Volume collected should be sufficient to ensure a representative sample, allow for replicate analysis (if required), and minimize waste disposal.
- 8.2. Samples are stored at room temperature, no preservative.
- 8.3. Samples should be analyzed within 28 days of collection.

9. QUALITY CONTROL

- 9.1. Initial Demonstration of Capability: All analysts must successfully complete 4 LCSs prior to the analysis of any samples. Calculate the average recovery and standard deviation of the recovery. If the analyte does not meet the acceptance criteria, the test must be repeated. Repeated failure of the test indicates the need for the laboratory to evaluate the analytical procedure and take corrective action.
- 9.2. Method Detection Limit (MDL): The MDL is determined annually as described in SOP-QA-0006, MDLs and IDLs, and S-Q-003.
- 9.3. Maximum Conductivity Threshold (MCT): The highest permitted conductance of an unknown sample matrix, measured prior to conducting the analysis, which is used to determine when sample matrix dilution is required. The conductance in the MCT/sample is proportional to the concentration of common anions present. The MCT and the Instrument Performance Check (IPC) contain perchlorate, as well as the common anions of chloride, sulfate, and carbonate. These common anions are known to elute into the perchlorate window and cause potential interference. After the MCT is determined, it must be confirmed in each batch by the IPC. The IPC must meet three criteria:
 - 9.3.1. Percent Difference of Area/Height ratio between the ICV and the IPC solution <25%.
 - 9.3.2. 80%-120% Perchlorate Recovery.
 - 9.3.3. Retention time shift <5% from ICV.
 - 9.3.4. Corrective action: Restart batch. If IPC fails repeatedly, MCT must be re-established.

- 9.4. Batch: A quality control batch is a set of up to 20 field samples that have the same matrix and are processed using the same procedures, reagents, and standards within a 30 hour time period. A MB, LCS and MS/SD are also part of the batch. An analysis batch must also include all QC samples, however they do not contribute to the maximum of 20 samples.

Note: A field sample from the original batch can be reanalyzed after the closing CCV/CCB if it is still within 30 hours of the start of the run. An ICCS, as well as a CCV/CCB must be analyzed first, and the run must close with another CCV/CCB within that 30-hour window.

- 9.5. One Method Blank (MB) must be processed with every batch of similar matrix, not to exceed twenty (20) samples. The method blank is an aliquot of laboratory reagent water processed in the same manner and at the same time as the associated samples. Corrective actions must be documented on a Non-Conformance memo, then implemented when target analytes are detected in the method blank above the reporting limit. Re-extraction of the blank, other batch QC and the affected samples are required when the method blank is deemed unacceptable.

9.5.1. For aqueous analyses, the ICB is evaluated as the MB.

9.5.2. For solid analyses, a blank is prepared with the batch.

- 9.6. A Laboratory Control Sample (LCS) must be processed with every batch of similar matrix, not to exceed twenty (20) samples. The LCS is an aliquot of laboratory matrix (e.g. water, Ottawa sand, sodium sulfate, etc.) spiked with analytes of known identity and concentration. The LCS must be processed in the same manner and at the same time as the associated samples. Corrective actions must be documented in a Non-Conformance memo, then implemented when recoveries of any spiked analyte is outside control limits provided in LIMS or by the client. Reextraction of the blank, other batch QC and all associated samples are required if the LCS is deemed unacceptable. See Policy QA-003-SAC for specific acceptance criteria.

9.6.1. For aqueous analyses, the ICV is evaluated as the LCS.

9.6.2. Solid LCSs are spiked with a concentration of 500 ug/kg. A blank is prepared with the batch and spiked just prior to analysis.

9.6.3. LCS/DCS recoveries must be 90-110 % with an RPD of $\leq 15\%$ for aqueous samples, and 75 – 125% with an RPD of $< 20\%$ for solid matrices.

- 9.7. A Matrix Spike/Matrix Spike Duplicate (MS/MSD or MS/SD) pair must be processed with every batch of similar matrix, not to exceed twenty (20) samples. An MS/MSD are aliquots of a selected field sample spiked with analytes of known identity and concentration. The MS/MSD pair must be processed in the same manner and at the same time as the associated samples. Spiked analytes with recoveries or precision outside control limits must be within control limits for the LCS. Corrective actions must be documented in a Non-Conformance memo, then implemented when recoveries of any spike analyte is outside control limits provided in LIMS or by the client. Re-extraction of the blank, LCS, the selected field sample and the MS/MSD may be required after evaluation and review.
- 9.7.1. Two aliquots of an aqueous sample are spiked with a concentration of 50 ug/L.
- 9.7.2. Solid samples are spiked with a concentration of 500 ug/kg. A sample duplicate is prepared with the batch and two aliquots are spiked just prior to analysis.
- 9.7.3. MS/SD recoveries must be 80 – 120% with RPD of <20 for aqueous, and 75 – 125% with RPD of <20% for solid matrices.
- 9.8. A duplicate control sample (LCSD or DCS) must be substituted when insufficient sample volume is provided to process an MS/MSD pair. The LCSD is evaluated in the same manner as the LCS. See Policy QA-003-SAC for specific acceptance criteria.
- 9.8.1. For aqueous samples, an additional ICV standard can be analyzed, or two 2 CCVs of identical concentration can be evaluated as the LCS/DCS.
- 9.9. The QC terms and criteria listed below are a combination of those specified by Method 314.0 and STL Sacramento standard QC requirements.

Acceptance Criteria and Corrective Actions-Perchlorate			
QC Type	Frequency	Acceptance Criteria	Corrective Action
Initial Calibration Curve	Calibrated initially, then monthly. Verified daily prior to analysis.	$r > 0.995$	Reanalyze once. If the problem persists, reprepare the standards, and recalibrate. If the problem persists, consult the supervisor for instrument repair.
ICV/REF/LCS-50ppb (Second Source Standard)	At start of every analytical sequence, following the initial calibration.	90%-110% Recovery	Reanalyze once. If the problem persists, reprepare the standards, reanalyze, and/or recalibrate.
ICB/CCB/MB	Directly following ICV/CCVs.	<1/2 Reporting Limit	Reanalyze once. If the problem persists, isolate the source of the problem and fix it. If the problem is isolated to the blank, reprepare, reanalyze and proceed. If the problem may have affected previous sample results (i.e. instrument failure, contaminated vials, etc.), reanalyze samples bracketed by the failed blank.
IPC/MCT-25ppb perchlorate, 600 ppm anions	1 per batch of 20 samples or fewer.	1. Percent Difference of Area/Height ratio between the ICV and the MCT solution <25% 2. 80%-120% Perchlorate Recovery 3. Retention time shift <5%	Restart analysis. If problem persists, MCT level may need to be reestablished.
ICCS-4ppb	At start of every analytical sequence, following the MCT.	75%-125% Recovery	Restart analysis. If baseline is noisy, attempt to reduce baseline noise. Recalibration may be necessary.
CCV-Alternate 50ppb/100ppb	After every 10 samples and at the end of the analytical sequence.	85%-115% Recovery	Reanalyze once. If the problem persists, isolate the source of the problem and fix it. If the problem is isolated to the standard (i.e. misspike, etc.), reprepare, reanalyze and proceed. If the problem may have affected previous sample results (i.e. instrument failure, contaminated vials, etc.), reanalyze samples bracketed by the failed standard.
MS/SD-50ppb aqueous	1 MS/MSD pair per batch of 20 samples or fewer.	80%-120% Recovery, 15%RPD	Reanalyze once. If reanalysis recovery fails but % RPD passes, accept data. If reanalysis passes, report reanalysis.
MS/SD-500ppb solid		75%-125% Recovery, 20%RPD	
MB-solid (ICB=MB for aqueous)	1 per batch of 20 samples or fewer	< Reporting limit	Reanalyze once. If problem persists, reprepare and reanalyze batch.
LCS - solid (ICV=LCS for aqueous)	1 per batch of 20 samples or fewer	75%-125%	Respike aliquot and reanalyze. If problem persists, reprepare and reanalyze batch.
Samples: (Conductivity of the sample must be measured and recorded prior to analysis).	Water-no preservative. Soil-10X 1 hour DI leach. 28 day hold time.	RLw=4ppb RLs=40ppb	Conductivity of the water aliquot must be less than the conductivity of the MCT/IPC. If higher, dilute prior to analysis.

10. CALIBRATION

- 10.1. Initial Instrument Calibration (ICAL): A minimum of five calibration standards that represent the linear range of the instrument are analyzed and used as the instrument calibration for a month. The initial calibration sequence is listed below:

Reagent Water
2 ppb Standard
4 ppb Standard
10 ppb Standard
25 ppb Standard
50 ppb Standard
100 ppb Standard

- 10.1.1. Frequency: Initially, then monthly, or as required due to failed ICV/CCV. Verify daily with an ICV.
- 10.1.2. Criteria: r value of 0.995 or better
- 10.1.3. Corrective Action for failed ICAL: Recalibrate. If ICAL fails again, check standards and remake as needed. For failed linear curve due to instrument failure, consult a Dionex service representative.
- 10.1.4. Retention time of samples and standards should be within 5% of that obtained during the initial calibration. If a shift of > 5% occurs, results can be used after filing an NCM, provided that the shift is confirmed by the daily QC. The instrument should be recalibrated prior to initiating a new analysis.

NOTE: A series of reagent water blanks are analyzed prior to the instrument calibration in order to verify that the instrument baseline is stable and peak to peak criteria is met. Peak to peak noise must be less than 0.015.

11. PROCEDURE

- 11.1. One time procedural variations are allowed only if deemed necessary in the professional judgment of the supervisor to accommodate variation in sample matrix, radioactivity, chemistry, sample size, or other parameters. Any variation in procedure shall be completely documented using a Nonconformance Memo and approved by a Technical Specialist and QA Manager. If contractually required, the client shall be notified. The Nonconformance Memo shall be filed in the project file.
- 11.2. Any unauthorized deviation from this procedure must also be documented as a nonconformance, with a cause and corrective action described. See SAC-QA-0023 for additional information on the established procedures for the identification and documentation of nonconformances and corrective actions.

11.3. Instrument Start-up

- 11.3.1. Use fresh reagent water from the Nanopure system to fill the External Water bottles (EBW1, EBW2 and EBW3).
- 11.3.2. Fill the Eluent bottle with 32.7mM NaOH.
- 11.3.3. Inspect all He connections.
- 11.3.4. Prime the pump. Refer to the instrument manual, if necessary.
- 11.3.5. Using the Peak Net workstation menu, access RUN mode. Under File, Load Method: newperchlorate.met. The current will change from 0 to 300 mA. The pump will start.
- 11.3.6. Ensure that water is flowing through the system. The water flow rate is determined by He pressure and can be adjusted by the analyst. Flow rate should be between 3-8 mL/minute, and can be measured by collecting water waste from the appropriate waste line for a specific period of time.
- 11.3.7. Let the instrument run until the baseline has stabilized. To monitor the baseline, under Run of the Run mode, select Baseline. In addition, observe the total baseline reading in uS at the CD20 screen.
 - 11.3.7.1. To monitor peak to peak noise level, fill a sample vial with reagent water and run. Access the Optimize menu. Select the appropriate chromatogram. Select a 1 minute portion of the baseline. Under the Operations menu, select Autothreshold. Press Measure. The criteria for baseline reading must be met prior to sample analysis. If the baseline shows erratic response or severe noise (uS reading fluctuates frequently), see section 4.4, or consult the instrument manual.
- 11.3.8. As soon as the backpressure is stable around 2400 psi, baseline total uS is <2 uS and the pk to pk noise is <0.015, the instrument is ready for analysis.

11.4. Sample Pretreatment

- 11.4.1. Measure the conductivity of the sample using a calibrated conductivity meter and record the readings in the appropriate instrument logbook. If the conductivity of the sample is greater than the conductivity of the MCT/IPC, dilute the sample prior to analysis. Measure and record the conductivity of

the diluted sample. The sample must be diluted to the point that the conductivity of the sample or diluted portion thereof is less than the conductivity of the MCT/IPC. The reporting limit associated with the diluted sample will increase in proportion to the dilution.

- 11.4.2. Filter colored or turbid samples prior to analysis.
- 11.4.3. Arrange standard and sample vials in the same order as below. Two water reagent water blanks are recommended prior to each analytical run to confirm a stable baseline.

ICV	@ 50 ppb (use as aqueous LCS)
ICB	
IPC (MCT)	@ 25 ppb, with 600 ppb mixed anions.
ICCS	@ 4 ppb
10 samples, including QC below	
LCS	@ 500 ppb (soils only)
MS	@ 50 ppb (waters), 500 ppb (soils)
MSD	@ 50 ppb (waters), 500 ppb (soils)
CCV	@ 100 ppb
CCB	
10 samples	
CCV	@ 50 ppb
CCB	

11.5. Sample Analysis

- 11.5.1. Build analysis schedule as noted above.
- 11.5.2. Access the Run Mode. Under the File menu, select Load schedule.
- 11.5.3. On the autosampler, make sure it is in “Run” mode.
- 11.5.4. Under the run menu, select Start.
- 11.5.5. Monitor run and noise level from time to time.
- 11.5.6. Monitor water and eluent levels while the run is in progress.

11.6. Instrument Shutdown

11.6.1. Access the Run Mode. Under File, load Method-pre-shutdown.met. This method shuts off the water. Next, load Method-shutdown.met. This will stop the pump and current.

-OR-

11.6.2. In the last two lines of the schedule, enter as the method pre-shutdown.met, followed by shutdown.met. This will automatically stop the water, eluent, and current flow at the close of the run.

11.7. Standard Conditions and Equipment

11.7.1. Ion Chromatograph: Dionex DX500

11.7.2. Sample Loop: 1 mL

11.7.3. Eluent: 32.7 mM NaOH

11.7.4. Eluent Flow: 1.5 mL/min

11.7.5. Columns: Dionex AG16, 4 mm / AS16, 4 mm

11.7.6. Suppressor: ASRS ULTRA, external water mode, 300 mA current

11.7.7. Detector: Suppressed Conductivity Detector, Dionex CD20

11.7.8. Pump: Dionex GP50

11.7.9. Peak to Peak Noise: <0.015

11.7.10. Background Conductivity: <2 uS

11.7.11. Typical System Backpressure: 2200 psi-2800 psi

11.7.12. Approximate Retention Time: 9.5 – 10.5 minutes

11.7.13. Allowable shift between calibrations-5%

11.7.14. Approximate Analysis Time-11.5 minutes

12. DATA ANALYSIS AND CALCULATIONS

12.1. Perchlorate Identification

- 12.1.1. Identification occurs when a peak matching the retention time of the reference standard is found at a concentration above the reporting limit, or above the MDL if J flags are required.
- 12.1.2. If the analyst is unsure of perchlorate in the sample due to matrix, retention time shifts, or other factors, the sample should be spiked, analyzed and evaluated. A split or shouldering peak is evidence of an interferant and should not be reported as perchlorate.
- 12.1.3. The experience of the analyst should weigh heavily in the interpretation of the chromatogram. For example, sample matrix or laboratory temperature fluctuation may result in a variance of retention times.
- 12.1.4. All manual or re-integration of chromatograms must be documented in accordance with Policy S-Q-004 and the STL Sacramento-specific addendum. Documentation includes, as a minimum, before and after copies of the chromatograms with a reference to the reason for re-integration.

12.2. Calibration Range

- 12.2.1. If the concentration of the perchlorate anion exceeds the working range as defined by the calibration standards, then the sample must be diluted and reanalyzed. The reporting limit must be raised accordingly.
- 12.2.2. Responses for the diluted sample must be at a minimum 3-5 times the level of the lowest standard.
- 12.2.3. It may be necessary to dilute samples due to matrix.

12.3. Calculations

- 12.3.1. Peak areas are used as a measure of response since they have been found to be more consistent than peak heights.
- 12.3.2. All sample concentrations are calculated based on a linear regression. The calculation is automatically performed by the instrument, based on the equation:

Equation 1

$$\text{Concentration} = A + BR$$

Where: A = Intercept
B = Slope
R = Response (in area)

Equation 2 Conc in Sample (ug/L) = Concentration (ug/L) x DF

Where: DF = Dilution Factor

Equation 3 Conc in Sample (ug/kg) = Concentration (ug/L) x (V₁/M_s) x DF

Where: DF = Dilution Factor

V₁ = Volume of Leachate (in L)

M_s = Mass of soil (in kg)

12.4. Reporting Requirements

- 12.4.1. When it is necessary to redraw baselines, both the original and the redraw must be saved in the data system as well as included in the data pack.
- 12.4.2. Reporting limits and units are described in Section 1.5.
- 12.4.3. Sample results are entered into a LIMS system in accordance with current QA policies.
- 12.4.4. Footnotes and anomalies when applicable must be included in the data pack and data reduction process. Exceeded holding times must be immediately communicated to the project managers and followed by an electronically filed non-conformance memo.

13. METHOD PERFORMANCE

- 13.1. The group/team leader has the responsibility to ensure that this procedure is performed by an associate who has been properly trained in its use and has the required experience.

- 13.2. Both prep and analytical chemists must pass the initial demonstration of capability as outlined by this facility. Each laboratory must make a one time initial demonstration of capability for each individual method. Demonstration of capability for both soils and water matrices is required. This requires analysis of QC check samples containing all of the standard analytes for the method. For some tests it may be necessary to use more than one QC check mix to cover all analytes of interest.
- 13.3. The laboratory must generate a valid method detection limit for each analyte of interest. The MDL must be below the reporting limit for each analyte. The procedure for determination of the method detection limit is given in 40 CFR Part 136, Appendix B, and further defined in SAC-QA-006 and policy S-Q-003.

14. POLLUTION PREVENTION

- 14.1. When feasible, technological changes have been implemented to minimize the potential for pollution of the environment.

15. WASTE MANAGEMENT

- 15.1. Waste generated in the procedure will be segregated and disposed of into the waste streams detailed in the facility hazardous waste management plan.
- 15.2. Samples and other solutions containing high concentrations of toxic materials must be disposed of according to the facility hazardous waste management procedures.

16. REFERENCES

- 16.1. EPA Method 314.0, Determination of Perchlorate in Drinking Water using Ion Chromatography, Revision 1.0, November 1999.
- 16.2. Dionex Application Note 134.
- 16.3. Dionex IonPac AS16 Anion – Exchange Column Literature.

17. MISCELLANEOUS

- 17.1. Deviations from reference method.
 - 17.1.1. Alternate matrices included.
 - 17.1.2. According to Method 314.0, MDLs are to be performed over at least a 3-day period. Instead, STL's MDL Policy, S-Q-003 will be followed.

Although this policy allows for MDLs to be performed over multiple days, it does not require it. As a result, MDLs will generally be analyzed during one analysis on one day.

- 17.2. Summary of modifications to SOP from previous revisions.
 - 17.2.1. The reporting limit was lowered to 4 ug/L for aqueous samples and 40 mg/kg for soil samples. The units were also corrected to read in ug instead of mg.
 - 17.2.2. The MDL check standard was removed, as it was an extra step that was not required by the method or STL's MDL policy.
 - 17.2.3. The sample duplicate was also removed. Per the method, it is necessary to run a sample duplicate, an LCSD, or an MSD per batch for precision monitoring. This requirement is met with the MSD.
 - 17.2.4. Stock standards are to be obtained commercially rather than made from salts when possible.
 - 17.2.5. The linear range was changed to 2 ug/L-100 ug/L.
 - 17.2.6. Additional acceptance criteria for the IPC were added to reflect method requirements.
 - 17.2.7. The level of the MCT was updated.
- 17.3. List of other SOPs cross-referenced in SOP.
 - 17.3.1. SOP SAC-QA-0041 Calibration and Calibration Check of Balances.
 - 17.3.2. Policy QA-008-SAC Data Recording Requirements.
 - 17.3.3. Policy QA-003-SAC Quality Control Program
 - 17.3.4. SOP SAC-WC-0009 Determination of Anions by Ion Chromatography
 - 17.3.5. SOP SAC-WC-0049 Deionized Leaching Procedure for General Chemistry Analyses.
 - 17.3.6. SAC-QA-0023, Nonconformance and Corrective Action System

17.3.7. SAC-QA-006, Method Detection Limits and Instrument Detection Limits

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Revision No. 2.0
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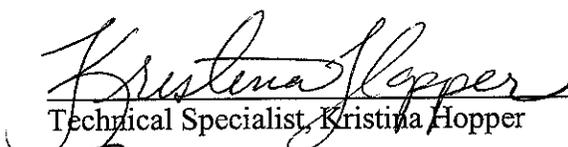


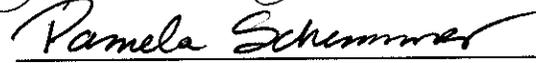
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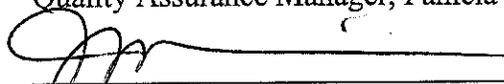
**TITLE: DEIONIZED WATER LEACHING PROCEDURE FOR
GENERAL CHEMISTRY ANALYSES**

(SUPERSEDES: SAC-WC-0049, REV. 1)

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1. SCOPE AND APPLICATION

- 1.1. This method is used for the preparation of samples for the analysis of water-soluble constituents by leaching with Deionized (D.I) water. Parameters that can be analyzed include chloride, sulfate, nitrate, nitrite, fluoride, alkalinity, specific conductivity, hexavalent chromium, perchlorate and other ions.
- 1.2. This method also covers soil extraction for ammonia analysis using 10% NaCl (acidified) instead of deionized water as the extraction buffer.
- 1.3. This method is applicable to soils, wastes, and other non-aqueous samples that are soluble in water.
- 1.4. The reporting limit depends on the analytical method used for the final determination. This prep method incorporates a 5X prep dilution. The prep factor can be adjusted based on the water absorbency of the sample. A 10X prep dilution is used for the determination of perchlorate.
- 1.5. The dynamic range depends on the analytical method used for final determination. The range may be extended by dilution of the leachate.
- 1.6. The analysis time depends on the analytical method used for final determination. The D.I. Leach prep takes approximately two hours per sample from initial weighing to final filtration. A number of samples can be prepared simultaneously following the standard batching protocols.

2. SUMMARY OF METHOD

- 2.1. A portion of a homogenized wet sample is leached with deionized water (DI Leach) for one hour, centrifuged, and filtered, depending on the nature of the sample. Aliquots of the leachate are preserved as appropriate for the parameters to be analyzed.
- 2.2. For ammonia, 10% NaCl (acidified to pH 2.5) is used for extraction instead of deionized water.

3. DEFINITIONS

- 3.1. Definitions of terms used in this SOP may be found in the glossary of the Laboratory Quality Manual (LQM).

4. INTERFERENCES

- 4.1. For alkalinity determination, leachates must be analyzed immediately after the leaching procedure is complete due to the possibility of calcium carbonate precipitation over time.
 - 4.1.1. Errors may result from the following reactions: peptization, hydrolysis, ion exchange, mineral dissolution, absorption, and other phenomena.
 - 4.1.2. Some samples such as dry drilling muds may soak up large volumes of water and prevent any liquid from being recovered. A smaller soil:water ratio must be used in these cases, such as 10X up to 100X prep factor.

5. SAFETY

Employees must abide by the policies and procedures in the Corporate Safety Manual, Radiation Safety Manual, Sacramento Supplement to the CSM, and this document. All work must be stopped in the event of a known or potential compromise to the health or safety of an associate. The situation must be reported **immediately** to a supervisor, the EH&S Staff, or a senior manager.

- 5.1. Specific Safety Concerns or Requirements
 - 5.1.1. 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.
 - 5.1.2. Eye protection that satisfies ANSI Z87.1, laboratory coat, and chemically resistant gloves must be worn while samples, standards, solvents, and reagents are being handled. Latex, PVC and nitrile gloves all provide adequate levels of protection against the chemicals used in this SOP.
 - 5.1.3. Exposure to chemicals must be maintained **as low as reasonably achievable**, therefore all samples must be opened, transferred and prepared in a fume hood. Solvent and waste containers will be kept closed unless transfers are being made.
 - 5.1.4. Laboratory procedures such as repetitive use of pipets, repetitive transferring of extracts, and manipulation of filled separatory funnels and other glassware represent a significant potential for repetitive motion or other ergonomic injuries. Laboratory associates performing these procedures are in the best position to realize when they are at risk for these types of injuries. Whenever a situation is found in which an employee is performing the same repetitive motion, the employee shall immediately bring this to the attention of their supervisor, manager, or the EH&S staff. The task will be analyzed to determine a better means of accomplishing it.

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
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. EQUIPMENT AND SUPPLIES

- 6.1. Centrifuge tubes, 50 mL capacity.
- 6.2. Analytical balance, 0.1 g capability.
- 6.3. Mechanical shaker.
- 6.4. Centrifuge.
- 6.5. Filtration apparatus. Vacuum, pressure, or gravity filtration may be used depending on the nature of the samples.
- 6.6. 0.45 μm filters, 47 mm - or acrodisk filters with plastic syringe attachment.

7. REAGENTS AND STANDARDS

- 7.1. Sulfuric acid, 18N or 1:1 ratio: Add slowly, while stirring, concentrated sulfuric acid (reagent grade) to an equal volume of deionized water. Allow to cool before transferring to a bottle for storage.

WARNING: ALWAYS ADD ACID TO WATER, NEVER WATER TO ACID!

- 7.2. Sodium hydroxide, 10N: Dissolve 40 g of sodium hydroxide in deionized water and dilute to 100 mL. Allow to cool before transferring to a bottle for storage.
- 7.3. Sodium chloride, 10% (acidified): Dissolve 100 g of NaCl in 800 mL of deionized water. Acidify with concentrated HCl to pH of 2.5. Dilute to 1L.
- 7.4. Deionized water, reagent grade.

8. SAMPLE COLLECTION, PRESERVATION AND STORAGE

- 8.1. Samples are to be collected in suitable wide-mouth containers.
- 8.2. Samples are to be stored at $4^{\circ} \pm 2^{\circ}\text{C}$.
- 8.3. Holding times have not been established for the soil samples for most methods. For most methods, holding time calculations begins after leaching. After the leaching is complete, the holding times for each parameter follows the holding time criteria for water samples for most tests. See the table below for specifics.

TABLE A – HOLD TIMES FOR LEACHATES

Analyte	Hold Time (from leaching to analysis, unless noted)	Preservation
Alkalinity	14 days (preferably 24 hours)	4+/- 2 degrees C
Specific Conductance	28 days (Note a)	4+/- 2 degrees C
Hexavalent Chromium	30 days to extract, 24 hours to analysis	4+/- 2 degrees C
Ammonia	28 days (Note a)	4+/- 2 degrees C (Note b)
NO ₂	48 hours	4+/- 2 degrees C
NO ₃	48 hours	4+/- 2 degrees C
OPO ₄	48 hours	4+/- 2 degrees C
Fluoride	28 days	4+/- 2 degrees C
Bromide	28 days	4+/- 2 degrees C
Chloride	28 days	4+/- 2 degrees C
Sulfate	28 days	4+/- 2 degrees C
NO ₂ + NO ₃ (unpreserved)	48 hours (Note a)	4+/- 2 degrees C
NO ₂ + NO ₃ (preserved)	28 days (Note a)	4+/- 2 degrees C (Note c)
Perchlorate	28 days (Note a)	None

Note a: Hold time is measured from date of sampling.

Note b: Verify pH is 2.5 or lower. If not, add 18N sulfuric acid to bring to 2.5 or lower.

Note c: Verify pH is 2 or lower. If not, add 18N sulfuric acid to bring to 2 or lower.

Note c: Verify pH is 2 or lower. If not, add 18N sulfuric acid to bring to 2 or lower.

9. QUALITY CONTROL

- 9.1. One method blank must be extracted with every process batch of similar matrix, not to exceed twenty (20) samples. The method blank is an aliquot of laboratory reagent water processed in the same manner and at the same time as the associated samples. Corrective actions must be documented on a Non-Conformance memo, and implemented when target analytes are detected in the method blank above the reporting limit. Re-extraction of the blank, other batch QC, and the affected samples are required when the method blank is deemed unacceptable. See Policy QA-003-SAC for specific acceptance criteria.
- 9.2. Duplicate blank leachate (for later use as an LCS): One duplicate blank must be extracted with every process batch of similar matrix, not to exceed twenty (20) samples. The duplicate blank is then spiked with analytes of known identity and concentration at the time of analysis. Corrective actions must be documented on a Non-Conformance memo, then implemented when recoveries of any spiked analyte is outside control limits provided on the LIMS or by the client.
- 9.3. Duplicate leachate (for later use as an MS/MSD): One duplicate sample must be leached, analyzed, and recorded with each batch of samples, not to exceed 20 samples. The duplicate leachate is then spiked with analytes of interest at time of analysis. If a duplicate analysis is requested, the relative percent difference (RPD) for the duplicate pair should be less than 20%. If the duplicate RPD is outside the control limit, impact on data will be assessed and narrated in the final report.
- 9.4. Acceptance criteria and corrective actions depend on the analytical methods used after sample prep.

10. PROCEDURE

- 10.1. One time procedural variations are allowed only if deemed necessary in the professional judgment of supervision to accommodate variation in sample matrix, radioactivity, chemistry, sample size, or other parameters. Any variation in procedure shall be completely documented using a Nonconformance Memo and is approved by a Technical Specialist and QA Manager. If contractually required, the client shall be notified. The Nonconformance Memo shall be filed in the project file.

- 10.2. Any unauthorized deviations from this procedure must also be documented as a nonconformance, with a cause and corrective action described.
- 10.3. Homogenize sample by thoroughly mixing the entire contents of the sample bottle with a spatula before taking a portion.
- 10.4. Weigh 10.0 g sample into a 50 mL centrifuge tube. Record the amount of sample used on the bench sheet. The centrifuge tube must be properly labeled with the sample ID, date of preparation, QC batch ID, initials of the prep analyst, and type of leachate. Perchlorate is prepared using 5.0 grams of sample. The soil to water ratio is 1:10.

Note: Sample weight, DI water volume, and size of bottle may be reduced as long as soil to water ratio is 1:5 and the final volume of leachate is sufficient for the tests required.

10.4.1. For Method Blanks, use Ottawa sand or similar matrix.

10.5. LCS Spike Levels

- 10.5.1. A blank is prepared in duplicate with each prep batch. At the analytical stage, one blank is analyzed as the method blank. The other is spike and analyzed as the LCS. See below for specific spike levels. Additionally, a sample is prepared in duplicate and spiked for MS/SD at the analytical stage.
- 10.5.2. For alkalinity and specific conductance, the LCS is a whole volume standard that is not spiked, but poured directly into tubes for analysis. No leachate spiking is necessary.
- 10.5.3. For hexavalent chromium – Add 0.2 mL of 5 ppm Cr(VI) standard to 9.8 mL of blank solution prepped with the batch. The final concentration is 0.10 mg/L. Be sure to divide by the weight.
- 10.5.4. For ammonia – Add 0.1 mL of 100 ppm NH₃ standard to .9 mL of blank solution prepped with the batch. The final concentration is 2.0 mg/L. Be sure to divide by the weight.
- 10.5.5. For general anions analyzed by ion chromatography (IC) Method 300.0 or 9056 – Add 0.5 mL of IC spiking solution to 4.5 mL of blank solution prepared with the batch. The final concentration is: 1 ppm for NO₂, NO₃; 2 ppm for OPO₄; 5 ppm for fluoride and bromide; 10 ppm for chloride and 10 ppm for sulfate. All have to be divided by the weight to calculate for spike level added in mg/kg units.

- 10.5.6. For nitrate, nitrite analyzed by automated colorimetry – Add 0.2 mL of 10 ppm NO₃/NO₂ standard to 4.8 mL of blank solution prepped with the batch. The final concentration is 0.40 mg/L. Remember to divide by the weight.
- 10.5.7. For perchlorate analyzed by ion chromatography method 314.0 – Add 25 uL of 10,000 ppb Perchlorate working standard to 5 mL of water from the blank prepared with the batch. The final concentration is 50 ppb. Remember to divide by the weight.
- 10.6. Add 50 g of deionized water.
- Note: For ammonia leachates, use 10% NaCl (acidified) in lieu of deionized water. Otherwise, prep factor and leaching procedure is the same.*
- 10.7. Cap each centrifuge securely.
- 10.8. Place the centrifuge tubes on the mechanical shaker. Agitate the samples at a rate of speed that maintains a constant state of agitated suspension. Leave on the shaker for one hour. Record start time on the bench sheets.
- 10.9. After one hour, remove the samples from the shaker. Record end time.
- 10.10. Separate the liquid phase by centrifuging the samples for 5 - 10 minutes, if needed. Apply filtration to the samples using a 0.45 µm filter, if needed. Treat the method blank in the same manner.
- 10.10.1. For hexavalent chromium only - if the sample is high in salts where it is extremely difficult to settle the particulates or filter them through the 0.45 µm filter, add 0.2 g of NaCl to the entire suspension. Shake for 1 minute, then centrifuge and finally filter prior to Cr⁺⁶ determination. Treat the method blank and duplicate leachate in the same manner. No preservation needed.
- 10.11. Preserve aliquots of the filtrate according to the analyses required. See Table A, Section 8.

11. DATA ANALYSIS AND CALCULATIONS

- 11.1. Divide the sample weight by the volume of water added to obtain the weight, normally a 0.2 weight is used:

$$W = \frac{\text{Mass of wet sample used (g)}}{\text{Final volume (mL) or mass (g) of water added to the wet sample}}$$

- 11.2. For final concentration of sample in mg/kg:

$$S, \text{ mg/kg} = [\text{Final concentration found in the leachate (mg/L)}/\text{weight}]$$

- 11.3. Reporting limit (RLs), in mg/kg:

$$\text{RLs, mg/kg} = (\text{RLw}) / (\text{W}) \times (\text{DF})$$

Where:

RLw = normal reporting limit of water samples (mg/L)

W = weight (to convert mg/L to mg/kg)

DF = dilution factor used at the analysis stage

12. METHOD PERFORMANCE

- 12.1. The group/team leader has the responsibility to ensure that this procedure is performed by an associate who has been properly trained in its use and has the required expertise.
- 12.2. This procedure by itself does not have any results. However, when calculating the results from the analysis of the leachate, report results in mg/Kg.

13. POLLUTION PREVENTION

- 13.1. All waste will be disposed of in accordance with Federal, State and Local regulations.
- 13.2. Where reasonably feasible, technological changes have been implemented to minimize the potential for pollution of the environment. Employees will abide by this method and the policies in section 13 of the Corporate Safety Manual for "Waste Management and Pollution Prevention."
- 13.3. Proportional reductions in sample and reagent volume are permitted in accordance with paragraph 10.4. This reduces the excess sample, waste and unused reagent that must be disposed of.

14. WASTE MANAGEMENT

The following waste streams are produced when this method is carried out.

- 14.1 Assorted test tubes, autovials, syringes, filter discs and extracted soil samples. Dump the solid waste into a contaminated lab trash bucket. When the bucket is full or at the end of the day, tie the plastic bag liner shut and put the lab trash into the steel collection drum in the H3 closet. When the drum is full or after no more than 75 days, move it to the waste collection area for shipment.

15. REFERENCES

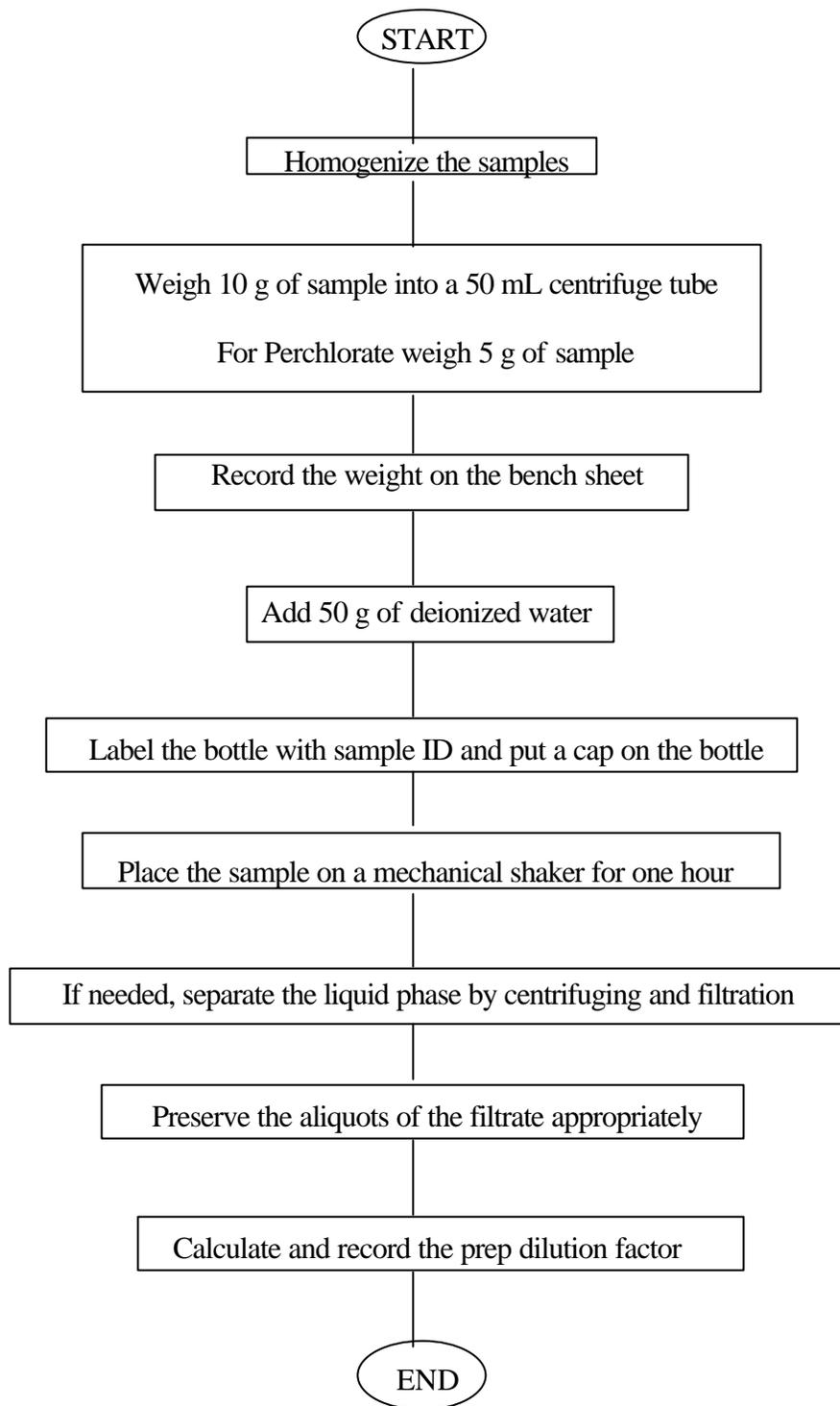
- 15.1. This procedure was adapted from Section 10-2.3., "Methods of Soil Analysis, Part 2, Chemical and Microbiological Properties," Second Edition, Edited by A.L. Page.
- 15.2. Method 300.0, "Determination of Inorganic Anions by Ion Chromatography," Methods for the Determination of Inorganic Substances in Environmental Samples, USEPA, August 1993. Section 11.7.
- 15.3. SW-846, Third Edition, Chapter 3, December 1996.
- 15.4. For Ammonia preparation, Methods for Determination of Inorganic Substances in Water and Fluvial Sediments, USGS, Book 5, Chapter A1, 1979. Method 1-6523-78., Nitrogen, ammonia, total in bottom material, colorimetric, extraction-indophenol, automated, Section 6.1, page 420.

16. MISCELLANEOUS

- 16.1. Deviations from reference method.
 - 16.1.1. The sample is not air-dried before prep. Results are adjusted based on dry weight if requested.
 - 16.1.2. Sodium hexametaphosphate is not used as a preservative. The preservative appropriate to the particular analyte is employed.
 - 16.1.3. A prep factor of 5X is used in lieu of a 10X prep factor as recommended in Method 300.0.
 - 16.1.4. For ammonia, the final volume is not adjusted at the end of the filtration stage. Instead the total final volume is added at the beginning of the extraction.
- 16.2. Summary of modifications to SOP from previous revisions.
 - 16.2.1. Format updated to reflect name change from Quanterra to STL Sacramento.
 - 16.2.2. Added table – Sample collection, preservation and storage
 - 16.2.3. Added perchlorate to list of parameters that can be prepared using DI leach.

16.2.4. The LCS is now spiked at the analytical stage.

16.3. Procedure flow diagram



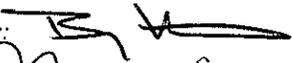
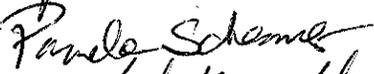
SOP Number:	SAC-WC-0049 Rev. 2	Change Form Number: 1
SOP Title:	DEIONIZED WATER LEACHING PROCEDURE FOR GENERAL CHEMISTRY ANALYSES	
SOP Sections Affected by Change:	Table A, Sections 5, 14, and 15	
Reason for Addition or Change:	Correct the holding time for hexavalent chromium, update to current EH&S Standard.	
Change Effective From [Date]:	12/1/04	
Change or Addition (Specific Section; use additional sheets if necessary.):	<p>Table A, amend the hexavalent chromium holding time as follows: 30 days to extract, 24 hours to analysis</p> <p>Section 5, change to read:</p> <p>5. SAFETY</p> <p>Employees must abide by the policies and procedures in the Corporate Safety Manual, Radiation Safety Manual, Sacramento Supplement to the CSM, and this document. All work must be stopped in the event of a known or potential compromise to the health or safety of an associate. The situation must be reported immediately to a supervisor, the EH&S Staff, or a senior manager.</p> <p>5.1 Specific Safety Concerns or Requirements</p> <p>5.1.1 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.</p> <p>5.1.3 Eye protection that satisfies ANSI Z87.1, laboratory coat, and chemically resistant gloves must be worn while samples, standards, solvents, and reagents are being handled. Latex, PVC and nitrile gloves all provide adequate levels of protection against the chemicals used in this SOP.</p> <p>5.1.4 Exposure to chemicals must be maintained as low as reasonably achievable, therefore all samples must be opened, transferred and prepared in a fume hood. Solvent and waste containers will be kept closed unless transfers are being made.</p> <p>5.1.5 Laboratory procedures such as repetitive use of pipets, repetitive transferring of extracts, and manipulation of filled separatory funnels and other glassware represent a significant potential for repetitive motion or other ergonomic injuries. Laboratory associates performing these procedures are in the best position to realize when they are at risk for these types of injuries. Whenever a situation is found in which an employee is performing the same repetitive motion, the employee shall immediately bring this to the attention of their supervisor, manager, or the EH&S staff. The task will be analyzed to determine a better means of accomplishing it.</p> <p>5.2 Primary Materials Used</p> <p>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.</p>	

SOP Number:	SAC-WC-0049 Rev. 2 Change Form Number: 1												
	<p>--Table of Materials --</p> <table border="0"> <thead> <tr> <th style="text-align: left;">Material (1)</th> <th style="text-align: left;">Hazards</th> <th style="text-align: left;">Exposure Limit (2)</th> <th style="text-align: left;">Signs and symptoms of exposure</th> </tr> </thead> <tbody> <tr> <td>Sodium Hydroxide</td> <td>Corrosive</td> <td>2 Mg/M3-Ceiling</td> <td>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.</td> </tr> <tr> <td>Sulfuric Acid</td> <td>CorrosiveOxidizerDehydratorPoisonCarcinogen</td> <td>1 Mg/M3-TWA</td> <td>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.</td> </tr> </tbody> </table> <p>1 – Always add acid to water to prevent violent reactions. 2 – Exposure limit refers to the OSHA regulatory exposure limit.</p> <p>-- End of Table --</p> <p>Section 13, change to read:</p> <p>13. Pollution Prevention</p> <p>13.1 All waste will be disposed of in accordance with Federal, State and Local regulations.</p> <p>13.2 Where reasonably feasible, technological changes have been implemented to minimize the potential for pollution of the environment. Employees will abide by this method and the policies in section 13 of the Corporate Safety Manual for “Waste Management and Pollution Prevention.”</p> <p>13.3 Proportional reductions in sample and reagent volume are permitted in accordance with paragraph 10.4. This reduces the excess sample, waste and unused reagent that must be disposed of.</p> <p>Section 14, change to read:</p> <p>14. Waste Management</p> <p>The following waste streams are produced when this method is carried out.</p> <p>14.1 Assorted test tubes, autovials, syringes, filter discs and extracted soil samples. Dump the solid waste into a contaminated lab trash bucket. When the bucket is full or at the end of the day, tie the plastic bag liner shut and put the lab trash into the steel collection drum in the H3 closet. When the drum is full or after no more than 75 days, move it to the waste collection area for shipment.</p>	Material (1)	Hazards	Exposure Limit (2)	Signs and symptoms of exposure	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	CorrosiveOxidizerDehydratorPoisonCarcinogen	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.
Material (1)	Hazards	Exposure Limit (2)	Signs and symptoms of exposure										
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.										
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Submitted by: Lisa Stafford

Date: 1/24/2005

Approved by: *

Technical Reviewer:		Date: 1/25/05
EH&S Signature:		Date: 1/25/05
QA Signature:		Date: 1/25/05
Management Signature:		Date: 2/8/05

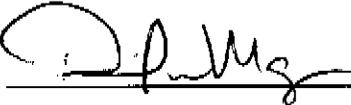
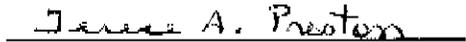
*Must be same signature authorities of SOP being revised.

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**TITLE: HIGH PERFORMANCE LIQUID CHROMATOGRAPHY
Nitroaromatics and Nitramines by SW-846 8330/8332**

Updated by:	Signature:	Date:
Sharon A. Newkirk HPLC Chemist		<u>4/4/05</u>

Approved by:	Signature:	Date:
Patti J. Gibson Section Manager, Organics Dept.		<u>4/5/05</u>
David W. Mazur Env. Health & Safety Coord.		<u>4/6/05</u>
Terese A. Preston Quality Manager		<u>4/6/05</u>

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1.0 SCOPE / APPLICATION

This Standard Operating Procedure (SOP) outlines the trace analysis of explosive residues by High Performance Liquid Chromatography (HPLC) using a UV detector. This SOP was written using SW-846 Methods 8330, 8332, and 8000B as references and is used to determine the concentration of the following compounds in a water, soil, sediment matrix.

ID	Compound	CAS No.*
HMX	Octahydro-1,3,5,7-tetranitro-1,3,5,7-tetrazocine	2691-41-0
RDX	Hexahydro-1,3,5-trinitro-1,3,5-triazine	121-82-4
1,3,5-TNB	1,3,5-Trinitrobenzene	99-35-4
1,3-DNB	1,3-Dinitrobenzene	99-65-0
Tetryl	Methyl-2,4,6-trinitrophenylnitramine	479-45-8
NB	Nitrobenzene	98-95-3
2,4,6-TNT	2,4,6-Trinitrotoluene	118-96-7
2,6-DNT	2,6-Dinitrotoluene	606-20-2
2,4-DNT	2,4-Dinitrotoluene	121-14-2
2-AM-DNT	2-Amino-4,6-dinitrotoluene	355-72-78-2
4-AM-DNT	4-Amino-2,6-dinitrotoluene	1946-51-0
2-NT	2-Nitrotoluene	88-72-2
4-NT	4-Nitrotoluene	99-99-0
3-NT	3-Nitrotoluene	99-08-1
NG	Nitroglycerine **	55-63-0
PETN	Pentaerythritol tetranitrate **	78-11-5

* Chemical Abstracts Service Registry Number

** Extraction and analysis are the same for these compounds, with the exception of the wavelength used for this analysis (sec.4.2.2)

On occasion, clients request slight modifications to this SOP. These modifications are addressed on a case-by-case basis with the range of accuracy (i.e., MDLs, linearity check or PT sample) verified prior to implementation. Any modifications would be written into a Quality Assurance Plan (QAP) and authorized via laboratory signature approval; and amended to the data packages case narrative.

1.1 Method Sensitivity

1.1.1 Method Detection Limits

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 Appendix B of 40 CFR 136, "Guidelines Establishing Test Procedures for the Analysis of Pollutants". 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.

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1.1.2 Reporting Limits

Reporting Limits (Attachment 1) are defined as the lowest concentration of an analyte determined by a given method in a given matrix that the laboratory feels can be reported with acceptable quantitative error or client requirements, values specified by the EPA methods or other project and client requirements. The laboratory maintains reporting limits that are higher than the MDL. Wherever possible, reporting is limited to values approximately 3-5x the respective MDL to ensure confidence in the value reported.

1.1.3 Definitions

Refer to Section 3.0 of the Laboratory's Quality Manual (LQM).

1.2 Summary of Method

This method provides instrument operating parameters for the detection of ppb levels of certain explosives in extracts of soil, sediment or water samples by HPLC.

All of the compounds listed above are either used in the manufacture of explosives or are the degradation products of compounds used for that purpose. For compounds other than these, or for other sample sources, the analyst must demonstrate the usefulness of the method by performing an MDL study, and collecting precision and accuracy data on actual samples.

1.2.1 Low-Level Water Method (Salting Out)

Aqueous samples of low concentration are prepared for analysis by a reverse salting-out extraction procedure with acetonitrile and sodium chloride.

1.2.2 High-Level Water Method (Option)

Direct injection of diluted and filtered water samples can be used for water samples of higher concentrations. (Reporting limits can be established).

1.2.3 Soil and Sediment Samples

Soil and sediment samples are extracted using acetonitrile in an ultrasonic bath.

1.2.4 Solid Phase Extraction (SPE)

Aqueous samples are prepared for analysis by passing sample through a solid-phase media and then eluted from media by using appropriate solvent.

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2.0 INTERFERENCES

- Solvents, glassware and other sample processing hardware may yield discrete artifacts and/or elevated baselines, causing misinterpretation of the chromatograms. All of these materials must be demonstrated to be free from interference under the conditions of the analysis by running method blanks.
- All glassware/equipment used in the preparation of standards (volumetric flasks, pipettes, beakers, eppendorf tips are rinsed well with the appropriate solvent and allowed to dry before use. After completion of standard preparation, unused amounts of standards are properly disposed of, and all glassware/equipment rinsed well with the appropriate solvent. Rinse/excess solvent is properly collected and disposed of. These procedures should eliminate positive interferences from these sources.
- All glassware used must be scrupulously clean prior to use. All re-usable glassware must be washed following procedures described in the laboratory glassware cleaning SOP (UQA-009). All glassware must also be rinsed at least 3 times with the appropriate solvent.
- Tetryl decomposes rapidly in methanol/water solutions, as well as with heat. All samples expected to contain Tetryl should **not** be exposed to temperatures above room temperature and not above 10°C when diluted for injection. It is imperative that the automatic liquid samplers employed have the capability of being chilled to <10°C to minimize Tetryl degradation while standards and samples are awaiting analysis.

3.0 SAFETY

Employees must abide by the policies and procedures in the Corporate Safety Manual, Radiation Safety Manual and this document.

3.1 Specific Safety Concerns or Requirements

- 2,4,6-TNT is the analyte most often detected in high concentrations in soil samples. Soil samples as high as 2% 2,4,6-TNT can be safely ground. Samples containing higher concentrations should **not** be ground. The project manager or client must provide information as to whether the samples are suspected to contain explosives at a level greater than 2%. Visual observation of soil samples taken from a site expected to contain explosives is also important. Lumps of material that have a chemical appearance should be suspect and not ground. Explosives are generally a very finely ground grayish-white material.
- Parts of the instrument can be hot. Care should be taken if the instrument needs to be adjusted internally.

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3.2 Primary Materials Used

The following is a list of the materials used in this method, which have a serious or significant hazard rating. **NOTE: This list does not include all materials used in the method. The table contains a summary of the primary hazards listed in the MSDS for each of the materials listed in the table.** A complete list of materials used in the method can be found in the reagents and materials section. Employees must review the information in the MSDS for each material before using it for the first time or when there are major changes to the MSDS.

Material	Hazards	Exposure Limit (2)	Signs and symptoms of exposure
Acetonitrile	Flammable Poison	40 ppm-TWA	Early symptoms may include nose and throat irritation, flushing of the face, and chest tightness. Prolonged exposure to high levels of vapors may cause formation of cyanide anions in the body.
Methanol	Flammable Poison Irritant	200 ppm-TWA	A slight irritant to the mucous membranes. Toxic effects exerted upon nervous system, particularly the optic nerve. Symptoms of overexposure may include headache, drowsiness and dizziness. Methyl alcohol is a defatting agent and may cause skin to become dry and cracked. Skin absorption can occur; symptoms may parallel inhalation exposure. Irritant to the eyes.
Methylene Chloride	Carcinogen Irritant	25 ppm-TWA 125 ppm- STEL	Causes irritation to respiratory tract. Has a strong narcotic effect with symptoms of mental confusion, light-headedness, fatigue, nausea, vomiting and headache. Causes irritation, redness and pain to the skin and eyes. Prolonged contact can cause burns. Liquid degrades the skin. May be absorbed through skin.

2 – Exposure limit refers to the OSHA regulatory exposure limit.

4.0 EQUIPMENT AND SUPPLIES

4.1 Supplies

- Disposable luer-lock filters - 0.20 um Teflon® filter
- Pipettes: 10-mL, 9-mL, 5-mL, 1-mL glass, volumetric, Class A
- Pasteur pipettes
- Disposable 10-mL syringe with Luer-Lock fitting
- 6x125mm screw-top (Teflon-lined) test tubes
- 1.5-mL amber autosampler vials
- Eppendorfs: 100 uL-1000 uL size (adjustable)
- Air-forced drying oven
- Volumetric flasks (glass, Class A): 5.0-mL and 10.0-mL
- 1.0 L Erlenmeyer flask
- 125-mL Erlenmeyer flask
- Magnetic stir bars
- Automatic magnetic stirrer
- Muffle furnace capable of 400°C

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- Temperature-controlled ultrasonic bath
- Water chiller recirculator capable of achieving <10°C
- Balance, ± 0.1 mg
- Vortex, VWR, Vortex Genie 2, or equivalent
- Vacuum Pump
- Strata SPE Manifold

4.2 HPLC System

- 4 - Agilent 1100 HPLCs equipped with UV detectors and autosampler with a refrigerated water circulator capable of chilling extracts to <10°C.

4.2.1 HPLC Columns

- Primary Column: Phenomenex Ultracarb (C18), 5u ODS (20); 250 x 4.6mm ID
- Confirmation Column: Phenomenex Luna 3u Phenyl-Hexyl 150 x 4.6mm ID

4.2.2 HPLC Conditions

Primary Column	Secondary Column
Phenomenex Ultracarb (C18), 250 x 4.6 mm ID, 5 micron particle size, normal phase.	Phenomenex Luna Phenyl Hexyl, 150x4.6mm ID, 3 micron particle size, reverse phase
Mobile Phase: 50% water / 45% MeOH / 5% ACN*	Mobile Phase: 59% 0.1% H3PO4/ 41% 9:1 MeOH / ACN *
Flow Rate: 1.0 mL/min*	Flow Rate: 1.0 mL/min*
Injection volume: 100 uL*	Injection Volume: 100 uL*
UV Detector: 254 nm**/**	UV Detector: 254 nm**/**
Column Temperature: 30°C	Column Temp.: 35°C
Range 0.100 Au	Range: 0.200 Au

* Conditions may be varied to achieve optimum separation.

** When analyzing for NG and PETN a wavelength of 210 nm must be used for C18 column and a wavelength of 214 nm for the Phenyl Hexyl column.

4.2.3 Data Collection

Each HPLC uses TurboChrom for data acquisition and Target for processing data.

5.0 REAGENTS AND STANDARDS

5.1 Reagents

- Laboratory pure water (Milli-Q)
- Methanol (MeOH) - HPLC grade or better
- Acetonitrile (ACN) - HPLC grade or better
- Sodium Chloride, NaCl, Reagent grade. Kilned at 400°C for 4 hours. Stored in glass bottles.

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5.2 Standards

Stock standards are purchased as solutions generally from Restek and Cerrilant. When available, standards with A2LA, CRADA or EPA certifications are purchased. If unavailable, the standards are verified against an alternate source.

These solutions are purchased at concentrations such that appropriate dilution's can be made to achieve desired concentrations. When making stock solutions for calibration, treat each explosive compound with caution.

The preparation of all standards and QC solutions must be properly documented. All standards and QC solutions must be labeled with the date of preparation, expiration date, analyst, and concentrations of analytes. Standards must be stored in glass containers with Teflon-lined lids at $4 \pm 2^{\circ}\text{C}$ protected from the light. Stock standards must be replaced after 1 year, or sooner if comparison with a check standard indicates a problem.

5.2.1 Parent Surrogate Solution: 20 ug/mL

The surrogate solution (1,2-Dinitrobenzene) is purchased from a vendor at a concentration of 1,000 ug/mL. The Parent Surrogate Solution is prepared by diluting 1-mL of surrogate with ACN to a final volume of 50-mL. This produces a final concentration of 20 ug/mL.

5.2.1.1 Working Surrogate Solution: 2 ug/mL

The Working Surrogate Solution is prepared by volumetrically diluting 5.0-mLs of the Parent Surrogate to 50.0-mLs of ACN (1/10 dilution), resulting in a concentration of 2.0-ug/mL. 600 μL s is added to water samples; and 1.0 mL is added to soil samples.

- **Label Information:** All standard labels must contain the date prepared, the date of expiration, the analyst name, and the standard number.
- **Storage / Life:** All standard and spikes must be stored in Teflon-sealed screw-capped bottles with minimal headspace at $4 \pm 2^{\circ}\text{C}$ and protected from light. This solution is valid for 30 days.

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5.2.2 Parent Spike Solution

[Laboratory Control Sample (LCS) and Matrix Spike (MS)/MS Duplicate (MSD)]

Two different explosive spike mixes are purchased from a vendor for the preparation of the working spike solution.

Mix #1	Mix #2
HMX	Tetryl
RDX	2,6-Dinitrotoluene
1,3,5-Trinitrobenzene	2-Nitrotoluene
1,3-Dinitrobenzene	3-Nitrotoluene
Nitrobenzene	4-Nitrotoluene
TNT	2-Amino-4,6-DNT
2,4-Dinitrotoluene	4-Amino-2,6-DNT

Their concentrations are 1,000-ug/mL. The Parent Spike is prepared by adding 1.0-mLs of Mix #1 and 2.0-mLs of Mix #2; and brought up to a final volume of 50-mL in ACN. The final concentrations are 20 ug/mL (Mix #1) and 40 ug/mL (Mix #2). Independently, PETN and NG are prepared in the same manner using 1.0-mL of each to a final volume of 25-mL giving a final concentration of 40 ug/mL.

5.2.2.1 Working Spike Solution: 2.0 ug/mL / 4.0 ug/mL

The Working Spike Solution is prepared by diluting 1.0 mL of the Parent Spike Solution to 10.0 mLs ACN (1/10 dilution). The final concentrations are 2.0 / 4.0 ug/mL (Attachment 2). 600 uLs are added to water samples and 1 mL is added to soil samples.

- **Label Information:** All standard labels must contain the date prepared, the date of expiration, the analyst name, and the standard number.
- **Storage / Life:** All standard and spikes must be stored in Teflon-sealed screw-capped bottles with minimal headspace at 4±2°C and protected from light. This solution is valid for 30 days.

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5.2.3 Parent Calibration Standards

Calibration standards are prepared in ACN. One of the concentration levels should be at, or below a concentration equivalent to the reporting limit. The remaining concentration levels should correspond to the expected range of concentrations found in real samples or should define the working range of the HPLC. Explosive standards are purchased individually from a vendor for the preparation of the intermediate solutions Mix #1 and Mix #2.

Mix #1	Mix #2
HMX	Tetryl
RDX	2,6-Dinitrotoluene
1,3,5-Trinitrobenzene	2-Nitrotoluene
1,3-Dinitrobenzene	3-Nitrotoluene
Nitrobenzene	4-Nitrotoluene
TNT	2-Amino-4,6-DNT
2,4-DNT	4-Amino-2,6-DNT

Their concentrations are 1,000 ug/mL. The Parent standard is prepared by adding 0.5-mL of Mix #1, 1.0-mL of Mix #2, and 0.5-mL of the surrogate. Dilute to a final volume of 50-mL in ACN. The resulting concentrations are listed in Attachment 2. NG and PETN are made independently using 1.0-mL of each and 0.5-mL of the surrogate to a final volume of 50-mL in ACN.

- **Label Information:** All standard labels must contain the date prepared, the date of expiration, the analyst name, and the standard number.
- **Storage / Life:** All standard and spikes must be stored in Teflon-sealed screw-capped bottles with minimal headspace at 4±2°C and protected from light. This solution is valid for 30 days.

5.2.3.1 Working Calibration Standards

6-concentration levels are prepared through dilution of the Parent Calibration Standard and are prepared fresh on the day of calibration. The resulting calibration range in relative concentrations ranges from 0.02/0.04 ng/uL through 1.0/2.0 ng/uL. Refer to Attachment 2 for a listing of the concentrations.

- **Label Information:** All standard labels must contain the date prepared, the date of expiration, the analyst name, and the standard number.
- **Storage / Life:** All standard and spikes must be stored in Teflon-sealed screw-capped bottles with minimal headspace at 4±2°C and protected from light. This solution is valid for 30 days.

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5.2.4 Continuing Calibration Verification (CCV)

The CCVs are alternating mid-level standards; and the concentrations are consistent with the cited Levels 4 and 5 concentrations listed in Attachment 2.

- Label Information: All standard labels must contain the date prepared, the date of expiration, the analyst name, and the standard number.
- Storage / Life: All standard and spikes must be stored in Teflon-sealed screw-capped bottles with minimal headspace at $4 \pm 2^{\circ}\text{C}$ and protected from light. This solution is valid for 6 mos..

5.2.5 Second Source Verification (SSV)

The SSV is second-source standard consistent with the Level 5 concentration listed in Attachment 2. (Some clients request the SSV at the Level 2 concentration).

- Label Information: All standard labels must contain the date prepared, the date of expiration, the analyst name, and the standard number.
- Storage / Life: All standard and spikes must be stored in Teflon-sealed screw-capped bottles with minimal headspace at $4 \pm 2^{\circ}\text{C}$ and protected from light. This solution is valid for 6 mos..

6.0 CALIBRATION (NON-DAILY)

Explosives are confirmed by use of a second column (Section 4.2.2). The acceptance criteria for this analysis is identical to that of the primary column.

6.1 Retention Time Windows

Before establishing retention time (RT) windows, make sure the HPLC system is within optimum operating conditions. Make 3-injections of a standard throughout the course of a 72-hour period. Serial injections over less than a 72-hour period may result in retention time windows that are too tight.

$\pm 3\text{X}$ Standard Deviation of the absolute RTs for each standard will be used to define the RT window; however, the experience of the analyst should weigh heavily in the interpretation of chromatograms.

The laboratory must calculate RT windows for each analyte on each LC column and whenever a new LC column is installed. The data must be retained by the laboratory and available for review.

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7.0 PROCEDURE

7.1 Quality Control Checks

Quality Controls	Frequency	Control Limit
Method Blank (MB)	1 in 20 or fewer samples	< Reporting Limit
Lab Control Sample (LCS)	1 in 20 or fewer samples	Statistical control limits ⁴
LCS Duplicate (LCD) ¹	1 in 20 or fewer samples	Statistical control limits
Matrix Spike (MS) ²	1 in 20 or fewer samples	Statistical control limits
MS Duplicate (MSD) ²	1 in 20 or fewer samples	Statistical control limits
Surrogate	every sample ³	Statistical control limits

¹ LCDs are performed only when insufficient sample is available to perform the MS/MSD or when requested by the client/project/contract.

² If not designated, the sample selection for MS/MSD are rotated among client samples so that various matrix problems may be noted and/or addressed.

³ Analytical and QC samples.

⁴ Internal statistical control limits are updated; refer to Attachment 1.

7.2 Sample Preservation and Storage

Samples are to be collected in glass containers with Teflon-lined lids. All samples are to be maintained at 4±2°C, in the dark, prior to and after extraction/analysis.

Matrix	Holding Time (VTS): (to Extract)	Holding Time: (Extraction to Analysis)
Soil / Sediment	14 days	40 days
Water	7 days	40 days

VTS: Verified Time of Sampling

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7.3 Sample Preparation

7.3.1 Reverse Salt-Out Extraction (Low - Level Waters)

7.3.1.1 Measure out 770 mL of sample (using a 1-liter graduate) and transfer it to a 1-liter narrow neck Erlenmeyer flask, or 1-liter volumetric flask. Add 251.3 g of NaCl (kilned at 400°C for 4 hours) to the sample. Add a stir bar and mix the contents at maximum speed on a magnetic stirrer until the salt is completely dissolved.

7.3.1.2 Using an adjustable microdispenser, add 600 uLs of working surrogate solution to each sample, MB, LCS, and MS/MSD. Add 600 uLs of working spike solution to the LCS and MS/MSD.

7.3.1.3 Continue the stirring motion and add 164 mLs HPLC grade ACN. Stir for 15 minutes. Allow the phases to separate for 10 minutes. Remove the top ACN layer (about 8 mLs) with a Pasteur pipette and transfer it to a 125 mL narrow neck Erlenmeyer flask, or a 100 mL volumetric flask.

7.3.1.4 Add 10 mLs of fresh ACN to the original sample and stir for 15 minutes. Allow the phases to separate for 10 minutes. Remove the top ACN layer and add to the Erlenmeyer containing the original ACN extract.

7.3.1.5 Add 84 mLs of saturated salt solution (325.1 g NaCl/1 L H₂O) to the 125 mL Erlenmeyer containing the ACN sample extract. Stir for 15 minutes. Allow to separate for 10 minutes.

7.3.1.6 Remove the top ACN layer and add it to a graduated test tube which accurately measures <10.0 mL. (It is imperative at this point to not allow the transfer of any saturated salt solution with the ACN.)

7.3.1.7 Add 1.0 mL fresh ACN to the Erlenmeyer and stir for 15 minutes. Allow to separate for 10 minutes. Remove the top ACN layer from the Erlenmeyer and add to the first extraction aliquot in the graduated test tube.

7.3.1.8 Adjust the final volume to 6 mLs. On occasion, the final volume ends up being greater than 6 mLs. Document the final volume appropriately.

7.3.1.9 Filter the extract through a 0.2 um Teflon filter prior to analysis.

7.3.1.10 All sample extracts and standards are diluted 1:1 with filtered Milli-Q water prior to analysis on the C18 column. All sample extracts and standards are diluted 1:2 with filtered Milli-Q water prior to analysis of the Phenyl Hexyl column.

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7.3.2 High-Level Waters (Option)

Sample filtration: Place a 5 mL aliquot of each water sample in a 16x125mm test tube. Add 5 mLs of ACN, shake thoroughly, and filter through a 0.2 um Teflon filter using a disposable syringe. Discard the first 3 mLs of filtrate, and retain the remainder in a Teflon-capped vial for HPLC analysis.

7.3.3 Soil and Sediment Samples

7.3.3.1 Dry a homogenized, representative portion of each soil sample in an air-forced drying oven being careful not to expose the sample to direct light - soils/sediments are dried to a constant weight. Add the MB soil (Ottawa sand) into the oven along with the samples. This will ensure no cross contamination occurs during the drying process.

Pulverize the dried sample with a mortar and pestle. It is imperative that the mortar and pestle be rinsed thoroughly between samples to prevent the possibility of cross contamination. With a spatula, remove any sticks, rocks or other extraneous material. Particle size should resemble soil passed through a 30 mesh sieve.

NOTE: See safety section regarding extraction of wet soils for high level samples.

7.3.3.2 Weigh a 2.0 g aliquot of each dry, pulverized, soil sample into a properly labeled, 16x125mm screw-top test tube. Add 1.0 mL of working surrogate solution to each sample, MB, LCS, and MS/MSD. Add 1.0 mL of working spike solution to each sample, LCS, and MS/MSD. Add ACN to achieve a final volume of 10.0 mLs. Cap with a Teflon-lined screw cap. Vortex each sample for 1 minute. Store the extra, pulverized sample in small, labeled vials for future use, if needed.

7.3.3.3 Place the test tubes (contained in a test tube rack) in an ultrasonic bath and extract for 18 hours.

7.3.3.4 To minimize Tetryl breakdown, the ultrasonic bath must be chilled to <10°C. This may be achieved by running a line from the water chiller recirculator into the bath to chill the bath to <10°C.

7.3.3.5 After sonication, centrifuge each sample tube to separate the soil from the extract. Place the supernatant in a disposable syringe and filter through a 0.2 micron Teflon-filter. Sample is then prepared for instrument as is Section 7.3.1.10

7.3.3.6 Solid Phase Extraction (SPE) Method 3535 (See Attachment 6).

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7.4 Calibration / Standardization

Before any instrument is used as a measurement device, the instrument response to known reference materials must be determined. The manner in which various instruments are calibrated depends on the particular type of instrument and its intended use. All sample measurements must be made within the calibration range of the instrument. Preparation of all reference materials used for calibration must be documented.

Calibration Controls	Sequence	Control Limit
Calibration Standards	5-point (minimum) curve	≤ 20% RSD
Cont. Cal. Verif. (CCV)	Prior to and after every 10 injections	±15% pred. response
RT Windows (RTW)	Initial CCV determines midpt. of RTW	+ 3X SD

All standards and samples must be allowed to equilibrate in the autosampler chiller prior to analysis.

All electronic equipment is allowed to warm up for 30-minutes. During this period, at least 15 void volumes of mobile phase are passed through the column (~20 min at 1.5 mL/min) and continued until the baseline is level.

7.4.1 Instrument Calibration

7.4.1.1 Prepare a minimum of 5 levels of calibration standards (Attachment 2). The calibration standards define the working range of the HPLC with the lowest standard being at or below the reporting limit.

7.4.1.2 Inject each calibration standard using the same sample introduction technique that will be used to introduce the actual samples into the HPLC. The ratio of the response to the amount injected, defined as the calibration factor (CF), can be calculated for each analyte at each standard concentration. If the percent standard deviation (%RSD) of the calibration factors is ≤ 20%, linearity through the origin can be assumed and the average response factor (RF) can be used for calculations. Alternatively, if a correlation coefficient of ≥0.995 is obtained, linear regression may be used for calculating compounds.

$$\text{Calibration Factor} = \frac{\text{Peak Area (or Height)}}{\text{Mass injected (nanograms)}}$$

$$\text{Response Factor (RF)} = \frac{\text{Concentration}}{\text{Peak Area (or Height)}}$$

7.4.1.3 The working calibration curve, or calibration factor, must be verified on each working day by injecting the alternating CCVs (mid-level standards). If the response for any analyte varies from the predicted response by more than ±15% Difference, a new calibration curve must be prepared for that analyte, unless maintenance can be performed which brings the instrument back into control.

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7.4.1.4 Calibration Evaluation and Acceptance Criteria

Refer to the STL Corporate Procedure, P-T-001, 'Selection of Calibration Points' (Attachment 3) or on STLs Intranet.

7.5 Preventive Maintenance

- Laboratory pure water must be filtered, using an all-glass apparatus, through 0.2um filter prior to use on the instrument.
- If peak splitting occurs, an increase in pressure is usually seen. The source of the increase is most likely a plugged purge valve frit or pre-column filter. The column itself may be the source of increased pressure. This is easily checked by substitution with a known good column. As a last resort the column's internal frit may be changed. Care should be taken not to disturb the column's packing. Disturbance of the packing may result in voids within the column and channeling.

7.6 Sample Analysis

7.6.1 Samples are analyzed in a set referred to as an analytical sequence. This sequence begins with the analysis of a CCV. If comparison of the CF from the CCV are within $\pm 15\%$ difference of the average CF from the calibration curve, then the analysis sequence may proceed with a MB, followed by the LCS and then the samples. If the CF of the CCV is greater than $\pm 15\%$ difference, a new calibration sequence must be analyzed.

7.6.2 A CCV must be injected after every 10 injections. The CF for each analyte to be quantitated must not exceed a 15% difference when compared to the initial calibration sequence. When this criterion is exceeded, inspect the HPLC system to determine the cause and perform whatever maintenance is necessary before re-analyzing the standard. If the CF still exceeds the 15% difference criteria, a new calibration sequence is required. All samples must be bracketed by standards that are within control.

7.6.3 If the response of any sample exceeds the linear range of the system, dilute the sample and re-analyze.

7.6.4 Establish daily RT windows for each analyte. Use the absolute RT for each analyte from the beginning of the sequence as the midpoint of the window for that day. The daily RT window equals the midpoint $\pm 3X$ the standard deviation (Sec. 6.1).

7.6.5 Tentative identification of an analyte occurs when a peak from a sample extract falls within the daily RT window. Confirmation is required on a second HPLC column. When at sufficient concentrations, GC/MS confirmation may be used.

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7.6.6 Validate the qualitative performance of the HPLC system by running the alternating mid-level CCVs throughout the analysis sequence to evaluate this criterion. If any of the standards fall outside their daily retention time window, the system is out-of-control. Determine the cause of the problem and correct it.

7.7 Manual Integration Policy

In each case where manual integrations have taken place, the operator must identify, initial and date the changes on the hardcopy. The following guidelines apply with further details available in STLs Corporate SOP for manual integrations (S-Q-004).

- Manual integrations should be consistent between all files integrated.
- Manual integrations should not be performed to meet QC criteria.
- Manual integrations are automatically flagged with an 'M' on the raw data.
- Excessive manual integrations may reflect an instrumental or methodological problem that should be addressed.

Manual integrations are most often performed for the following reasons:

- Assignment of correct peak that was mis-identified by the data system.
- Incomplete auto-integration due to high level of target compound detected.
- Incomplete auto-integration due to background interference.
- Incorrect auto-integration due to co-elution or near co-elution of compounds.
- Missed peaks.

All integrations are reviewed by the analyst. All chromatograms and reports are printed after any integrations take place and are routinely included in the data packages. Manual integrations may be documented within the narrative (if so required), however, reference to this and the Corporate Manual Integration SOP will be used for explanations and any further documentation beyond initials and dates will not be done.

7.8 DOCUMENTATION

7.8.1 Instrument Run Logs

The analysis of samples and standards is documented within each instrument-specific run log (Attachment 4), and must be completed for each day's analysis.

7.8.2 Traceability of Standards

Upon receipt or preparation, each standard is entered into LabNet (LIMS) and is issued a unique ID# based upon the type and sequential order in which the item was received. Further information entered into the database includes the manufacturer, lot #, the date received or prepared, the expiration date, volume/weight received; concentration; preparation details (if applicable), initials of the recording analyst, and the description of the item (i.e., XXXX Stock Solution – LCS/MS). Once the record is created, a unique label is printed and affixed to the appropriate standard bottle.

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7.8.3 Data Review

Analytical data goes through a 200% review cycle. The analyst and a trained data reviewer perform the reviews according to the criteria established on the data review checklist (Attachment 5). Upon the first 100% review, the checklist is initialed and dated as reviewed. The package, with its checklist, comments and any CARs, is submitted to the section manager or peer reviewer for a second review. Once again, the checklist is initialed and dated by the second reviewer. The completed data review checklist remains on file with the original data

8.0 QUALITY CONTROL

8.1 QC Summary

8.1.1 At least one MB and LCS will be included in each laboratory lot of 20 or fewer samples. The MB will be examined to determine if contamination is being introduced in the laboratory. The MB and LCS must be carried through all stages of the sample preparation and measurement steps. The results of these are tabulated by the QA department to generate in-house control limits.

8.1.2 Accuracy will be measured by the percent recovery (%R) of the LCS. The recovery must be in range, as determined by statistical analysis, in order to be considered acceptable. Additionally, %R will be plotted on control charts to monitor method accuracy.

8.1.3 Precision will be measured by the reproducibility of the MSs and will be calculated as Relative Percent Difference (RPD). If MSs were not analyzed, reproducibility will be measured using the LCS/LCD. Results must agree within statistical control limits in order to be considered acceptable.

8.1.4 Surrogate compounds will be added to every analytical and QC sample to measure the performance of the analysis. Results must agree within statistical control limits in order to be considered acceptable.

8.1.5 Each time an analytical sequence is started, the standards must be evaluated to determine if the chromatographic system is operating properly. The analyst should consider--Do the peaks look normal?, is the response obtained comparable to the response from previous calibrations? Careful examination of the standard chromatograms can indicate whether the column is still good, the injector is leaking, etc....

8.1.6 The laboratory must maintain records to document the quality of the data generated. When results of the LCS indicate irregular method performance, a quality control check standard should be analyzed to confirm that the measurements were performed in an in-control mode of operation.

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8.1.7 Before analysis of any samples, the analyst should demonstrate, through the analysis of a MB that interference from the analytical system, glassware and reagents are under control.

8.1.8 If any changes are made to the chromatographic system, recalibration of the system must take place.

8.1.9 Required Instrument QC

- The method requires that the %RSD vary by <20% when comparing calibration factor to determine if a five (or more) point calibration is linear through the origin. If the %RSD is <20%, the average CF from the calibration can be used to quantitate the samples. Alternatively, linear regression can be used to quantitate the samples; however, a correlation coefficient of 0.995 should be achieved prior to using the curve.
- CCVs must be within $\pm 15\%$ difference from the average CF of the linearity. If the limit is exceeded, corrective action must be taken to correct the problem, or the sequence must be started over. All samples should be bracketed by acceptable CCVs and all analytical runs are closed-out with a CCV. There are situations where if samples are clean, and a CCV displays an increase in sensitivity, the samples don't have to be re-analyzed. This issue must be dealt with on a case-by-case situation and must be documented and approved prior to reporting any data.
- RT windows must be established.
- All continuing standards must fall within their daily RT windows.
- For every batch of samples (≤ 20 samples/batch), a MB, LCS, and MS/MSD must be performed. Also, every sample, MB, LCS, MS/MSD must be spiked with the surrogates.
- Limits used for spike recoveries are statistically generated limits, or limits which have been specifically requested by the client (refer to project QAP).

8.2 Corrective Action

When an out-of-control situation occurs, the analysts must use his/her best analytical judgment and available resources when determining the action to be taken. The out-of-control situation may or may not be caused by more than one problem. The analyst should seek the help of his/her supervisor, QA personnel, or other experienced staff if he/she are uncertain of the cause of the out-of-control situation and the corrective action. The analysis must not be resumed until the source of the problem and an in-control status is attained. All samples associated with the out-of-control situation must be reanalyzed. Out-of-control data must never be released without approval of the section manager, QA personnel, or the laboratory manager.

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Listed below are steps that MUST be taken when an out-of-control situation occurs:

- demonstrate that all the problems creating the out-of-control situation were addressed,
- document the problem and the action which was taken to correct the problem on a corrective action report (CAR) form,
- document on the CAR that an in-control situation has been achieved; and
- receive approval (signature) of the section manager, QA personnel, or the laboratory manager prior to release of any analytical data associated with the problem.

NOTE: See your section manager if at any time the analyst is uncertain as to what actions to take or how to perform suggested maintenance; or if something unusual is happening that is not described in this section. Costly damage can result to the instrument, detector, or column if maintenance is not performed correctly.

8.2.1 Calibration Curve

- Reanalyze the standard curve.
- Prepare new stock and/or working standards.

8.2.2 Continuing Calibration Verification (CCV)

- Repeat CCV to verify proper preparation.
- Prepare new CCV from original stock.
- Check for instrument drift.
- Recalibrate with new standard curve and repeat all samples since the previous in-control CCV.
- Prepare new stock and/or working standards.

8.2.3 Laboratory Control Sample (LCS)

If the LCS is low

- Re-inject the extract to ensure the error wasn't an injection error.
- Determine the source of the error, re-extraction of the entire set may be necessary. Initiate a Sample Discrepancy Report (SDR) so the project manager or section manager can determine if re-extraction is required.

If the LCS is high

- Re-inject the extract to ensure the error wasn't an injection error.
- Check for source of possible contamination, re-extraction of the entire set may be necessary.
- Initiate an SDR so the project manager or section manager can determine if re-extraction is required. If all samples are non-detects, this situation may be able to be narrated.

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8.2.4 Method Blank (MB)

The MB should not have any target compounds present above the reporting limit.

- Re-inject the MB to verify that the contamination is not within the chromatographic system.
- Determine the level of contamination in the MB and in the associated samples. If the associated samples are either non-detects, or have the same target compound as the MB at a level >10 times what was in the MB, the data may be acceptable to report.
- Initiate an SDR immediately, so that the project manager or section manager can determine if re-extraction is necessary.

8.2.5 Matrix Spike/Matrix Spike Duplicate (MS/MSD)

- Re-inject the extracts to ensure the error wasn't with the injection.
- If both the MS/MSD are biased low, or both are biased high, and the RPD is within control, sample matrix may be assumed as the cause.
- Otherwise, initiate an SDR so that the appropriate actions can be taken. Re-extraction may be required.

8.2.6 Retention Time Windows

Initial RT windows must be established following the procedure described in Section 6. Daily windows are established (Section 7.6.4) at the beginning of each analytical sequence. If a continuing standard has any analytes that are outside of their daily windows, corrective actions must be taken before continuing.

- Evaluate the data for usability based on a comparison with the standards run during the analytical sequence.
- Consider the RTs for the surrogates and spiked compounds analyzed before and after the sample in question.
- Check the instrument for leaks, check flows, and pressures.
- Document using a CAR.

8.2.7 Surrogate Spike

The recoveries for the surrogate(s) should be within statistical control limits.

- Check calculations and spike preparation for documentable errors.
- If the surrogate recoveries in the MB and LCS are within the control limits, then sample matrix effects are the most likely cause. However, any samples with surrogate recoveries significantly below the control limits with, no visible chromatographic cause, should be reanalyzed to determine if an injection error was the cause for the low recovery.

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- If the surrogate recoveries in the associated MB and LCS are not within control limits, and the samples are within the holding time, then re-extract all associated samples.
- If the samples are outside the holding time, then contact the project manager using and SDR.

Unless otherwise directed, samples will not be re-analyzed out of hold time and data will be submitted with appropriate narration.

8.2.8 Continuing Calibration Verification (CCV)

Alternating mid-level CCVs are run after every 10 sample injections. The response factors of the CCV should not vary from the average response factor of the initial calibration by more than $\pm 15\%$. If any of the target compounds fail this criteria, then the standard is considered to be non-compliant.

- Re-analyze the standard.
- If the standard is still non-compliant, then prepare a new standard.
- If the new standard is non-compliant, then recalibrate the instrument with a new curve.
- Any samples bracketed by a standard that does not meet this criteria, must be reanalyzed.
- Notify the project manager of any non-compliance using the SDR.

Unless directed otherwise, samples will not be analyzed outside of holding time and the data will be submitted with appropriate narration.

9.0 DATA ANALYSIS AND CALCULATIONS

9.1 Concentration (ppb) =
$$\frac{[(A_1)(A)(V_1)(D)]}{[(A_2)(V_i)(V_s)]}$$

Where:

A_1 = response for the analyte in the sample (area or height)

A = amount of standard injected (ng)

A_2 = average CF for the linearity (area or height, which ever was used for A_1)

V_1 = volume of extract injected

D = dilution factor

V_t = volume of total extract (uL)

V_s = volume/weight of sample extracted.

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9.2 **%RSD** = $\frac{\text{Standard Deviation}}{\text{Mean}} \times 100$

Where:

$$\text{Std. Dev.} = \sqrt{\frac{\sum_{i=1}^n (x_i - \bar{x})^2}{n - 1}}^{\frac{1}{2}}$$

Where:

x_i = each individual value used to calculate the mean
 \bar{x} = the mean of n values
n = the total number of values

9.3 **% Difference** = $\frac{\text{Avg. CF} - \text{CF}}{\text{Avg. CF}} \times 100$

Where:

Avg. CF = Average CF from initial linearity
CF = CF from the analysis of the verification standard

9.4 **Surrogate % Recovery** = $\frac{Q_d}{Q_a} \times 100$

Where:

Q_d = Quantity determined by analysis
 Q_a = Quantity added to sample/blank

9.5 **Spike %Recovery** = $\frac{\text{SR}}{\text{SA}} \times 100$

Where:

SR = Spike Result
SA = Spike Added

9.6 **Accuracy (%R)** = $\frac{(A_T - A_O)}{A_F} \times 100$

Where:

A_T = Total amount recovered in the fortified sample
 A_O = Amount recovered in the unfortified sample
 A_F = Amount added to sample

9.7 **Precision (RPD)** = $\frac{|B_1 - B_2|}{(B_1 + B_2) / 2} \times 100$

Where:

B_1 = % Recovery MS (or LCS)
 B_2 = % Recovery MSD (or LCD)

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

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10.0 WASTE MANAGEMENT AND POLLUTION CONTROL

All waste will be disposed of in accordance with Federal, State and Local regulations. Where reasonably feasible, technological changes have been implemented to minimize the potential for pollution of the environment. Employees will abide by this method and the policies in section 13 of the Corporate Safety Manual for "Waste Management and Pollution Prevention."

10.1 Waste Streams Produced by the Method

The following waste streams are produced when this method is carried out.

- Solid sample waste will be placed in the "Non-Hazardous" waste buckets.
- Acetonitrile/Methanol/Water mixture will be collected in approved containers and poured into the drum labeled "Flammable Solvent" waste using a funnel to reduce splashing.
- Expired and single component standards will be turned over to the EHSC or Waste Technician.

11.0 METHOD PERFORMANCE CRITERIA

Refer to Sections 1, 6, 7 and 8.

12.0 REFERENCES

Refer to Section 1.0

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13.0 ATTACHMENTS

Table 1. Retention Times

- Attachment 1. Example: Reporting Limits, MDLs, and Statistical Control Limits
- Attachment 2. Standards; Working Spike and Working Surrogate Concentrations
- Attachment 3. STL Corporate Procedure, P-T-001, 'Selection of Calibration Points'
- Attachment 4. Example: Analysis Run Log / Maintenance Log
- Attachment 5. Example: Data Review Checklist
- Attachment 6. Solid Phase Extraction (SPE) Method 3535

<u>Historical File:</u>	Revision 00: 09/09/94	Revision 05: 05/14/99
	Revision 01: 09/28/94	Revision 06: 04/18/00
	Revision 02: 10/24/96	Revision 07: 03/28/02
	Revision 03: 06/17/97	Revision 08: 02/17/04
	Revision 04: 12/30/98	Revision 09: 03/30/05

Revision 09; Reasons for Change:

- Annual Review
- Added 1.2.4 SPE section
- Section 2.0 added bullet for glassware
- 3.2 Added MeCl to list
- 4.1 added to supplies
- 5.2 Updated vendors
- 5.2.2 updated amounts used
- 6.0 removed statement about co-elutions
- 7.3.1.10 updated columns
- 7.3.3.5 refer to section 7.3.1.10 for sample instrument prep
- 7.3.3.6 added section for SPE method 3535
- Added Attachment 6, SPE Method 3535

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Table 1.

Example: Retention Times (minutes)

Compound	C18 Column	Phenyl Hexyl Column
HMX	4.48	17.50
RDX	6.71	12.28
1,3,5-Trinitrobenzene	9.15	11.54
1,3-Dinitrobenzene	11.07	10.14
Nitrobenzene	12.44	8.84
2,4,6-Trinitrotoluene	15.24	15.68
Tetryl	13.25	15.68
2,4-Dinitrotoluene	17.95	12.64
2-Amino-4,6-Dinitrotoluene	16.50	13.70
2,6-Dinitrotoluene	17.49	12.64
4-Amino-2,6-Dinitrotoluene	15.84	13.38
2-Nitrotoluene	20.96	10.83
4-Nitrotoluene	22.63	10.83
3-Nitrotoluene	24.34	11.12
1,2-Dinitrobenzene (surrogate)	9.55	12.06
Nitroglycerine**	14.60	*
PETN**	29.4	*

*To be determined

**Analyzed at a wavelength of 210 nm

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Attachment 1.

Example: Reporting Limits, MDLs, and Statistical Control Limits

Method Limit Report using report code 8330 Explosives by 8330 (HPLC) (8330)

Test Long Description	TMX	Units	Limits							
			MDL	RL	LCSLL	LCSUL	LCSRPD	SLL	SUL	
1,2-Dinitrobenzene (surr)		ug/L							66	144
1,3-Dinitrobenzene		ug/L	0.033	0.16	79	115	20			
1,3,5-Trinitrobenzene		ug/L	0.039	0.16	77	130	20			
2-Amino-4,6-Dinitrotoluene		ug/L	0.035	0.31	87	117	20			
2-Nitrotoluene		ug/L	0.082	0.31	77	113	20			
2,4,6-TNT		ug/L	0.036	0.16	75	145	20			
2,4-Dinitrotoluene		ug/L	0.032	0.31	81	116	20			
2,6-Dinitrotoluene		ug/L	0.071	0.31	85	115	20			
3-Nitrotoluene		ug/L	0.137	0.31	80	112	20			
4-Amino-2,6-Dinitrotoluene		ug/L	0.074	0.31	87	117	20			
4-Nitrotoluene		ug/L	0.082	0.31	76	113	20			
HMX		ug/L	0.122	0.31	82	116	20			
MNX		ug/L	0.064	0.16	74	117	20			
Nitrobenzene		ug/L	0.032	0.16	81	106	20			
RDX		ug/L	0.077	0.16	86	124	20			
Tetryl		ug/L	0.065	0.39	84	118	20			
1,2-Dinitrobenzene (surr)		ug/L							66	144
1,3-Dinitrobenzene		ug/L	0.033	0.16	79	115	20			
1,3,5-Trinitrobenzene		ug/L	0.039	0.16	77	130	20			
2-Amino-4,6-Dinitrotoluene		ug/L	0.035	0.31	87	117	20			
2-Nitrotoluene		ug/L	0.082	0.31	77	113	20			
2,4,6-TNT		ug/L	0.036	0.16	75	145	20			
2,4-Dinitrotoluene		ug/L	0.032	0.31	81	116	20			
2,6-Dinitrotoluene		ug/L	0.071	0.31	85	115	20			
3-Nitrotoluene		ug/L	0.137	0.31	80	112	20			
4-Amino-2,6-Dinitrotoluene		ug/L	0.074	0.31	87	117	20			
4-Nitrotoluene		ug/L	0.082	0.31	76	113	20			
HMX		ug/L	0.122	0.31	82	116	20			
MNX		ug/L	0.064	0.16	74	117	20			
Nitrobenzene		ug/L	0.032	0.16	81	106	20			
RDX		ug/L	0.077	0.16	86	124	20			
Tetryl		ug/L	0.065	0.39	84	118	20			

Method Limit Report using report code 8330

Explosives by 8330 (HPLC) (8330)

Test Long Description	TMX	Units	Limits							
			MDL	RL	LCSLL	LCSUL	LCSRPD	SLL	SUL	
1,2-Dinitrobenzene (surr)	Solid	ug/Kg							80	121
1,3-Dinitrobenzene	Solid	ug/Kg	5.0	100.	86	112	30			
1,3,5-Trinitrobenzene	Solid	ug/Kg	7.0	100.	82	125	30			
2-Amino-4,6-Dinitrotoluene	Solid	ug/Kg	6.4	200.	90	112	30			
2-Nitrotoluene	Solid	ug/Kg	17.0	200.	88	114	30			
2,4,6-TNT	Solid	ug/Kg	14.7	100.	67	152	30			
2,4-Dinitrotoluene	Solid	ug/Kg	7.9	100.	87	114	30			
2,6-Dinitrotoluene	Solid	ug/Kg	10.0	200.	90	112	30			
3-Nitrotoluene	Solid	ug/Kg	11.6	200.	89	115	30			
4-Amino-2,6-Dinitrotoluene	Solid	ug/Kg	85.6	200.	88	119	30			
4-Nitrotoluene	Solid	ug/Kg	30.2	200.	86	114	30			
HNX	Solid	ug/Kg	30.2	200.	86	117	30			
MNX	Solid	ug/Kg	23.0	100.	70	130	30			
Nitrobenzene	Solid	ug/Kg	7.7	100.	90	109	30			
RDX	Solid	ug/Kg	33.3	200.	90	115	30			
Tetryl	Solid	ug/Kg	117.	250.	60	130	30			

Method Limit Report using report code 8330

Explosives by 8330 (HPLC) (8330)

Test Long Description	TMX	Units	Limits							
			MDL	RL	LCSLL	LCSUL	LCSRPD	SLL	SUL	
1,2-Dinitrobenzene (surr)	TCLP	Le ug/L							62	159
1,3-Dinitrobenzene	TCLP	Le ug/L	0.16	0.16	79	115	20			
1,3,5-Trinitrobenzene	TCLP	Le ug/L	0.16	0.16	77	130	20			
2-Amino-4,6-Dinitrotoluene	TCLP	Le ug/L	0.31	0.31	87	117	20			
2-Nitrotoluene	TCLP	Le ug/L	0.31	0.31	77	113	20			
2,4,6-TNT	TCLP	Le ug/L	0.16	0.16	75	145	20			
2,4-Dinitrotoluene	TCLP	Le ug/L	0.31	0.31	77	123	20			
2,6-Dinitrotoluene	TCLP	Le ug/L	0.31	0.31	85	115	20			
3-Nitrotoluene	TCLP	Le ug/L	0.31	0.31	80	112	20			
4-Amino-2,6-Dinitrotoluene	TCLP	Le ug/L	0.31	0.31	87	117	20			
4-Nitrotoluene	TCLP	Le ug/L	0.31	0.31	76	113	20			
HMX	TCLP	Le ug/L	0.31	0.31	82	116	20			
NNX	TCLP	Le ug/L	0.16	0.16	74	117	20			
Nitrobenzene	TCLP	Le ug/L	0.16	0.16	81	106	20			
RDX	TCLP	Le ug/L	0.16	0.16	86	124	20			
Tetryl	TCLP	Le ug/L	0.39	0.39	84	118	20			

Method Limit Report using report code 8330

Explosives by 8330 (HPLC) (8330)

Test Long Description	TMX	Units	Limits							
			MDL	RL	LCSSL	LCSUL	LCSRPD	SLL	SUL	
1,2-Dinitrobenzene (surr)	SPLP	Le ug/L							62	159
1,3-Dinitrobenzene	SPLP	Le ug/L	0.16	0.16	79	115	20			
1,3,5-Trinitrobenzene	SPLP	Le ug/L	0.16	0.16	77	130	20			
2-Amino-4,6-Dinitrotoluene	SPLP	Le ug/L	0.31	0.31	87	117	20			
2-Nitrotoluene	SPLP	Le ug/L	0.31	0.31	77	113	20			
2,4,6-TNT	SPLP	Le ug/L	0.16	0.16	75	145	20			
2,4-Dinitrotoluene	SPLP	Le ug/L	0.31	0.31	77	123	20			
2,6-Dinitrotoluene	SPLP	Le ug/L	0.31	0.31	85	115	20			
3-Nitrotoluene	SPLP	Le ug/L	0.31	0.31	80	112	20			
4-Amino-2,6-Dinitrotoluene	SPLP	Le ug/L	0.31	0.31	87	117	20			
4-Nitrotoluene	SPLP	Le ug/L	0.31	0.31	76	113	20			
HMX	SPLP	Le ug/L	0.31	0.31	82	116	20			
MXN	SPLP	Le ug/L	0.16	0.16	74	117	20			
Nitrobenzene	SPLP	Le ug/L	0.16	0.16	81	106	20			
RDX	SPLP	Le ug/L	0.16	0.16	86	124	20			
Tetryl	SPLP	Le ug/L	0.39	0.39	84	118	20			

Method Limit Report using report code 8330

Explosives by 8330 (HPLC) (8330)

Test Long Description	TMX	Units	Limits							
			MDL	RL	LCSLL	LCSUL	LCSRPD	SLL	SUL	
1,2-Dinitrobenzene (surr)	Wipe	ug/Wipe							80	121
1,3-Dinitrobenzene	Wipe	ug/Wipe	1.0	1.0	86	112	30			
1,3,5-Trinitrobenzene	Wipe	ug/Wipe	1.0	1.0	82	125	30			
2-Amino-4,6-Dinitrotoluene	Wipe	ug/Wipe	2.0	2.0	90	112	30			
2-Nitrotoluene	Wipe	ug/Wipe	2.0	2.0	88	114	30			
2,4,6-TNT	Wipe	ug/Wipe	1.0	1.0	67	152	30			
2,4-Dinitrotoluene	Wipe	ug/Wipe	1.0	1.0	87	114	30			
2,6-Dinitrotoluene	Wipe	ug/Wipe	2.0	2.0	90	112	30			
3-Nitrotoluene	Wipe	ug/Wipe	2.0	2.0	89	115	30			
4-Amino-2,6-Dinitrotoluene	Wipe	ug/Wipe	2.0	2.0	88	119	30			
4-Nitrotoluene	Wipe	ug/Wipe	2.0	2.0	86	114	30			
HMX	Wipe	ug/Wipe	2.0	2.0	86	117	30			
MXM	Wipe	ug/Wipe	1.0	1.0	70	130	30			
Nitrobenzene	Wipe	ug/Wipe	1.0	1.0	90	109	30			
RDX	Wipe	ug/Wipe	2.0	2.0	90	115	30			
Tetryl	Wipe	ug/Wipe	2.5	2.5	60	130	30			

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Attachment 2.

Example: Standards, Working Spike, and Working Surrogate Concentrations

STL CHICAGO
NITROAROMATICS AND NITROAMINES BY HPLC
CALIBRATION STANDARDS (ug/mL)

COMPOUND	LEVEL 1	LEVEL 2	LEVEL 3	LEVEL 4	LEVEL 5	LEVEL 6
HMX	0.020	0.050	0.100	0.200	0.400	1.00
RDX	0.020	0.050	0.100	0.200	0.400	1.00
1,3,5-TNB	0.020	0.050	0.100	0.200	0.400	1.00
1,2-DNB	0.020	0.050	0.100	0.200	0.400	1.00
1,3-DNB	0.020	0.050	0.100	0.200	0.400	1.00
TETRYL	0.040	0.100	0.200	0.400	0.800	2.00
NITROBENZENE	0.020	0.050	0.100	0.200	0.400	1.00
2,4,6-TNT	0.020	0.050	0.100	0.200	0.400	1.00
2-AM-4,6-DNT	0.040	0.100	0.200	0.400	0.800	2.00
4-AM-2,6-DNT	0.040	0.100	0.200	0.400	0.800	2.00
2,4-DNT	0.020	0.050	0.100	0.200	0.400	1.00
2,6-DNT	0.040	0.100	0.200	0.400	0.800	2.00
2-Nitrotoluene	0.040	0.100	0.200	0.400	0.800	2.00
3-Nitrotoluene	0.040	0.100	0.200	0.400	0.800	2.00
4-Nitrotoluene	0.040	0.100	0.200	0.400	0.800	2.00
MX	0.020	0.050	0.099	0.198	0.396	0.99
PETN*	0.040	0.100	0.200	0.400	0.800	2.00
Nitroglycerine*	0.040	0.100	0.200	0.400	0.800	2.00

*Compounds are not part of routine 8330 list and would require a separate analytical run to report

SPIKE CONCENTRATIONS:

HMX	2.0 ug/mL
RDX	2.0
1,3,5-TNB	2.0
1,3-DNB	2.0
TETRYL	4.0
NITROBENZENE	2.0
2,4,6-TNT	2.0
2-AM-4,6-DNT	2.0
4-AM-2,6-DNT	4.0
2,4-DNT	2.0
2,6-DNT	4.0
2-NITROTOLUENE	4.0
3-NITROTOLUENE	4.0
4-NITROTOLUENE	4.0
MX	5.0
Petn	1.0
Nitroglycerin	1.0

SURROGATE:

	2.0 ug/mL
1,2-DNB	

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Attachment 3.

STL Corporate Procedure, P-T-001, 'Selection of Calibration Points'

Approvals and Signatures

Senior Vice President &
Chief Operating Officer:



Dr. Keith C. Wheatstone

Date: 10/6/2004

Vice President,
Client and Operations Services:



Dr. Charles W. Carter

Date: 10/8/2004

Technology Director:



Dr. Richard Burrows

Date: 9/8/2004

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1.0 PURPOSE

This policy describes Severn Trent Laboratories (STL) requirements for determination of the number of points, and removal of points from calibration curves.

2.0 SCOPE

Applies to all multi-level initial calibrations.

3.0 **POLICY**

- 3.1 If the number of data points required for an initial calibration is defined in the method, Quality Assurance (QA) plan, published report, or previously approved Standard Operating Procedure (SOP) that is what will be used.
- 3.2 In the cases not defined in Section 1, the number of data points will be determined by the technical director based on the Data Quality Objectives (DQOs) for precision and accuracy to be met by the method.

Examples:

- A. Need to analyze a new pesticide in water and a published method does not exist. The data will be used to screen samples by UV-HPLC at a waste site for further remediation, using DQOs that require precision/accuracy of $\pm 50\%$.

The Technical director selects 2 data points to represent the range of the expected concentration of pesticide and based on 4 Laboratory Control Samples (LCS), the recoveries ranged from 78-104%. Therefore, 2 data points are sufficient for initial calibration for this method.

NOTE: Calibration curves with less than 3 points should only be used after discussion with the client that the data quality objectives will be met.

- B. Same compound as above but being measured in laboratory for meeting regulatory limit of 0.05 mg/L in water. Precision and accuracy of $\pm 20\%$ required.

A five-point calibration is used, based on similar requirements in published methods with similar objectives and the high level of precision and accuracy required.

As noted above for methods where technical director selects the number of data points to meet DQOs for precision and accuracy, the 4 LCS used in the demonstration of capability will be used to assure those DQOs are met. The SOP will then be approved by the Quality Assurance (QA) Manager.

3.3 **Removal of Points from a Calibration Curve**

- 3.3.1 Removal or replacement of levels from the middle of a calibration (i.e., levels other than the highest or lowest) is not permitted unless an injection or instrument problem confined to that point can be clearly documented as described below. Removal of points for individual analytes from levels other than the highest and lowest is not permitted in any event.
- 3.3.2 If the analyst can document that a level is not valid because of an injection or instrument problem confined to that run, the level may be excluded if the curve still has sufficient levels, or the run may be repeated once only. The whole level (all compounds) must be

removed or replaced. The curve is evaluated with the level removed or replaced. If the curve still fails to meet criteria, then corrective action must be taken and the whole curve reanalyzed. Corrective action may include, but is not limited to, instrument maintenance and/or re-preparation of standards.

3.3.3 One of the following conditions must be satisfied to allow removal or replacement of a level:

- The data file is corrupted and unusable or the run is interrupted before completion.
- The analyst observes and documents a problem such as leaking of a purge vessel.
- For internal standard methods, the recovery of the internal standards is less than 70% or greater than 130% of the recovery in the other standards, or the amount of analyte recovered is less than 70% or greater than 130% of the expected values (indicating an improperly made up standard).
- For external standard methods, the unit response of the analyte is less than 70% or greater than 130% of the average unit response for the analyte in the other calibration standards (indicating an improperly prepared standard or bad injection).

3.3.4 When using autosamplers with discrete sample pathways for different samples (such as 16 port purge and trap autosamplers) the level to be replaced must be reanalyzed on the same port or that port must be excluded from sample analysis until corrective action is performed and verified by successful analysis of a continuing calibration standard on that port.

3.3.5 The reason for replacing the level **must** be documented in the run log. The fact that the curve passes criteria with the level removed is **not** alone sufficient evidence to document an injection or instrument problem confined to the level.

3.3.6 Removal of the highest or lowest levels is permitted, but the calibration range must be adjusted accordingly. If the lowest level is removed then the reporting limit is raised to be equivalent to the lowest level used in the calibration curve. In any event the number of levels remaining in the calibration must be at least that required by the method.

3.3.7 Removal of the highest or lowest point is permitted on a compound specific basis. This may be necessary when strongly responding and poorly responding analytes are included in the same standard mix at the same level. Each compound must have at least the minimum number of calibration levels required by the method.

4.0 **RESPONSIBILITIES**

All STL associates utilizing methods involving multi-point calibrations are required to follow this policy.

5.0 ATTACHMENTS

Not Applicable.

6.0 REVISION HISTORY

Revision 3: Updated by Richard Burrows, Technology Director; 9/8/2004.
Section 3.2: Amended 'NOTE'.
Section 3.3.3, bullet #4, text reworded for clarification.

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Attachment 4.

Example: Analysis Run Log / Maintenance Log

Extraction Date: _____
 Solvent: _____

**STL Chicago
 Explosives Extraction Record**

Page No.: _____
 Analyst Initials: _____

LabNet Batch No.: _____

Matrix: a. Water b. Soil
 c. Other: _____

Extraction Method: SW-846 8330 / 8332

	STL #	Sample ID	ICOC	Initial Volume (mLs) / Weight (g)	Final Volume (mLs)	Multipliers		
						Surr.	Spike	Split
1								
2								
3								
4								
5								
6								
7								
8								
9								
10								
11								
12								
13								
14								
15								
16								
17								
18								
19								
20								
21								
22								
23								
24								

Note: Soil samples are dried prior to the taking of a sample aliquot for extracting.

Comments: _____

Surrogate: _____ Volume: _____ Std. ID#: _____
 LCS/MS Solution: _____ Volume: _____ Std. ID#: _____
 LCS/MS Solution: _____ Volume: _____ Std. ID#: _____

Analyst Signature: _____ Date: _____

Reviewer Signature: _____ Date: _____

Analysis Custody Record

Sample(s)	Date/Time Out	Date/Time In	Analyst	Sample(s)	Date/Time Out	Date/Time In	Analyst

Routine/Informal Maintenance includes: Periodically check all pump seals for leaks.

Date of Maintenance: _____	Analyst: _____	Entry No.: _____
<input type="checkbox"/> Changed Purge Valve Frit	<input type="checkbox"/> Changed Column	
<input type="checkbox"/> Changed Pre-Column Filter	<input type="checkbox"/> Repair / Replaced Pump Seal(s)	
<input type="checkbox"/> Flushed Column with _____	<input type="checkbox"/> Other (explain)	
<input type="checkbox"/> Changed Lamp		
Explain/Actions Taken: _____		

Demonstration of Control:		
<input type="checkbox"/> CCV analyzed and in-control.	<input type="checkbox"/> Other (explain)	
<input type="checkbox"/> Samples Reanalyzed.		
Explain: _____		

Date of Maintenance: _____	Analyst: _____	Entry No.: _____
<input type="checkbox"/> Changed Purge Valve Frit	<input type="checkbox"/> Changed Column	
<input type="checkbox"/> Changed Pre-Column Filter	<input type="checkbox"/> Repair / Replaced Pump Seal(s)	
<input type="checkbox"/> Flushed Column with _____	<input type="checkbox"/> Other (explain)	
<input type="checkbox"/> Changed Lamp		
Explain/Actions Taken: _____		

Demonstration of Control:		
<input type="checkbox"/> CCV analyzed and in-control.	<input type="checkbox"/> Other (explain)	
<input type="checkbox"/> Samples Reanalyzed.		
Explain: _____		

Reviewer Signature: _____

Date: _____
CHI-22-18-018/A-01/01

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Attachment 5.

Example: Data Review Checklist

**STL Chicago
EGC / HPLC / GC VOA DATA REVIEW CHECKLIST**

Site Name: _____ Primary Reviewer: _____ Review Date: _____
 JOB Number: _____ Secondary Reviewer: _____ Review Date: _____
 No. of Samples/Matrix: a) _____ WATER b) _____ SOIL c) _____ TCLP / SPLP d) _____ Other (_____)
 Review Level: a) Full b) Cursory Method: a) GC VOA _____ b) GC _____ c) HPLC _____ d) Other (_____)
 Extr. Method: a) SW 3510 b) SW 3520 c) SW 3550 d) SW 3580 e) SW 3541 f) SW 5030 /5035 g) CLP _____ h) Other (_____)

TASK	CAR's _____ Std. Traceability _____	PRI REV	SEC REV	COMMENTS
LAB CIRON:	1) Matches Raw Data (Form 4, 8)			
	2) Samples which were Re-Analyzed or Re-Extracted were Re-Logged into LabNet			
	3) Sample Hold Times were Met			
	4) Proper Prep Links were created			
PROJ. REQ.MET:	1) List of Compounds			
	2) Sample Detection Limits Met			
	3) Method Blank Detection Limits Met			
	LabNet Batch Status Report Displays Data at RVWD Status Incomplete JOB Status Report reveals no Outstanding Data			
FORM 1:	1) Matches Quant Report			
	2) Matches LabNet Report			
FORM 2: Surrogate Recoveries Within Limits	Statistical Limits _____ Method Limits _____ Project Limits _____ (S-F10 used to Clone By Project)			
FORM 3: MS/MSD Recoveries Acceptable	Statistical Limits _____ Method Limits _____ Project Limits _____ (S-F10 used to Clone By Project)			
FORM 3: BS Recoveries Acceptable	Statistical Limits _____ Method Limits _____ Project Limits _____ (S-F10 used to Clone By Project)			
	Initial Calibration Criteria Met			
	Daily Calibration (CCV) Criteria Met			
Form 10:	1) Retention Window (RT) Criteria Met			
	2) Concentrations Correct			
	Correct Usage of FLAGS			
RAW DATA:	1) Raw Data Verified/Complete			
	2) Raw Data Matches Forms			
NARRATIVE:	1) Holding Times			
	2) Method References			
	3) % Recoveries / RPD's			
	4) Analytical Difficulties/Typos			

Client: _____ JOB#: _____ Test: _____

Reviewer(1): _____ Date: _____

Reviewer(2): _____ Date: _____

COC/Deliverable

Reviewer 1 Reviewer 2

____ ____ Chain-of-custody has been checked to ensure that the proper analyte list is reported.
____ ____ All special project requirements are met, i.e., control limits, forms, etc...

Comments: _____

Updating Results in Labnet

____ ____ Reagent codes are correct.
____ ____ Batch test results match quant. reports.
____ ____ Batch cloned for project limits.
____ ____ Proper prep links were created (including TCLP link).
____ ____ Each required target compound displays "0" when data is reported.
____ ____ Client information has been checked for correct list, reporting limits, special requirements.
____ ____ Job Notes (CTRL F12) for the job have been reviewed.

Form 1/Associated Data

____ ____ Observe chromatograms, check for obvious chromatography errors.
____ ____ Check form information for correctness (JOB #, reporting limit, dilution factor, matrix, flags, etc...)
____ ____ Check values reported. In an "E" is present, there must be another dilution; if a value is reported, review the chromatogram; if a "J" is reported, review the chromatogram; if an "I" is reported, there must be another dilution run.
____ ____ Check that all JOB #'s on the chromatograms match quant report and data on quant report is correct.
____ ____ Check that confirmation chromatograms agree with quantitation chromatograms.
____ ____ All method blanks are clean.
____ ____ All OPC file information is present and correct (matrix, final volume, %sol present for soils).
____ ____ Before and after chromatograms are present for all Manual integrations and are dated/initialed by analyst.

Comments: _____

Form 2/Associated Data

____ ____ Requested surrogates were used.
____ ____ All surrogate recoveries are within control limits. If not, proper corrective actions have been taken and are documented. [Samples with surrogates outside of control limits may not require re-analysis but must be evaluated, flagged, and discussed in the case narrative. If not specified by the client, both (if applicable) surrogate recoveries must be within statistical control limits. Surrogates associated with the method blank and blank spikes should be within control.]
____ ____ Confirm all samples and QC for that batch are represented.
____ ____ Confirm correct matrix.
____ ____ Correct surrogate concentration used for calculating % Recovery. Verify calculations.

Comments: _____

Form 3/Associated Data

Reviewer 1 Reviewer 2

- ____ ____ Blank spike and blank spike duplicate have the proper batch number, and the proper matrix
- ____ ____ All blank spike/blank spike duplicate recoveries and RPDs are within control limits. If not, proper corrective actions have been taken and documented. Flag outliers for the Case Narrative and provide documentation.
- ____ ____ Confirm that the matrix spike and matrix spike duplicate data correlates with the unspiked analysis.
- ____ ____ All matrix spike/matrix spike duplicate recoveries and RPDs are within control limits. If not, proper corrective actions have been taken and documented. Flag outliers for the Case Narrative.
- ____ ____ Confirm correct matrix and sample ID.
- ____ ____ Correct spike concentration used for calculating % Recovery. Verify calculations.

Comments: _____

Form 4/Associated Data

- ____ ____ Check all header information, extraction date, analysis date and time, instrument and column IDs.
- ____ ____ Confirm all samples associated with the blank are present.
- ____ ____ Confirm all blank summaries are present for all samples/matrices in the data package.
- ____ ____ Confirm proper matrix and extraction method.
- ____ ____ Sulfur cleanup performed (not applicable to some methods)

Comments: _____

Endrin/DDT Breakdown

- ____ ____ All breakdown criteria is met (<15% for DDT and/or Endrin) for 608 and 8081A only.

Comments: _____

Initial Calibration/Associated Data (Form 6)

- ____ ____ Method SW-846: All % RSDs are <20% (5-pt. minimum)
- ____ ____ Method 40CFR: All %RSDs are <10% (3-pt. minimum).
- ____ ____ Recalculate a few %RSDs.
- ____ ____ All header information is correct (times, dates).
- ____ ____ Second Source Verification (SSV) is in control (85%-115%)

Comments: _____

Continuing Calibration Verification/Associated Data (Form 7) - Standards that are outside the control limits must be approved by the Section Manager prior to the reporting of any data.

- ____ ____ All header information is correct. Dates and times of initial standards and continuing standards are correct.
- ____ ____ All retention times are within their windows, those that are outside are marked for Case Narrative.
- ____ ____ Ave. CFs match from ICAL (Form 6) Ave CFs.
- ____ ____ All %Ds are within control limits. If not, proper corrective actions have been taken and documented.
- ____ ____ All Form 7's associated with the samples and QC are present.

Comments: _____

Form 10/Associated Data

Reviewer Reviewer
1 2

- ____ Confirm that all analytes which are reported as being detected on the Form 1 are present.
- ____ Confirm that all retention times of analytes reported as positive hits are within their retention time window, and if they aren't, why the analyst reported them must be documented in the comment section.
- ____ The GC columns are marked Y/N for quantitation/confirmation (applicable to 8330 only).

Comments: _____

Additional Data

- ____ All necessary runlogs are present and contain the proper sequences.
- ____ All necessary extraction records are present.
- ____ All required holding times were met for samples dilutions and QC.
- ____ Chronology of data is correct.
- ____ Dates and times of analysis are correct.
- ____ Verify samples are quantitated using the proper ICAL.

Comments: _____

____ RG LabChron/Report Review Initial/Date _____

Project: _____ Job #: _____ Method: _____

Reviewer (1): _____ Date: _____

Reviewer (2): _____ Date: _____

Sublist: _____
Instruments (Primary/Confirmation): _____
Cleanups: _____
CAR (Y/N): _____

Target Review

Reviewer 1 Reviewer 2

- _____ _____ Chromatography is acceptable.
- _____ _____ Chromatograms are scaled properly.
- _____ _____ All peaks are labeled properly.
- _____ _____ All initial calibrations are within control limits ($\leq 20\%$ RSD; Correlation Coefficient ≤ 0.995).
- _____ _____ Second Source Verification is in control (85% - 115%).
- _____ _____ All continuing calibrations are within control limits ($\pm 15\%$ difference).
- _____ _____ All retention times are within their windows.
- _____ _____ All method blanks are clean.
- _____ _____ Calculations verified.
- _____ _____ Verify samples are quantified using the proper ICAL.
- _____ _____ Before and after chromatograms produced for all manual integrations.

Comments: _____

Updating Results in LabNet

- _____ _____ Reagent codes are correct.
- _____ _____ Batch test results match quant reports.
- _____ _____ Batch cloned for project limits.
- _____ _____ Proper prep links were created (including TCLP link).
- _____ _____ Each required target compound displays "0" when data is reported.
- _____ _____ Client information has been checked for correct list, reporting limits, special requirements.
- _____ _____ Job notes (CTRL F12) for the job have been reviewed.

Comments: _____

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Attachment 6.

Solid Phase Extraction (SPE) Method 3535

EPA Method 3535
Explosives from aqueous samples

I. Materials

Strata SPE: SDBL 500 mg/ 6mL

Part Number: 8B-S014-HCH

Conditioning Solvent: Methylene Chloride (DCM), Acetonitrile, DI Water

Wash Solvent: DI Water

Extraction Solvent: Acetonitrile

II. Solid Phase Extraction Method

A. Condition: Slowly pass through the cartridge (3-6ml /min)

1. Rinse with DCM (about 18mL) with no vacuum.
2. 1 column volume of Acetonitrile (about 6mL)
3. 1 column volume of DI water (about 6mL)

B. Load Sample:

For processing large volume samples greater than 6mL; refer to Phenomenex Strata publication entitled "*Processing Large Volume Samples with Strata SPE Cartridges*" located on next page.

C. Wash:

Wash the sample with three column volumes (about 18mL) of DI Water. Dry thoroughly for 30-60 seconds to insure removal of aqueous wash.

D. Elute: (about 2 minutes)

1. Rinse the sample bottle with 5mL of Acetonitrile:MeOH 80:20 and decant into the syringe barrel reservoir.
2. Engage vacuum until the Acetonitrile just begins to drip through the sorbent, then stop and allow remaining volume of Acetonitrile to drain through by gravity into the collection tube. Apply vacuum to get the final drops.

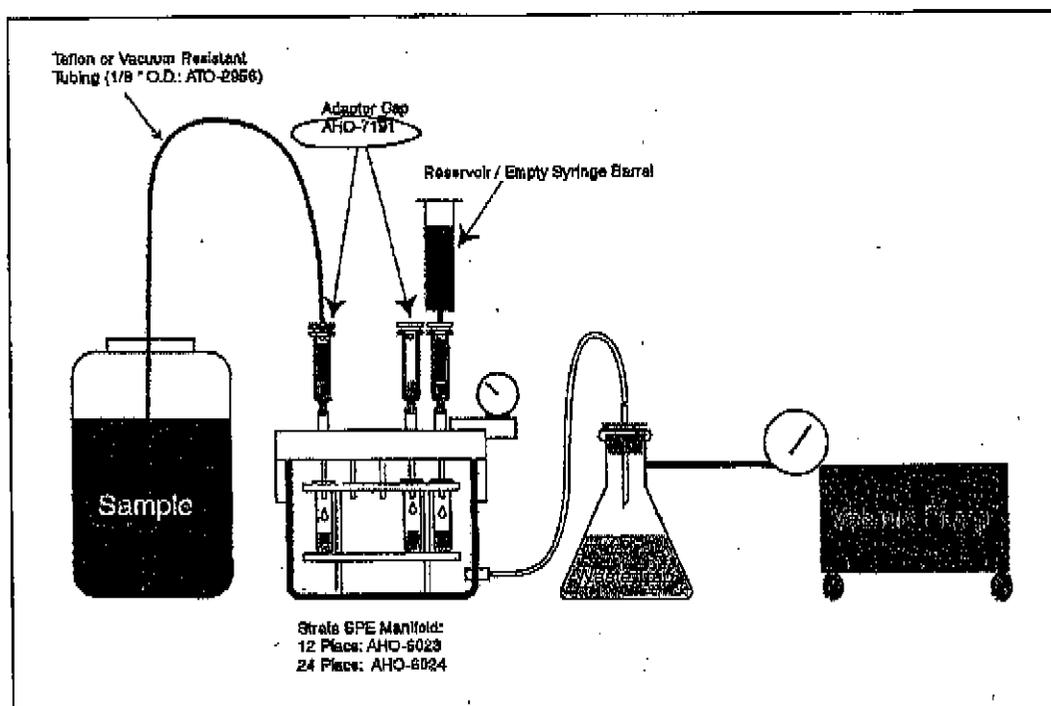
See Section 7.3.1.10 for preparation of sample for analysis.

Processing Large Volume Samples with Strata SPE Cartridges

A major benefit of SPE is its ability to concentrate trace amounts of analyte from very dilute, large volume samples. Unfortunately these samples are often several times larger than the reservoir capacity of the SPE cartridge. Several strategies have proven most useful in dealing with these large volume samples.

1. Apply the sample to the cartridge in multiple aliquots.
2. Attach a separate, large-volume reservoir to the top of the SPE cartridge using an adapter cap.
3. Connect the SPE cartridge directly to the sample reservoir using a piece of tubing. The applied vacuum creates a suction that draws the sample from the sample reservoir through the tubing, into the SPE cartridge and through the sorbent bed. Vacuum tight connection between the tubing and the SPE cartridge adapter is critical.

Reminder: A cartridges' retention capacity is strictly related to the mass of sorbent packed into the cartridge and is unaffected by the so-called "reservoir" volume capacity of the cartridge. For example, a 500 mg / 3 ml tube has the exact same retention capacity as a 500 mg / 6 ml, or a 500 mg / 12 ml.

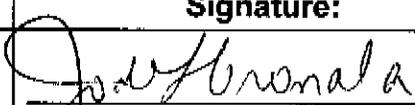
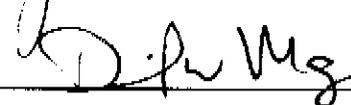
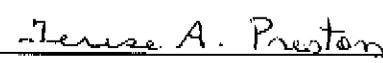


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**TITLE: Metals Analysis
 Mercury by EPA Methods 245.1/245.5; SW-846 7470A/7471A;
 and U.S. EPA CLP Document No. ILM04.0**

Updated by: George O. Klee Senior Analyst, Mercury	Signature: 	Date: 3/22/05
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Approved by: Jodi L. Gromala Section Manager, Metals Dept.	Signature: 	Date: 3/22/05
David W. Mazur Env. Health & Safety Coord.		3/22/05
Terese A. Preston Quality Manager		3/22/05

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1.0 SCOPE / APPLICATION

This Standard Operating Procedure (SOP) outlines the digestion and analytical procedure for the determination of the mercury concentration in aqueous and non-aqueous media. This SOP was written using EPA 600/4-79-020 Methods 245.1 and 245.5; SW-846, 3rd Edition, Methods 7470A/7471A; and U.S. EPA CLP Document No. ILM04.0 as references.

On occasion, clients request slight modifications to this SOP. These modifications are addressed on a case-by-case basis with the range of accuracy (i.e., MDLs, linearity check or PT sample) verified prior to implementation. Any modifications would be written into a Quality Assurance Plan (QAP), authorized via laboratory signature approval, and mentioned in the data package's case narrative.

1.1 Method Sensitivity

1.1.1 Method Detection Limits

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 Appendix B of 40 CFR 136, "Guidelines Establishing Test Procedures for the Analysis of Pollutants". 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.

1.1.2 Instrument Detection Limits

Instrument Detection Limits (IDLs) are performed quarterly for each element by the metals laboratory for each instrument as specified in CLP. These limits are used to gauge instrument sensitivity and when routinely evaluated, instrument performance without the introduction of method variance can be determined.

1.1.3 Reporting Limits

Reporting Limits are defined as the lowest concentration of an analyte determined by a given method in a given matrix that the laboratory feels can be reported with acceptable quantitative error or client requirements, values specified by the EPA methods or other project and client requirements. The laboratory maintains reporting limits that are higher than the MDL. Wherever possible, reporting is limited to values approximately 3-5x the respective MDL to ensure confidence in the value reported. Client specific requests for reporting to the IDL or MDL are special circumstances not to be confused with the previous statement.

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Matrix	Reporting Limit¹	CRDL²
Water	0.2 ug/L	0.2 ug/L
Soil	0.017 mg/kg	0.1 mg/kg

¹ Reporting Limit is used for EPA Method 245.1 and SW-846 7470A/7471A. Reporting Limits may vary depending on sample volume/size, dilution factors, and changes in the MDL.

² CRDL (Contract Required Detection Limit) is used for U.S. EPA CLP ILM04.0.

1.1.4 Definitions

Refer to Section 3.0 of the Laboratory's Quality Manual (LQM, Revision 03).

1.2 Summary of Method

This flameless cold vapor AA procedure is a physical method based on the absorption of radiation at 253.7 nm by mercury vapor. The mercury is reduced to the elemental state and swept from solution and passed through a cell of a double beam AA. Absorbance is a function of mercury concentration.

2.0 INTERFERENCES

- Chloride, sulfide and certain volatile organic materials.

3.0 SAFETY

Employees must abide by the policies and procedures in the Corporate Safety Manual, Radiation Safety Manual and this document.

3.1 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.

3.2 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.

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Material (1)	Hazards	Exposure Limit (2)	Signs and symptoms of exposure
Mercury (1,000 ppm in Reagent)	Oxidizer Corrosive Poison	0.1 Mg/M3 Ceiling (Mercury Compounds)	Extremely toxic. Causes irritation to the respiratory tract. Causes irritation. Symptoms include redness and pain. May cause burns. May cause sensitization. Can be absorbed through the skin with symptoms to parallel ingestion. May affect the central nervous system. Causes irritation and burns to eyes. Symptoms include redness, pain, and blurred vision; may cause serious and permanent eye damage.
Sulfuric Acid	Corrosive Oxidizer Dehydrator Poison	1 Mg/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.
Nitric Acid	Corrosive Oxidizer Poison	2 ppm-TWA 4 ppm-STEL	Nitric acid is extremely hazardous; it is corrosive, reactive, an oxidizer, and a poison. Inhalation of vapors can cause breathing difficulties and lead to pneumonia and pulmonary edema, which may be fatal. Other symptoms may include coughing, choking, and irritation of the nose, throat, and respiratory tract. Can cause redness, pain, and severe skin burns. Concentrated solutions cause deep ulcers and stain skin a yellow or yellow-brown color. Vapors are irritating and may cause damage to the eyes. Contact may cause severe burns and permanent eye damage.
Hydrochloric Acid	Corrosive Poison	5 ppm-Ceiling	Inhalation of vapors can cause coughing, choking, inflammation of the nose, throat, and upper respiratory tract, and in severe cases, pulmonary edema, circulatory failure, and death. Can cause redness, pain, and severe skin burns. Vapors are irritating and may cause damage to the eyes. Contact may cause severe burns and permanent eye damage.
Potassium Permanganate	Oxidizer	5 Mg/M3 for Mn Compds.	Causes irritation to the respiratory tract. Symptoms may include coughing, shortness of breath. Dry crystals and concentrated solutions are caustic causing redness, pain, severe burns, brown stains in the contact area and possible hardening of outer skin layer. Diluted solutions are only mildly irritating to the skin. Eye contact with crystals (dusts) and concentrated solutions causes severe irritation, redness, and blurred vision and can cause severe damage, possibly permanent.
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.
<p>1 – Always add acid to water to prevent violent reactions. 2 – Exposure limit refers to the OSHA regulatory exposure limit.</p>			

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4.0 EQUIPMENT AND SUPPLIES

- 2 – Leeman Labs Model PS200 Automated Mercury Analyzer
- Class A volumetric glassware
- Eppendorf pipettes

5.0 REAGENTS AND STANDARDS

5.1 Reagents

5.1.1 Miscellaneous Reagents

- Hydrochloric Acid [HCl], Concentrated
- Nitric Acid [HNO₃], Concentrated
- Sulfuric Acid [H₂SO₄], Concentrated
- Deionized (DI) Water, Type II

5.1.2 Sodium Chloride-Hydroxylamine Hydrochloride Solution

Dissolve 240 g of sodium chloride and 240 g of hydroxylamine hydrochloride in sufficient DI water to make 2-liters of solution.

- Life of Reagent: 1 Year
- Storage Requirements: None

5.1.3 Stannous Chloride Solution

Dissolve 100 g of stannous chloride in 10% hydrochloric acid to make 1-liter of solution.

- Life of Reagent: 1 Month
- Storage Requirements: None

5.1.4 Potassium Permanganate, 5%

Dissolve 175 g of potassium permanganate into 3.5-liters of DI water.

- Life of Reagent: 1 Year
- Storage Requirements: None

5.1.5 Potassium Persulfate, 5%

Dissolve 175 g of potassium persulfate into 3,500 mLs of DI water.

- Life of Reagent: 1 Year
- Storage Requirements: None

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5.2 Standards All standards are prepared in Class A volumetric flasks.

5.2.1 Standard Stock Solution I; 1,000 ppm

A 1,000 ppm concentrated mercury standard is purchased from an outside supplier.

- Life of Standard: 1 Year
- Storage Requirements: None

5.2.2 Working Standard Solution I; 100 ppb

To a 1.0 L volumetric flask filled with ~800 mLs DI water, transfer 100 uLs of Stock Solution I to the flask using a 100 uL Eppendorf pipette. Add 2.5 mLs conc. nitric acid as a preservative. Dilute to volume with DI Water. Invert and mix to insure complete mixture.

*For use in spiking Matrix Spikes, CRAs & the Standard Curve.

- Life of Standard: 24 Hours
- Storage Requirements: None

5.2.2.1 Working Standard Solution IA; 25 ppb

To a 100 mL volumetric flask filled with ~80 mLs DI water, transfer 25 uLs of Working Standard Solution I (Item 5.2.2) to the flask using an Eppendorf pipette. Dilute to volume with DI Water. Invert and mix to insure complete mixture.

*For use in spiking Matrix Spikes, CRAs & the Standard Curve in the Hot Block Digester

- Life of Standard: 24 Hours
- Storage Requirements: None

5.2.3 Standard Stock Solution II; 1,000 ppm

Purchased from an outside supplier as a 1,000 ppm solution and is from an alternate source than that of Standard Stock Solution I (Rgt. 5.2.1).

- Life of Standard: 1 Year
- Storage Requirements: None

5.2.4 Working Standard Solution II; 200 ppb

To a 1.0 L volumetric flask filled with ~800 mLs DI water, add 2.5 mLs concentrated nitric acid (as a preservative) and 200 uLs of Standard Stock Solution II to the flask (using a 200 uL Eppendorf pipette). Dilute to volume with DI water and invert several times to mix.

*For use in spiking the ICV/CCV and LCS.

- Life of Standard: 24 Hours
- Storage Requirements: None

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5.2.4.1 Working Standard Solution IIA; 50 ppb

To a 100 mL volumetric flask filled with ~80 mLs DI water, add 25 uLs of Working Standard Solution II (Item 5.2.4) to the flask using an Eppendorf pipette. Dilute to volume with DI Water. Invert and mix to insure complete mixture.

*For use in spiking the ICV/CCV and LCS in the Hot Block Digester

- Life of Standard: 24 Hours
- Storage Requirements: None

5.2.5 Working Standards for Mercury in Water

Standard (ug/L)	mLs of Working Soln. I or IA	Final Volume (mLs) Water Bath	Final Volume (mLs) Hot Block
Blank	0.0	100	25
0.2	0.2	100	25
0.5	0.5	100	25
1.0	1.0	100	25
3.0	3.0	100	25
5.0	5.0	100	25
CRA (0.2 ug/L)	0.2	100	25
Matrix Spike (1.0 ug/L)	1.0	100	25

Standard (ug/L)	mLs of Working Soln. II or IIA	Final Volume (mLs) Water Bath	Final Volume (mLs) Hot Block
Init. Cal. Verif. (ICV) (2.0 ug/L)	1.0	100	25
Cont. Cal. Verif. (CCV) (1.0 ug/L)	0.5	100	25
Lab Control Sample (LCS) (2.0 ug/L)	1.0	100	25

CLP Standard (ug/L)	mLs of Working Soln. II or IIA	Final Volume (mLs) Water Bath	Final Volume (mLs) Hot Block
Init. Cal. Verif (ICV) (2.0 ug/L)	1.0	100	25
Cont. Cal. Verif. (CCV) (1.0 ug/L)	0.5	100	25

NOTE: ILM04.0 requires the ICV and CCV to be at different levels.

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5.2.6 Working Standards for Mercury in Soil (Block Digestion)

Standard (ug/L)	mLs of Working Soln. I	Final Volume (mLs) Hot Block
Blank	0.00	50
0.2	0.10	50
0.5	0.25	50
1.0	0.50	50
3.0	1.50	50
5.0	2.50	50
CRA (0.2 ug/L)	0.10	50
Matrix Spike (1.0 ug/L)	0.50	50

Standard (ug/L)	mLs of Working Soln. II	Final Volume (mLs) Hot Block
Init. Cal. Verif. (ICV) (2.0 ug/L)	0.50	50
Cont. Cal. Verif. (CCV) (1.0 ug/L)	0.25	50
Lab Control Sample (LCS) (2.0 ug/L)	0.50	50

CLP Standard (ug/L)	mLs of Working Soln. II	Final Volume (mLs) Hot Block
Init. Cal. Verif. (ICV) (2.0 ug/L)	0.50	50
Cont. Cal. Verif. (CCV) (1.0 ug/L)	0.25	50

NOTE: ILM04.0 requires the ICV and CCV to be at different levels.

6.0 CALIBRATION (NON-DAILY)

All calibration procedures are performed on a daily basis. Refer to Section 7.4 for details.

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7.0 PROCEDURE

7.1 Quality Control Checks

The following Quality Control samples are performed with each batch of samples. Refer to Section 8.0 for additional details.

QC Sample	Frequency ¹	Control Limits
Method Blank (MB)	1 in 20 samples	<ul style="list-style-type: none"> • < Reporting Limit (EPA / SW-846) • < CRDL (CLP)
LCS	1 in 20 samples	<ul style="list-style-type: none"> • 80-120% Recovery (EPA 245.5 / SW-846 / CLP) • 85-115% Recovery (EPA 245.1)
Matrix Duplicate (MD) ²	1 in 20 samples	<ul style="list-style-type: none"> • 20 RPD unless the sample conc. is <5x RL, then ± RL. (EPA / SW-846) • 20 RPD unless the sample conc. is <5x CRDL, then ± CRDL. (CLP)
Matrix Spike (MS) MS Duplicate (MSD) ²	1 in 20 samples	<ul style="list-style-type: none"> • 75 – 125% Recovery unless the sample concentration > spike level by 4x (EPA 245.5 / SW-846 / CLP) • 70 – 130% Recovery (EPA 245.1) • > 50% Recovery; if <50% Recovery, Method of Standard Additions (MSA) is required (TCLP)

¹ Drinking waters by EPA 245.1; and CLP analyses are analyzed at a frequency of 1 in 10 samples.

² The sample selection for MS/MSD or MS/MD, where appropriate, are rotated among client samples so that various matrix problems may be noted and/or addressed. MD's are performed only when requested by the client/project/contract. The MS/MSD are the routinely performed matrix QC indicators.

7.2 Sample Preservation 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 request. Listed below are the holding times and preservations for the referenced programs.

Program	Preservation ¹	Holding Time ²
SDWA	pH < 2, Cool 4 ± 2°C	28 days VTS ³
CWA	pH < 2, Cool 4 ± 2°C	28 days VTS
RCRA	pH < 2, Cool 4 ± 2°C	28 days VTS
CLP	pH < 2, Cool 4 ± 2°C	26 days VTSR ⁴

¹ Waters are preserved with nitric acid at pH <2; Soils are preserved at Cool 4 ± 2°C.

² Holding times include digestion and analysis.

³ VTS: Verified Time of Sampling.

⁴ VTSR: Verified Time of Sample Receipt.

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7.3 Sample Preparation

7.3.1 Mercury Water Digestion Procedure - EPA Method 245.1 / CLP ILM04.0

Item	Full Scale (Water Bath)	Hot Block
Sample Volume	100 mLs	25 mLs
Reaction Vessel	BOD Bottle, 300 mLs	Sample Vials, 50 mLs
Sulfuric Acid (conc.)	5 mLs	1.25 mLs
Nitric Acid (conc.)	2.5 mLs	0.625 mLs
Potassium Permanganate, 5% Sol. (W/V)	15 mLs	3.75 mLs
Potassium Persulfate, 5% Sol. (W/V)	8 mLs	2 mLs
Preparation	2 hrs. @ 90 – 95°C, Cool	2 hrs. @ 90 - 95°C, Cool
Hydroxylamine Addition	6 mLs	1.5 mLs
Total Volume	136.5 mLs	34.125 mLs

NOTE: The sample should remain purple for 15 minutes after adding the potassium permanganate. If the sample does not maintain the purple color, a second addition of potassium permanganate is added to all samples of the batch to maintain the purple color.

Proceed with the Stannous Chloride addition.

7.3.2 Mercury Water Digestion Procedure - SW-846 Method 7470A

Item	Full Scale (Water Bath)	Hot Block
Sample Volume	100 mLs	25 mLs
Reaction Vessel	BOD Bottle, 300 mLs	Sample Vials, 50 mLs
Sulfuric Acid (conc.)	5 mLs	1.25 mLs
Nitric Acid (conc.)	2.5 mLs	0.625 mLs
Potassium Permanganate, 5% Sol. (W/V)	15 mLs	3.75 mLs
Potassium Persulfate, 5% Sol. (W/V)	8 mLs	2 mLs
Preparation	2 hrs. @ 90-95°C, Cool	2 hrs. @ 90 - 95°C, Cool
Hydroxylamine Addition	6 mLs	1.5 mLs
Total Volume	136.5 mLs	34.125 mLs

NOTE: The sample should remain purple for 15 minutes after adding the potassium permanganate. If the sample does not maintain the purple color, a second addition of potassium permanganate is added to all samples of the batch to maintain the purple color.

Proceed with the Stannous Chloride addition.

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7.3.3 Mercury Soil Digestion Procedure - SW-846 Method 7471A

NOTE: Three aliquots of soils (~0.2 g) are combined and digested as one sample.

Item	Full Scale (Water Bath)	Hot Block
Sample Weight	~ 0.6 – 0.7 grams	~ 0.6 – 0.7 grams
Reaction Vessel	BOD Bottle, 300 mLs	Digestion Vessel
DI Water, Type II	5 mLs	2.5 mLs
Aqua Regia [3:1 HCl (conc.) to HNO ₃ conc.]	5 mLs	2.5 mLs
Preparation	2 min. @ 90-95°C, Cool	2 min. @ 90-95°C, Cool
DI Water, Type II	50 mLs	25 mLs
Potassium Permanganate, 5% Sol. (W/V)	15 mLs	7.5 mLs
Preparation	30 min. @90-95°C, Cool	30 min. @90-95°C, Cool
Hydroxylamine Addition	6 mLs	3 mLs
Total Volume	Dilute to 100 mLs	50 mLs

NOTE: The sample should remain purple for 15 minutes after adding the potassium permanganate. If the sample does not maintain the purple color, a second addition of potassium permanganate is added to all samples of the batch to maintain the purple color.

Proceed with the Stannous Chloride addition.

7.3.4 Mercury Soil Digestion Procedure - EPA Method 245.5 / CLP ILM04.0

Item	Full Scale (Water Bath)
Sample weight	0.2 - 0.3 grams
Reaction Vessel	BOD bottle, 300 mLs
Sulfuric Acid (conc.)	5 mLs
Nitric Acid (conc.)	2.5 mLs
Preparation	2 min. @ 90 -95°C, Cool
DI Water, Type II	50 mLs
Potassium Permanganate, 5% Sol. (W/V)	15 mLs
Potassium Persulfate, 5% Sol. (W/V)	8 mLs
Preparation	30 min. @ 90 - 95°, Cool
Hydroxylamine Addition	6 mLs
Total Volume	Dilute to 100 mLs

NOTE: The sample should remain purple for 15 minutes after adding the potassium permanganate. If the sample does not maintain the purple color, a second addition of potassium permanganate is added to all samples of the batch to maintain the purple color.

Proceed with the Stannous Chloride addition.

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7.4 Calibration / Standardization

Before the instrument is used as a measurement device, the instrument response to known reference materials must be determined. All sample measurements must be made within this linear range of the instrument.

Standard	Frequency	Control Limit
Calibration Curve	Initially	Corr. Coeff. > 0.995
ICV	After the Calibration Curve	<ul style="list-style-type: none"> • 90 – 110% Recovery (EPA 245.5 / SW-846 / CLP) • 95 – 105% Recovery (EPA 245.1)
ICB	After the ICV	<ul style="list-style-type: none"> • < Reporting Limit (EPA / SW-846) • < CRDL (CLP)
CRA	After ICB	• No established limits.
CCV	Every 10 readings; end of each run	<ul style="list-style-type: none"> • 90 – 110% Recovery (EPA / CLP) • 80 – 120% Recovery (SW-846) (Note: The LabNet (LIMS) QC criteria code for the CCV is set at the default limit of 90-110% for all methods.)
CCB	Every 10 readings; End of each run	<ul style="list-style-type: none"> • < Reporting Limit (EPA / SW-846) • < CRDL (CLP)

7.4.1 Calibrating the System

The instrument must be calibrated before samples are analyzed.

To perform a calibration, go to the WinHg Runner and select the 'STANDARD' tab. Select the standards to be used by clicking on the S1, S2, S3, S4, S5 and S6 buttons. To set the number of replicates, click on the Rep1 button. Click on the 'Std Auto' button and the calibration will begin.

Reviewing the Calibration:

Go to the Database application by clicking the 'DB' button on the toolbar. Click on the 'Cal Curve' tab. Calibration data can be accepted by clicking the 'Accept' button.

7.4.2 Check Standards

This option allows for the verification that the calibration has not drifted. The Check Standards are placed in the tray with the samples to allow for AutoRun:

Positions 1-5: ICV, ICB, CRA, MB, LCS
Positions 11-12: CCV, CCB

To check standard concentrations:

- From the Main Menu, select CALIBRATION and then select CHECK STANDARDS. The check standard screen will appear.

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- Type 1 for a check standard blank. Enter, in units specified on the standards page, the range of acceptance.
- Type 2 for check standards cup 2. Type the concentration and Enter. Type the percent acceptance and Enter.
- Repeat this for up to seven check standards.
- From Main Menu, select AUTOSAMPLES, then select SETUP and then check Enter the C1 frequency (e.g., 5/EPA protocol)
- Halt: Enter Y if the instrument should halt after an unacceptable check standard. Enter N for an alert only. Macros can be written to automatically recalibrate and rerun samples if check standards fall outside specifications.

7.5 Preventive Maintenance

The instrument requires some routine daily maintenance as well as some scheduled and non-scheduled periodic maintenance. All maintenance will be recorded in the instruments maintenance logbook. The following maintenance schedule lists the various maintenance procedures and when they should be performed. Each of these procedures is described in the following sections.

7.5.1 Maintenance Schedule

Equipment	Schedule
Drying Tube	Must be changed daily.
Pump Tubing	Weekly, or as needed.
Lamp	Replace as needed (avg. 4 mos. - 1 yr.).
Optical Cell	Clean as needed (typically monthly).
Liquid Gas Separator	Replace every 1-3 yrs., as needed.
Internal Tubing	Should not require replacement under normal circumstances.

7.5.2 Packing and Changing the Drying Tube

Under normal use, the drying tube must be changed each morning before analyzing samples. (The drying tube is located on the front panel on the left side of the instrument) Several tubes can be packed at one time and stored in an airtight container for a ready supply.

To pack a tube, plug one end with quartz wool, pour in magnesium perchlorate to fill tube, and plug the other end with quartz wool.

To change a tube, slightly loosen the nuts that hold the tube in at either end and slide the used tube out of the fittings. Slide a fresh tube into the fittings and tighten the fittings with your fingers to make a gas-tight seal.

To clean a tube, remove the quartz wool and the magnesium perchlorate. Either dispose of as a solid waste or dissolve in water and dispose of as a liquid waste. Clean the tube with ordinary laboratory glassware cleaner and dry thoroughly.

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7.5.3 Replacing and Conditioning Pump Tubing

Pump Tubing should be replaced weekly or when it shows signs of wear. There are four pump tubes: two for drainage, one for sample, and one for reductant. Each tube is fed through a pump cassette which then clamps onto the pump head. Slide a tube through the plastic clips at the bottom of a cassette until the plastic tab is secure. Hold the tube taut, slide the loaded cassette onto the pump head, and lock the clamp up. Repeat for the remaining tubes, then connect the tubes ends.

For optimal performance, run DI water through new tubes for one hour to exercise them before using them for running samples. To do this, select INSTRUMENT from the Main Menu and then select OPERATION.

The INSTRUMENT:OPERATION screen will appear. Set the Pump Rate flow to the standard rate for 5 mL/min (Type R and M and 5 Enter). Wait for one hour and then connect the tubing to the appropriate fluids.

NOTE: This procedure only needs to be done once, when the tubes are new and unused.

7.5.4 Replacing the Lamp

The mercury lamp has a life of about 2000 hours, between four months and a year of use. The lamp needs to be replaced if the relative absorbance of a standard has changed significantly while the optical cell is clean. If the lamp is suspected, it is faster to replace the lamp and recalibrate than to clean the optical cell.

NOTE: Before installation, clean the new lamp quartz with methanol and wipe it dry. Do not get finger prints on the lamp and do not face the printing on the lamp toward the optical cell.

- Turn off the lamp (press the blue button on the front of the instrument).
- Remove the front panel of the instrument (lift up and out).
- Remove the optical assembly.
- Remove the two screws on the lamp housing and take off the lamp cover.
- Twist the lamp 90° and slide it straight out.
- Insert the new lamp and rotate it 90° in the reverse direction to secure it in place. Make sure that the lettering on the lamp will be facing to the left of the instrument when it has been reinstalled. If it is not, remove the lamp and reinsert it correctly.
- Replace the optical assembly.

7.5.5 Cleaning the Optical Cell

If the relative absorbance of standards differs significantly from that of previous calibrations, the optical cell (located inside the front panel) may be dirty and must be cleaned:

- Turn the lamp and the power off and remove the front panel by lifting it up and out.

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- Remove the optics clamps, disconnect the detector, and rotate and lift out the assembly. Disconnect the gas lines.
- Remove the six screws holding the lamp spacer and the detector spacer onto the optical cell.
- Inspect the two ends with the lenses. If the external surface of the lenses appear to be the only contaminant, then clean. To clean use methanol. Install if no other cleaning is necessary.
- Disassemble the optical cell (using the allen wrench provided on the inside of the front cover) by removing (in order) the screws, lens, and gasket at each end.
- Carefully clean the inside of the cell with laboratory glassware cleaner, taking care not to scratch the inside surfaces. Rinse thoroughly, first with water and then with DI water. Dry the cell in the oven (free of contaminants) for one hour at approximately 40 - 50°C.
- Clean the lenses with laboratory glassware cleaner and rinse thoroughly with hot tap water. Flush lightly with methanol and dry by air or vacuum oven (maximum 50°C).
- Replace the gaskets (this is recommended although not required unless the gasket shows signs of wear) and reassemble the optical cell. Cleaning of the gaskets should only be done with DI water.

7.5.6 Replacing the Liquid Gas Separator

- The liquid gas separator (transparent block on the chemical panel) should only need to be replaced once every one to three years, depending on the amount of use it receives.
- To replace the separator, shut off the gas and liquid flow and flush the tubing with DI Water for safety purposes. Disconnect the four lines and remove the two screws. Remove the unit from the system, screw on a new one, reconnect the four lines, and turn the gas and liquid flow back on.

7.5.7 Replacing Internal Tubing

Internal gas and Teflon tubes should last indefinitely and should not need to be replaced. Periodically inspect all tubing for restrictions or blockages. If tubing should need to be replaced, do so one piece at time to avoid any confusion while making connections.

7.6 Sample Analysis

7.6.1 Preparing the System

The following procedures must be performed each morning before warming up the system:

- Change the drying tube. Refer to maintenance, Section 7.5 for instructions.

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- Release the clamps and check the pump tubing for wear. Under normal use, the tubes will need to be replaced once a week. To replace the tubing, refer to maintenance, Section 7.5 for instructions.
- Check the reductant volume and refresh, if needed.
- Clean the rinse tank using standard lab cleaning practices, add fresh rinse.
- If the lamp has been off then turn on the lamp power and allow the lamp to warm up for at least 45 minutes.
- Start up the system.

7.6.2 Start-up Procedures

The start-up routine used will depend on the current state of the system. If it is in Overnite mode, use the Warmstart macro (15 minute warm-up). If the system has been completely powered down, run the Coldstart macro instead (3 hour warm-up).

7.6.3 Software Setup

- In order to run samples, enter all necessary information regarding the protocol, sample ID's, calibration values, and autosampler parameters into the software. This information is entered into a series of screens which are accessed from the Main Menu. (Display the Main Menu at any time by pressing the F1 key)
- Perform each of the following steps in sequence to set up the software. When these steps have been completed, the instrument will be able to run samples automatically.

NOTE: The steps below comprise the basic daily software setup sequence. The software also contains numerous advanced functions. Refer to the PS Series Reference Guide for a detailed description of the many other keys and functions available for use with this system.

7.6.4 Name the Protocol

From the 'Runner', click the toolbar button labeled 'DB' on the 'Database' command button on the 'Main' tab. From the 'Database', select 'File' from the pull down menu. Select 'New Protocol'. Enter a name in the 'Protocol Name' dialog box that appears. Select one of the check boxes in the 'Protocol type' group. This ensures the correct analytical conditions for the concentration range desired.

7.6.5 Name the Folder

Data set name is added from the WinHg Runner. Click on 'File' and select 'New Dataset'. 'Add New Batch' will pop up when you hit 'Enter'. New batch can also be added from the sample tab on the Runner.

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7.6.6 Verify Values and Integration Times

Check to make sure that all values and integration times are correct for running the samples:

- From the Main Menu, select PROTOCOL, then select SET Values. The Set Values screen appears.
- For normal operation, enter the following values (as illustrated below):

Number of Integrations:	1
Uptake time	10
Weight	N
Dilution	N
Percent Recovery	N

- Press F1 to return to the Main Menu.

7.6.7 Enter values for on/off's, times, and gains

All the information entered on the PROTOCOL tab on the WinHg database.

7.6.8 Enter the Calibration Standard Concentrations:

From the WinHg Database, select the 'Line Info' tab. Calibration standards and concentrations are entered here.

7.6.9 Reset the Calibration Intensity Data

The calibration can be reset from 2 different places. One option is from the WinHg Runner. Click the standards tab, then click on the 'New Cal Reset' button and 'OK'. Another option is from the WinHg Database. Click on the 'Cal Curve' tab and click the 4 buttons in the reset box.

7.6.10 Set the Autosampler Rinse Time

Pump rinse times and rates are setup in the protocol through the WinHg Database tab. Rinse is set at 50 seconds and the uptake is set at 10 seconds.

7.6.11 Set up the Racks

To launch the Rack Edit application, either click the 'Rack Editor' button the Runner 'Main' tab or click the Autosampler rack icon on the toolbar.

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7.6.12 Define start-to finish sample sequence

Click on the 'Sample' tab on the WinHg Runner. In the 'Autosampler run' group, click on the combo box for Station 1 and select the rack to be run. Using the spin boxes, set the starting and ending cups. Click on 'Run Auto' to begin analysis.

7.6.13 Running Samples

NOTE: Optimum Concentration Range = 0.2 ug/L - 5 ug/L

Once the rack and ranges are put in on the 'Autosample run' box on the sample tab, click the 'Run Auto' button to begin analysis.

7.6.14 Shutdown Procedures

There are two methods for shutting down the instrument. Under routine operation, when the system is used daily, only the lamp is shut off (system power remains on) and the Overnite routine is used to put the unit into a "sleep mode". If the system is to be completely turned off and not used for an extended period of time, or if it is to be shipped or moved, use the long-term Shutdown routine instead. These two methods are described below. For weekends or periods of "sleep" greater than 24 hours it is recommended to turn off the mercury lamp using the blue button.

NOTE: Before shutting down the instrument, the system must have beeped to indicate completion of the last procedure, and the word "Idle" should appear in the "State field in the top left of the displayed screen.

7.6.15 Short-Term (Overnite Macro)

Return to the WinHg Runner and click on the 'Control' tab. In the gas group, click on the 'Off' button, then in the 'Pump Group', click the 'Stand by' button. This will allow the instrument to autcycle to keep tubing clear of clogs.

7.6.16 Long-Term (Shutdown Macro)

For long-term shutdown, exit the software by selecting 'File' and then 'Exit' on the WinHg Runner pull-down menu.

7.7 Documentation

7.7.1 Instrument Run-Log

The analysis of samples and standards is documented within the instrument run log (Attachment 1) and supported by the instrument print-out. The runlog must be completed for each days analysis.

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7.7.2 Traceability of Standards

Custom made and single element stock standard solution which are traceable to NIST or EPA are purchased. On receipt, each standard is recorded in LabNet (LIMS) and is issued a unique source ID#. The manufacturer, lot #, date received, expiration date, date of verification and the initials of the recording analyst are entered into the system.

7.7.3 Data Review

Analytical data goes through a 200% review cycle. The analyst and a trained data reviewer perform the reviews according to the criteria established on the data review checklist (Attachment 2). Upon the first 100% review, the checklist is initialed and dated as reviewed. The package, with its review sheet, comments and any corrective action reports (CARs) is submitted to the supervisor, section manager, or peer reviewer for a second review. Once again, the checklist is initialed and dated by the second reviewer. The completed checklist remains on file with the original data.

8.0 QUALITY CONTROL

8.1 QC Summary

The laboratory generates annual statistically generated control limits and these can be used when requested by the client, contract or QAPP. These limits are based on the successive analysis of LCSs.

8.1.1 Calibration curve must be composed of a minimum of a blank and 5-standards. A least square fit linear calibration curve must have a minimum correlation coefficient of 0.995, which must be reported with the raw data.

8.1.2 ICV and ICB will be performed at the beginning of an analytical sequence. The ICV must not vary more than - a) 10% for EPA 245.5, SW-846 & CLP methods or b) 5% for EPA 245.1 method from its true value and must be prepared from a different source than the calibration curve standards.

Calibration verification will be performed with a CCV and CCB every 10 samples and at the end of the analysis. The CCV must not vary more than a) 20% for SW-846 methods or b) 10% for EPA & CLP methods from its true value and must be prepared from a different source than the calibration curve standards. (Note: The LabNet (LIMS) QC criteria code for the CCV is set at the default limit of 90-110% for all methods.) The CCB must be < Reporting Limit (EPA / SW-846) and < CRDL (CLP).

8.1.3 Dilute samples if they are more concentrated than the highest standard or if they fall on the plateau of a calibration curve (dilute with a digested blank containing all reagents, or repeat the analysis using a smaller sample volume).

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8.1.4 A minimum of one MB must be analyzed per sample batch to determine if contamination has occurred

8.1.5 An LCS will be included with each batch of 10 (drinking waters and EPA 245.1) or 20 (EPA 245.5, SW-846 or CLP) samples. The analyzed result must not vary more than 20% from the true value. For EPA Method 245.1, the LCS acceptance limits are 85-115%.

8.1.6 Matrix spike and duplicate samples are analyzed with each batch of 10 (drinking waters and EPA 245.1) or 20 (EPA 245.5, SW-846 or CLP) samples.

8.2 Corrective Actions

When an out-of-control situation occurs, the analysts must use his/her best analytical judgment and available resources to determine the corrective action to be taken. The out-of-control situation may be caused by more than one variable. The analyst should seek the assistance of his/her immediate supervisor, section manager, QA personnel, or other experienced staff if he/she is uncertain of the cause of the out-of-control situation. The test must not be resumed until the source of the problem and an in-control status is attained. All samples associated with the out-of-control situation should be reanalyzed. Out-of-control data must never be released without approval of the supervisor, section manager, project manager, QA personnel or the laboratory manager.

Listed below are steps that must be taken when an out-of-control situation occurs:

- demonstrate that all the problems creating the out-of-control situation were addressed
- document the problem and the action which was taken to correct the problem on a CAR
- document on the CAR that an in-control has been achieved and receive approval (signature) of the supervisor, section manager, QA personnel, or the laboratory manager prior to the release of any analytical data associated with the problem.

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QC Indicator	Suggested Corrective Actions
Calibration Curve	<ul style="list-style-type: none"> • reanalyze the standard curve; • prepare a new stock and/or working standards; • check the reagents/solutions and prepare fresh if necessary.
Initial Calibration Verification (ICV)	<ul style="list-style-type: none"> • repeat ICV to verify proper preparation; • prepare new ICV from original stock; • recalibrate with a new standard curve; • prepare new stock and/or working standards; • check reagents/solutions and prepare fresh if necessary.
Initial Calibration Blank (ICB)	<ul style="list-style-type: none"> • prepare new ICB to verify proper preparation; • verify that the instrument base-line is stable and perform necessary maintenance, cleaning, etc.. to achieve stability; • determine the source of contamination by the process of elimination, carryover from a previous analysis or reagent contamination and correct the problem; • check reagents/solutions and prepare fresh if necessary; • correct for any contamination and reanalyze ICB and any associated samples.
Laboratory Control Sample (LCS)	<p><u>If LCS is low:</u></p> <ul style="list-style-type: none"> • reanalyze LCS to verify that it is out-of-control; • determine the source of error within the preparation procedure, repeat the sample set, write a CAR. <p><u>If the LCS is high:</u></p> <ul style="list-style-type: none"> • reanalyze LCS to verify that it is out-of-control; • determine the source of error within the preparation procedure, repeat the sample set; • determine if the high result is due to contamination; • check for contamination of reagents, LCS stock solution, or preparation area; • correct for contamination, reanalyze.
Method Blank (MB)	<ul style="list-style-type: none"> • reanalyze the MB to verify that it is beyond the reporting limit; • determine the source of contamination; • determine if the high result is due to contamination; • check for contamination of reagents or preparation area; • correct for contamination, reanalyze set; • in the extreme case where all samples in the set are at least 10X > the MB, reanalysis will not be required. However, a CAR and approval will be necessary.
Matrix Duplicate (MD)	<ul style="list-style-type: none"> • the sample must be reprocessed and reanalyzed; • if the reanalysis results in data that is still out of the control limit, then the sample will be ticked with a "N"; • regardless of the outcome of the reanalysis, a CAR will be written and approved by the Section Manager.
Matrix Spike (MS)	<ul style="list-style-type: none"> • the sample must be reprocessed and reanalyzed; • if the reanalysis results in data that is still out of the control limit, then the sample will be ticked with a "N"; • regardless of the outcome of the reanalysis, a CAR will be written and approved by the Section Manager.

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QC Indicator	Suggested Corrective Actions
Continuing Calibration Verification (CCV)	<ul style="list-style-type: none"> • repeat CCV to verify proper preparation; • prepare new CCV from original stock; • check for instrument base-line drift or a change in one or more of the reagents; • check reagents/solutions and prepare fresh if necessary; • recalibrate with a new standard curve and repeat all samples since the previous in control CCV; • never dispose of any samples until you are sure that all QC, especially the CCV, are within the control limits.
Continuing Calibration Blank (CCB)	<ul style="list-style-type: none"> • prepare new CCB to verify proper preparation; • verify that the instrument base-line is stable and/or perform necessary maintenance, cleaning, etc.. to achieve stability; • determine the source of contamination by the process of elimination, carryover from a previous analysis or reagent contamination and correct the problem, • check reagents/solutions and prepare fresh if necessary; • correct for any contamination and reanalyze CCB and any associated samples; • never dispose of any samples until you are sure that all QC, especially the CCB are within the control limits.
Summary	<ul style="list-style-type: none"> • If any of the ICV, ICB, CCV or CCB results are out-of-control for any element, the instrument is restandardized and the samples associated with the out-of-control elements are reanalyzed. • If the MB or LCS are out-of-control for any element, the samples are redigested. An exception is if the sample concentrations are $\geq 10X$ the MB contamination, the results are reported as is. • If any of the MD or MS results are out-of-control, a reanalysis is performed if there is sufficient sample. If there is insufficient sample, or the reanalysis is still out-of-control, the client is notified of the poor results via a case narrative that is sent with the data report. • CARs are available for out-of-control MB, LCS, MS and MD problems. These forms are completed by the analyst performing the analysis. The forms are then reviewed and signed by the supervisor or section manager. The signed forms are kept on file within the laboratory department and are used to prepare the case narrative (if applicable).

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9.0 DATA ANALYSIS AND CALCULATIONS

Perform a linear regression or quadratic fit analysis of the calibration standard results. Compare sample results to the curve to determine the mercury concentration.

9.1 Water $\text{ug/L Hg} = \text{ug/L} \times \text{Dilution Factor}$ (Where L = Final digestate volume)

9.2 Soil $\text{mg/kg Hg} = \frac{(\text{ug/L}) \times L \times \text{Dilution Factor}}{\text{wt(g)} \times \text{fraction solids}}$

(Where L = Final digestate volume)

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

9.3 Accuracy $\%R = \frac{(A_T - A_O)}{A_F} \times 100$

Where:

A_T = Total amount recovered in fortified sample

A_O = Amount recovered in unfortified sample

A_F = Amount added to sample

9.4 Precision $\text{RPD} = \frac{|C_1 - C_2|}{(C_1 + C_2)/2} \times 100$

Where:

C_1 = First measurement value

C_2 = Second measurement value

10.0 WASTE MANAGEMENT AND POLLUTION PREVENTION

All waste will be disposed of in accordance with Federal, State and Local regulations. Where reasonably feasible, technological changes have been implemented to minimize the potential for pollution of the environment. Employees will abide by this method and the policies in section 13 of the Corporate Safety Manual for "Waste Management and Pollution Prevention."

10.1 Waste Streams Produced by this Method

- Waste from this process goes into the "Corrosive Wastewater" wastestream.
- Single component standards should not be mixed into the waste streams unless approved by the Waste Coordinator. All standards with Hazardous constituents will be turned in to the waste technician for disposal.

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11.0 METHOD PERFORMANCE CRITERIA

Refer to Sections 1, 6, 7 and 8.

12.0 REFERENCES

Refer to Section 1.0.

13.0 ATTACHMENTS

Attachment 1: Example: Instrument Maintenance Log and LabNet Forms

Attachment 2: Example: Data Review Checklist

<u>Historical File:</u>	Revision 00: 10/03/90	Revision 06: 03/16/00
	Revision 01: 08/09/91	Revision 07: 05/23/01
	Revision 02: 03/19/93	Revision 08: 09/06/02
	Revision 03: 10/18/95	Revision 09: 03/29/04
	Revision 04: 01/24/97	Revision 10: 03/22/05
	Revision 05: 03/31/99	

Reasons for Change, Revision 10:

- Annual Review –
- Maintenance Log added as attachment
- Soil RL changed to reflect hot block final volume of 50 mLs.

U:\QC\SOP\ME\UME-245.1.DOC

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Attachment 1:

Example: Instrument Maintenance Log and LabNet Forms

STL Chicago
PS200 Leeman Mercury Analyzer – HG3
Instrument Maintenance Log

Page No. _____

	Date/Initials						
<i>Daily Maintenance:</i>							
Change Drying Tube							
Clean and Refill Rinse Tank							
Clean Sample Tip							
Check/Clean Optical Cell (Clean when reference intensity is <300,000)							

<i>Weekly Maintenance:</i>							
Change Pump Tubing							
Change Activated Carbon and Quartz Wool							

Comments: _____

****Any Maintenance/Repair/Part Replacement performed that is not listed above must be documented in the Comments sections****

Reviewer Signature: _____

Date: _____

SW846 Digestion (Hg)		Status.....: RVWD	User Name.....: gok	Location Code...: 57222
Method Code...: HGSWD		Batch Date...: 03/14/05	QC Code.....:	Equipment Code.: HG3
Batch Code...: 143878		Batch Time...: 1442	Calc Code.....: PREPFO	Import Code.....:
SAMPLE: Grp Pos	Sample ID	Dilution	TEST POS Date / Time	D I G E S T I O N
1 1	__s_s1_M04KSTK001__		3/10/05 1630	0
1 2	__s_s2__		3/10/05 1630	0
1 3	__s_s3__		3/10/05 1630	0
1 4	__s_s4__		3/10/05 1630	0
1 5	__s_s5__		3/10/05 1630	0
1 6	__s_s6__		3/10/05 1630	0
1 7	__s_MB__		3/10/05 1630	0
1 8	__s_LCS_M04LSTK010_7		3/10/05 1630	0
1 9	234659_2_s__		3/10/05 1630	0
1 10	234670_1_s__		3/10/05 1630	0
1 11	234670_2_s__		3/10/05 1630	0
1 12	234783_1_s__		3/10/05 1630	0
1 13	234783_2_s__		3/10/05 1630	0
1 14	234822_1_s__		3/10/05 1630	0
1 15	234822_13_s__		3/10/05 1630	0
1 16	234833_1_s__		3/10/05 1630	0
1 17	234842_1_s__		3/10/05 1630	0
1 18	234842_1_s_MD__17		3/10/05 1630	0
1 19	234842_1_s_MS_M04KSTK001_17		3/10/05 1630	0
1 20	234842_1_s_MSD_M04KSTK001_17		3/10/05 1630	0
1 21	234842_2_s__		3/10/05 1630	0
1 22	234842_3_s__		3/10/05 1630	0
1 23	234842_4_s__		3/10/05 1630	0
1 24	234842_5_s__		3/10/05 1630	0
1 25	234842_6_s__		3/10/05 1630	0
1 26	234855_4_0__		3/10/05 1630	0
1 27	234855_8_s__		3/10/05 1630	0

SW846 Digestion (Hg)

Report Date: 3/22/05 11:41

Method Code...: HGSWD	Batch Date...: 03/14/05	QC Code.....:	Equipment Code.: HG3
Batch Code...: 143878	Batch Time...: 1442	Calc Code.....: PREPFO	Import Code.....:
Status.....: RVWD	User Name...: gok	Location Code...: 57222	

BATCH:	Item	Description	Description Information
	1	Analyst:	GEORGE KLEE JR.
	2	Reviewer:	
	3	Equipment ID:	1173
	4	Wavelength: 253.7nm Cell length: 20.5cm	
	5	Water Bath Temp: Initial(Limits 90C-95C)	
	6	Water Bath Temp: Final	
	7	Block Digestor Temp: Initial (90C-95C)	955
	8	Thermometer ID: Correction Factor:	1173 +2
	9	Repipettor Volume Check:	OK
	10	HNO3 Lot#:	A22035
	11	HCL Lot#:	5587 A06A22
	12	H2SO4 Lot#:	5557 A2A0A08
	13	KMnO4 Lot#:	7056 x49655
	14	SnCl2-H2O Lot#:	A40600
	15	NH2OH-HCL Lot#:	Y28599
	16	K2S2O8 Lot#:	T44H13
	17	NaCl Lot#:	43234351
	18	Date Sample Prepped:	03/10/05
	19	Prep Time In:	1630
	20	Prep Time Out:	1700

SAMPLE:	Grp	Pos	Sample ID	Dilution	DIGHG Text	MLI mL	MLF mL	WEIGHT g	PREPF N/A
	1	1	__S_S1_M04KSTK001__		Complete		50	0.60	83.3333
	1	2	__S_S2__		Complete		50	0.60	83.3333
	1	3	__S_S3__		Complete		50	0.60	83.3333
	1	4	__S_S4__		Complete		50	0.60	83.3333
	1	5	__S_S5__		Complete		50	0.60	83.3333
	1	6	__S_S6__		Complete		50	0.60	83.3333
	1	7	__S_MB__		Complete		50	0.60	83.3333
	1	8	__S_LCS_M04LSTK010_7		Complete		50	0.60	83.3333
	1	9	234659_2_s__		Complete		50	0.60	83.3333
	1	10	234670_1_s__		Complete		50	0.60	83.3333
	1	11	234670_2_s__		Complete		50	0.60	83.3333

SW846 Digestion (Hg)

Report Date: 3/22/05 11:41

Method Code...: HGSWD	Batch Date...: 03/14/05	QC Code.....:	Equipment Code.: HG3
Batch Code...: 143878	Batch Time...: 1442	Calc Code.....: PREPFO	Import Code.....:
Status.....: RVWD	User Name....: gok	Location Code..: 57222	

SAMPLE:	Grp	Pos	Sample ID	Dilution	DIGHG Text	MLI mL	MLF mL	WEIGHT g	PREPF N/A
1	12		234783_1_s		Complete		50	0.60	83.3333
1	13		234783_2_s		Complete		50	0.60	83.3333
1	14		234822_1_s		Complete		50	0.60	83.3333
1	15		234822_13_s		Complete		50	0.60	83.3333
1	16		234833_1_s		Complete		50	0.60	83.3333
1	17		234842_1_s		Complete		50	0.60	83.3333
1	18		234842_1_s_MD_17		Complete		50	0.60	83.3333
1	19		234842_1_s_MS_M04KSTK001_17		Complete		50	0.60	83.3333
1	20		234842_1_s_MSD_M04KSTK001_17		Complete		50	0.60	83.3333
1	21		234842_2_s		Complete		50	0.60	83.3333
1	22		234842_3_s		Complete		50	0.60	83.3333
1	23		234842_4_s		Complete		50	0.60	83.3333
1	24		234842_5_s		Complete		50	0.60	83.3333
1	25		234842_6_s		Complete		50	0.60	83.3333
1	26		234855_4_0		Complete		50	0.60	83.3333
1	27		234855_8_s		Complete		50	0.60	83.3333

SAMPLE:	Grp	Pos	Sample ID	Dilution	DLFAC N/A	VOL mL			
1	1		_s_s1_M04KSTK001_		1.0000	50			
1	2		_s_s2		1.0000	50			
1	3		_s_s3		1.0000	50			
1	4		_s_s4		1.0000	50			
1	5		_s_s5		1.0000	50			
1	6		_s_s6		1.0000	50			
1	7		_s_MB		1.0000	50			
1	8		_s_LCS_M04LSTK010_7		1.0000	50			
1	9		234659_2_s		1.0000	50			
1	10		234670_1_s		1.0000	50			
1	11		234670_2_s		1.0000	50			
1	12		234783_1_s		1.0000	50			
1	13		234783_2_s		1.0000	50			
1	14		234822_1_s		1.0000	50			
1	15		234822_13_s		1.0000	50			

SWB46 Digestion (Hg)

Report Date: 3/22/05 11:41

SAMPLE:		Grp Pos	Sample ID	Dilution	DLFAC N/A	VOL mL			
Method Code..:	HGSWD	Batch Date...:	03/14/05	QC Code.....:		Equipment Code.:	HG3		
Batch Code...:	143878	Batch Time...:	1442	Calc Code.....:	PREPFO	Import Code.....:			
Status.....:	RVWD	User Name....:	gok	Location Code..:	57222				
1	16		234833_1_s__		1.0000	50			
1	17		234842_1_s__		1.0000	50			
1	18		234842_1_s_MD__17		1.0000	50			
1	19		234842_1_s_MS_M04KSTK001_17		1.0000	50			
1	20		234842_1_s_MSD_M04KSTK001_17		1.0000	50			
1	21		234842_2_s__		1.0000	50			
1	22		234842_3_s__		1.0000	50			
1	23		234842_4_s__		1.0000	50			
1	24		234842_5_s__		1.0000	50			
1	25		234842_6_s__		1.0000	50			
1	26		234855_4_0__		1.0000	50			
1	27		234855_8_s__		1.0000	50			

Mercury (CVAA) Solids		Status.....: RVMD	User Name.....: gok	Location Code...: 57222
Method Code...: 7471		Batch Date....: 03/14/05	QC Code.....: METHG	Equipment Code.: HG3
Batch Code....: 143880		Batch Time....: 1456	Calc Code.....:	Import Code.....:
SAMPLE: Grp Pos	Sample ID	Dilution	TEST CODE Date / Time	H G
1 1	__S_S1_M04KSTK001_		3/11/05 1320	
1 2	___S_S2___		3/11/05 1322	
1 3	__S_S3__		3/11/05 1324	
1 4	__S_S4__		3/11/05 1326	
1 5	__S_S5__		3/11/05 1328	
1 6	__S_S6__		3/11/05 1331	
1 7	___ICV_M04LSTK010_		3/11/05 1333	0
1 8	___ICB__		3/11/05 1336	0
1 9	___CRA_M04KSTK001_		3/11/05 1338	0
1 10	__S_MB__		3/11/05 1340	0
1 11	__S_LCS_M04LSTK010_10		3/11/05 1342	0
1 12	234659_2_s___		3/11/05 1344	0
1 13	234670_1_s___		3/11/05 1347	0
1 14	234670_2_s___		3/11/05 1350	0
1 15	234783_1_s___		3/11/05 1353	0
1 16	234783_2_s___		3/11/05 1356	0
1 17	___CCV_M04LSTK010_		3/11/05 1403	0
1 18	___CCB__		3/11/05 1406	0
1 19	234822_1_s___		3/11/05 1408	0
1 20	234822_13_s___		3/11/05 1411	0
1 21	234833_1_s___		3/11/05 1414	0
1 22	234842_1_s___		3/11/05 1416	0
1 23	234842_1_s_MD__22		3/11/05 1419	0
1 24	234842_1_s_MS_M04KSTK001_22		3/11/05 1421	N
1 25	234842_1_s_MSD_M04KSTK001_22		3/11/05 1424	N
1 26	234842_2_s___		3/11/05 1427	0
1 27	234842_3_s___		3/11/05 1429	0
1 28	234842_4_s___		3/11/05 1432	0

3/22/05 11:38

Mercury (CVAA) Solids		Status.....: RVWD	User Name.....: gok	Location Code...: 57222
Method Code...: 7471		Batch Date....: 03/14/05	QC Code.....: METHG	Equipment Code.: HG3
Batch Code....: 143880		Batch Time....: 1456	Calc Code.....:	Import Code.....:
SAMPLE: Grp Pos	Sample ID	Dilution	TEST POS Date / Time	H G
1 57	__CCV_M04LSTK010__		3/11/05 1552	0
1 58	__CCB__		3/11/05 1554	0
1 59	234873_13_s__		3/11/05 1556	0
1 60	234873_14_s__		3/11/05 1559	0
1 61	234873_15_s__		3/11/05 1601	0
1 62	234873_16_s__		3/11/05 1603	0
1 63	234873_17_s__		3/11/05 1605	0
1 64	234873_18_s__		3/11/05 1607	0
1 65	234873_19_s__		3/11/05 1609	0
1 66	__CCV_M04LSTK010__		3/11/05 1612	0
1 67	__CCB__		3/11/05 1614	0

Mercury (CVAA) Solids

Report Date: 3/22/05 11:38

Method Code...: 7471		Batch Date...: 03/14/05		QC Code.....: METHG		Equipment Code.: HG3						
Batch Code...: 143880		Batch Time...: 1456		Calc Code.....:		Import Code.....:						
Status.....: RVWD		User Name....: gok		Location Code...: 57222								
Grp	Smp	Sample ID	Pos	Test	Result	Known	Original	Alternate	QC Res	F	QC Res	F
1	7	___ICV_M04LSTK010_	1	HG	2.034483	1000000			102			
1	8	___ICB_	1	HG	0.0866							
1	9	___CRA_M04KSTK001_	1	HG	0.284826	1000			142			
1	10	___S_MB_	1	HG	0.113689							
1	11	___S_LCS_M04LSTK010_10	1	HG	2.007891	1000000	0.113689		100			
1	17	___CCV_M04LSTK010_	1	HG	0.863191	1000000			86			
1	18	___CCB_	1	HG	-0.0336							
1	23	234842_1_S_MD_22	1	HG	0.833016		0.69038		18.7			
1	24	234842_1_S_MS_M04KSTK001_22	1	HG	1.182848	1000	0.69038		49	N		
1	25	234842_1_S_MSD_M04KSTK001_22	1	HG	1.246989	1000	0.69038	1.182848	56	N	13.3	
1	30	___CCV_M04LSTK010_	1	HG	0.932352	1000000			93			
1	31	___CCB_	1	HG	-0.08577							
1	36	___CCV_M04LSTK010_	1	HG	0.919853	1000000			92			
1	37	___CCB_	1	HG	-0.12177							
1	38	___S_MB_	1	HG	-0.03533							
1	39	___S_LCS_M04LSTK010_38	1	HG	2.358481	1000000	-0.03533		118			
1	41	234873_1_S_MD_40	1	HG	0.082625		0.114578				0.03195	
1	42	234873_1_S_MS_M04KSTK001_40	1	HG	1.146921	1000	0.114578		103			
1	43	234873_1_S_MSD_M04KSTK001_40	1	HG	1.161093	1000	0.114578	1.146921	105		1.9	
1	45	___CCV_M04LSTK010_	1	HG	1.0616	1000000			106			
1	46	___CCB_	1	HG	0.006509							
1	57	___CCV_M04LSTK010_	1	HG	1.082989	1000000			108			
1	58	___CCB_	1	HG	0.001828							
1	66	___CCV_M04LSTK010_	1	HG	1.116589	1000000			112			
1	67	___CCB_	1	HG	0.01655							

Mercury (CVAA) Solids

Report Date: 3/22/05 11:38

Method Code...: 7471	Batch Date...: 03/14/05	QC Code.....: METHG	Equipment Code.: H63
Batch Code...: 143880	Batch Time...: 1456	Calc Code.....:	Import Code.....:
Status.....: RVWD	User Name....: gok	Location Code...: 57222	

SAMPLE:	Grp	Pos	Sample ID	Dilution	HG ug/L			
1	1		S_S1_M04KSTK001_		7661			
1	2		S_S2_		14169			
1	3		S_S3_		23962			
1	4		S_S4_		43168			
1	5		S_S5_		114901			
1	6		S_S6_		199699			
1	7		ICV_M04LSTK010_		2.034483			
1	8		ICB_		0.0866			
1	9		CRA_M04KSTK001_		0.284826			
1	10		S_MB_		0.113689			
1	11		S_LCS_M04LSTK010_10		2.007891			
1	12		234659_2_S_		1.448405			
1	13		234670_1_S_		2.340857			
1	14		234670_2_S_		0.359034			
1	15		234783_1_S_		2.759537			
1	16		234783_2_S_		0.869309			
1	17		CCV_M04LSTK010_		0.863191			
1	18		CCB_		-0.0336			
1	19		234822_1_S_		2.209909			
1	20		234822_13_S_		0.434706			
1	21		234833_1_S_		0.640463			
1	22		234842_1_S_		0.69038			
1	23		234842_1_S_MD_22		0.833016			
1	24		234842_1_S_MS_M04KSTK001_22		1.182848			
1	25		234842_1_S_MSD_M04KSTK001_22		1.246989			
1	26		234842_2_S_		0.348261			
1	27		234842_3_S_		0.114421			
1	28		234842_4_S_		0.202565			
1	29		CCV_M04LSTK010_		0.777818			
1	30		CCV_M04LSTK010_		0.932352			
1	31		CCB_		-0.08577			
1	32		234842_5_S_		0.367872			
1	33		234842_6_S_		0.03284			
1	34		234855_4_O_		-0.1178			
1	35		234855_8_S_		1.30522			
1	36		CCV_M04LSTK010_		0.919853			
1	37		CCB_		-0.12177			
1	38		S_MB_		-0.03533			
1	39		S_LCS_M04LSTK010_38		2.358481			
1	40		234873_1_S_		0.114578			
1	41		234873_1_S_MD_40		0.082625			
1	42		234873_1_S_MS_M04KSTK001_40		1.146921			
1	43		234873_1_S_MSD_M04KSTK001_40		1.161093			
1	44		234873_2_S_		0.164023			
1	45		CCV_M04LSTK010_		1.0616			
1	46		CCB_		0.006509			
1	47		234873_3_S_		0.217888			
1	48		234873_4_S_		0.084429			
1	49		234873_5_S_		0.250965			
1	50		234873_6_S_		0.177437			
1	51		234873_7_S_		0.066937			
1	52		234873_8_S_		0.081945			
1	53		234873_9_S_		0.184523			
1	54		234873_10_S_		0.237133			
1	55		234873_11_S_		0.192995			
1	56		234873_12_S_		0.314321			
1	57		CCV_M04LSTK010_		1.082989			
1	58		CCB_		0.001828			
1	59		234873_13_S_		0.20382			
1	60		234873_14_S_		0.318243			
1	61		234873_15_S_		0.035794			
1	62		234873_16_S_		0.360394			
1	63		234873_17_S_		0.199872			
1	64		234873_18_S_		0.127966			

Mercury (CVAA) Solids

Report Date: 3/22/05 11:38

Method Code...: 7471	Batch Date...: 03/14/05	QC Code.....: METHG	Equipment Code.: HG3
Batch Code...: 143880	Batch Time...: 1456	Calc Code.....:	Import Code.....:
Status.....: RVWD	User Name.....: gok	Location Code..: 57222	

SAMPLE:	Grp Pos	Sample ID	Dilution	HG ug/L				
1	65	234873_19_S__		0.300018				
1	66	__CCV_M04LSTK010_		1.116589				
1	67	__CCB__		0.01655				

**STL CHICAGO
LABORATORY STANDARD OPERATING PROCEDURE**

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Attachment 2.

Example: Data Review Checklist

STL Chicago
INORGANIC CLP / LEVEL IV DATA REVIEW CHECKLIST

Site Name: _____ Primary Reviewer: _____ Review Date: _____
 JOB Number: _____ Secondary Reviewer: _____ Review Date: _____
 No. of Samples/Matrix: a) WATER _____ b) SOIL _____ c) TCLP / SPLP _____ d) OTHER _____

Metals List: a) TAL b) PP c) TCLP d) Other (_____)

Report Level: IDL = a) CLP b) Non-CLP c) MDL d) Other _____ CRDL = a) CLP b) Client c) Default RL d) Other _____

TASK: CAR's _____	PRI REV	SEC REV	COMMENTS
LAB CHRON: 1) Matches COC			
2) Proper Prep Links: S-F6 (Routine) S-F9 (TCLP/SPLP)			
3) Sample Hold Times Met			
Cyanide Reported on Forms	Y/N		Method: a) CLP b) SW846 9010B/9014
Initial / Continuing Calibration Criteria Met (CRA/CRI requirements met if applicable)			
FORM 1: Matches Report LabNet Report Units / Test Matrix Match Form 1's Dilutions due to interference's resulted in elevated RL's			
FORM 3: Method Blanks < CRDL			
FORM 5A: MS Recoveries Acceptable Default Limits _____ Statistical Limits _____ Project Limits _____ (S-F10 used to Clone By Project)			N
FORM 5B: PDS Performed			
FORM 6: Duplicate RPD Acceptable Default Limits _____ Statistical Limits _____ Project Limits _____ (S-F10 used to Clone By Project)			*
FORM 7: LCS Recoveries Acceptable Default Limits _____ Statistical Limits _____ Project Limits _____ (S-F10 used to Clone By Project)			^
FORM 8: MSA Analysis Performed			S
GFAA - Analytical Spike (AS) Recoveries Acceptable			W
GFAA - Repeat Analytical Recovery <40%			E
GFAA - Duplicate Injection Precision Met			M
FORM 9: Serial Dilution (SD) Acceptable			E
FORM 14's Correct			
RAW DATA: Complete (Match Batches to LabChron) a) Instr. Raw Data clearly displays the LabNet Batch number and includes the "Batch Worksheet" Report b) Prep Raw Data displays the LabNet Batch Number and includes the "Batch Worksheet" Report or "Raw Data" Report			

STL Chicago
Mercury Data Review Checklist: Automated CV (PS 200)

III. Data Documentation (continued)

Analyst Reviewer

- 4. Matrix Spike outside the control limits:
 - a. **CLP QC:** No corrective action required, the sample is ticked appropriately.
 - b. **Std. QC: A CAR must be written** and the Section Manager or Unit Leader must make the decision as to whether re-digestion is required.
 - c. If MSA is performed; check the calculation.
- 5. Sample Duplicate outside the control limits:
 - a. **CLP QC:** Normally no corrective action required, and the result is ticked appropriately.
 - b. **Std. QC: A CAR must be written** and the Section Manager or Unit Leader must make the decision as to whether redigestion is required.
- 6. The sample data and QC is recorded in the databook in the order in which they were analyzed. **All** unused data is clearly identified.
- 7. Standard Traceability is correctly documented.
- 8. Data Report accurately reflects the documentation in the Databook and the LIMS Spreadsheet.
- 9. The analyst's full signature is required on the following:
 - a. Instrument Data Report
 - b. Databook
 - c. Data Review Checklist
 - d. Print out LabNet Pages, Raw Data, QC, and RunLog
 - e. Samples needing copying are clearly marked
- 10. All unused portions of the data page are Z'd out.
- 11. Proper Corrective Action Documentation for any out of control situation is clearly identified.

IV. Miscellaneous

Analyst Reviewer

- 1. Is Sample Prep Linked?
- 2. Is TCLP Linked? (Shift F9 from the start page)
- 3. Did all dilutions carry over for MD, MS, MSD (where applicable)?
- 4. Did all prep and analysis matrices match up?

Comments: _____

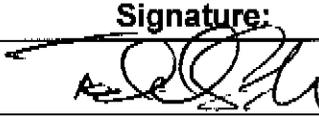
Analyst Signature: _____ Date: _____

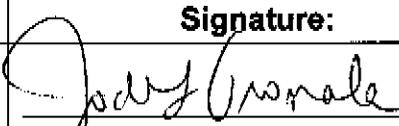
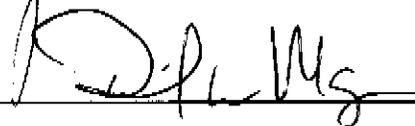
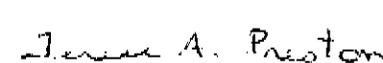
Reviewer Signature: _____ Date: _____

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**TITLE: Metals Analysis
 Trace Inductively Coupled Argon Plasma by SW-846 6010B
 (Simultaneous Operation)**

Updated by: Todd D. Smith Senior Analyst	Signature: 	Date: 1-4-05
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Approved by: Jodi L. Gromala Supervisor, Metals Dept.	Signature: 	Date: 1-4-05
David W. Mazur Env. Health & Safety Coord.		1-5-05
Terese A. Preston Quality Manager		1/5/05

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1.0 SCOPE / APPLICATION

This Standard Operating Procedure (SOP) outlines the guidelines for determining metal concentrations by Trace Inductively Coupled Argon Plasma (ICAP) Emission Spectrometry - Simultaneous Operation. This SOP was written using U.S. EPA SW-846 "Test Methods for Evaluating Solid Waste", Third Edition, Method 6010B as a reference.

On occasion, clients request slight modifications to this SOP. These modifications are addressed on a case-by-case basis with the range of accuracy (i.e., MDLs, linearity check or PT sample) verified prior to implementation. Any modifications would be written into a Quality Assurance Plan (QAP), authorized via laboratory signature approval, and mentioned in the data package's case narrative.

1.1 Method Sensitivity

1.1.1 Method Detection Limits

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 Appendix B of 40 CFR 136, "Guidelines Establishing Test Procedures for the Analysis of Pollutants". 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.

1.1.2 Instrument Detection Limits

Instrument Detection Limits (IDLs) are performed on a quarterly basis for each element and for each instrument (as specified in CLP). These limits are used to gauge instrument sensitivity and when routinely evaluated, instrument performance without the introduction of method variance can be determined.

1.1.3 Reporting Limits

Reporting Limits are defined as the lowest concentration of an analyte determined by a given method in a given matrix that the laboratory feels can be reported with acceptable quantitative error or client requirements, values specified by the EPA methods or other project and client requirements. The laboratory maintains reporting limits that are higher than the MDL. Wherever possible, reporting is limited to values ~3-5x the respective MDL to ensure confidence in the value reported. Client specific requests for reporting to the MDL are special circumstances not to be confused with the previous statement. Refer to Table 1 for element wavelength and reporting limits.

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1.1.4 Definitions

Refer to Section 3.0 of the Laboratory's Quality Manual (LQM).

1.2 Summary of Method

ICAP is a technique for the analysis of soluble or digested samples for metal concentrations using atomic emission spectrometry. All matrices, including water, TCLP extracts, wastes, soils, sludges and sediments, require digestion prior to analysis. The instrument is capable of analyzing simultaneously 29 different elements on a sample.

2.0 INTERFERENCES

Spectral, Physical and Chemical Interferences are the three main interferences that are commonly present on the ICAP.

2.1 Spectral Interferences

Mainly caused by continuous background wavelength, stray light from a high concentration element or overlap of a spectral line from another element. The ICAP can correct for the first two types of interferences by using background correction adjacent to the wavelength. Spectral overlap can be corrected by monitoring the interfering wavelength and computer correcting the results for the false concentration. The values used to correct are known as Inter-Element Correction Factors or IEC's.

2.2 Physical Interferences

Usually associated with the sample uptake and nebulization processes. These interferences can usually be eliminated by using a peristaltic pump which assures a constant sample uptake rate. If a sample is extremely viscous or contains a very high dissolved solids concentration, a dilution of the sample may be required to assure a constant and smooth nebulization rate.

2.3 Chemical Interferences

Normally not significant on the ICAP. These interferences include ionization effects and molecular compound formation. Chemical interferences are highly dependent on the sample matrix type and the element.

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Trace ICP can have some ionization effects caused by torch positioning. To eliminate these effects, Cesium is added to the internal standard solution (100 mLs / 1-Liter). Most interferences can be corrected by ensuring a constant sample uptake rate and by using the correcting abilities of the computer. If severe interferences are suspected, an alternate method such as Graphite Furnace Atomic Absorption (GFAA) can be used or to verify the ICAP results.

3.0 SAFETY

Employees must abide by the policies and procedures in the Corporate Safety Manual, Radiation Safety Manual and this document.

3.1 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.
- Parts of the instrument can be extremely hot. Care should be taken if the instrument needs to be adjusted internally.
- Proper ventilation is required due to sample fumes and extreme heat generation (RF generator and plasma) and plasma emissions. People with medical conditions that may respond to ozone emissions should exercise caution.

3.2 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
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.

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Material (1)	Hazards	Exposure Limit (2)	Signs and Symptoms of Exposure
Hydrochloric Acid	Corrosive Poison	5 ppm- Ceiling	Inhalation of vapors can cause coughing, choking, inflammation of the nose, throat, and upper respiratory tract, and in severe cases, pulmonary edema, circulatory failure, and death. Can cause redness, pain, and severe skin burns. Vapors are irritating and may cause damage to the eyes. Contact may cause severe burns and permanent eye damage.
1 – Always add acid to water to prevent violent reactions.			
2 – Exposure limit refers to the OSHA regulatory exposure limit.			

4.0 EQUIPMENT AND SUPPLIES

4.1 Instrumentation

3 - Thermo Jarrell Ash ICAP 61E Trace Analyzer. These instruments are simultaneous ICAP's which currently have 31 analytical wavelengths. Additional wavelengths may be added as required.

The instruments are operated via desktop computers and Thermo Jarrell Ash software (Version 6.2). They also come equipped with a peristaltic pump for sample uptake and an autosampler.

4.2 Supplies

- Volumetric Flasks (Class A): 100 mLs; 200 mLs; 1000 mLs
- Eppendorf Pipettes, varying volumes

5.0 REAGENTS AND STANDARDS

5.1 Reagents

- Milli-Q Water
- *Concentrated Nitric Acid (HNO₃) - InstraPure
- *Concentrated Hydrochloric Acid (HCl) - InstraPure

*Purchased from a vendor.

5.2 Standards and QC Solutions

All stock standards and QC solutions are purchased from an outside supplier in aqueous form. Two types of standards are used: single element and custom mixed standards. Single element standards are available for most elements at a 1,000 mg/L concentration. The shelf life of all purchased solutions are as stated by the manufacturer and are listed in LabNet (LIMS).

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5.2.1 Calibration Standards

Prepared with Milli-Q water that has been acidified with 1% HNO₃ and 5% HCl. The calibration standards are prepared daily as follows:

A. Calibration Blank

Add ~500 mLs of Milli-Q water to a 1-L Class A volumetric flask. Repipette 10 mLs conc. HNO₃ and 50 mLs conc. HCl into the flask. Dilute to volume with Milli-Q water and mix thoroughly.

B. Calibration Standards (Refer to Attachment 1 for element concentrations)

Standard	Preparation
S1	<ul style="list-style-type: none"> • Add ~50 mLs of Milli-Q water to a 200 mL Class A volumetric flask. • Re-pipette 2 mLs conc. HNO₃ into the flask. • Re-pipette 10 mLs conc. HCl into the flask. • Using Eppendorf pipettes, add 2.0 mLs each of: RFW-ICPT-STD-1B RFW-ICPT-STD-1C RFW-ICPT-STD-1D • Dilute to volume with Milli-Q water and mix thoroughly.
S1A	<ul style="list-style-type: none"> • Add ~50 mLs of Milli-Q water to a 200 mL Class A volumetric flask. • Re-pipette 2 mLs conc. HNO₃ into the flask. • Re-pipette 10 mLs conc. HCl into the flask. • Using Eppendorf pipettes, add 0.8 mLs each of: RFW-ICPT-STD-1B RFW-ICPT-STD-1C RFW-ICPT-STD-1D • Dilute to volume with Milli-Q water and mix thoroughly.
S1B	<ul style="list-style-type: none"> • Add ~50 mLs of Milli-Q water to a 200 mL Class A volumetric flask. • Re-pipette 2 mLs conc. HNO₃ into the flask. • Re-pipette 10 mLs conc. HCl into the flask. • Using Eppendorf pipettes, add 1.0 mLs each of: RFW-ICPT-STD-1B RFW-ICPT-STD-1C RFW-ICPT-STD-1D • Dilute to volume with Milli-Q water and mix thoroughly.
S2	<ul style="list-style-type: none"> • Add ~50 mLs of Milli-Q water to a 200 mL Class A volumetric flask. • Re-pipette 2 mLs conc. HNO₃ into the flask. • Re-pipette 10 mLs conc. HCl into the flask. • Using Eppendorf pipettes, add 2.0 mLs each of: RFW-ICPT-STD-2A RFW-ICPT-STD-2B RFW-ICPT-STD-3 • Dilute to volume with Milli-Q water and mix thoroughly.

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Standard	Preparation
S2A	<ul style="list-style-type: none"> • Add ~50 mLs of Milli-Q water to a 200 mL Class A volumetric flask. • Re-pipette 2 mLs conc. HNO₃ into the flask. • Re-pipette 10 mLs conc. HCl into the flask. • Using Eppendorf pipettes, add 0.8 mLs each of: RFW-ICPT-STD-2A RFW-ICPT-STD-2B RFW-ICPT-STD-3. • Dilute to volume with Milli-Q water and mix thoroughly.
S2B	<ul style="list-style-type: none"> • Add ~50 mLs of Milli-Q water to a 200 mL Class A volumetric flask. • Re-pipette 2 mLs conc. HNO₃ into the flask. • Re-pipette 10 mLs conc. HCl into the flask. • Using Eppendorf pipettes, add 1.0 mL each of: RFW-ICPT-STD-2A RFW-ICPT-STD-2B RFW-ICPT-STD-3 • Dilute to volume with Milli-Q water and mix thoroughly.

5.2.2 QC Solutions (Refer to Attachment 2 for element concentrations.)

Prepared with Milli-Q water that has been acidified with 1% HNO₃ and 5% HCl. All QC Solutions are recorded in the intermediate standard traceability logbook.

QC Solution	Preparation (In a 1-L Class A volumetric flask filled w/ ~500 mLs of Milli-Q water, add the following for each QC Solution)
Initial Calibration Verification (ICV)	<ul style="list-style-type: none"> • 10 mLs conc. HNO₃ • 50 mLs conc. HCl. • 8 mLs of CCV Soln. A • 8 mLs of CCV Soln. A1 • 8 mLs CCV Soln. B • 1.84 mLs of 10,000 ug/mL Ca • 1.6 mLs of 10,000 ug/mL Na, Fe • 1.68 mLs of 10,000 ug/mL Mg • 3.6 mLs of 10,000 ug/mL K, Al • Dilute to volume with Milli-Q water and mix thoroughly.
Continuing Calibration Verification (CCV)	<ul style="list-style-type: none"> • 10 mLs conc. HNO₃ • 50 mLs conc. HCl. • 10 mLs of CCV Soln. A • 10 mLs of CCV Soln. A1 • 10 mLs of CCV Soln. B • 2.3 mLs of 10,000 ug/mL Ca • 2.0 mLs of 10,000 ug/mL Na, Fe • 2.1 mLs of 10,000 ug/mL Mg • 4.5 mLs of 10,000 ug/mL K, Al • Dilute to volume with Milli-Q water and mix thoroughly.

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QC Solution	Preparation (In a 1-L Class A volumetric flask filled w/ ~500 mLs of Milli-Q water, add the following for each QC Solution):
CRI [Contract Required Detection Limit (CRDL) Standard for ICAP]	<ul style="list-style-type: none"> • 10 mLs conc. HNO₃ • 50 mLs conc. HCl • 40 uLs of Cd Intermediate Std. * • 80 uLs of Be Intermediate Std. * • 10 uLs of 10,000 ug/mL Fe • 10 uLs of 1,000 ug/mL Co, Se, Ag, Sr, Ti, V, Pb • 20 uLs of 10,000 ug/mL Ca, Mg • 20 uLs of 1,000 ug/mL As, Cu, Cr, Mn, Ni, Ba, Mo, Tl, Zn • 40 uLs of 10,000 ug/mL Al • 40 uLs of 1,000 ug/mL Sb, Sn • 200 uLs of 10,000 ug/mL Na • 100 uLs of 10,000 ug/mL K • 100 uLs of 1,000 ug/mL B, Bi • 400 uLs of 1,000 ug/mL Si • Dilute to volume with Mill-Q water and mix thoroughly. <p>* Cd Intermediate = 1:10 dilution of 1,000 ppm Cd. * Be Intermediate = 1:10 dilution of 1,000 ppm Be</p>
Interferent Check Standard (ICSA)	<ul style="list-style-type: none"> • 10 mLs conc. HNO₃ • 50 mLs conc. HCl • 100 mLs of CLP Interferent A Solution • Dilute to volume with Milli-Q water and mix thoroughly
Interferent Check Standard (ICSAB)	<ul style="list-style-type: none"> • 10 mLs conc. HNO₃ • 50 mLs conc. HCl • 100 mLs of CLP Interferent A Solution • 10 mLs of CLPP-ICS-B4 • Dilute to volume with Milli-Q water and mix thoroughly.

6.0 CALIBRATION (NON-DAILY)

6.1 Linear Range Analysis Standard (LRS)

LRS calibration is performed quarterly that covers the anticipated range of measurement. The expected recovery limit for this verification standard is 95-105%. This is used to verify linearity and document the upper limit of the calibration range for each element. At least one of the calibration standards will be at or near the reporting limit. The calibration curve generated must have a correlation coefficient of ≥ 0.995 in order to consider the responses linear over that range. All samples found to be above the ICAP linear range are diluted and re-analyzed until the concentration falls within the instruments linear range.

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6.2 Inter-Element Correction (IEC)

Correction factors for spectral interference due to Al, Ca, Fe, and Mg will be determined at least annually for all wavelengths used for each analyte reported or any time the ICAP is adjusted in any way that may affect the IECs. Correction factors for spectral interferences other than Al, Ca, Fe, and Mg are recommended and are performed as needed and documented with the instrument records.

7.0 PROCEDURE

7.1 Quality Control Checks

The following section summarize the quality control (QC) samples associated with ICAP analysis.

QC Sample	Frequency	Control Limit ¹
Method Blank (MB)	1 per 20 samples	< Reporting Limit
Lab Control Sample (LCS) ²	1 per 20 samples	80 – 120 %
Matrix Spike (MS) ^{3,6}	1 per 20 samples	75 – 125 %
MS Duplicate (MSD) ^{3,6}	1 per 20 samples	75 – 125 %; 20 RPD
Duplicates (MD) ^{4,6}	1 per 20 samples	20 RPD
Serial Dilution (5x) ⁵	1 per 20 samples	+ 10% of the original result

¹ Refer to Section 8 for additional details.

² LCS Duplicate (LCD) is performed only when required by the client or project.

³ If sample concentration is $\leq 4X$ spike level, 75-125%; if sample concentration is $> 4X$ spike level, no control range. If TCLP matrix spike is $< 50\%$, Standard Addition must be performed.

⁴ If $\geq 5X$ reporting limit, 20 RPD; if $< 5X$ reporting limit \pm reporting limit; if $<$ reporting limit no control range.

⁵ If the analyte concentration is $>10X$ the MDL, results should agree within $\pm 10\%$ of the original sample result.

⁶ The sample selection for matrix QC, if not specified by the client or on the chain-of-custody, is rotated among client samples so that various matrix problems may be noted and/or addressed...pre-determined by the digestion department.

7.2 Sample Preservation 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.

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Matrix	Holding Time	Preservation	Reference
Waters	180 days	HNO ₃ , pH < 2; Cool 4 + 2°C	40 CFR Part 136.3
Soils	180 days	Cool 4 + 2°C	N/A

¹ Inclusive of digestion and analysis.

7.3 Sample Preparation

The most commonly used digestion procedures are SW-846 Methods 3010A (waters) and 3050B (soils). Refer to USP-3000 for details on sample digestion. The samples are received in the metals laboratory as 25, 50 or 100 mL final volumes.

7.4 Calibration / Standardization

7.4.1 Instrument Set Up

Set up the instrument with the proper operating conditions as defined in the TJA instrument manual. The instrument must be allowed to become thermally stable (~1-hour) prior to profiling and calibration. The instrument is profiled using a 1-ppm Arsenic standard (S1) by aspiration and selecting the automatic profile feature from the TJA software. The peak position reading should be within +/- 0.1. If the reading is acceptable, record the peak area in the logbook & rinse. If the reading is > +/- 0.1, set the micrometer to the adjusted vernier position given by the instrument and profile again to verify. Record the peak area in the logbook and rinse. The instrument is now ready to calibrate.

7.4.2 Standardization

Before any instrument is used as a measurement device, the instrument response to known reference materials must be determined. All sample measurements must be made within the linear range of the instrument.

The instrument is standardized using a calibration blank and 3 calibration standards, which consist of 6 multi-element solutions. The results are given in intensities. Minimum requirement is a blank and a standard.

Standard	Frequency	Control Limit
Calibration Curve	Initially	Corr. Coeff. \geq 0.995
High Standards (S1, S2)	After the Calibration Curve	+ 5% of the Known Conc.
Initial Cal. Verif. (ICV)	After the Calibration Curve	+ 10% of the Known Conc.
Initial. Cal. Blank (ICB)	After the ICV	< Reporting Limit
CRI	Daily, every 8 hrs. thereafter	None Required
ICSA / ICSB	Daily, every 8 hrs. thereafter	+ 20% of the Known Conc.
Cont. Cal. Verif. (CCV)	Every 10 reading; End of each run	\pm 10% of the Known Conc.

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Standard	Frequency	Control Limit
Cont. Cal. Blank (CCB)	Every 10 readings; End of each run	≤ Reporting Limit

7.5 Preventive Maintenance

The required preventive maintenance is listed in the preventive maintenance logbooks which are kept at the instruments. All maintenance is recorded in these logbooks along with the date and the signature of the analyst performing the maintenance. The instruments are under a full service contract with the manufacturer for all major repairs.

7.5.1 Daily Maintenance

Includes changing the pump tubing for consistent sample uptake and a visible check of the waste container to make sure that it doesn't overflow.

7.5.2 Weekly Maintenance

Includes checking the air filters on the back of the instrument for excessive dust buildup, and checking the tip of the torch for excessive buildup of material.

7.5.3 Monthly Maintenance

Includes cleaning and checking the water re-circulator for proper fluid level, cleaning the spray chamber.

7.6 Sample Analysis

7.6.1 Analytical Run

After the instrument is standardized (Section 7.4.2), an analytical run is initiated. The first run of the day would proceed as follows:

- S1,S2 Reanalysis of calibration standard as a sample
- ICV Initial Calibration Verification
- ICB Initial Calibration Blank
- CRI Spiked Blank Sample
- ICSA Interferent Check Standard A
- ICSB Interferent Check Standard B
- CCV Continuing Calibration Verification
- CCB Continuing Calibration Blank
- MB (1) Method Blank
- LCS (2) Laboratory Control Sample
- Sample (3)
- Sample (4) Serial Dilution (L)

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- Sample (5) Matrix Duplicate (MD)
- Sample (6) Matrix Spike (MS)
- Sample (7) Matrix Spike Duplicate (MSD)
- Sample (8)
- .
- Sample X (10)
- CCV Continuing Calibration Verification
- CCB Continuing Calibration Blank

If the CCV and CCB results are acceptable, the run may continue without restandardization. If any of the post-run QC is out of control, or close to being out of control, the instrument is restandardized before analyzing the next batch. Any samples with elements associated with an out of control CCV or CCB will be reanalyzed.

7.7 Documentation

7.7.1 Instrument Run-Log

The analysis of samples and standards is documented within the instrument run log (Attachment C), which must be for each days analysis, and is supported by the instrument print-out.

7.7.2 Traceability of Standards

Custom made and single element stock standard solution which are traceable to NIST or EPA are purchased. Upon receipt, each standard is entered into LabNet and is issued a unique source ID#. The manufacturer, lot #, date received, expiration date, date of verification and the initials of the recording analyst are also entered.

7.7.3 Data Review

Analytical data goes through a 200% review cycle. The analyst and a trained data reviewer perform the reviews according to the criteria established on the data review checklist (Attachment D). Upon the first 100% review, the checklist is initialed and dated as reviewed. The package, with its review sheet, comments and any Corrective Action Reports (CARs) are submitted to the supervisor or peer reviewer for a second review. Once again, the checklist is initialed and dated by the second reviewer. The completed data review form remains on file with the original data.

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8.0 QUALITY CONTROL

8.1 QC Summary

NOTE: The following laboratory acceptance criteria are set at default control limits. Statistical limits are generated on an annual basis from cumulative LCS data and can be implemented when specified by the client, contract, or QAP.

8.1.1 Method Blank (MB)

At least one MB and one LCS will be included in each digestion batch of 20 samples. Regardless of the matrix being processed, the LCS and MB will be in an aqueous media. The MBs are analyzed to determine if contaminants are being introduced into the sample via the sample preparation procedures.

8.1.2 Laboratory Control Sample (LCS)

The LCS is analyzed to determine the accuracy of the digestion process.

Accuracy will be measured by the percent recovery (%R) of the LCS. The recovery must be within $\pm 20\%$ of the known concentration. If the LCS results are outside these control limits, all samples in the preparation set must be redigested and reanalyzed. Refer to Attachment E for element concentrations.

8.1.3 Matrix Duplicate (MD)

A duplicate sample will be prepared at a frequency of 5% (1 in 20 samples). A 20 RPD is set as the acceptance limits.

8.1.4 Matrix Spike (MS) / Matrix Spike Duplicate (MSD)

The MS / MSD will be prepared at a frequency of 5% (1 in 20 samples). The recovery must be within 75–125%. (Exception allowed if the sample concentration exceeds 4 times the spike added concentration.)

TCLP - If the MS recovery is $< 50\%$ and the concentration does not exceed the regulatory limit or the sample concentration is within 20% of the regulation level, the Method of Standard Addition (MSA) is required. Three aliquots of the sample are spiked at 50%, 100% and 150% of the sample concentration or, if the sample concentration is $< RL$, the MSA is at 50%, 100% and 150% of the MS level. The data is subjected to linear regression whereas the concentration of the unknown is the x-intercept and the correlation coefficient value must be ≥ 0.995 .

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8.1.5 Serial Dilution

A Serial Dilution (5X) will be prepared from the digestate at a frequency of 5% (1 in 20 samples). If the concentration is >50 times the MDL, results should agree within +/- 10% of the original results.

8.2 Corrective Action

When an out-of-control situation occurs, the analysts must use his/her best analytical judgment and available resources to determine the corrective action to be taken. The out-of-control situation may be caused by more than one variable. The analyst should seek the assistance of his/her supervisor, QA personnel, or other experienced staff if he/she are uncertain of the cause of the out-of-control situation. The analysis must not be resumed until the source of the problem and an in-control status is attained. All samples associated with the out-of-control situation should be reanalyzed. Out-of-control data must never be released without approval of the supervisor, or QA personnel.

The following steps that must be taken when an out-of-control situation occurs:

- demonstrate that all the problems creating the out-of-control situation were addressed;
- document the problem and the action which was taken to correct the problem on a CAR;
- document on the CAR that an in-control has been achieved; and
- receive approval (signature) of the supervisor or QA personnel prior to the release of any analytical data associated with the problem.

QC Indicator	Suggested Corrective Actions
Calibration Curve	<ul style="list-style-type: none"> • reanalyze the standard curve; • prepare a new stock and/or working standards; • check the reagents/solutions and prepare fresh if necessary.
ICV	<ul style="list-style-type: none"> • repeat the ICV to verify proper preparation; • prepare a new ICV from original stock; • recalibrate with a new standard curve; • prepare a new stock and/or working standards; • check the reagents/solutions and prepare fresh if necessary.
ICB	<ul style="list-style-type: none"> • prepare a new ICB to verify proper preparation; • verify that the instrument base-line is stable and perform necessary maintenance, cleaning, etc.. to achieve stability; • determine the source of contamination by process of elimination, carryover from a previous analysis or reagent contamination and correct the problem; • check the reagents/solutions and prepare fresh if necessary; • correct for any contamination and reanalyze the ICB and any associated samples.

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LABORATORY STANDARD OPERATING PROCEDURE**

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QC Indicator	Suggested Corrective Actions
LCS	<p><u>If the LCS is low:</u></p> <ul style="list-style-type: none"> • reanalyze the LCS and all samples in the set for the failed analyte(s) to confirm that it is out of control. • If continued out of control, redigest and reanalyze the set. • Write a CAR. <p><u>If the LCS is high:</u></p> <ul style="list-style-type: none"> • reanalyze the LCS and all samples in the set for the failed analyte(s) to confirm that it is out of control. • check for contamination of reagents, LCS stock solution, or in the preparation area; • correct for contamination, redigest and re-analyze the set; • Write a CAR.
MB	<ul style="list-style-type: none"> • reanalyze the MB to verify that it is beyond the reporting limit; • determine the source of contamination; • determine if a high value is due to contamination; • check for contamination of reagents or in the preparation area; • correct for contamination, reanalyze the set; • in the extreme case where all samples in the set are at least 10x > the MB or < RL, reanalysis will not be required; however, a CAR will be written and approved by the supervisor or section manager.
MD	<ul style="list-style-type: none"> • a CAR will be written and approved by the supervisor or section manager.
MS / MSD	<ul style="list-style-type: none"> • a CAR will be written and approved by the supervisor or section manager.
Serial Dilution (L)	<ul style="list-style-type: none"> • prepare a new serial dilution to verify proper preparation; • a CAR will be written and approved by the supervisor or section manager.
CCV	<ul style="list-style-type: none"> • repeat the CCV to verify proper preparation; • prepare a new CCV from the original stock; • check for instrument base-line drift or a change in one or more of the reagents; • check the reagents/solutions and prepare fresh if necessary; • recalibrate with a new standard curve and repeat all samples since the previous in control CCV; • never dispose of any samples until you are sure that all QC are within the control limits.
CCB	<ul style="list-style-type: none"> • check reagents/solutions to verify proper preparation and prepare fresh if necessary; • verify that the instrument base-line is stable and/or perform necessary maintenance, cleaning, etc., to achieve stability; • correct for any contamination (carryover from a previous analysis or reagent contamination) and reanalyze the CCB and any associated samples; • never dispose of any samples until you are sure that all QC are within the control limits.

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QC Indicator	Suggested Corrective Actions
Additional CAs	<ul style="list-style-type: none"> • If any of the ICV, ICB, ISA, ISB, CCV or CCB results are out-of-control for any element, the instrument is restandardized and the samples associated with the out-of-control elements are reanalyzed. • If the MB or LCS are out of control for any element, the samples are redigested. An exception is if the sample concentrations are $\geq 10X$ the MB contamination or $< RL$. In this case, the results are reported as is. • If any of the MD or MS/MSD results are out of control, the client is notified of the poor results via a case narrative that is sent with the data report. • CARs are completed by the analyst performing the analysis. The forms are then reviewed and signed by the supervisor or section manager. The signed forms are filed with the original data and a copy is kept on file in the Metals Department.

9.0 DATA ANALYSIS AND CALCULATIONS

The sample results are stored in a data file on the desktop computer. The data is transferred over to LabNet and edited there. This system helps to eliminate transcription errors, since data is not entered by hand.

9.1 Accuracy

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

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

9.2 Precision (RPD)

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

9.3 Concentration mg/kg or L = $\frac{C \times V \times D}{W}$

Where:

C = sample concentration in extract (ppm)

V = Volume of extract (mL)

D = Dilution Factor

W = Weight/Volume of sample aliquot extracted (grams or mLs)

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

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10.0 WASTE MANAGEMENT AND POLLUTION PREVENTION

All waste will be disposed of in accordance with Federal, State and Local regulations. Where reasonably feasible, technological changes have been implemented to minimize the potential for pollution of the environment. Employees will abide by this method and the policies in section 13 of the Corporate Safety Manual for "Waste Management and Pollution Prevention."

10.1 Waste Streams Produced by the Method

The following waste streams are produced when this method is carried out.

- Waste from this procedure will enter the "Corrosive Wastewater" wastestream.

11.0 METHOD PERFORMANCE CRITERIA

Refer to Sections 1.0, 7.0 and 8.0.

12.0 REFERENCES

Refer to Section 1.0.

13.0 ATTACHMENTS

Table 1. Element and Reporting Limits
Attachment 1. Standard Stock Solutions
Attachment 2. Stock QC Solutions
Attachment 3. Example: Analysis Run Log / Maintenance Log
Attachment 4. Example: Data Review Form
Attachment 5. Known Digested Quality Control

Historical File: Revision 00: 02/11/98 Revision 05: 10/30/03
 Revision 01: 01/29/99 Revision 06: 01/03/05
 Revision 02: 03/20/00
 Revision 03: 06/29/01
 Revision 04: 09/13/02

Reasons for Revision: Revision 06:

- Annual Review -- Maintenance Log added as attachment.

U:\QC\SOP\Metals\UME-6010B.doc

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Table 1.

Element and Reporting Limits

Element	ICAP 61E (ICP3)	ICAP 61E (ICP4)	ICAP 61E (ICP5)	Reporting Limits ¹	
	Wavelength (nm)	Wavelength (nm)	Wavelength (nm)	Waters (mg/L)	Soils (mg/kg)
Al	308.2	308.2	308.2	0.2	20
Sb	206.8	206.8	206.8	0.02	2
As	189.0	189.0	189.0	0.01	1
Ba	493.4	493.4	493.4	0.01	1
Be	313.0	313.0	313.0	0.004	0.4
Bi	223.0	223.0	N/A	0.05	5
B	249.6	249.6	249.6	0.05	5
Ca	317.9	317.9	317.9	0.1	10
Cd	226.5	226.5	226.5	0.002	0.2
Cr	267.7	267.7	267.7	0.01	1
Co	228.6	228.6	228.6	0.005	0.5
Cu	324.7	324.7	324.7	0.01	1
Fe	271.4	271.4	271.4	0.05	5
Pb	220.3	220.3	220.3	0.005	0.5
Mg	279.0	279.0	279.0	0.1	10
Mn	257.6	257.6	257.6	0.01	1.0
Mo	202.0	202.0	202.0	0.01	1
Ni	231.6	231.6	231.6	0.01	1
K	766.4	766.4 / 404.7	766.4	0.5 / 10	50 / 1,000
Se	196.0	196.0	196.0	0.01	1
Si	288.1	288.1	288.1	0.2	20
Ag	328.0	328.0	328.0	0.005	0.5
Na	330.2	330.2	330.2 / 588.9	1	100
Sr	421.5	NA	421.5	0.005	0.5
Tl	190.8	190.8	190.8	0.01	1
Sn	189.9	189.9	189.9	0.02	2
Ti	334.9	337.2	334.9	0.005	0.5
V	292.4	292.4	292.4	0.005	0.5
Y ²	371.0	371.0	371.0	N/A	N/A
Zn	213.8	206.2	206.2	0.02	2

¹These are routine Trace ICAP reporting limits (RL). Lower RLs are available and can be used per client request. RLs will vary depending on sample size/volume, dilution factors, dry weight reporting for soils, and changes in MDLs.

²Y is used as an internal standard and is introduced continuously to all samples (including standards and QC samples) via the peristaltic pump at an approximate concentration of 5 ppm.

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Attachment 1.

Standard Stock Solutions

Vendor	Stock Name	Element	Conc. (mg/L)	S1A	S1B	S1	S2A	S2B	S2
Inorganic Ventures	RFW-ICPT-STD-1B	Sb	100	0.4	0.5	1			
		Mo	100	0.4	0.5	1			
		Si	100	0.4	0.5	1			
		Sn	100	0.4	0.5	1			
		Ti	100	0.4	0.5	1			
Inorganic Ventures	RFW-ICPT-STD-1C	Al	1,000	4	5	10			
		Fe	1,000	4	5	10			
		K	1,000	4	5	10			
		Na	1,000	4	5	10			
		Li	800	2	4	8			
		Mg	800	2	4	8			
		Ca	400	1.6	2	4			
Inorganic Ventures	RFW-ICPT-STD-1D	As	100	0.4	0.5	1			
		Ba	100	0.4	0.5	1			
		Be	100	0.4	0.5	1			
		Bi	100	0.4	0.5	1			
		B	100	0.4	0.5	1			
		Cd	100	0.4	0.5	1			
		Cr	100	0.4	0.5	1			
		Cu	100	0.4	0.5	1			
		Pb	100	0.4	0.5	1			
		Ni	100	0.4	0.5	1			
		Se	100	0.4	0.5	1			
		Ag	100	0.4	0.5	1			
		Sr	100	0.4	0.5	1			
		Tl	100	0.4	0.5	1			
Zn	100	0.4	0.5	1					
Inorganic Ventures	RFW-ICPT-STD-2A	Al	10,000				40	50	100
		K	10,000				40	50	100
Inorganic Ventures	RFW-ICPT-STD-2B	Ca	5,000				20	25	50
		Fe	5,000				20	25	50
		Mg	5,000				20	25	50
		Na	5,000				20	25	50
Inorganic Ventures	RFW-ICPT-STD-3	Pb	2,000				8	10	20
		Mn	1,000				4	5	10
		V	1,000				4	5	10

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Attachment 2.

Example of Stock QC Solutions

Vendor	Stock Name	Element	Conc. (mg/L)	ICV (mg/L)	CCV (mg/L)
High Purity	CCV Solution A	As	50	0.4	0.5
		B	50	0.4	0.5
		Ba	50	0.4	0.5
		Be	50	0.4	0.5
		Bi	50	0.4	0.5
		Cd	50	0.4	0.5
		Co	50	0.4	0.5
		Cr	50	0.4	0.5
		Cu	50	0.4	0.5
		Ni	50	0.4	0.5
		Pb	50	0.4	0.5
		Se	50	0.4	0.5
		Fe	500	20	25
		Mn	500	4	5
		V	500	4	5
		Tl	50	0.4	0.5
		Zn	50	0.4	0.5
Sr	50	0.4	0.5		
High Purity	CCV Solution A2	Ca	200	20	25
		Li	400	---	---
		Na	500	20	25
		Al	500	40	50
		Mg	400	20	25
		K	500	40	50
High Purity	CCV Solution B	Ag	50	0.4	0.5
		Sb	50	0.4	0.5
		Mo	50	0.4	0.5
		Si	50	0.4	0.5
		Sn	50	0.4	0.5
		Ti	50	0.4	0.5
Ultra	Single Elements * spiked on top of custom mixes.	Al	10,000	40	50
		Ca	10,000	20	25
		Fe	10,000	20	25
		Na	10,000	20	25
		K	10,000	40	50
		Mg	10,000	20	25

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**Attachment 2.
(continued)
Examples of Stock QC Solutions**

Vendor	Stock Name	Element	Conc. (mg/L)	CRI Conc. (mg/L)
Inorganic Ventures	Beryllium	Be	1,000	0.008
	Chromium	Cr	1,000	0.02
	Cobalt	Co	1,000	0.01
	Copper	Cu	1,000	0.02
	Manganese	Mn	1,000	0.02
	Nickel	Ni	1,000	0.02
	Silver	Ag	1,000	0.01
	Vanadium	V	1,000	0.01
	Zinc	Zn	1,000	0.02
	Antimony	Sb	1,000	0.04
	Arsenic	As	1,000	0.02
	Cadmium	Cd	1,000	0.004
	Lead	Pb	1,000	0.01
	Selenium	Se	1,000	0.01
Thallium	Tl	1,000	0.02	
Inorganic Ventures	Calcium	Ca	10,000	0.2
	Potassium	K	10,000	1.0
	Magnesium	Mg	10,000	0.2
	Sodium	Na	10,000	2.0
	Iron	Fe	10,000	0.1
	Aluminum	Al	10,000	0.04
	Barium	Ba	1,000	0.02
	Boron	B	1,000	0.1
	Bismuth	Bi	1,000	0.1
	Molybdenum	Mo	1,000	0.02
	Silicon	Si	1,000	0.4
	Tin	Sn	1,000	0.04
	Strontium	Sr	1,000	0.01
Titanium	Ti	1,000	0.01	

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**Attachment 2.
(continued)
Stock QC Solutions**

Vendor	Stock Name	Element	Conc. (mg/L)	ICSA Conc. (mg/L)
Inorganic Ventures	CLP Interferents "A" Solution	Al	5,000	500
		Ca	5,000	500
		Mg	5,000	500
		Fe	2,000	200
				ICSB Conc. (mg/L)
Inorganic Ventures	CLP Interferent A Solution	Al	5,000	500
		Ca	5,000	500
		Mg	5,000	500
		Fe	2,000	200
Inorganic Ventures	CLPP-ICS-B4	Cd	100	1
		Ni	100	1
		Zn	100	1
		Sb	60	0.6
		Ba	50	0.5
		Be	50	0.5
		Co	50	0.5
		Cr	50	0.5
		Cu	50	0.5
		Mn	50	0.5
		V	50	0.5
		Ag	20	0.2
		As, Tl	10	0.1
Pb, Se	5	0.05		

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Attachment 3.

Example: Analysis Runlog / Maintenance Log

STL Chicago
TJA Trace ICAP (61E) Analysis Log – ICP3

Page No. _____

Date	Initials	File Name	Dig. Set	Int. Std	Sample Nos.	Parameters	Comments
				As =			
				Y =			
				As =			
				Y =			
				As =			
				Y =			
				As =			
				Y =			
				As =			
				Y =			
				As =			
				Y =			
				As =			
				Y =			

Reviewed by: _____ Date: _____

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**STL Chicago
TJA Trace ICAP (61E) - ICP3
Instrument Maintenance Log**

Page No. _____

	Date/Initials						
Daily Maintenance:							
Check/Change Pump Tubing							
Check Waste Container							

Weekly Maintenance:							
Clean Air Filters							
Check Torch for buildup (Note Cleaning)							
Check/Change Printer Ribbon							

Monthly Maintenance:							
Check/Refill Recirculator							
Check Nebulizer/Spray Chamber							

Comments: _____

Any Maintenance/Repair/Part Replacement performed that is not listed above must be documented in the Comments sections

Reviewer Signature: _____

Date: _____

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Attachment 4.

Example: Data Review Checklist

STL Chicago
INORGANIC CLP / LEVEL IV DATA REVIEW CHECKLIST

Site Name: _____ Primary Reviewer: _____ Review Date: _____
 JOB Number: _____ Secondary Reviewer: _____ Review Date: _____
 No. of Samples/Matrix: a) WATER _____ b) SOIL _____ c) TCLP / SPLP _____ d) OTHER: _____

Metals List: a) TAL b) PP c) TCLP d) Other (_____)

Report Level: IDL = a) CLP b) Non-CLP c) MDL d) Other _____ CRDL = a) CLP b) Client c) Default RL d) Other _____

TASK : CAR's _____	PRI REV	SEC REV	COMMENTS
LAB CHRON: 1) Matches COC			
2) Proper Prep Links: S-F6 (Routine) S-F9 (TCLP/SPLP)			
3) Sample Hold Times Met			
Cyanide Reported on Forms	Y/N		Method: a) CLP b) SW846 9010B/9014
Initial / Continuing Calibration Criteria Met (CRA/CRI requirements met if applicable)			
FORM 1: Matches Report LabNet Report Units / Test Matrix Match Form 1's Dilutions due to interference's resulted in elevated RL's			
FORM 3: Method Blanks < CRDL			
FORM 5A: MS Recoveries Acceptable Default Limits _____ Statistical Limits _____ Project Limits _____ (S-F10 used to Clone By Project)			N
FORM 5B: PDS Performed			
FORM 6: Duplicate RPD Acceptable Default Limits _____ Statistical Limits _____ Project Limits _____ (S-F10 used to Clone By Project)			*
FORM 7: LCS Recoveries Acceptable Default Limits _____ Statistical Limits _____ Project Limits _____ (S-F10 used to Clone By Project)			^
FORM 8: MSA Analysis Performed			S
GFAA – Analytical Spike (AS) Recoveries Acceptable			W
GFAA – Repeat Analytical Recovery <40%			E
GFAA – Duplicate Injection Precision Met			M
FORM 9: Serial Dilution (SD) Acceptable			E
FORM 14's Correct			
RAW DATA: Complete (Match Batches to LabChron) a) Instr. Raw Data clearly displays the LabNet Batch number and includes the "Batch Worksheet" Report b) Prep Raw Data displays the LabNet Batch Number and includes the "Batch Worksheet" Report or "Raw Data" Report			

**STL Chicago
ICAP Metals Data Review Checklist**

Instrument ID: ICP 3 ICP 4 ICP 5

Filename: _____

Analyst Initial(s): _____

LabNet Batch No.: _____

Copies: _____

QC Type: a. CLP b. Standard c. TCLP d. Drinking Waters e. Solubles

I. Calibration:

Analyst	Reviewer	
<input type="checkbox"/>	<input type="checkbox"/>	1. Verification of standard traceability and expiration (daily).
<input type="checkbox"/>	<input type="checkbox"/>	2. Calibration is clearly documented:
<input type="checkbox"/>	<input type="checkbox"/>	a. Instrument is calibrated using a Blank and three Calibration Standards. The correlation coefficient must be >0.995.
<input type="checkbox"/>	<input type="checkbox"/>	b. Reanalysis of the top calibration standard as a sample. Control limits are 95 - 105%. (Run once daily prior to sample analysis).
<input type="checkbox"/>	<input type="checkbox"/>	3. Calibration Verification: (10% Frequency):
<input type="checkbox"/>	<input type="checkbox"/>	a. ICV/CCV: Std./CLP – Recovery 90-110% EPA 200.7 (ICV) – Recovery 95-105%
<input type="checkbox"/>	<input type="checkbox"/>	b. ICB/CCB: Std. QC: < RL; CLP QC: < CRDL; SW-846 QC: < 3x MDL. (custom criteria code)
<input type="checkbox"/>	<input type="checkbox"/>	4. CLP QC: An Initial & Final for each sample analysis run:
<input type="checkbox"/>	<input type="checkbox"/>	a. CRI - 2x RL; No Limit Set
<input type="checkbox"/>	<input type="checkbox"/>	b. ISA/ISAB - 80-120% Recovery
<input type="checkbox"/>	<input type="checkbox"/>	5. Std. QC: Analyzed at the beginning of the day and every 8 hours thereafter:
<input type="checkbox"/>	<input type="checkbox"/>	a. CRI: 2x CRDL; No Limit Set
<input type="checkbox"/>	<input type="checkbox"/>	b. ISA/ISAB: 80-120% Recovery
		Refer to Run #:

Note: CLP QC requires the use of the IDL for calculating % Recoveries and Reporting Limits.
 Standard QC requires the use of the RL for calculating % Recoveries and Reporting Limits.

II. Sample Analysis:

Analyst	Reviewer	
<input type="checkbox"/>	<input type="checkbox"/>	1. Each Prep Batch consists of a maximum of 20 samples of a similar matrix:
<input type="checkbox"/>	<input type="checkbox"/>	a. Prep Batches must be clearly identified
<input type="checkbox"/>	<input type="checkbox"/>	b. 1 Prep Blank CLP - < CRDL; Std. QC - < RL TCLP - < TCLP Reporting Limit
<input type="checkbox"/>	<input type="checkbox"/>	c. 1 LCS Std./CLP - 80-120% Rec.; EPA 200.7 - 85-115% Rec.
<input type="checkbox"/>	<input type="checkbox"/>	d. 1 Duplicate Std. - RPD or RSD limits are 20%; Unless the sample conc. is <5x RL then ± RL applies; for CLP + CRDL applies. EPA 200.7 - 10% Frequency
<input type="checkbox"/>	<input type="checkbox"/>	e. 1 Matrix Spike Std./CLP - 75-125% Rec.; Unless the sample conc. exceeds the spike conc. by 4x; EPA 200.7 - 70-130% Rec.; 10% Frequency
<input type="checkbox"/>	<input type="checkbox"/>	f. Analytical MS TCLP - >50% (MSA performed if <50% recovery)
<input type="checkbox"/>	<input type="checkbox"/>	g. Serial Dilution 1 per 20 samples; 10% Difference Limit
<input type="checkbox"/>	<input type="checkbox"/>	h. A post-digestion spike (PMS) must be performed for CLP (75-125%) and 200.7 (85-115%) if the above limits are not met, (CLP - except for Ag, Na, Ca, K, and Mg for waters and soils, and Al and Fe for soils only).
<input type="checkbox"/>	<input type="checkbox"/>	i. Turbidity Checked: EPA 200.7 Drinking Water (< 1 NTU; no prep required).

STL Chicago ICAP Metals Data Review Checklist

II. Sample Analysis (continued):

Analyst	Reviewer	
		2. A Corrective Action Report (CAR) must be written for any out of control situations, clearly stating the problem and action to be taken:
		a. CAR included with original data run
		b. CAR with corrective action results included with the corrective action run.

III. Data Documentation

Analyst	Reviewer	
		1. Raw Data:
		a. Unused data is clearly identified.
		b. All crossed out data is initialed and dated.
		c. Out of control QC is clearly identified.
		d. Any data that has a tick (S, I, H or L) is commented on with appropriate action taken.
		e. The first page of the run must have the filename; instrument; and analyst's signature
		2. Run Log:
		a. Unused data is clearly identified.
		b. All cross outs are initialed and dated.
		c. Analyst's Signature is required.
		3. LabNet:
		a. Worksheet and data pages are printed.
		b. Unused data is clearly identified.
		c. All cross-outs are initialed and dated.
		d. First page must have the filename, instrument identification; analyst signature.
		e. Samples needing copying are clearly marked.
		f. Label Sample ID with the LabNet Batch their in.

III. Miscellaneous

Analyst	Reviewer	
		1. Is Sample Prep Linked?
		2. Is TCLP Linked? (Shift F9 from the start page)
		3. Did all dilutions carry over for MD, MS, MSD (where applicable)?
		4. Did all prep and analysis matrices match up?

Comments:

Analyst Signature: _____ Date: _____

Reviewer Signature: _____ Date: _____

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Attachment 5.

Known Digested QC Values (mg/L)

Element	LCS/Spike	TCLP Spike
Al	2	---
Sb	0.5	---
As	0.1	5
Ba	2	100
Be	0.05	---
Bi	0.5	---
B	1	---
Cd	0.05	1
Ca	10	---
Cr	0.2	5
Co	0.5	---
Cu	0.25	0.25
Fe	1	---
Pb	0.10	5
Mg	10	---
Mn	0.5	---
Mo	1	---
Ni	0.5	0.5
P	0.5	---
K	10	---
Se	0.10	1
Si	5	---
Ag	0.05	1
Na	10	---
Sr	1	---
Tl	0.10	---
Sn	1	---
Ti	1	---
V	0.5	---
Zn	0.5	---

Default Control Limits

LCS: 80 - 120%

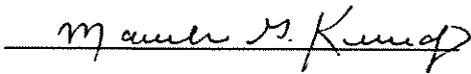
Spike: 75 - 125%

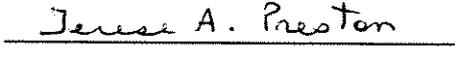
TCLP Spike: >50%

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**TITLE: QUALITY ASSURANCE
 Sample Discrepancy Reports / Resubmitted Data Reports /
 Corrective Action Reports (SDR / RDR / CAR)**

Reviewed by:	Signature:	Date
Marilyn J. Krueding Quality Assurance Specialist		9/23/04

Approved by:	Signature:	Date
Terese A. Preston Quality Manager		9/22/04

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1.0 SCOPE / APPLICATION

This Standard Operating Procedure (SOP) describes the laboratory process for responding to client complaints, sample-related discrepancies, and analytical non-conformances. The documentation process that is used to address the issue, its resolution, actions taken and final approval are described within this SOP. The documentation mechanism includes the following forms:

- 1) Sample Discrepancy Report (SDR)
- 2) Resubmitted Data Request (RDR)
- 3) Corrective Action Report (CAR).

This SOP provides instructions for the completion of these forms.

2.0 DISCUSSION

The SDR, RDR and CAR are communication vehicles for documenting events within the laboratory and decisions related to those events. This documentation provides the laboratory and client with information in which to recreate the situation and to understand the circumstances that led to its ultimate resolution. Review of these reports can be a diagnostic quality tool for measuring system or process performance.

An SDR is generated prior to the release of data to the client. It is applicable for any sample-related situation noted during sample receipt, sample analysis, and data reporting. The SDR is initiated either by the sample custodian, project manager (PM), data management personnel, section manager (SM), analyst, or Quality Assurance (QA) personnel. All information discrepancies associated with the sample Chain-of-Custody (COC) must be documented with an SDR and/or LabNet Job Note with reference made to the discrepancy on the sample receipt checklist.

The RDR is used after the client has received the analytical report and their specifications, expectations, and/or client satisfaction were not achieved. RDRs are prepared when:

- a client requests re-evaluation of already submitted data
- a client requests additional information originally omitted from data package
- a client complaint requires a formal laboratory response

The RDR is initiated either by the project management (PM) or data management personnel, however, section managers, Quality Assurance (QA) personnel or anyone with direct customer contact can initiate this process. An RDR may also be initiated by the laboratory when an error has been identified internally.

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The CAR is prepared prior to the reporting of the data for any quality-related non-conformance to requirements, excluding sample specific situations in which an SDR is completed. The CAR can be initiated by anyone, but is typically initiated by the analyst, section manager, or QA personnel. Typical 'non-conformance to requirements' documented with a CAR are:

- Analytical Quality Control criteria deviation
- Regulatory method deviation
- Laboratory policy or procedure deviation
- Certification/agency program requirement deviation
- Contract deviation

3.0 Sample Discrepancy Report

An SDR (Attachment 1), electronic SDR (U/Groups/Everyone/SDR_Template.doc), or email version (Attachment 1a) is initiated at the time the discrepancy is observed, prior to the release of sample data. The individual (Initiator) identifying the discrepancy completes the header information indicating the client, analyses, samples affected and project related information. The type of deficiency and an explanation of the details surrounding the deficiency are recorded. The SDR is then transferred to the project manager (PM) for coordinating or approving of the action plan. The PM may need to contact the client to further develop the action plan or to obtain client approval for the action plan recommended by the laboratory. Documentation of this client contact and approval process is required either on the SDR or on an attachment to the SDR (e-mail response or letter). If the PM is not available, submit the SDR to the QA department for assistance in formulating an action plan. It is critical that sufficient time be given to the PM to determine the best course of action to meet the client's needs. The laboratory must not lose sight of the ultimate 'end use' of the client's data. The SDR is composed of six (6) sections which are described further below.

3.1 Initiator

The initiator documents the SDR with his or her name, date of the occurrence, pertinent client, sample, and data deliverable information. The initiator, PM or data management section manager will complete the contact information for forwarding effective documents.

3.2 Type of Sample Discrepancy

In this section, the initiator typically checks off or describes the type of sample discrepancy and lists specific concerns. The initiator signs/dates this section of the SDR in recognition that all the information is complete and accurate to the best of their knowledge. Upon completion of this section, the PM is then given the SDR.

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3.3 PM Establish Action Plan

The PM, with assistance from any of the operating sections and or QA, will develop an action plan and indicate the steps for resolution of the discrepancy. The required personnel and specific actions to be taken are listed on the SDR. Data management personnel will route the SDR to the appropriate people indicated for action plan responsibilities. Upon completion of the requested task, each person will initial and date the actions taken and route the package to the next department for which action is required.

3.4 PM Final Approval

Once all actions are completed and before the analytical report is delivered to the client, the PM will review the SDR, assuring that all actions were completed. If all corrective actions are acceptable, the PM will sign the 'Final Approval' section. The SDR is scanned for inclusion in the client data report if PM indicates such routing. The electronic file is stored on the LAN (T:/RepGen/Sent/JobNumber.pdf or JobNumberMisc.pdf). The original SDR is then routed to the QA department.

3.5 Receipt of Copies

Sample Log-in or Data management personnel distribute copies of the SDR to those people indicated by the PM. All actions taken are to be documented on the original SDR. This routing occurs through the SM and PM signature baskets located in the data management section.

3.6 Quality Assurance (QA)

All completed SDRs are routed to the QA department after scanning and placement in its respective Job folder. SDRs are periodically reviewed by QA personnel to help identify systematic or recurring problems. Once identified, goals are established with the objective of reducing or eliminating the problem. To accomplish this, action plans are developed by the QA personnel in cooperation with the laboratory staff. Implementation is scheduled and monitoring to determine if the action taken was effective in resolving the problem. This activity would be listed as a 'preventive action measure' or 'Quality System improvement item', which is summarized in the monthly QA Reports. Any SDR involving missed holding times (HTs) are, on a monthly basis, evaluated, and the information entered onto a missed HT tracking spreadsheet. This information is tabulated and summarized for inclusion in the monthly QA Reports. (U/Groups/Everyone/Holdtime.xls)

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4.0 RESUBMITTED DATA REQUEST (RDRs)

RDRs are initiated for client requested review of data, requests for additional information or client complaints, which require a formal response (Attachment 2). The individual receiving the request completes the header information indicating the client samples affected and project related information. The type of deficiency and an explanation of the details are recorded. An action plan must be determined and clearly defined on the RDR. Depending on the nature of the problem, consultation with other sections such as project management, QA, data management, accounting, or operating sections may be needed to develop and approve an action plan. The RDR is composed of seven (7) sections, which are described further below.

An RDR may also take the form of an e-mail, (preferably the formatted electronic RDR - Attachment 2a) The request is typically initiated via e-mail by the client or data validator to the PM requesting a correction, additional information or clarification of data that was submitted. As with a paper RDR, an action plan must be determined and routed to appropriate personnel for completion. It is critical that the electronic request for data correction clearly outlines the actions required and taken by the laboratory just as if the RDR Form were used.

An RDR tracking number must be assigned to each RDR at the time that it is initiated. The RDR Tracking sheet is located in the All Public directory on the LAN. (U\Groups\Everyone\RDR_Tracking.xls) The initiator of the RDR assigns the next available tracking number to the RDR prior to routing it for completion. All completed RDR's are turned into the data management section leader, who on a monthly basis insures that the RDR Tracking table is complete, and that a completion is date entered on the spreadsheet. A check is also performed to ensure that a pdf file exists for the each RDR located in its associated job number folder on the LAN (T\Groups\RepGen\Sent\Job NumberREV_.pdf). The revision and number is appended to the job number.

4.1 Initiator

The initiator documents the RDR with his/her name, date of the occurrence, pertinent client, sample, and data deliverable information and assigns a tracking number to it. The initiator, PM or data management section manager will complete the contact information for forwarding the completed response.

4.2 Type of Data Deficiency

In this section, the initiator describes the type of data deficiency and lists specific concerns in the 'Explanation of Details' section. The initiator signs/dates this section of the RDR to indicate who initiated it and recognition that all the information is complete and accurate to the best of their knowledge.

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4.3 Action Plan

This section documents the 'action plan' developed by the PM, reporting staff, section manager, or QA for the deficiency previously identified in detail. The necessary personnel and detailed actions to be taken are listed. Upon completion of the requested task, each person will initial and date this section, attach any corrected item, and route it to the next person listed on the action plan. Please note that if the corrected item is to replace a page from the original report, the page number it is replacing needs to be written on the bottom right hand corner of the page. If it is to be added as an additional page, the page it is to follow in the original report is added to the page with a letter designation starting with 'A', 'B', etc. for as many pages as is necessary.

4.3.1 Data Management

The RDR tracking number is used to gauge the progress and turnaround time of the designated action plan. This tracking list is maintained by the data management section, project management and QA and is available to all lab personnel on the laboratory's LAN. (U:/Groups/Everyone/RDR Tracking.xls) (Attachment 2b)

The data management section will assist in the routing of the RDR, which occurs through the Section Manager and PM signature baskets located in the data management section. They will also facilitate the submittal of the final product to the client and the documentation within LabNet how the data was submitted to the client in the form of Job Notes.

4.3.2 Project Manager (PM)

Once all actions are completed and before the report correction or other subsequent actions items are delivered to the client, the PM for the associated project will review the RDR, assuring that all actions were completed and then, if acceptable, will sign the 'Final Approval' section. The RDR and subsequent corrections are then scanned and stored electronically on the LAN (T:/RepGen/Sent/Job NumberREV_.pdf) with the identification of Job number followed by revision number.

4.3.3 Quality Assurance (QA)

Completed RDRs are routed to the QA department. RDRs are reviewed on a monthly basis. A summary and details of this review is included in the monthly QA report and is used to help resolve systematic or recurring problems. Quality goals can be established, action plans developed and implemented for the vital few recurring or systemic problems. This problem identification process and corrective action planning is identical to that outlined in section 3.6 for the SDRs.

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5.0 CORRECTIVE ACTION REPORTS (CARs)

CARs are prepared for any quality related non-conformance to requirements other than sample specific client requirements, which should be addressed with an SDR. When CARs are initiated by the analyst or section manager, the form typically follows the example identified in Attachment 3. However, to expedite reporting, each laboratory section has specific formats to meet their needs and that contain the four basic reporting requirements.

CARs may be produced in a tabular or a custom format to deal with audit/assessment-identified non-conformance, or for other situations which identify more than a single non-conformance item. All corrective action reports will consist of four (4) basic elements. These are described further below.

5.1 Initiator

The initiator documents the CAR with his or her name, date of the occurrence, associated method or application and the client data that is directly affected by the non-conformance.

5.2 Description of the Non-conformance

The initiator details the type of non-conformance; lists specific concerns; and signs/dates this section of the CAR. This signature indicates that all the information is complete and accurate to the best of their knowledge. However, if the information is transmitted via e-mail, memo, or other format (i.e., tabular or audit report) indicating the initiator/date, then this information is sufficient and a signature is not required. The CAR may be routed for the development of an action plan to their immediate supervisor, when the situation is such that no clear guidance is listed in the associated method SOP.

5.3 Action Steps to Resolve the Non-Conformance

This section documents the "action plan" developed for the discrepancy or in the case of a request for response, such as an audit, this space may be blank and requested to be completed by an appropriate party. After resolution of the issue, the appropriate person will sign their response. However, if the information is transmitted via e-mail, memo, or other format (i.e., tabular or audit report) indicating the initiator/date, then this information is sufficient and a signature is not required.

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5.4 Acknowledgment of Return-to-Control

In the case of an individual non-conformance, after all actions are complete, the analyst or supervisor will review the actions and document the return-to-control. If return-to-control has not been demonstrated, an SDR may be necessary to obtain further corrective action to meet specific project requirements. If the issue is a multiple non-conformance such as an audit report, the QA section or appropriate witness will assess the situation to assure that the corrective action has been effectively implemented.

6.0 PREVENTIVE ACTION

The quality assurance section uses various sources of information to determine preventive action measures. Proficiency Testing (PT) studies, CARs, SDRs, RDRs, internal and external audit reports, and other quality records are reviewed. Vital issues, suitable for preventive action recourse, can be determined by any laboratory staff member and submitted to the QA section.

The QA section summarizes vital issues in the monthly QA report. Potential corrective action steps are determined for the various vital issues and resources are directed to those issues which directly impact the quality of analytical data or laboratory services. The monthly quality assurance report is distributed and submitted to laboratory management for review and acknowledgment of corrective and preventive measures to be taken.

The monthly QA report is also an information vehicle used to document the effectiveness of corrective actions, status of tasks and new vital issues. This vehicle provides a continuous improvement loop to ensure that the application of actions and controls are effective. Also, it implicitly enacts a plan-do-check-act process loop with regard to preventive as well as corrective actions.

Since monthly QA reports, as well as the preventive information sources, are subsequently reviewed and summarized in the Quality Systems Management Review (UQA-002), the ultimate effectiveness of the preventive action process is again reviewed on a broader time scale.

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7.0 ATTACHMENTS

Attachment 1: Example: Sample Discrepancy Report (SDR) Form
Attachment 1a: Example: Electronic (e-mail) SDR
Attachment 2: Example: Resubmitted Data Request (RDR) Form
Attachment 2a: Example: Electronic (e-mail) RDR
Attachment 2b: Example: RDR Tracking Sheet
Attachment 3: Example: Corrective Action Report (CAR) Form

Historical File: Revision 00: 12/12/94 Revision 06: 04/02/02
 Revision 01: 04/10/96 Revision 07: 09/25/03
 Revision 02: 05/16/96 Revision 08: 09/22/04
 Revision 03: 02/24/98
 Revision 04: 07/19/99
 Revision 05: 08/07/00

Reasons for Change: Revision 08:

- Annual Review - Update to clarify the use of LabNet Job Notes and the Sample Receipt Checklist for documenting sample discrepancies upon receipt of the samples at the laboratory.
- Clarification of the process used to evaluate and develop action plans for SDRs and RDRs that appear to be systematic or recurring errors.

U:\QC\SOP\QA\SOP-029.DOC

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Attachment 1.

Example: Sample Discrepancy Report (SDR) Form

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Attachment 1a.

Electronic (e-mail) Sample Discrepancy Report (SDR)

Sample Discrepancy Report (SDR)

Client:
Contact:
Project:
Job:
Date:

Deficiency/Discrepancy:

Action Plan:

•

Richard Wright
Project Manager
STL Chicago
2417 Bond Street
University Park, IL 60466
708-534-5200
708-534-5211 fax
rwright@stl-inc.com

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Attachment 2.

Example: Resubmitted Data Request (RDR) Form

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Attachment 2a.

**Example:
Electronic (e-mail) Resubmitted Data Request (RDR) Format**

Resubmitted Data Request (RDR)

Client:
Contact:
Project:
Job:
RDR #:
Date:

Analysis in Question:
Explain Problem:

Date CA Needed:

Action Plan:

- Include whether changes in results should be PDF'd, HC sent, EDD updated.

*Richard Wright, Project Manager, STL Chicago, 2417 Bond Street, University Park, IL 60466
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Attachment 2b.

Example: Resubmitted Data Request (RDR) Tracking Summary

RDR Tracking Form-Active

Required Actions:	Check	Initials/Date	Additional Comments
Raw Data re-submittal Required			
Reprint Report			
Repint QC Report			
Revise Case Narrative			
Revise EDD			

RDR Tracking #	Date Initiated	Client	PM	Job Number	Unit of Error	Description of Error	Indicate (x) How Correction is to be Sent					Requested Due Date	Completion Date
							PDF	FAX	Hardcopy	EDD	Other		
1926													

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Attachment 3.

Example: Corrective Action Report (CAR) Form



LABORATORY QUALITY MANUAL

STL Chicago
 2417 Bond Street
 University Park, Illinois 60466-3182
 (708) 534-5200

Approved by (Signature / Date):

Michael J. Healy

 Michael J. Healy
 Laboratory Director

Terese A. Preston 6/4/04

 Terese A. Preston
 Quality Assurance Manager

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STL

Vision

STL will be the recognized industry leader for environmental analysis.



Mission

Through the innovation and dedication of our people, together with the quality of our systems, we will deliver levels of performance that delight our clients, retain the confidence of our stakeholders and enable the profitable growth of our business.

Severn Trent Laboratories

1.0 Introduction, Purpose, and Scope

1.1 STL Overview

STL Chicago (STL) is a part of Severn Trent Laboratories, a major group of U.S. based companies. The companies are owned by Severn Trent, plc, an international provider of water and wastewater services headquartered in Birmingham, UK.

STL is a full-service environmental laboratory that provides quality comprehensive and integrated professional analytical services effectively and efficiently. A broad range of environmental testing services are offered that span a variety of matrices including aqueous, saline, solid, tissue and drinking water.

Associated with this activity are services to assure client requirements are known, communicated and satisfactorily addressed, and a deliverables package presenting the analytical results. The laboratory provides expert personnel for supervision, technical consultation, and project review for effective planning and implementation of analytical assignments.

STL operates under the regulations and guidelines of the following federal programs:

- ◆ Air Force Center for Environmental Excellence (AFCEE)
- ◆ US Army Corp of Engineers, Hazardous, Toxic and Radioactive Waste (USACE HTRW)
- ◆ Clean Water Act (CWA)
- ◆ Comprehensive Environmental Response, Compensation, and Liability Act (CERCLA)
- ◆ Navy Facilities Engineering Service Center (NFESC)
- ◆ National Pollution, Discharge, and Elimination System (NPDES)
- ◆ Occupational Safety and Health Administration (OSHA)
- ◆ Resource Conservation and Recovery Act (RCRA)
- ◆ Safe Drinking Water Act (SDWA)
- ◆ Toxic Substances Control Act (TSCA)

STL also provides services under various state and local municipal guidelines. A current table of analytical services, list of certifications and general service listing is presented on the MySTL webpage or available from the laboratory. www.stl-inc.com

1.2 Quality Assurance Policy

It is STL's policy to:

- ◆ Provide high quality, consistent, and objective environmental testing services that meet all federal, state, and municipal regulatory requirements.
- ◆ Generate data that are scientifically sound, legally defensible, meet project objectives, and are appropriate for their intended use.
- ◆ Provide STL clients with the highest level of professionalism and the best service practices in the industry.
- ◆ Build continuous improvement mechanisms into all laboratory, administrative, and managerial activities.
- ◆ Maintain a working environment that fosters open communication with both clients and staff and ensures data integrity.

1.3 Management Commitment to Quality Assurance

STL management is committed to providing the highest quality data and the best service in the environmental testing industry. To ensure that the data produced and reported by STL meet the requirements of its clients and comply with the letter and spirit of municipal, state and federal regulations, STL maintains a quality system that is clear, effective, well communicated, and supported at all levels in the company.

Line organizations verify that specifications are achieved; QA organizations assist and provide oversight and verification of processes through planning, reviews, audits, and surveillances. The quality objectives are derived from this Laboratory Quality Manual (LQM), Standard Operating Procedures (SOPs) and Work Instructions.

1.4 Purpose

The purpose of the LQM is to describe STL's Quality System and to outline how that system enables all employees to meet the Quality Assurance (QA) policy. This LQM also describes specific QA activities and requirements and prescribes their frequencies. Roles and responsibilities of management and laboratory staff in support of the Quality System are also defined in this LQM.

1.5 Scope

This LQM is specific to STL Chicago's quality systems and laboratory operation's. All other STL locations have LQMs under the Corporate Quality Management Plan (QMP) or the Corporate QMP itself.

The laboratory is committed to ensuring that resources are available and deployed to meet client expectations. This includes gathering project information prior to sample receipt to ensure client expectations will be met with respect to:

- ◆ Sampling containers;
- ◆ Analytical methods employed;

- ◆ Accuracy and precision;
- ◆ Reporting limits;
- ◆ Personnel qualifications, training, and experience;
- ◆ Calibration and quality control measures employed;
- ◆ Regulatory requirements;
- ◆ Report contents;
- ◆ Supporting documentation, records and evidence; and
- ◆ Review of data

1.6 Servicing

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

The laboratory has established procedures for performing and verifying that client servicing meets requirements. Typical services provided are:

- ◆ Sample Containers/Supplies – *Container Management: Process Operation* (UCM-001)
- ◆ Project QAP preparation – *Project Planning Process* (UPM-003)
- ◆ Regulatory advisory functions – *Project Planning Process* (UPM-003)
- ◆ Consulting – *Project Planning Process* (UPM-003)

Regulatory and advisory functions are addressed under the same procedures used for project planning.

2.0 References

The following references were used in preparation of this document and as the basis of the STL Quality System:

EPA Guidance for Preparing Standard Operating Procedures (SOPs), EPA QA/G-6, US EPA, Office of Environmental Information, EPA/240/B-01/004, March 2001.

EPA Requirements for Quality Management Plans, EPA QA/R-2, US EPA, Office of Environmental Information, EPA/240,B-01/002 March 2001.

EPA Requirements for Quality Assurance Project Plans, EPA QA/R-5, US EPA, Office of Environmental Information, EPA/240/B-01/003, March 2001.

EPA Quality Manual for Environmental Programs, 5360 A1, US EPA Office of Environmental Information – Quality Staff, May 2000.

General Requirements for the Competence of Testing and Calibration Laboratories, ISO/IEC 17025, December 1999.

Good Automated Laboratory Practices, Principles and Guidance to Regulations for Ensuring Data Integrity in Automated Laboratory Operations with Implementation Guidance, EPA 2185, US EPA Office of Information Resources Management, August 1995.

Air Force Center for Environmental Excellence (AFCEE) Quality Assurance Project Plan (QAPP), Version 3.1, August 2001.

National Environmental Laboratory Accreditation Conference, Constitution, Bylaws, and Standards, EPA 600/R-00/084, US EPA Office of Research and Development, June 2000.

Navy Installation Restoration Laboratory Quality Assurance Guide, Interim Guidance Document, Naval Facilities Engineering Service Center (NFESC), February 1996.

Navy Installation Restoration Chemical Data Quality Manual, Navy IR CDQM, Special Publication SP-2056-ENV, September 1999.

Department of Defense Quality Systems Manual for Environmental Laboratories, Version 1, October 2000.

Shell for Analytical Chemistry Requirements, US Army Corps of Engineers, EM 200-1-3, Appendix I, February 2001

This LQM was written to comply with the National Environmental Laboratory Accreditation Conference (NELAC) standards. Refer to Table 1 for a cross-section comparison of this LQM to the NELAC standards.

Table 1.

Correlation of QAPP Sections with NELAC 5.5.2 Quality Manual Requirements

NELAC Chapter 5.5.2 Quality Manual	Laboratory Quality Manual Section
a. Quality policy statement, including objectives and commitments	1.2 Quality Assurance Policy 4.2.1 Objectives of the Quality System
b. Organization and management structure	4.1 Organization and Management
c. Relationship between management, technical operations, support services and the quality systems	4.1.2 Roles and Requirements 4.2 Quality System
d. Records retention procedures; document control procedures	4.3 Document Control 4.12.2 Record Retention
e. Job descriptions of key staff and references to job descriptions of other staff	4.1.2 Roles and Requirements
f. Identification of laboratory approved signatories	4.1 Organization and Management
g. Procedures for achieving traceability of measurements	5.5 Measurement Traceability
h. List of all test methods under which the laboratory performs its accredited testing	5.3.1 Method Selection
i. Mechanisms for assuring the laboratory reviews all new work to ensure that it has the appropriate facilities and resources before commencing such work	4.4.2 Project-Specific Quality Planning

Table 1.

Correlation of QAPP Sections with NELAC 5.5.2 Quality Manual Requirements

NELAC Chapter 5.5.2 Quality Manual	Laboratory Quality Manual Section
j. Reference to the calibration and/or verification test procedures used	5.3.4 Method Verification 5.3.5 Method Validation & Verification Activities 5.3.6 Data Reduction & Review 5.4.3 Equipment Verification and Calibration
k. Procedures for handling submitted samples	4.7.1 Sample Acceptance Policy 5.7 Sample Handling, Transport and Storage
l. Reference to the major equipment and reference measurement standards used as well as the facilities and services used in conducting tests	1.6 Servicing 4.1.1 Laboratory Facilities 4.6 Purchasing Services & Supplies 5.2 Facilities 5.4.2 Equipment Maintenance 5.4.3 Equipment Verification and Calibration
m. Reference to procedures for calibration, verification and maintenance of equipment	5.4.2 Equipment Maintenance 5.4.3 Equipment Verification and Calibration
n. Reference to verification practices including inter-laboratory comparisons, proficiency testing programs, use of reference materials and internal QC schemes	5.8.1 Proficiency Testing 5.8.2 Control Samples
o. Procedures for feedback and corrective action whenever testing discrepancies are detected, or departures from documented procedures occur	4.8 Complaints 4.9 Control of Non-Conformances 4.10 Corrective Action 4.11 Preventive Action 5.8.6 Permitting Departures from Documented Procedures
p. Laboratory management arrangements for exceptionally permitting departures from documented policies and procedures	4.4.1 Contract Review 4.4.2 Project-Specific Quality Planning 5.8.6 Permitting Departures from Documented Procedures
q. Procedures for dealing with complaints	4.8 Complaints
r. Procedures for protecting confidentiality and proprietary rights	4.7.2 Client Confidentiality and Proprietary Rights
s. Procedures for audits and data review	4.13 Internal Audits 4.14 External Audits 5.3.6 Data Reduction and Review
t. Process/procedures for establishing that personnel are adequately experienced in duties they are expected to carry out and are receiving any needed training	5.1.2 Training
u. Ethics policy statement developed by the laboratory and training personnel in their ethical & legal responsibilities	5.1.3 Ethics Policy
v. Reference to procedures for reporting analytical results	5.3 Test Methods 5.3.6 Data Reduction and Review 5.9 Project Reports
w. Table of contents, listing reference, glossaries and appendices	TOC Table of Contents Appendix List of Cited SOPs and Work Instructions

3.0 Terms and Definitions

Accuracy: The degree of agreement between a measurement and true or expected value, or between the average of a number of measurements and the true or expected value.

Audit: A systematic evaluation to determine the conformance to specifications of an operational function or activity.

Batch: Environmental samples, which are prepared and/or analyzed together with the same process, using the same lot(s) of reagents. A preparation batch is composed of 1 to 20 environmental samples of a similar matrix, meeting the above mentioned criteria. Where no preparation method exists (e.g., volatile organics, water), the batch is defined as environmental samples that are analyzed together with the same process and personnel, using the same lots of reagents, not to exceed 20 environmental samples. An analytical batch is composed of prepared environmental samples, extracts, digestates or concentrates that are analyzed together as a group. An analytical batch can include prepared samples originating from various environmental matrices and can exceed 20 samples.

Chain of Custody (COC): A system of documentation demonstrating the physical possession and traceability of samples.

Comprehensive Environmental Response, Compensation and Liability Act (CERCLA/Superfund): Legislation (42 U.S.C. 9601-9675 et seq., as amended by the Superfund Amendments and reauthorization Act of 1986 (SARA), 42 U.S.C. 9601 et seq.

Compromised Sample: A sample received in a condition that jeopardizes the integrity of the results. See Section 4.7.1 for a description of these conditions.

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.

Confirmation: Verification of the presence of a component using an additional analytical technique. These may include second column confirmation, alternate wavelength, derivatization, mass spectral interpretation, alternative detectors, or additional cleanup procedures.

Corrective Action: Action taken to eliminate the causes of an existing non-conformance, defect or other undesirable situation in order to prevent recurrence.

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.

Demonstration of Capability (DOC): Procedure to establish the ability to generate acceptable accuracy and precision.

Detection Limit Check Standard (DLCK): A non-processed standard spiked at approximately ½ the method reporting limit. Used in conjunction with the MRL Check standard in LGC analysis.

Equipment Blank (EB): A portion of the final rinse water used after decontamination of field equipment; also referred to as Rinsate Blank and Equipment Rinsate.

Extraction Blank (EB1, EB2, EB3): A blank that has been taken through the extraction procedure such as TCLP/SPLP; 5035, AVS/SEM.

Document Control: The act of ensuring that documents (electronic or hardcopy and revisions thereto) are proposed, reviewed for accuracy, approved for release by authorized personnel, distributed properly and controlled to ensure use of the correct version at the location where the prescribed activity is performed.

Federal Insecticide, Fungicide and Rodenticide Act (FIFRA): Legislation under 7 U.S.C. 135 et seq., as amended.

Federal Water Pollution Control Act (Clean Water Act, CWA): Legislation under 33 U.S.C. 1251 et seq., Public Law 92-50086 Stat. 816.

Field Blank (FB): A blank matrix brought to the field and exposed to field environmental conditions.

Field Duplicate (FD): Duplicate field-collected sample.

Field of Testing (FOT): A field of testing is based on NELAC's categorization of accreditation based on program, matrix and analyte.

Good Laboratory Practices (GLP): Formal regulations for performing basic laboratory operations outlined in 40 CFR Part 160 and 40 CFR Part 729 and required for activities performed under FIFRA and TSCA.

Holding Time: The maximum time that a sample may be held before preparation and/or analysis as promulgated by regulation or as specified in a test method.

Instrument Blank: A blank matrix that is the same as the processed sample matrix (e.g. extract, digestate, condensate) and introduced onto the instrument for analysis.

Internal Chain of Custody (COC): An unbroken trail of accountability that ensures the physical security of samples, data and records. Internal COC refers to additional documentation procedures implemented within the laboratory that includes special sample storage requirements, and documentation of all signatures and/or initials, dates, and times of personnel handling specific samples or sample aliquots.

Instrument Detection Limit (IDL): The minimum amount of a substance that can be measured with a specified degree of confidence that the amount is greater than zero using a specific instrument. The IDL is associated with the instrumental portion of a specific method only, and sample preparation steps are not considered in its derivation. The IDL is a statistical estimation at a specified confidence interval of the concentration at which the relative uncertainty is $\pm 100\%$. The IDL represents a range where qualitative detection occurs on a specific instrument. Quantitative results are not produced in this range.

Laboratory Control Sample (LCS): A blank matrix spiked with a known amount of analyte(s), processed simultaneously with, and under the same conditions as, samples through all steps of the analytical procedure.

Laboratory Quality Manual (LQM): A document stating the quality policy, quality system and quality practices of the laboratory. The LQM may include by reference other documentation relating to the laboratory's quality system.

Limit of Detection (LOD): The minimum amount of a substance that an analytical process can reliably detect.

Matrix: The substrate of a test sample. Common matrix descriptions are defined in Table 2.

Table 2. Matrix Descriptions

Matrix	Description
Aqueous	Aqueous sample excluded from the definition of Drinking Water or Saline/Estuarine source. Includes surface water, groundwater, effluents, leachates and wastewaters.
Drinking Water	Aqueous sample that has been designated a potable water source.
Saline	Aqueous sample from an ocean or estuary, or other salt-water source such as the Great Salt Lake.
Liquid	Liquid with <15% settleable solids.
Solid	Soil, sediment, sludge, ash, paint chips, filters, wipes or other matrices with >15% settleable solids.
Waste	A product or by-product of an industrial process that results in a matrix not previously defined (i.e., drum liquid or oils).
Tissue	Sample of a biological origin such as fish tissue, shellfish, or plant material. Such samples shall be grouped according to origin.

Matrix Duplicate (MD): Duplicate aliquot of a sample processed and analyzed independently; under the same laboratory conditions; also referred to as Sample Duplicate; Laboratory Duplicate.

Matrix Spike (MS): Field sample to which a known amount of target analyte(s) is added.

Matrix Spike Duplicate (MSD): A replicate matrix spike.

Method Blank (MB): A blank matrix processed simultaneously with, and under the same conditions as, samples through all steps of the analytical procedure.

Method Detection Limit (MDL): 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 measurement system. The MDL is a statistical estimation at a specified confidence interval of the concentration at

which the relative uncertainty is $\pm 100\%$. The MDL represents a range where qualitative detection occurs using a specific method. Quantitative results are not produced in this range.

Method Detection Limit Check (MDLCK): A standard that is processed with the MDL Study that is spiked at approximately $\frac{1}{2}$ the low standard or reporting limit in the method.

Method Reporting Limit Check (MRL): A standard that is not processed, is spiked at approximately 2x the low standard or reporting limit. This standard check is used in conjunction with the LCG analysis.

Non-conformance: An indication, judgment, or state of not having met the requirements of the relevant specifications, contract, or regulation.

Precision: An estimate of variability. It is an estimate of agreement among individual measurements of the same physical or chemical property, under prescribed similar conditions.

Preservation: Refrigeration and/or reagents added at the time of sample collection to maintain the chemical, physical and/or biological integrity of the sample.

Proficiency Testing: Determination of the laboratory calibration or testing performance by means of inter-laboratory comparisons.

Proficiency Test (PT) Sample: A sample, the composition of which is unknown to the analyst, that is provided to test whether the analyst/laboratory can produce analytical results within specified performance limits. Also referred to as Performance Evaluation (PE) Sample.

Proprietary: Belonging to a private person or company.

Quality Assurance (QA): An integrated system of activities involving planning, quality control, quality assessment, reporting and quality improvement to ensure that a product or service meets defined standards of quality with a stated level of confidence.

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.

Quality Control (QC): The overall system of technical activities, the purpose of which is to measure and control the quality of a product or service.

Quality Control (QC) Sample: A control sample, generated at the laboratory or in the field, or obtained from an independent source, used to monitor a specific element in the sampling and/or testing process.

Quality Management Plan (QMP): A formal document describing the management policies, objectives, principles, organizational authority, responsibilities, accountability, and implementation plan of an agency, organization or laboratory to ensure the quality of its product and the utility of the product to its users.

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/QC.

Quantitation Limit (QL): The minimum amount of a substance that can be quantitatively measured with a specified degree of confidence and within the accuracy and precision guidelines of a specific measurement system. The QL can be based on the MDL, and is generally calculated as 3-5 times the MDL, however, there are analytical techniques and methods where this relationship is not applicable. Also referred to as Practical Quantitation Level (PQL), Estimated Quantitation Level (EQL), Limit of Quantitation (LOQ).

Raw Data: Any original information from a measurement activity or study recorded in laboratory notebooks, worksheets, records, memoranda, notes, or exact copies thereof and that are necessary for the reconstruction and evaluation of the report of the activity or study. Raw data may include photography, microfilm or microfiche copies, computer printouts, magnetic/optical media, including dictated observations, and recorded data from automated instruments. Reports specifying inclusion of "raw data" do not need all of the above included, but sufficient information to create the reported data.

Record Retention: The systematic collection, indexing and storing of documented information under secure conditions.

Reference Standard: A standard, generally of the highest metrological quality, available at a given location from which measurements made at that location are derived.

Reporting Limit (RL): The level to which data is reported for a specific test method and/or sample. The RL is generally related to the QL. The RL must be minimally at or above the MDL.

Resource Conservation and Recovery Act (RCRA): Legislation under 42 U.S.C. 321 et seq. (1976).

Safe Drinking Water Act (SDWA): Legislation under 42 U.S.C. 300f et seq. (1974), Public Law 93-523.

Sampling and Analysis Plan (SAP): A formal document describing the detailed sampling and analysis procedures for a specific project.

Selectivity: The capability of a measurement system to respond to a target substance or constituent.

Sensitivity: The difference in the amount or concentration of a substance that corresponds to the smallest difference in a response in a measurement system using a certain probability level.

Spike: A known amount of an analyte added to a blank, sample or sub-sample.

Standard Operating Procedure (SOP): A written document which details the method of an operation, analysis or action whose techniques and procedures are thoroughly prescribed and which is accepted as the method for performing certain routine or repetitive tasks.

Storage Blank: A blank matrix stored (2-weeks) with field samples of a similar matrix (volatiles only) that measures storage contribution to any source of contamination. OR A blank matrix stored with field samples of a similar matrix.

Systems Audit: A thorough, systematic, on-site, qualitative review of the facilities, equipment, personnel, training, procedures, record keeping, data validation, data management, and reporting aspects of a total measurement system.

Test Method: Defined technical procedure for performing a test.

Toxic Substances Control Act (TSCA): Legislation under 15 U.S.C. 2601 et seq., (1976).

Traceability: The property of a result of a measurement that can be related to appropriate international or national standards through an unbroken chain of comparisons.

Trip Blank (TB): 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.

Verification: Confirmation by examination and provision of evidence against specified requirements.

4.0 Management Requirements

The organizational chart of STL is presented in Figure 1. Corporate employees are located at various STL facilities as outlined in the organizational structure. The organizational chart of STL Chicago is presented in Figure 2.

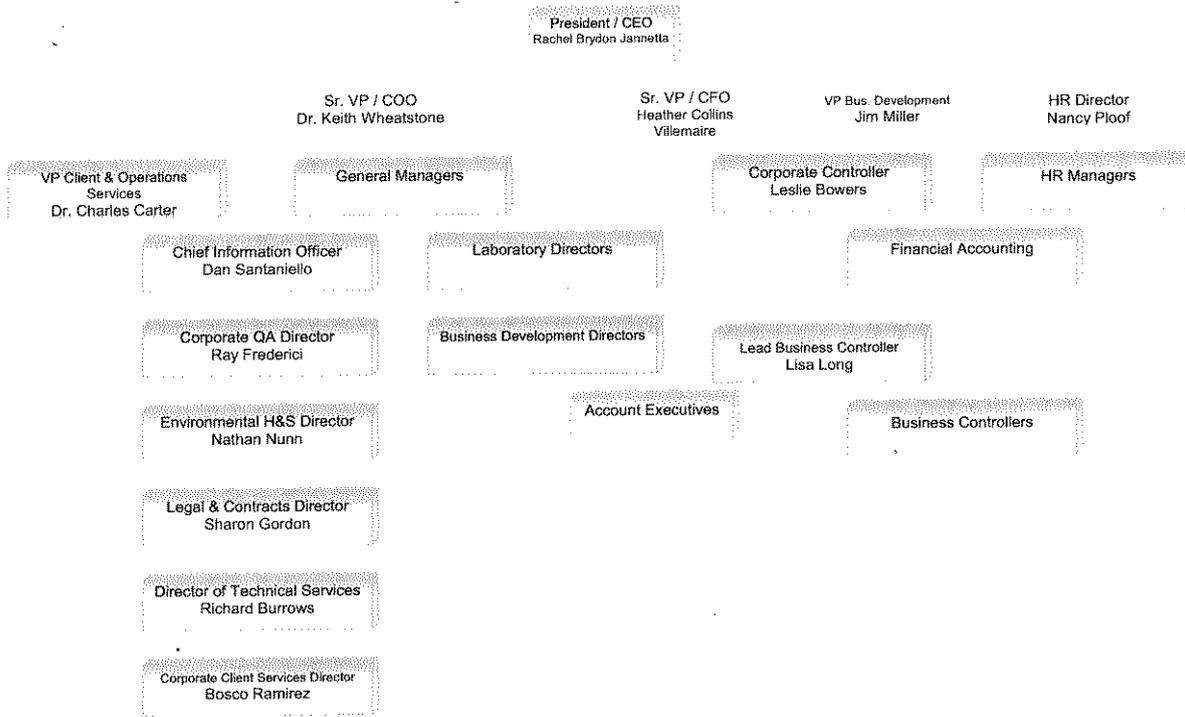
4.1 Organization and Management

The Laboratory Director and Quality Assurance Manager are responsible and have the signature authority for approving and implementing this plan. Additional signatory authorities for the approval of work and release of reports are defined in the *Signature Authority SOP* (UQA-030).



STL

Severn Trent Laboratories, Inc.
Management Structure
January 1, 2004



COMPANY CONFIDENTIAL AND PROPRIETARY

Figure 1. STL Organization Chart

STL Chicago Laboratory Quality Manual

UQA-LQM

Revision No. : 03

Revision Date: 06/03/2004

Effective Date: 06/07/2004

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STL Chicago Operations

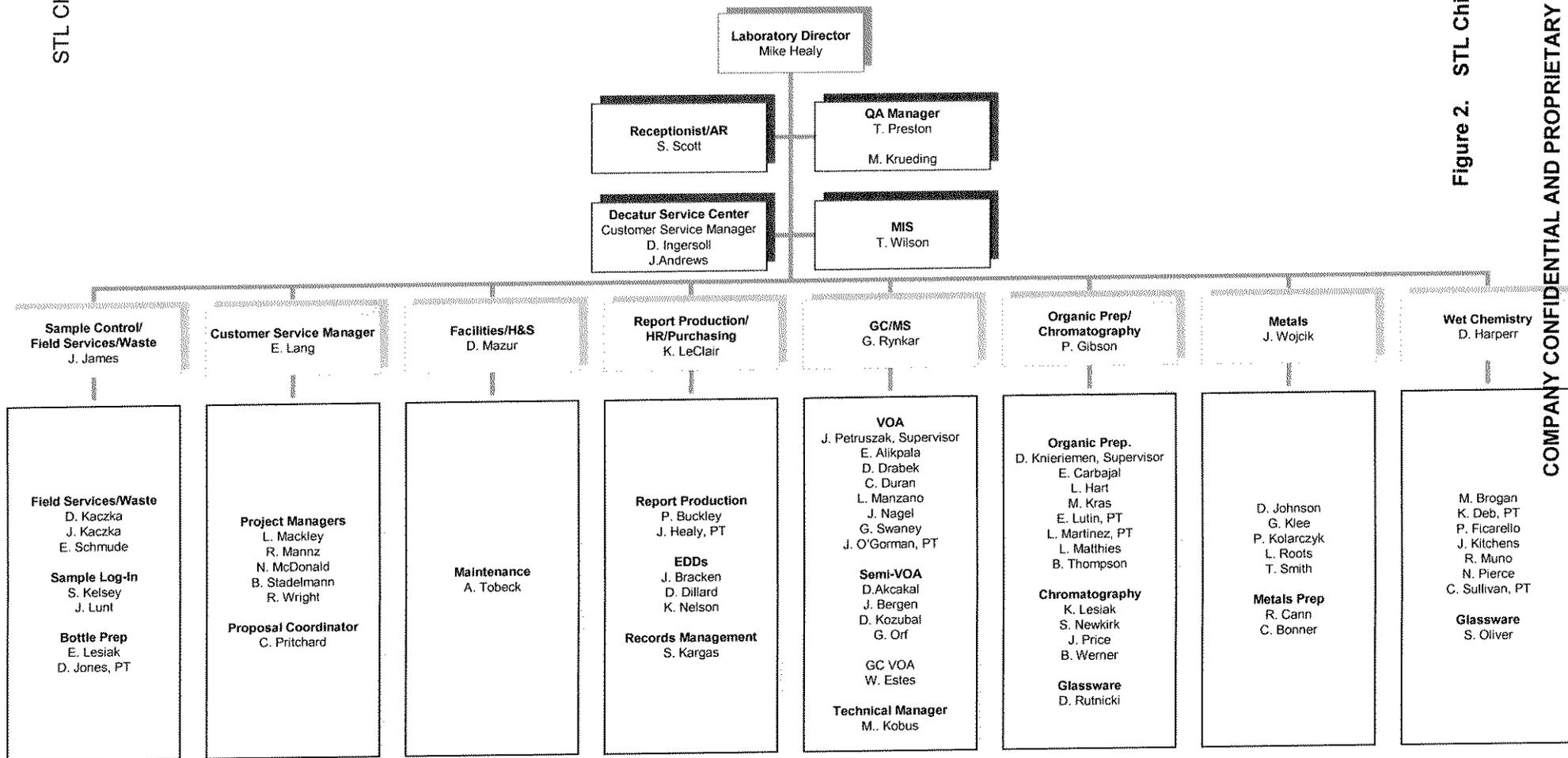


Figure 2. STL Chicago Organizational Chart

COMPANY CONFIDENTIAL AND PROPRIETARY

4.1.1 Laboratory Facilities

The laboratory is located in University Park, IL, which is approximately 30 miles south of Chicago, and is staffed by 84 professionals. The laboratory is comprised of 51,000 square feet of state-of-the-art commercial laboratory and office space and houses both inorganic and organic operations. The facility is divided into separate work areas to facilitate sample throughput. These areas include the following:

- ◆ Sample receipt and refrigerated storage
- ◆ Organic sample preparation
- ◆ Glassware preparation
- ◆ Metals digestion
- ◆ Wet chemistry laboratory
- ◆ Instrumentation laboratories

The main instrumentation laboratory is equipped with state-of-the-art instrumentation and sufficient duplicate equipment to provide back-up service for most major systems. A listing of laboratory equipment and instrumentation is referenced as Work Instruction No. CHI-22-09-103. Table 3 is a summary of the major laboratory instruments.

Table 3. Major Equipment List

GC	GC/MS	AA	ICP	CVAA	HPLC	AutoAnalyzer	IC	TOC	TOX
15	14	3	3	2	6	2	2	2	2

Each of these areas has separate heating, ventilation, and air conditioning systems. Non-destructive gas chromatographic detectors and GC/MS rotary pumps are vented out of the instrumentation through charcoal filters.

4.1.2 Roles and Responsibilities

The specific duties and responsibilities of the Laboratory Director, Quality Assurance Manager, Project Managers, Technical Managers, Sample Management Coordination, Data Management Section Manager, Quality Assurance Specialist, Health and Safety Coordinator/Waste Management, Information Technology Manager, and Chemists/Technicians are as follows.

In the absence of any one individual, the staff or assistant within each department is professionally skilled in the ability to administer the function of the administrator or support personnel. This will allow for the continuance of the day-to-day operations of the laboratory.

4.1.2.1 Laboratory Director

The ultimate responsibility for the generation of reliable laboratory data rests with the Laboratory Director, who is accountable to his General Manager and oversees the daily operations of the laboratory. The Laboratory Director's responsibilities include allocation of personnel and resources, setting goals and objectives for both the business and employees, achieving the financial, business and quality objectives of STL. Furthermore, to see that all tasks performed in the laboratory are conducted according to the requirements of this LQM, the Project Technical Profile and/or the appropriate QAPP; and to assure that the quality of service provided complies with the project's requirements.

The Laboratory Director has the authority to affect those policies and procedures to ensure that only data of the highest level of excellence are produced. As such, the Laboratory Director supports a QA Section which has responsibilities independent from sampling and analysis.

The Laboratory Director, with the assistance of the Quality Assurance Manager, has the overall responsibility for establishing policies that ensure the quality of analytical services meet our clients expectations. These policies are defined in this LQM.

4.1.2.2 Quality Assurance Manager

The Quality Assurance (QA) Manager has the full-time responsibility to evaluate the adherence to policies and to assure that systems are in place to produce the level of quality defined in this LQM. The QA Manager is responsible for the approval of IDL/MDL studies, method validation studies, IDOC and CDOC evaluations, the annual review of statistical control limits, data package inspections, and LIMS system method development, validation and maintenance. In addition, the QA Manager assists in the preparation, compilation, and submittal of quality assurance plans; reviews program plans for consistency with organizational and contractual requirements and advises appropriate personnel of deficiencies. The QA Manager is assisted by the QA Specialist in the maintenance of QA records, certifications, accreditations, internal and external audits, corrective action procedures, management of the laboratory's PT Program, and maintenance of training documentation.

The QA Manager shall have the final authority to accept or reject data, and to stop work in progress in the event that procedures or practices compromise the validity and integrity of analytical data. The QA Manager is available to any employee at the facility to resolve data quality or ethical issues. The QA Manager must address any data integrity issue identified internally or externally, establish a corrective action plan and resolve the issue to the client's satisfaction. Issues that involve data recall must be discussed with the Corporate Quality Director Ray Frederici. The QA Manager shall be independent of laboratory operations and has an indirect reporting relationship to the QA Director.

4.1.2.3 Project Managers

The laboratory recognizes the importance of efficient project management. The laboratory Project Managers (PM) are responsible for preparing the Project Technical Profile which summarizes QA/QC requirements for the project, maintaining the laboratory schedule, ensuring that technical

requirements are understood by the laboratory, and advising the Laboratory, QA and Technical Managers of all variances. The laboratory Project Manager will provide technical guidance and the necessary laboratory-related information to the preparer of project-specific QAPPs and provide peer review of the final document to ensure accuracy of the laboratory information.

4.1.2.4 Technical Managers

The Technical Managers are the Laboratory Director, laboratory Section Managers and the QA Manager. They are as follows:

- ◆ Michael J. Healy, Laboratory Director, BS Environmental Biology,
- ◆ 22 years laboratory experience.
- ◆ Terese A. Preston, Quality Assurance Manager, BA Biology,
- ◆ 20 years laboratory experience.
- ◆ Diane L. Harper, Inorganics Section Manager, MA Biology,
- ◆ 24 years laboratory experience.
- ◆ Jodi L. Wojcik, Metals Section Manager, BS Biology,
- ◆ 18 years laboratory experience.
- ◆ Patti J. Gibson, Chromatography/Organic Extractions Section Manager, BS Biology,
- ◆ 15 years laboratory experience.
- ◆ Gary L. Rynkar, GC/MS Section Manager, BS Environmental Biology,
- ◆ 16 years laboratory experience.

All of these managers report to the Laboratory Director and serve as the technical experts on assigned projects, provide technical liaison, assist in resolving any technical issues within the area of their expertise; and implement established policies and procedures to assist the Laboratory Director in achieving section goals. The Technical Managers are responsible for ensuring that their personnel are adequately trained to perform analyses; that equipment and instrumentation under their control is calibrated and functioning properly; that system and performance audits are performed on an as-needed basis; provide input and review in the development and implementation of project-specific QA/QC requirements; and for providing the critical review of proposal and project work for programs as directed by the Laboratory Director. The Technical Managers coordinate these activities with the project management and quality assurance sections.

4.1.2.5 Sample Management Coordination

The Project Manager is designated as the Sample Management Coordination for any work subcontracted under their management. The Project Manager verifies each subcontracting request to ensure that special client restrictions are not jeopardized (e.g., samples must be analyzed by the receiving affiliated or network laboratory and must maintain specific certification(s)). The Project Manager is also responsible for verifying the credentials; establishing the service agreement; ensuring data review; and invoicing of all laboratory subcontractors. The Project Manager discusses any deficiencies or anomalies with the subcontractor prior to reporting any data to the client.

4.1.2.6 Data Management Section Manager

The Data Management Section Manager is responsible for coordinating receipt of all data from the various service groups within the laboratory, reviewing data for compliance to laboratory QC criteria and/or criteria in the Project Technical Profile, and ensuring that data are reported in a timely manner and in the proper format.

4.1.2.7 Quality Assurance Specialist

The QA Specialist is responsible for QA documentation and involvement in the following activities:

- ◆ Assist the QA Manager in performing the annual internal laboratory audits, compiling the evaluation, and coordinating the development of an action plan to address any deficiency identified.
- ◆ Facilitate external audits, coordinating with the QA Manager and Laboratory Staff to address any deficiencies noted at the time of the audit and subsequently presented in the final audit report.
- ◆ Assist the QA Manager in the preparation of new SOP's and in the maintenance of existing SOPs, coordinating annual reviews and updates.
- ◆ Manages the performance testing (PT) studies, coordinates follow up studies for failed analytes and works with QA Manager and Laboratory Staff to complete needed corrective action reports.
- ◆ Personnel training records review and maintenance.
- ◆ Document control maintenance.
- ◆ Assists the Quality Manager and Project Management Group in the review of program plans for consistency with organizational and contractual requirements. Summarize and convey to appropriate personnel anomalies or inconsistencies observed in the review process.
- ◆ Manages certifications and accreditations.
- ◆ Monitors for compliance the following QA Metrics: Temperature Monitoring of refrigeration units and incubators; thermometer calibrations; balance calibrations; eppendorf/pipette calibrations; and proper standard/reagent storage.
- ◆ Periodic checks on the proper use and review of instrument logs.
- ◆ Initiate the Mint-miner data file review process for organic instrumentation. Maintain tracking sheet of activity.
- ◆ Initiate the annual Instrument review.
- ◆ Assist in the technical review of data packages which require QA review.

4.1.2.8 Health and Safety Coordinator / Waste Management

The Health and Safety Coordinator is responsible for the safety and well-being of all employees while at the laboratory. This includes, but is not limited to, administering the Corporate Safety Manual that complies with federal regulations, MSDS training and review, conducting laboratory safety orientation and tours for all new employees, providing instructions on safety equipment, cleaning up laboratory spills, and instructing personnel of laboratory procedures for emergency situations. The Health and Safety Coordinator is on-call 24-hours a day, 7-days a week for all laboratory situations.

The Health and Safety Coordinator responsibilities additionally include waste management of laboratory generated hazardous waste in accordance with appropriate regulations. This includes maintenance of required documentation, such as waste manifests, segregation of waste in accordance with requirements, and training of personnel in proper segregation of waste.

4.1.2.9 Information Technology Manager

The overall role of the Information Technology (IT) Manager is to enhance laboratory productivity through improved information access, flow, and security. For information to be of greatest value, it must be readily accessible and reliable. It is the responsibility of the IT Manager to provide software tools that allow quick and user friendly access to that information, while at the same time controlling access to that information to those that have the need and proper authority.

Information flow can be enhanced through automation. Automation is the minimization of human intervention in a process. Reduction in human intervention can result in significant error reductions and time savings. The IT Manager assists the laboratory in automation by providing hardware and software solutions to help minimize human intervention in data collection, processing, and storage.

The IT Manager is responsible for providing data security by controlling access, as mentioned above, and for providing for disaster recovery. Data stored on the central Laboratory Information Management System (LIMS, a.k.a., LabNet) is the direct responsibility of the IT Manager. No fewer than two copies of all data should exist at any time so that lost or destroyed data can always be retrieved from an alternate source. These copies may consist of data within the system and on magnetic tape in the case of live data, or two copies on magnetic tape for archived data. Data stored electronically in other departments is the direct responsibility of those departments. However, the IT Manager is responsible for providing procedures and training to all laboratory operations, as appropriate, to assist in making backup copies of local data within the respective operating unit.

STL has established procedures for IT management:

- ◆ *Internet Use Policy – P-I-001*
- ◆ *Electronic Mail Use – P-I-002*
- ◆ *Computer Systems Account and Naming Policy – P-I-003*
- ◆ *Computer Systems Password Policy – P-I-004*
- ◆ *Software Licensing Policy – P-I-005*
- ◆ *Virus Protection Policy – P-I-006*

4.1.2.10 Chemists / Technicians

Any effective laboratory quality assurance/quality control program depends on the entire organization, including management and every individual on the laboratory staff. The initial review for acceptability of analytical results rests with the analysts conducting the various tests. Observations made during the performance of an analytical method may indicate that the analytical system is not in control. Analysts must use quality control indicators to assure that the method is in-control before reporting results.

4.2 Quality System

Organizational support for implementing the quality system and achieving the quality objectives is derived from this LQM, SOPs and Work Instructions. Within these documents, management with executive responsibilities ensures that the quality policy is understood, implemented, and maintained at all levels of the organization. The development and implementation of appropriate accountabilities, duties, and authority by organizational positions are clearly delineated. Line organizations achieve and verify that specifications are achieved; QA organizations assist and provide oversight and verification of processes through planning, reviews, audits, and surveillances. Top management leadership, support and direction ensures that the policies and procedures are appropriately implemented.

4.2.1 Objectives of the Quality System

The goal of the quality system is to ensure that business operations are conducted with the highest standards of professionalism in the industry.

To achieve this goal, it is necessary to provide our clients with not only scientifically sound, well documented, and regulatory compliant data, but also to ensure that we provide the highest quality service available in the industry with uncompromising data integrity. A well-structured and well-communicated quality system is essential in meeting this goal. The laboratory's quality system is designed to minimize systematic error, encourage constructive, documented problem solving, and provide a framework for continuous improvement within the organization.

As stated in Section 1.3, this LQM, Work Instructions and the SOPs themselves are the basis and outline for our quality and data integrity system and contains requirements and general guidelines under which the laboratory conducts our operations. In addition, other documents may be used by the laboratory to clarify compliance with quality system or other client requirements. As you read this LQM, you will note SOP or Work Instruction numbers in parenthetical text. These numbers refer to the laboratory procedure(s) associated with the subject item. A table listing these quality system policies and procedures is appended to this document.

The QA Manager and QA Specialist are responsible for implementing and monitoring the Quality System. The QA Manager reports to the Laboratory Director on the performance of the quality system for review and continuous improvement. The QA Manager has sufficient authority, access to work areas, and organizational freedom (including sufficient independence from cost and schedule considerations) to:

- ◆ Initiate action to prevent the occurrence of any nonconformities related to product, process and quality system,
- ◆ Identify and record any problems affecting the product, process and quality system,
- ◆ Initiate, recommend, or provide solutions to problems through designated channels,
- ◆ Verify implementation of solutions, and
- ◆ Assure that further work is stopped or controlled until proper resolution of a non-conformance, deficiency, or unsatisfactory condition has occurred and the deficiency or unsatisfactory condition has been corrected.

The QA Manager reports where appropriate action can be affected. However, should a situation arise where acceptable resolution of identified problems cannot be agreed upon at the laboratory level, direct access to STL's Corporate Quality Director is available. This provides laboratory QA personnel non-laboratory management support, if needed, to ensure that QA policies and procedures are enforced.

The QA Manager or QA Specialist conducts annual LQM training for all laboratory and administrative personnel to ensure their familiarity with the quality documentation and the implementation of the policies and procedures in their work.

4.3 Document Control

The laboratory maintains procedures to control documents and analytical data. Since intensive data is generated and this is our primary product, document control is inherently segregated from data control, as described further in Sections 4.3.1 and 4.3.2.

4.3.1 Document Control Procedure

Security and control of documents are necessary to ensure that confidential information is not distributed and that all current copies of a given document are from the latest applicable revision (*Document Control*; UQA-006). Unambiguous identification of a controlled document is maintained by identification of the following items in the document header: Document Number, Revision Number, Effective Date, and Number of Pages. Document control may be achieved by either electronic or hardcopy distribution.

Controlled documents are authorized by the QA Department and are marked as either "Controlled" or "Uncontrolled" and records of their distribution are kept by the QA Department. Controlled status is defined as the continuous distribution of document updates. Uncontrolled status is defined as the single distribution of the current SOP. Document updates are not distributed to uncontrolled status holders. For tracking purposes, a control copy number is assigned to documents distributed with a controlled status. All copy numbers are written or typed in red to easily identify the SOP as a controlled copy.

4.3.1.1 Document Revision

Changes to documents occur when a procedural change warrants a revision of the document. When an approved revision of a controlled document is ready for distribution, obsolete copies of the document are replaced with the current version of the document. The previous revision of the controlled document is stamped "ARCHIVED COPY" and is filed by the QA Specialist in the QA library. Only the most current revision is maintained electronically.

SOPs are updated on a 12-18 month basis, which is tracked by an established review schedule (*Approved SOP Listing*; CHI-22-09-SOP List). These reviews are conducted by the creator of the SOP and/or Department Manager, QA Specialist and/or QA Manager, and the Health and Safety Coordinator, all of whom provide the approval signature for each SOP.

4.3.2 Data Control

All raw data, such as bound logbooks, instrument printouts, magnetic tapes, electronic data, as well as final reports, are retained for a minimum period of 5 years. Such data may be maintained longer, as defined by client and project requirements. The procedure for archiving records and client or project specific requirements is contained in the *Record Retention and Purging SOP* (UDM-002).

Raw data and reports are documented and stored in a manner in which they are easily retrievable. The procedure for maintaining raw data records is briefly described below:

- ◆ Instrument print-outs for conventional inorganic parameters are filed by LabNet Batch Number. Inorganic Metals are filed by Instrument and Filename. Generally, current year and previous year documents are kept on file in the laboratory sections.
- ◆ All raw data, for example, instrument print-outs and logbooks, are maintained in an on-site and secured storage area.
- ◆ The computer information is backed up on tape daily, and stored in a secured and temperature/humidity controlled environment to maintain the integrity of the electronic information in the event of system failure. Copies of all back-up tapes are maintained in secured off-site locations.
- ◆ All copies of client final reports are maintained electronically (e.g., Adobe Acrobat).

4.4 Request, Tender, and Contract Review

4.4.1 Contract Review

For many environmental sampling and analysis programs, testing design is site or program specific and does not necessarily "fit" into a standard laboratory service or product. It is STL's intent to provide both standard and customized environmental laboratory services to our clients. To ensure project success, technical staff performs a thorough review of technical and QC requirements contained in contracts. Contracts are reviewed for adequately defined requirements and STL's capability to meet those requirements.

All contracts entered into by the laboratory are reviewed for the client's requirements in terms of compound lists, test methodology requested, sensitivity, accuracy, and precision requirements. 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 review also includes the laboratory's capabilities in terms of turnaround time, capacity, and resources to provide the services requested, as well as the ability to provide the documentation, whether hardcopy or electronic. If the laboratory cannot provide all services but intends to subcontract such services, whether to another STL facility or to an outside firm, this will be documented and discussed with the client prior to contract approval.

Any contract requirement or amendment to a contract communicated to STL verbally is documented and confirmed with the client in writing. Any discrepancy between the client's requirements and STL's capability to meet those requirements is resolved in writing before

acceptance of the contract. Contract amendments, initiated by the client and/or STL, are documented in writing for the benefit of both the client and STL.

All contracts, QAPPs, Sampling and Analysis Plans (SAPs), contract amendments, and documented communications become part of the permanent project record as defined in Section 4.12.1.

4.4.2 Project-Specific Quality Planning

Communication of contract specific technical and QC criteria is an essential activity in ensuring the success of site specific testing programs. To achieve this goal, STL assigns a Project Manager (PM) to each client. The PM is the first point of contact for the client. It is the PM's responsibility to ensure that project specific technical and QC requirements are effectively evaluated and communicated to the laboratory personnel before and during the project (*Project Planning Process*; UPM-003). QA department involvement may be needed to assist in the evaluation of custom QC requirements.

PM's are the direct client contact and they ensure resources are available to meet project requirements. Although PM's do not have direct reports or staff in production, they coordinate opportunities and work with laboratory management and supervisory staff to ensure that the 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 (e.g., LabNet Project Notes) 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 (UPM-002)* or to the supervisory staff during *Production Meetings (UPM-004)*. These meetings provide direction to the laboratory staff in order to maximize production and client satisfaction, while maintaining quality. In addition, the LabNet Project Notes are associated with each sample batch (e.g., Job) as a reminder upon sample receipt and analytical processing.

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

Such changes are also communicated to the laboratory through the management Production Meetings which are conducted three times per week (T,W,Th). Such changes are updated to the LabNet Project Notes and are introduced to the managers at these meetings. The laboratory staff is then introduced to the modified requirements via the Project Manager or the individual laboratory section manager. After the modification is implemented into the laboratory procedure, documentation of the modification is made in the case narrative of the data report(s).

STL strongly encourages our clients to visit the laboratory and hold formal or informal sessions with employees in order to effectively communicate ongoing client needs as well as project specific details for customized testing programs.

4.4.3 Data Quality Objectives

Data quality objectives (DQO) are qualitative and quantitative statements used to ensure the generation of the type, quantity, and quality of environmental data that will be appropriate for the intended application. Typically, DQOs are identified before project initiation and during the development of a QAPPs and SAPs. The analytical DQOs addressed in this section are precision, accuracy, representativeness, completeness, and comparability.

The components of analytical variability (uncertainty) can be estimated when QC samples of the right types and at the appropriate frequency are incorporated into the measurement process of the laboratory. STL incorporates numerous QC samples to obtain data for comparison with the analytical DQOs and to ensure that the measurement system is functioning properly. The control samples and their applications, described in Section 5.8.2, are selected based on regulatory, method- or client-specific requirements. Analytical QC samples for inorganic and organic analyses may include calibration blanks, instrument blanks, method blanks, LCS, calibration standards, MS, MSD, MD, surrogate spikes, and yield monitors.

The DQOs discussed below ensure that data are gathered and presented in accordance with procedures appropriate for its intended use, that the data is of known and documented quality, and are able to withstand scientific and legal scrutiny.

4.4.3.1 Precision

Precision is an estimate of variability. It is an estimate of agreement among individual measurements of the same physical or chemical property, under prescribed similar conditions. Precision is expressed either as Relative Standard Deviation (RSD) for greater than two measurements or as Relative Percent Difference (RPD) for two measurements. Precision is determined, in part, by analyzing data from LCSs, MS, MSD, and MD. A description of these control samples is provided in Section 5.8.2.

Precision also refers to the measurement of the variability associated with the entire process, from sampling to analysis. Total precision of the process can be determined by analysis of duplicate or replicate field samples and measures variability introduced by both the laboratory and field operations.

4.4.3.2 Accuracy

Accuracy is the degree of agreement between a measurement and the true or expected value, or between the average of a number of measurements and the true or expected value. It reflects the total error associated with a measurement.

Both random and systematic errors can affect accuracy. For chemical properties, accuracy is expressed either as a percent recovery (R) or as a percent bias (R - 100). Accuracy is determined, in part, by analyzing data from LCSs, MS and MSD.

Accuracy and Precision objectives employed by the laboratory are as defined in the CERCLA's Inorganic and Organic Statements of Work (SOW); statistically-derived control limits; or default limits as listed in each respective method SOP.

4.4.3.3 Representativeness

Representativeness is the degree to which data accurately and precisely represent a characteristic of a population, a variation in a physical or chemical property at a sampling point, or an environmental condition. Data representativeness is primarily a function of sampling strategy; therefore, the sampling scheme must be designed to maximize representativeness. Representativeness also relates to ensuring that, through sample homogeneity, the sample analysis result is representative of the constituent concentration in the sample matrix. STL makes every effort to analyze an aliquot that is representative of the original sample, and to ensure the homogeneity of the sample before sub-sampling.

4.4.3.4 Completeness

Completeness is defined as the percentage of measurements that are judged valid or useable. Factors negatively affecting completeness include the following: sample leakage or breakage in transit or during handling, loss of sample during laboratory analysis through accident or improper handling, improper documentation such that traceability is compromised, or sample result is rejected due to failure to conform to QC specifications. A completeness objective of greater than 90% of the data specified by the statement of work is the goal established for most projects.

4.4.3.5 Comparability

Comparability is a measure of the confidence with which one data set can be compared to another. To ensure comparability, all laboratory analysts are required to use uniform procedures (e.g., SOPs) and a uniform set of units and calculations for analyzing and reporting environmental data.

A measure of inter-laboratory comparability is obtained through the laboratory's participation in proficiency testing (PT) programs established with Water Supply (WS), Water Pollution (WP), Solid Waste (SW), and Underground Storage Tank (UST) programs. In addition, the laboratory employs the use of NIST or EPA traceable standards, when available, to provide an additional measure of assurance of the comparability of data.

Project representativeness and comparability are dependent upon the sampling plan on a project specific basis, and are therefore not covered in this LQM. Assessment of site and collection representativeness and comparability is performed by the field engineer.

4.4.3.6 Additional DQOs

Method Detection Limits

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 Appendix B of 40 CFR 136, "Guidelines Establishing Test Procedures for the Analysis of Pollutants". 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. (UQA-017)

For the performance of non-routine methods, e.g., client/contract requirement, MDLs or Method Validation Studies will be completed on an as needed basis. The turnaround time for such studies will be as determined by the client and Project Manager. Such studies will be reviewed and approved by the client and/or regulatory agency prior to project implementation.

Instrument Detection Limits

There are a number of ways to determine Instrument Detection Limit (IDL) sensitivity (e.g., signal-to-noise ratio; precision of the low-level standard; lowest calibration curve point or the IDL study defined within CLP). The method and means in which IDLs are determined are documented and maintained in the QA department for each individual instrument.

IDLs are generated for each element by the metals laboratory quarterly via each instrument as specified in CLP. These limits are used to gauge instrument sensitivity and when routinely evaluated, instrument performance without the introduction of method variance can be determined. (UQA-010)

Reporting Limits

Reporting Limits are defined as the lowest concentration of an analyte determined by a given method in a given matrix that the laboratory feels can be reported with acceptable quantitative error or client requirements, values specified by the EPA methods or other project and client requirements. The laboratory reporting limits are further related and verified by the lowest point on a calibration curve. Because of the high level of quantitative error associated with determinations at the level of the MDL, the laboratory maintains reporting limits higher than the MDL. Wherever possible, reporting is limited to values approximately 2-5x the respective MDL to ensure confidence in the value reported. Client specific requests for reporting to the IDL or MDL are special circumstances not to be confused with the previous statement. Data evaluated down to the MDL/IDL is qualified as estimated with a 'J' for organic analyses and a 'B' for inorganic analyses on the data report.

MDL studies are performed annually, and reporting limits are assessed. If the MDL does not meet the routine laboratory reporting limit or the method specified limit, it is repeated or the laboratory reporting limit is reassessed. If the laboratory continually demonstrates that the method reporting limits are not achieved, equipment, technique, and the method are reviewed to assure optimal performance or appropriate action is taken.

4.5 Subcontracting

Subcontracting is arranged with the documented consent of the client, in a timely response which shall not be unreasonably refused. All QC guidelines specific to the client's analytical program are transmitted to the subcontractor and agreed upon before sending the samples to the subcontract facility. Proof of holding required certifications from the subcontract facility are maintained in the project records. Where applicable, the specific QC guidelines, QAPPs, and/or SAPs are transmitted to the subcontract laboratory. Samples are subcontracted under formal Chain of Custody (COC).

Subcontract laboratories may receive an on-site audit by a representative of STL's QA staff if it is deemed appropriate by the QA Manager. The audit involves a measure of compliance with the required test method, QC requirements, as well as any special client requirements (e.g., Technical Profile and LabNet Project Notes). STL may also perform a paper audit of the subcontractor, which would entail reviewing the LQM, the last two PT studies, and a copy of any recent regulatory audits with the laboratory's responses.

Intra-company subcontracting may also occur between STL facilities. Intra-company subcontracting within STL is arranged with the documented consent of the client (e.g., QAPP). The originating laboratory is responsible for communicating all technical, quality, and deliverable requirements as well as other contract needs. STL has implemented a standard form for Intra-laboratory subcontracting, refer to the following document for specific details: *Work Sharing Process – Policy No.: S-C-001*.

Project reports from both STL and external subcontractors are not altered and are included in their original form in the final project report provided by STL. This clearly identifies the data as being produced by a subcontractor facility. All data, as required in Section 5.9.4, is included.

4.6 Purchasing Services and Supplies

Evaluation and selection of suppliers and vendors is performed, in part, on the basis of the quality of their products, their ability to meet the demand for their products on a continuous and short term basis, the overall quality of their services, their past history, and competitive pricing. This is achieved through evaluation of objective evidence of quality furnished by the supplier, which can include certificates of analysis, recommendations, and proof of historical compliance with similar programs for other clients. To ensure that quality critical consumables and equipment conform to specific requirements, all purchases from specific vendors are approved by a member of the supervisory or management staff.

Chemical reagents, solvents, glassware, and general supplies are ordered as needed to maintain sufficient quantities on hand. Purchasing guidelines for equipment and reagents meet with the requirements of the specific method and testing procedures for which they are being purchased. The measurements for evaluation and selection of suppliers; the acceptance of supplies and services; and certificates of conformance are described in the procurement SOP (*Procurement Quality Assurance Process; UQA-020*).

4.6.1 Solvent and Acid Lot Verification

Pre-purchase approval is performed for solvents and acids purchased in large quantities unless a certificate of conformance has been furnished. These may include acetone, ethyl ether, hexane, methylene chloride, nitric acid, hydrochloric acid, sulfuric acid, and hydrogen peroxide. Each lot of incoming supplies requiring pre-approval is checked against the previously approved lot number. If the lot number is not approved, the lot is refused. If the lot number is an approved lot number, it is accepted and documented. Solvents and acids are pre-tested in accordance with STLs Corporate *Testing Solvents and Acids* procedure (S-T-001) for all of the STL laboratories.

4.7 Service to the Client

4.7.1 Sample Acceptance Policy

Samples are considered "compromised" if the following conditions are observed upon sample receipt:

- ◆ Cooler and/or samples are received outside of temperature specification.
- ◆ Samples are received broken or leaking.
- ◆ Samples are received beyond holding time.
- ◆ Samples are received without appropriate preservation.
- ◆ Samples are received in inappropriate containers.
- ◆ COC does not match samples received.
- ◆ COC is not properly completed or not received.
- ◆ Breakage of any Custody Seal.
- ◆ Apparent tampering with cooler and/or samples.
- ◆ Headspace in volatiles samples.
- ◆ Seepage of extraneous water or materials into samples.
- ◆ Inadequate sample volume.
- ◆ Illegible, impermanent, or non-unique sample labeling.

When "compromised" samples are received, it is documented on the hardcopy COC, the LabNet Sample Receipt Checklist and on a Sample Discrepancy Report (SDR); and the client is contacted for instructions. If the client decides to proceed with the analysis, the project report will clearly indicate any of the above conditions and the resolution.

4.7.2 Client Confidentiality and Proprietary Rights

Data and sample materials provided by the client or at the client's request, and the results obtained by STL, shall be held in confidence (unless such information is generally available to the public or is in the public domain or client has failed to pay STL for all services rendered or is otherwise in breach of the terms and conditions set forth in the STL and client contract) subject to any disclosure required by law or legal process. Technical, business and proprietary information provided by a client and data/information generated by the laboratory are restricted for the use within the laboratory for purposes of accomplishing the project. Client information is not to be used on other

projects or revealed except in conjunction with project work to anyone outside the laboratory without permission of the client.

STL's reports, and the data and information provided therein, are for the exclusive use and benefit of client, and are not released to a third party without written consent from the client (*Client Confidentiality*; UQA-004).

4.8 Complaints

STL believes that effective client complaint handling processes have important business and strategic value. Listening to and documenting client's concerns captures 'client knowledge' that helps to continually improve processes and outpace the competition. Implementing a client complaint handling process also provides assurance to the data user that the laboratory will stand behind its data, service obligations and products.

Client inquiries, complaints or noted discrepancies are documented, communicated to management, and addressed promptly and thoroughly. The investigation of the cause, resolution and authorization of corrective action is documented [*Sample Discrepancy Report (SDR)*, *Resubmitted Data Request (RDR)*, *Corrective Action Report (CAR)*; UQA-029].

Client complaints are documented by the employee receiving the complaint. The documentation can take the form of a Resubmitted Data Request (RDR) or in a format specifically designed for that purpose (e.g., phone conversation record or e-mail). The Laboratory Director, Project Manager and/or QA Manager are informed of client complaints and assist in resolving the complaint.

The RDR is used after the client has received the analytical report and their specifications, expectations, or client satisfaction was not achieved. RDRs are prepared when clients request re-evaluation of submitted data, when additional information is requested or for general complaints.

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

The number and nature of client complaints is reported by the QA Manager to the 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 *Quality Systems Management Review* (UQA-002).

4.9 Control of Non-conformances

Non-conformances include any out of control occurrence. Non-conformances may relate to client specific requirements, procedural requirements, or equipment issues. All non-conformances in the laboratory are documented at the time of their occurrence on Corrective Action Reports (CARs) specifically formatted for each department or on a SDR.

All non-conformances that affect a sample and/or sample data become part of the affected project's permanent record. When appropriate, reanalysis is performed where QC data falls outside of specifications, or where data appears anomalous. If the reanalysis comes back within established tolerances, the results are approved. If the reanalysis is still outside tolerances, further reanalysis or consultation with the Section Manager, Project Manager or QA Manager for direction may be required. All records of reanalysis are kept with the project files.

Where non-conformances specifically affect a client's sample and/or data, the client is informed and action must be taken. Action can take the form of reporting and flagging the data, and including a description of the non-conformance in the project narrative.

4.10 Corrective Action

To consistently achieve technical and regulatory requirements, the laboratory data must be supported by an effective corrective action system. The system must be capable of isolating and rectifying both random and systematic errors. Identification of systematic errors, or errors that are likely to occur repetitively due to a defect or weakness in a system, is particularly valuable in maintaining an environment of continuous improvement in laboratory operations.

Mechanisms used to ensure problem definition include SOPs; internal and external audits and surveillances; and regular laboratory management meetings. When evaluation of performance against established criteria for good laboratory practices shows a condition that could adversely affect the quality of services provided, corrective action is initiated.

Any employee in STL can initiate a corrective action. The initial source of corrective action can also be external to STL (i.e., corrective action due to client complaint, regulatory audit, or PT(s)). When a problem that requires corrective action is identified, the following items are identified by the initiator on the corrective action report: the nature of the problem, the name of the initiator, and the date. If the problem affects a specific client project, the PM is informed immediately.

All corrective actions, whether immediate or long-term, will comprise the following steps to ensure a closed-loop corrective action process:

- ◆ Define the problem.
- ◆ Assign responsibility for investigating the problem.
- ◆ Determine a corrective action to eliminate the problem.
- ◆ Assign, and obtain commitment to, responsibility for implementing the corrective action.
- ◆ Implement the correction.
- ◆ Assess the effectiveness of the corrective action and verify that the corrective action has eliminated the problem.

4.10.1 Immediate Corrective Action

Immediate corrective actions to correct or repair non-conforming equipment and systems are generally initiated in response to adverse conditions identified through QC procedures. The analyst has relatively quick feedback that a problem exists, e.g., calibration does not meet or QC check samples exceed allowable criteria, and can take immediate action to repair the system.

The initial responsibility to monitor the quality of a function or analytical system lies with the individual performing the task or procedure. DQOs are evaluated against laboratory-established or against method or client specified QA/QC requirements. If the assessment reveals that any of the QC acceptance criteria are not met, the analyst must immediately assess the analytical system to correct the problem. When the appropriate corrective action measures have been defined and the analytical system is determined to be "in-control" or the measures required to put the system "in-control" have been identified and scheduled, the problem and resolution or planned action is documented in the appropriate logbook or CAR. Data generated by an analytical system that is determined to be out-of-control must never be released without approval of the Section Manager, QA Manager, Laboratory Director, Project Manager and client notification.

When an acceptable resolution cannot be met or data quality is negatively affected, the analyst will notify their Section Manager and initiate an SDR. If an SDR is required, it is routed for proper authorizations and direction. Proper authorization and direction is given by the Project Manager and/or QA Manager. Based upon the circumstances and judgment of the Project Manager, the client may be notified of the situation.

Data generated concurrently with an out-of-control system will be evaluated for usability in light of the nature of the deficiency. If the deficiency does not impair the usability of the results, data will be reported and the deficiency will be noted in the case narrative. Where sample results may be impaired, the Project Manager is notified by a written SDR and appropriate corrective action (e.g., reanalysis) is taken and documented.

A CAR documents analytical problems at the bench level. This form allows for the documentation of the out-of-control situation, actions undertaken to correct the problem and a return-to-control status. All CARs are signed/dated by the respective laboratory Section Manager.

The QA Manager has the authority to stop the analysis, e.g., failure to meet method or project requirements, and to hold all analyses of samples affected by an out-of-control situation. The method cannot be restarted without appropriate documentation leading to the QA Manager's approval and sign-off.

4.10.2 Long-term Corrective Action

Long-term corrective action is generally initiated due to QA issues, which are most often identified during internal and external audits (Sections 4.13 & 4.14). Typically, a deeper investigation into the root cause of the nonconformance is warranted, and the problem may take much longer to identify and resolve. Staff training, method revision, replacement of equipment, and LabNet reprogramming are examples of long-term corrective action.

4.10.3 Responsibility and Closure

The Section Manager is responsible for correcting out-of-control situations, placing highest priority on this endeavor. Associated corrective actions, once verified for effectiveness, are incorporated into standard practices. Ineffective actions will be re-evaluated until acceptable resolution is achieved. Section Managers are accountable to the Laboratory Director to ensure final acceptable resolution is achieved.

The QA Department also may implement a special audit (Section 4.13). The purpose of inclusion of the corrective action process in both routine and special audits is to monitor the implementation of the corrective action and to determine whether the action taken has been effective in overcoming the issue identified.

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. This provides laboratory QA personnel non-laboratory management support, if needed, to ensure that QA policies and procedures are enforced.

4.11 Preventative Action

The laboratory's preventive action programs improve, or eliminate potential causes of nonconforming product and/or nonconformance to the quality system. This preventive action process is a proactive continuous process improvement activity which can be initiated by clients, employees, business providers, and affiliates. The QA section 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.

Preventive action opportunities may be identified from information obtained through activities related to but not limited to the corrective action process, performance evaluation program, internal audits, management review, and/or market trends, industry trends and competitive comparisons.

Established standard practices for preventive action are included in the *Preventive Action Measures* SOP (UQA-019); the *SDR / RDR / CAR* SOP (UQA-029) and the *Quality System Management Review* SOP (UQA-002). These procedures describe the information sources used to detect, analyze, and eliminate potential causes of nonconformities and to ensure effective implementation of solutions.

4.12 Records

4.12.1 Record Types

Record types are described in Table 4.

4.12.2 Record Retention

Data reports are filed electronically as .pdf files by sample job number. Hardcopy COC files are maintained and are filed in Job Number order.

Laboratory data, project management files, QA records (e.g., PT scores/corrective actions; MDLs/IDLs, statistical analysis, QAPPs, etc.), Human Resources information, etc., are compiled by date order. The same procedure is followed both in current and archived hardcopy storage.

Upon archiving, a *Records Management Form* (CHI-22-05-032) is prepared for each storage box of records. This form documents the department, department manager, contents (description and dates), term of retention (e.g., no. of years) and an assigned identification number. The original of this form is maintained with the data management department with a carbon copy filed within the storage box. Upon purging of records, the individual department managers sign the original form as confirmation for the destruction of the associated data. This signature indicates that the laboratory has maintained the information for the required amount of time and is no longer required to store it.

Table 5 outlines the laboratory's standard record retention time. 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, QC, or Administrative Records, the retention time is calculated from the date the record is formally retired. Records related to the programs listed in Table 6 have lengthier retention requirements and are subject to the requirements in Section 4.12.3.

Table 4. STL Record Types

Raw Data	Controlled Documents	QC Records	Project Records	Administrative Records
See Section 3. Terms and Definitions	LQMs/ QAPPs	Audits/ Responses	COC Documentation	Accounting
	QMP (Corporate)	Certifications	Contracts and Amendments	Corporate Safety Manual, Permits, Disposal Records
	SOPs	SDRs/RDRs	Correspondence	Employee Handbook
		Logbooks*	QAPP	Personnel files, Employee Signature & Initials, Training Records
		Method & Software Validation, Verification	SAP	
	Work Instructions	Standards Certificates	Telephone Logbooks	Technical and Administrative Policies
		MDL/IDL/IDC Studies	E-mails	
		PTs	Electronic Data Report	
	Statistical Evaluations			

*Examples of Logbook types: Maintenance, Instrument, Preparation (standard and samples), Standard and Reagent Receipt, Archiving, and Balance Calibration.

Table 5. STL Record Retention

Record Type		Archival Requirement *
Raw Data	All* (Electronic Data Reports (.pdf & EDD))	5 Years from completion
Controlled Documents	All*	5 Years from document retirement date
QC	All*	5 Years from archival
Project	All*	5 Year from project completion
Administrative	Personnel/Training	Indefinitely
	Accounting	10 years

* Exceptions listed in Table 6.

4.12.3 Programs with Longer Retention Requirements

Some regulatory programs have longer record retention requirements than the laboratory's standard record retention time. These are detailed in Table 6 with their retention requirements and client-specific requirements are listed in the *Record Retention and Purging* SOP (UDM-002). In these cases, the longer retention requirement is implemented and noted in the archive. 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 6. Special Record Retention Requirements

Program	Retention Requirement
Colorado – Drinking Water	10 years
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
Massachusetts – Drinking Water	10 years
Michigan Department of Environmental Quality – all environmental data	10 years
Minnesota – Drinking Water	10 years
Navy Facilities Engineering Service Center (NFESC)	10 years
OSHA - 40 CFR Part 1910	30 years
Pennsylvania – Drinking Water	10 years
TSCA – 40 CFR Part 792	10 years after publication of final test rule or negotiated test agreement

4.12.4 Archives and Record Transfer

Archives are indexed such that records are accessible on either a project or temporal basis. Archives are protected against fire, theft, loss, deterioration, and vermin. Electronic records are protected from deterioration caused by magnetic fields and/or electronic deterioration. Access to archives is controlled and documented.

STL ensures that all records are maintained as required by the regulatory guidelines and per this LQM upon facility location change or ownership transfer. Upon facility location change, all archives are retained by STL in accordance with this LQM. Upon ownership transfer, all final test reports generated by the laboratory will be submitted to the clients if not previously provided. Any further record retention requirements will be addressed in the ownership transfer agreement and the responsibility for maintaining archives will be clearly established.

In the event that the laboratory is closed, all final test reports generated by the laboratory will be submitted to the clients if not previously provided. All records will then be transferred to STL's corporate record storage location. All boxes and contents will be appropriately labeled with the dates of destruction (Refer to Tables 5 and 6) and managed in accordance their policies.

4.13 Internal Audits

Quality assurance audits and surveillances are conducted to assess the performance of laboratory systems in meeting technical, regulatory and client requirements; and to evaluate the operational details of the QA program (*Internal Audits*; UQA-013). They provide a means for management to be apprised of, and to respond to, a potential problem before it actually impacts the laboratory operations. They also are a mechanism for ensuring closure of corrective actions resulting from external audits.

4.13.1 Audit Types and Frequency

A number of types of audits are performed at STL. These audit types and frequency are categorized in Table 7.

Table 7. Audit Types and Frequency

Audit Type	Performed by	Frequency
Systems	QA Department or Designee	Annual
Data Authenticity	QA Department or Designee	Data Report Review: As necessary to ensure an effective secondary review process and to meet special program independent review objectives Analyst Data Audits: 100% of all analysts annually
Electronic		Electronic Data Audits: 100% of all organic instruments
Special	QA Department or Designee	As Needed

4.13.2 Systems Audits

Systems audits are technical in nature and are conducted on an ongoing basis by the QA Manager or the QA Specialist. Systems audits cover all departments of the facility, both operational and support. The review consists of laboratory systems, procedures, documentation and issues noted in external audits.

The audit report is issued by the QA Manager or QA Specialist within 21 calendar days of the audit. The audit report is addressed to the department Section Manager and copied to the QA department and the Laboratory Director.

Written audit responses are required within 30 calendar days of the audit report issue. A maximum of one calendar month is given to address any recommended corrective actions. The audit response is directed to all individuals copied on the audit report. Where a corrective action may require longer than a calendar month to complete, the target date for the corrective action implementation is stated and evidence of the corrective action is submitted to the QA Department in the agreed upon time frame.

4.13.3 Data Audits

Data audits are focused to assess the level of customer service, SOP compliance, regulatory compliance, accuracy and completeness of test results and reports, documentation, and adherence to established QC criteria, laboratory SOPs, technical policy, and project specific QC criteria.

The QA Department provides feedback and/or corrections and revisions to project reports where necessary. Records of the data audits are kept, and the frequency of data audits is included in the monthly QA report. In performing data audits, it is essential that data be assessed in terms of differentiating between systematic and isolated errors. Upon noting anomalous data or occurrences in the data audits, the QA Department is responsible for seeking clarification from the appropriate personnel, ascertaining whether the error is systematic or an isolated error, and overseeing correction and/or revision of the project report if necessary. Errors found in client project reports are revised and the revision sent to the client (Section 4.8). The QA Department is also responsible for assisting in the corrective action process where a data audit leads to identification of the need for permanent corrective action.

The frequency of data auditing may also be dependent upon specific clients and regulatory programs. All active laboratory logbooks and QC files are subject to periodic audits/ surveillances by the QA personnel.

4.13.3.1 Data Authenticity Audits

Data authenticity audits shall be performed on 100% of all analysts by the QA department or a designee independent from laboratory operations. Performing data authenticity checks will typically include verifying raw data, evaluating calculation tools and independently reproducing the final results and comparing it to the hardcopy on randomly selected batches of data. The QA Manager will report the percentage of analysts reviewed (for the year) in the monthly QA report and should average about 8% per month.

4.13.3.2 Electronic Data Audits

Electronic data audits are performed on 100% of all organic instruments by the QA department or a designee independent from the operations. This may include Mint Miner® scanning of randomly selected batches of electronic data followed by a chromatography system review. The QA manager will report the percentage of instruments reviewed (for the year) in the monthly QA report and should average about 8% of instruments per month. Electronic data audits include spot-checking of manual integrations by QA personnel in order to determine that the manual integration is appropriate and documented according to Section 5.3.6.1.

4.13.4 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, proficiency testing results, data audits, systems

audits, validation comments, or regulatory audits. Special audits are focused on a specific issue, and report format, distribution, and timeframes are designed to address the nature of the issue.

4.14 External Audits

STL is routinely audited by clients and external regulatory authorities – both government and non-government. Whether the audit is scheduled or unannounced, full cooperation with the audit team is provided by the laboratory and administrative staff. STL recommends that the audits be scheduled with the QA Department so that all necessary personnel are available on the day of the audit.

4.15 Management Reviews

4.15.1 QA Reports to Management

A monthly QA report is prepared by QA Manager and forwarded to the Laboratory Director, Project Managers, Section (Technical) Managers and the Corporate Quality Director. The reports include statistical results that are used to assess the effectiveness of the quality system. The format of the monthly report is shown in Figure 3.

4.15.2 Quality Systems Management Review

A quality systems management review is performed at least annually by the QA Manager. This review ensures that the laboratory's quality system is adequate to satisfy the laboratory's policies and practices, government requirements, certification, accreditation, approval requirements, and client expectations. Quality systems management reviews are accomplished through the evaluation and revision of this LQM, monthly quality assurance reporting and goal setting.

Management reviews of specific quality system elements may be performed through continuous improvement activities, monthly QA reports, process changes, SOP revisions, and/or audit reports/responses. Documentation of these reviews are not required unless it is inherent in the review mechanism (e.g., approval signatures on SOP revisions).

4.15.3 Monthly QA Report and Metrics

By the 3rd day of the month, the QA manager prepares a monthly QA report. The report is sent to the Laboratory Director and Corporate Quality Director. The report contains a narrative summary and metrics spreadsheet. At a minimum, the report content contains the items listed below (Figure 3). During the course of the year, the Laboratory Director, General Manager or Corporate Quality Director may request that additional information be added to the report.

Figure 3. Monthly QA Report Format

1	Audits
	External System Audits Internal System Audits Internal Training Record Audits Internal Data Audits
2	Revised Reports / Client Complaints / Client Compliments
	Revised Reports (RDR) Client Complaints Client Compliments
3	Certification Changes
	Certification Status Losses / Revocations
4	Proficiency Testing
	Study participation PT scores PT failures History of failures
5	SOP Status
	SOPs totals summarized by manager On-Time percentages calculated for SOPs < 1 year
6	Project/QAPP Review Status
7	Holding Time Violations
8	Monthly QA Report Metrics
	Summarize metrics in template provided by the Corporate Quality Director

5.0 Technical Requirements

5.1 Personnel

5.1.1 General

STL management believes that its highly qualified and professional staff is the single most important aspect in assuring the highest level of data quality and service in the industry. The staff consists of professionals and support personnel that include the following positions:

- Laboratory Director
- QA Manager
- Health & Safety Coordinator / Waste Management
- Project Manager
- Information Technology Manager
- Department Section Manager (Technical Manager)
- Analyst
- Sample Custodian
- Technician
- Quality Assurance Specialist
- Data Review Specialist

In order to ensure that employees have sufficient education and experience to perform a particular task, job descriptions are developed for all personnel (Section 4.1.2).

5.1.2 Training

STL is committed to furthering the professional and technical development of employees at all levels. 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 STL employees are outlined in Table 8.

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. The QA department, in conjunction with the Human Resources coordinator and Section Supervisor are responsible for maintaining the documentation of these activities.

Each laboratory section maintains documentation associated with analytical training (e.g., training records, document control). The QA department maintains documentation of initial and continued method proficiency for laboratory instrumentation and for each analyst. This documentation is represented in the following forms: MDLs, IDMPs, IDOCs, CDOCs, PT Sample results, Instrument QC and Batch QC Control Charts. This information is available to managers and staff for planning and evaluation.

The Human Resource coordinator maintains documentation and attestation forms on employment status & records; benefit programs; time keeping/payroll; and employee conduct (e.g., ethics). This information is maintained in the employee's secured personnel file.

The following evidence items are on file for each technical employee:

- ◆ Initial Demonstration of Capability (IDOC) for each method.
- ◆ Attestation that the employee has read and understood the latest version of the laboratory's quality documentation.
- ◆ The employee has read and understood the latest, approved version of all test methods and/or SOPs for which the employee is responsible.
- ◆ Annual evidence of Continued Demonstration of Capability (CDOC) that may include, but is not limited to, successful analysis of a blind sample on the specific test method or a similar test method; an annual DOC of four successive and acceptable LCSs.
- ◆ An Ethics Agreement signed by each staff member (renewed each year).
- ◆ A Confidentiality Agreement signed by each staff member (renewed each year).

Table 8. STL Employee Minimum Training Requirements

Specialty	Experience
General Chemistry and Instrumentation	Six months
Gas Chromatography	One year
Atomic Absorption	One year
Mass Spectrometry	One year
Spectra Interpretation	Two years

Required Training	Time Frame ¹	Employee Type
Environmental Health & Safety	Month 1	All
Ethics – Corporate Overview	Week 1	All
Ethics	Month 1	All
Data Integrity	Month 1	Technical and PMs
Ethics Refresher	Annually	All
Quality Assurance	Quarter 1	All
Initial Demonstration of Capability (IDOC)	Prior to unsupervised method Performance	Technical

¹ From the date of initial employment unless otherwise indicated.

The quality assurance training includes an overview of regulatory programs and program goals, a review of the ethics statement, and group discussions about data integrity and data misrepresentation.

When an analyst does not meet these requirements, they can perform a task under the supervision of a qualified analyst, peer reviewer or section 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.

IDOCs (Initial Demonstration of Method Capability) are performed by the analysis of four replicate QC samples. Results of successive LCS analyses can be used to fulfill the IDOC requirement, however, LCSs performed over several batches is desirable. The accuracy and precision, measured as average recovery and standard deviation (using n-1 as the population), of the 4 replicate results are calculated and compared to those in the test method (where available). If the test method does not include accuracy and precision requirements, the results are compared to target criteria set by the laboratory. The laboratory sets the target criteria such that they reflect the DQOs of the specific test method or project. An IDOC Certification Statement is recorded and maintained in the employee's training file. Tabulated results summary and raw data are completed and signed by the analyst and section manager with the proper entries made onto the analysts training record. The data is submitted to the QA department for approval and entry into the master IDOC spreadsheet and for filing. Figure 4 shows an example of an *IDOC Certification Statement*. (CHI-22-09-271)

On an annual basis, the analyst's method capabilities must be evaluated. The requirement that a CDOC (Continued Demonstration of Capability) be completed for each method currently being analyzed must be presented for approval to the QA department. (e.g. *Yearly Method Capability Review Work Instruction-Wet Chemistry: CHI-22-09-279*)

Further details of the laboratory's training program are described in the Laboratory Training SOP (UQA-014).

Figure 4. Demonstration of Capability Certification Statement

Demonstration of Capability Certification Statement		
Date: STL Chicago 2417 Bond Street University Park, IL 60466		
Analyst Name: _____		
SOP No.: _____		
Method No.: _____		
Description: _____		
Matrix: _____		
Effective Date: _____		
We the undersigned certify that:		
1.	The analyst identified above, using the cited test method(s), which is in use at this laboratory for the analysis of samples under the National Environmental Laboratory Accreditation Program, have met the Demonstration of Capability.	
2.	The test method(s) was performed by the analyst identified on this certification.	
3.	A copy of the reference method and laboratory-specific SOP(s) are available for all personnel on-site.	
4.	The data associated with the demonstration capability are true, accurate, complete and self-explanatory.	
5.	All raw data (including a copy of this certification form) necessary to reconstruct and validate these analyses have been retained at the laboratory, and that the associated information is well organized and available for review by authorized assessors.	
_____ Technical Manager	_____ Signature	_____ Date
_____ Quality Assurance Manager	_____ Signature	_____ Date

5.1.3 Ethics Policy

Establishing and maintaining a high ethical standard is an important element of a Quality System. In order to ensure that all personnel understand the importance the company places on maintaining high ethical standards at all times; STL has established an Ethics Policy (P-L-006) and an Ethics Agreement (Figure 5). Each employee signs the Ethics Agreement, signifying agreed compliance with its stated purpose on an annual basis.

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

Ethics is also a major component of STL's quality and data integrity systems. Each employee is trained in ethics within thirty days of hire and quality training within three months of hire. Annual ethics refresher training will be provided. Employees are trained as to the legal and environmental repercussions that result from data misrepresentation. A data integrity hotline is maintained by STL and administered by the Corporate Quality Director.

Figure 5. STL Ethics Agreement

I understand that STL is committed to ensuring the highest standard of quality and integrity of the data and services provided to our clients. I have read the Ethics Policy of the Company.

With regard to the duties I perform and the data I report in connection with my employment at the Company, I agree that:

- I will not intentionally report data values that are not the actual values obtained;
- I will not intentionally report the dates, times, sample or QC identification, or method citations of data analyses that are not the actual dates, times, sample or QC identifications, or method citations;
- I will not intentionally misrepresent another individual's work;
- I will not intentionally report data values that do not meet established quality control criteria as set forth in the Method and/or Standard Operating Procedures, or as defined by Company Policy;
- I agree to inform my Supervisor of any accidental reporting of non-authentic data by me in a timely manner; and I agree to inform my Supervisor of any accidental or intentional reporting of non-authentic data by other employees; and
- If a supervisor or a member of STL management requests me to engage in or perform an activity that I feel is compromising data validity or quality, I will not comply with the request and report this action immediately to a member of senior management, up to and including the President of STL.

As a STL employee, I understand that I have the responsibility to conduct myself with integrity in accordance with the ethical standards described in the Ethics Policy. I will also report any information relating to possible kickbacks or violations of the Procurement Integrity Act, or other questionable conduct in the course of sales or purchasing activities. I will not knowingly participate in any such activity and will report any actual or suspected violation of this policy to management.

The Ethics Policy has been explained to me by my supervisor or at a training session, and I have had the opportunity to ask questions if I did not understand any part of it. I understand that any violation of this policy subjects me to disciplinary action, which can include termination. In addition, I understand that any violation of this policy which relates to work under a government contract or subcontract could also subject me to the potential for prosecution under federal law.

EMPLOYEE SIGNATURE: _____ Date: _____
 Supervisor/Trainer: _____ Date: _____

5.2 Facilities

The laboratory is a secure facility with controlled and documented access. Access is controlled by various measures including locked doors, electronic access cards, security codes, and a staffed reception area. All visitors sign in and are escorted by STL personnel while at the facility. The laboratory is locked at all times, unless a receptionist is present to monitor building access (e.g., between the hours of 8:00 a.m. and 5:00 p.m. Monday through Friday).

The facility is designed for efficient, automated high-quality operations. The laboratory is equipped with Heating, Ventilation, and Air Conditioning (HVAC) systems appropriate to the needs of environmental testing laboratories. Environmental conditions in the facility, such as hood flow, are routinely monitored and documented.

The facility 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. STL also provides and requires the use of protective equipment including safety glasses, protective clothing, gloves, etc..

5.3 Test 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.

5.3.1 Method Selection

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 in a Technical Profile and within LabNets Project Notes feature. 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.

Most of the test methods performed at STL originate from test methods published by a regulatory agency such as the US EPA and other state and federal regulatory agencies. These include, but are not limited to, the following published compendiums of test methods. A listing of methods in which the laboratory is capable of performing is listed in laboratory's *Methods Capabilities Work Instruction* (CHI-22-09-255).

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.

Method 1664, Revision A: N-Hexane Extractable Material (HEM; Oil and Grease) and Silica Gel Treated N-Hexane Extractable Material (SGT-HEM); Non-polar Material) by Extraction and Gravimetry, EPA-821-R-98-003, February 1999.

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.

NIOSH Manual of Analytical Methods, 4th ed., August 1994.

Statement of Work for Inorganics Analysis, ILM04.0, USEPA Contract Laboratory Program Multi-media, Multi-concentration.

Statement of Work for Organics Analysis, OLM04.2 and OLC02.1, USEPA Contract Laboratory Program, Multi-media, Multi-concentration.

Standard Methods for the Examination of Water and Wastewater, 18th/19th/20th 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 (SW-846), Third Edition, September 1986, Final Update I, July 1992, Final Update IIA, August 1993, Final Update II, September 1994; Final Update IIB, January 1995; Final Update III, December 1996.

Annual Book of ASTM Standards, American Society for Testing & Materials (ASTM), Philadelphia, PA.

The laboratory reviews updated versions to all the aforementioned references for adaptation based upon capabilities, instrumentation, etc., and establishes an implementation schedule. As such, the laboratory strives to perform only the latest versions of each approved method.

5.3.2 SOPs

STL maintains an *Approved SOP Listing* (CHI-22-09-SOP) for both Method and Process SOPs. Method SOPs are maintained to describe a specific test method. Process SOPs are maintained to describe function and processes not related to a analytical testing (e.g., administrative procedures).

Method SOPs contain the following information:

Title Page with Document Name, Document Number, Revision Number, Effective Date, Page Numbers and Total # of Pages, Authorized Signatures, Dates and Proprietary Information Statement (Figure 6).

- | | |
|---|--|
| <ol style="list-style-type: none"> 1. Identification of Test Method 2. Applicable Matrix 3. Scope and Application, including test analytes 4. Summary of the Test Method 5. Reporting Limits 6. Definitions 7. Interferences 8. Safety 9. Equipment and Supplies 10. Reagents and Standards 11. Sample Collection, Preservation and Storage 12. Quality Control | <ol style="list-style-type: none"> 13. Calibration and Standardization 14. Procedure 15. Calculations 16. Method Performance 17. Pollution Prevention 18. Data Assessment and Acceptance Criteria for Quality Control Measures 19. Corrective Actions for Out-of-Control Data 20. Contingencies for Handling Out-of-Control or Unacceptable Data 21. Waste Management 22. References 23. Tables, Diagrams, Flowcharts and Validation Data |
|---|--|

Process SOPs contain the following information:

Title Page with Document Name, Document Number, Revision Number, Effective Date, Page Numbers and Total # of Pages, Authorized Signatures, Dates and Proprietary Information Statement (Figure 6).

1. Scope
2. Summary
3. Definitions
4. Responsibilities
5. Procedure
6. References
7. Tables, Diagrams, and Flowcharts

The QA Department is responsible for maintenance of SOPs, archival of SOP historical revisions, maintenance of an SOP index, and records of controlled distribution. SOPs, at a minimum, undergo annual review (12-18 months). Where an SOP is based on a published method, the laboratory maintains a copy of the reference method.

Figure 6. Proprietary Information Statement

This documentation has been prepared by Severn Trent Laboratories (STL) solely for STL's own use and the use of STL's customers in evaluating its qualifications and capabilities in connection with a particular project. The user of this document agrees by its acceptance to return it to STL upon request and not to reproduce, copy, lend, or otherwise disclose its contents, directly or indirectly, and not to use it for any other purpose other than that for which it was specifically provided. The user also agrees that where consultants or other outside parties are involved in the evaluation process, access to these documents shall not be given to said parties unless those parties also specifically agree to these conditions.

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SOP Change Form

The SOP Change Form is used for implementation, documentation, and authorization of changes to SOPs (*SOP Change Protocol*; UQA-032). Immediate changes in SOPs may be necessary to accommodate improvements; to implement acceptable changes in practices; or to correct potential errors in the existing version. The reason for the change will be identified and a detailed description of the procedure change will be presented. Since this form will become part of the referenced SOP, until such time that the SOP is updated, it must be legible and comprehensible. The Change Form must provide an exact description and identify the affected sections.

Once this form is completed and changes are authorized, it becomes an official part of the SOP for which it revises, and is subject to all document control and records management policies.

5.3.3 Method Validation

Laboratory developed methods are validated and documented according to the procedure described in Section 5.3.5.

5.3.4 Method Verification

Method verification is required when a validated standard test method or a method modification is implemented. The level of activity required for method verification is dependent on the type of method being implemented, or on the level of method modification and its affect on a method's robustness. Method modification often takes advantage of a method's robustness, or the ability to make minor changes in a method without affecting the method's outcome.

It is the responsibility of the section manager to present to the QA manager all applicable method validation studies for review and approval. The documented approval by the section manager and QA manager must be applied to all applicable validation records before the method is released for use. Method verification may require some, but not all, of the activities described in Section 5.3.5.

5.3.5 Method Validation and Verification Activities

Before analyzing samples by a particular method, method validation and/or method verification must occur. A complete validation of the method is required for laboratory developed methods. While method validation can take various courses, the following activities can be required as part of method validation. Method validation records are designated QC records and are archived accordingly.

Determination of Method Selectivity

Method selectivity is demonstrated for the analyte(s) in the specific matrix or matrices. In some cases, to achieve the required selectivity for an analyte, a confirmation analysis is required as part of the method.

Determination of Method Sensitivity

Sensitivity can be both estimated and demonstrated. Whether a study is required to estimate sensitivity depends on the level of method development required when applying a particular measurement system to a specific set of samples. Where estimations and/or demonstrations of sensitivity are required by regulation or client agreement, such as the procedure in 40 CFR Part 136 Appendix B, under the Clean Water Act, these shall be followed. The laboratory determines MDLs are described in Section 4.4.3.6 and within UQA-017 and the corporate procedure S-Q-003.

Relationship of Limit of Detection (LOD) to the Quantitation Limit (QL)

An important characteristic of expression of sensitivity is the difference in the LOD and the QL. The LOD is the minimum level at which the presence of an analyte can be reliably concluded. The QL is the minimum level at which both the presence of an analyte and its concentration can be reliably determined. For most instrumental measurement systems, there is a region where semi-quantitative data is generated around the LOD (both above and below the estimated MDL or LOD) and below the QL. In this region, detection of an analyte may be confirmed but quantification of the analyte is unreliable within the accuracy and precision guidelines of the measurement system. When an analyte is detected below the QL, and the presence of the analyte is confirmed by meeting the qualitative identification criteria for the analyte, the analyte can be reliably reported, but the amount of the analyte can only be estimated. If data is to be reported in this region, it must be done so with a qualification that denotes the semi-quantitative nature of the result.

Determination of Interferences

A determination that the method is free from interferences in a blank matrix is performed.

Determination of Range

Where appropriate, a determination of the applicable range of the method may be performed. In most cases, range is determined and demonstrated by comparison of the response of an analyte in a curve to established or targeted criteria. The curve is used to establish the range of quantitation

and the lower and upper values of the curve represent upper and lower quantitation limits. Curves are not limited to linear relationships.

Demonstration of Capability

DOCs are performed prior to method performance.

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.

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 Appendix describing the specific differences in the new method is acceptable in place of a separate SOP.

Continued Demonstration of Method Performance

Continued demonstration of Method Performance is addressed in the SOP. Continued demonstration of method performance is generally accomplished by batch specific QC samples such as LCS and Method Blanks.

5.3.6 Data Reduction and Review

Analytical data are entered/downloaded directly into LabNet or recorded on pre-formatted bench sheets that are paginated and bound into laboratory logbooks. These logbooks are issued and controlled by the laboratory's QA Section. A unique document control code is assigned to each book to assure that chronological record keeping is maintained. Analytical data may be electronically stored as a secure .pdf file to which the analyst applies an electronic signature.

Analytical data is referenced to a unique sample identification number for internal tracking and reporting. Both LabNet entries and logbook pages contain the following information, as applicable: analytical method, analyst, date, sequential page number, associated sample numbers, standard concentrations, instrument settings, and raw data. Entries are in chronological order and maintained so as to enable reconstruction of the analytical sequence.

The analyst is responsible for entering / recording all appropriate information, and for signing and dating all logbook entries daily. All entries and logbook pages are reviewed for completeness by a supervisor, peer reviewer or the analyst themselves. Data review checklists document the analytical review of the LabNet entries, logbook and associated QC indicators. Copies of instrument outputs (chromatograms, mass spectra, etc..) are maintained on file or electronically with the analyst's signature/initials and date.

5.3.6.1 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 section manager or alternate analyst prior to updating the data in LabNet. 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 STL Corporate SOP entitled *Acceptable Manual Integration Practices* (S-Q-004).

Copies of all raw data and the calculations used to generate the final results, such as bound logbooks, are retained on file for a minimum of 5 years or as otherwise requested by the client/project.

Calculations and data reduction steps for various methods are summarized in the respective analytical SOPs or program requirements.

5.3.6.2 Data Review

All data, regardless of regulatory program or level of reporting, are subject to a thorough review process. The individual analyst continually reviews the quality of the data through calibration checks, quality control sample results and performance evaluation samples. Data review is initiated by the analyst during, immediately following, and after the completed analysis.

All levels of the review are documented on Data Review Checklists that are specific to each laboratory section.

GC Extractables/HPLC:	CHI-22-17-034
GC Volatiles:	CHI-22-19-003
GC/MS Volatiles and Semivolatiles:	CHI-22-20-038
Metals:	CHI-22-14-004, CHI-22-14-005, CHI-22-14-006
Wet Chemistry:	CHI-22-12-014

Primary Review

The primary review is often referred to as a "bench-level" review. In most cases, the analyst who generates the data (e.g., logs in, prepares and/or analyzes the samples) is the primary reviewer. In some cases, an analyst may be reducing data for samples run by an auto-sampler set up by a different analyst. In this case, the identity of both the analyst and the primary reviewer is identified in the raw data.

One of the most important aspects of primary review is to make sure that the test instructions are clear, and that all project specific requirements have been understood and followed.

Once an analysis is complete, the primary reviewer ensures, where applicable, that:

- ◆ Sample preparation information is complete, accurate, and documented.
- ◆ Calculations have been performed correctly.
- ◆ Quantitation has been performed accurately.
- ◆ Qualitative identifications are accurate.
- ◆ Manual integrations are appropriate.
- ◆ Data flags to indicate manual integrations are recorded.
- ◆ Manual integrations are authorized by a date and signature or initials of primary analyst.
- ◆ Client specific requirements have been followed.
- ◆ Method and process SOPs have been followed.
- ◆ Method QC criteria have been met.
- ◆ QC samples are within established limits.
- ◆ Dilution factors are correctly recorded and applied.
- ◆ Non-conformances and/or anomalous data have been properly documented and appropriately communicated.
- ◆ COC procedures have been followed.
- ◆ Primary review is documented by date and initials/signature of primary analyst.

Any anomalous results and/or non-conformances noted during the Primary Review are documented on the Data Review Checklist and on an SDR; and are communicated to the Section Manager and the Project Manager for resolution. Resolution can require sample reanalysis, or it may require that data be reported with a qualification. Non-conformances are documented per Section 4.9.

Secondary Review

The secondary review is also a complete technical review of a data and is performed by the Section Manager, analyst or data specialist. The secondary review is documented on the same Data Review Checklist as the primary review.

The following items are reviewed:

- Qualitative Identification
- Quantitative Accuracy
- Calibration
- QC Samples
- Method QC Criteria
- Adherence to method and process SOPs
- Accuracy of Final Client Reporting Forms
- Manual Integrations – Minimal requirement is to spot-check raw data files for manual integration, as verified by date and initials or signature of secondary data reviewer. Some regulatory programs require 100% secondary review of manual integrations.
- Completeness
- Special Requirements/Instructions

If problems are found during the secondary review, the reviewer must work with the appropriate personnel to resolve them. If changes are made to the data, such as alternate qualitative identifications, identifications of additional target analytes, re-quantitation, or re-integration, the secondary reviewer must contact the laboratory analyst and/or primary reviewer of the data so that the primary analyst and/or reviewer is aware of the appropriate reporting procedures.

Completeness Review

The completeness review includes the generation of a project narrative and/or cover letter which outlines anomalous data and non-compliances using project narrative notes and SDRs or CARs (non-compliance reports) generated during the primary and secondary review. The completeness review addresses the following items:

- Is the project report complete?
- Does the data meet with the client's expectations?
- Were the data quality objectives of the project met?

Are QC outages and/or non-conformances approved and appropriately explained in the narrative notes?

The laboratory Section Manager(s), Data Management personnel and the Project Manager contribute to the completeness review.

5.3.7 Data Integrity and Security

This section details those procedures that are relevant to computer systems that collect, analyze, and process raw instrumental data, and those that manage and report data.

Security and Traceability

Access to the laboratory's LabNet system, STL's proprietary LIMS, that collects, analyzes, and processes raw instrumental data, and those that manage and report data is both controlled and recorded. System users are granted access levels that are commensurate with their training and responsibilities.

Control of the system is accomplished through limitation of access to the system by users with the education, training and experience to perform the task knowledgeably and accurately. System users are granted privileges that are commensurate with their experience and responsibilities.

Computer access is tracked by using unique login names and passwords for all employees that have access to the computer system. Entries and changes are documented with the identity of the individual making the entry, and the time and date. Where a computer system is processing raw instrumental data, the instrument identification number as described in Section 5.4.1 is recorded. The system has the capability of maintaining audit trails to track entries and changes to the data. This function is activated on any computer system that has that capability (e.g., Target).

Verification

All the LabNet software programs have been verified prior to use and prior to the implementation of any version upgrades. Verification involves assessing whether the computer system accurately performs its intended function. Verification generally is accomplished by comparing the output of the program with the output of the raw data manually processed, or processed by the software being replaced. The verification of LabNet software programs are conducted by the QA manager with the assistance of the section managers and unit leaders. The QA manager documents the approval of the program verifications. All records of the verification are retained as QC records.

Validation

Software validation involves documentation of specifications and coding as well as verification of results. Software validation is performed by the QA manager on all in house programs. (LabNet) Records of validation include original specifications, identity of code, printout of code, software name, software version, name of individual writing the code, comparison of program output with specifications, and verification records as specified above. Records of validation are retained as QC records.

The QA manager must retain documentation of the validation process as defined above. The QA manager is the sole LabNet Methods Administrator at the laboratory and has the responsibility to validate any LabNet methods, calculations or criteria codes prior to use for sample analysis.

Auditing

STLs LabNet System Managers continually review the control, security, and tracking of IT systems and software.

Version Control

The laboratory maintains copies of outdated versions of software and associated manuals for all software in use at the laboratory for a period of 5 years from its retirement date. The associated hardware, required to operate the software, is also retained for the same time period.

5.4 Equipment

5.4.1 Equipment Operation

STL is committed to routinely updating and automating instrumentation. The laboratory maintains state of the art instrumentation to perform the analyses within the QC specifications of the test methods. The laboratory maintains an Equipment Tracking Form (CHI-22-09-068) for each piece of equipment and instrumentation that documents the following information:

- ◆ Identity
- ◆ Date In Service
- ◆ Manufacturer's Name, Model Number, Serial Number
- ◆ Current Location
- ◆ Preventative Maintenance Schedule

All equipment is subject to rigorous checks upon its receipt, upgrade, or modification to establish that the equipment meets with the selectivity, accuracy, and precision required by the test method for which it is to be used. All manufacturer's operations and maintenance manuals are kept up to date and accessible for the use of the equipment operator. Documentation of equipment usage is maintained using analytical run and maintenance logbooks.

5.4.2 Equipment Maintenance

STL employs a system of preventative maintenance in order to ensure system up time, minimize corrective maintenance costs and ensure data validity. All routine maintenance is performed as recommended by the manufacturer and may be performed by an analyst, instrument specialist or outside technician. Maintenance logbooks are kept on all major pieces of equipment in which both routine and non-routine maintenance is recorded.

Any item of equipment or instrumentation that has been subjected to overloading or mishandling, provides suspected results, has been shown by verification or otherwise to be defective, is new or not been used for an extended period of time, is taken out of services and tagged as "DO NOT USE INSTRUMENT". The tag is signed/dated by the person removing the item from service and noted as to the reason of in-operation (*Instrument and Equipment Out-of-Service Tagging*; UQA-012).

Any instrumentation that is brought back on-line must have MDLs and DOCs performed and have acceptance within prescribe criteria; or calibrated by a certified agency (e.g., balances or Class S weights) and tagged as being within calibration specifications; and proven to provide consistent measurements (e.g., refrigerators, eppendorf pipettes, ovens).

The return to analytical control following instrument repair is documented in the maintenance logbook. Maintenance logbooks are retained as QC records. Notation of the date and maintenance activity is recorded each time service procedures are performed. Maintenance logbooks are retained as QA records.

Table 9. Major Equipment Maintenance

Instrument	Procedure	Frequency
Hewlett Packard GC/MS	Ion gauge tube degassing Pump oil-level check Pump oil changing Analyzer bake-out Analyzer cleaning Resolution adjustment COMPUTER SYSTEM AND PRINTER: Air filter cleaning Change data system air filter Printer head carriage lubrication Paper sprocket cleaning Drive belt lubrication	As required Monthly Annually As required As required As required As required As required As required As required As required
Gas Chromatograph	Compare standard response to previous day or since last initial calibration Check carrier gas flow rate in column Check temp. of detector, inlet, column oven Septum replacement Check system for gas leaks with SNOOP Check for loose/frayed wires and insulation Bake injector/column Change/remove sections of guard column Replace connectors/liners Change/replace column(s)	Daily Daily via use of known compound retention Daily As required W/cylinder change as required Monthly As Required As Required As Required As Required
Electron Capture Detector (ECD)	Detector wipe test (Ni-63) Detector cleaning	Semi-annually As required
Flame Ionization Detector (FID)	Detector cleaning	As required
Photoionization Detector (PID)	Change O-rings Clean lamp window	As required As required
HPLC	Change guard columns Change lamps Change pump seals Replace tubing Change fuses in power supply Filter all samples Change autosampler rotor/stator	As required As required Semi-annually or as required As required As required Daily As required
Balances	Class "S" traceable weight check Clean pan and check if level Field service	Daily, when used Daily At least Annually
Conductivity Meter	0.01 M KCl calibration Conductivity cell cleaning	Daily As required
Turbidimeter	Check light bulb	Daily, when used

Maintenance contracts are held on specific pieces of equipment where outside service is efficient, cost-effective, and necessary for effective operation of the laboratory. Table 9 lists STL's major equipment and the suggested maintenance procedures.

Table 9. Major Equipment Maintenance

Instrument	Procedure	Frequency
AA (Graphite Furnace)	Clean lens and furnace head Replace windows Check or change cuvette Check & drain compressor drain Clean atomizer cell/furnace hood Nebulizer cleaned/dried Check/change marble stones Clean filters Change graphite tube/platform Empty waste container Remove carbon tube and check wear Check sample introduction probe	Daily As required Daily Daily Daily Weekly or as required Weekly Weekly As required Daily Daily Daily
Leeman Mercury Analyzer	Check tubing for wear Fill rinse tank with 10% HCl Insert clean drying tube filled with Magnesium Perchlorate Fill reductant bottle with 10% Stannous Chloride	Daily Daily Daily Daily
ICP	Check pump tubing Check liquid argon supply Check fluid level in waste container Check filters Clean or replace filters Check torch Check sample spray chamber for debris Clean and align nebulizer Check entrance slit for debris Change printer ribbon Replace pump tubing	Daily Daily Daily Weekly As required Daily Monthly Monthly Monthly As required As required
UV-Vis Spectrophotometer	Clean ambient flow cell Precision check/alignment of flow cell Wavelength verification check	As required As required Semi-annually
Auto Analyzers	Clean sampler Check all tubing Clean inside of colorimeter Clean pump well and pump rollers Clean wash fluid receptacle Oil rollers/chains/side rails Clean optics and cells	Daily Daily Daily Quarterly Weekly Weekly Quarterly

Table 9. Major Equipment Maintenance

Instrument	Procedure	Frequency
Deionized/Distilled Water	Check conductivity Check deionizer light Monitor for VOA's System cleaning Replace cartridge & large mixed bed resins	Daily Daily Daily As required As required
Drying Ovens	Temperature monitoring Temperature adjustments	Daily As required
Refrigerators/ Freezers	Temperature monitoring Temperature adjustment Defrosting/cleaning	Daily As required As required
Vacuum Pumps/ Air Compressor	Drained Belts checked Lubricated	Weekly Monthly Semi-annually
pH/Specific Ion Meter	Calibration/check slope Clean electrode	Daily As required
BOD Incubator	Temperature monitoring Coil and incubator cleaning	Daily Monthly
Centrifuge	Check brushes and bearings	Every 6 months or as needed
Water baths	Temperature monitoring Water replaced	Daily Monthly or as needed

5.4.3 Equipment Verification and Calibration

All equipment is calibrated prior to use (Initial Calibration) to establish its ability to meet the QC guidelines contained in the test method for which the instrumentation is to be used. All sample measurements are made within the calibrated range of the instrument and in compliance with method requirements. The calibration data, which includes instrument conditions and standard concentrations, is documented in pre-formatted instrument runlogs or within LabNet itself. The preparation of all reference materials used for calibration is documented via LabNet.

Once an instrument is calibrated, ongoing instrument calibration is demonstrated (Continuing Calibration) at the appropriate frequency as defined in the test method. Refer to the STL Corporate Policy *Selection of Calibration Points* (P-T-001), for guidance on using calibration data. Any instrument that is deemed to be malfunctioning is clearly marked and taken out of service. When the instrument is brought back into control, acceptable performance is documented.

5.4.3.1 Instrument Calibration

Specific instrument calibration procedures for various instruments are summarized further in this section, and detailed in the respective analytical methods. Typically, more than one analytical method is available for an analysis. These various methods and other program requirements (e.g., U.S. EPA CLP, AFCEE, NFESC, USACE, QAPPs, contracts, etc.) may specify different calibration

requirements. Therefore, calibration details as specified in the respective laboratory SOPs, Technical Profiles, QAPP, program requirements, and contracts supersede the general instrument calibration procedures are described further in Table 10. Complete details are provided in each method SOP.

Table 10. Minimum Instrument Calibration Procedures

Technique	Activity	Minimum Requirements
Metals (ICAP)	Initial Calibration	<p>Following a period of time sufficient to warm up the instrument, the ICP is calibrated prior to each analytical run or minimally every 24 hours. Calibration standards are prepared from reliable reference materials and contain all metals for which analyses are being conducted. Working calibration standards are prepared fresh daily.</p> <p>Quarterly, multi-concentration calibration is performed to document linearity. On a day-to-day basis, 4 calibration standards (blank, high standard, 50% standard, and 20% standard) are analyzed. Prior to an analytical run, the instrument is calibrated using three standards. An Initial Calibration Verification (ICV) standard is analyzed immediately after standardization, followed by an Initial Calibration Blank (ICB). The ICV is from a source other than that used for initial calibration and the ICB must be free of target analytes at and above the value to be reported or appropriate corrective action must be taken. ICP Interference Check Samples (ICSA/ICSAB) are analyzed at the frequency described in each method SOP.</p>
	Continuing Calibration	<p>The initial calibration is verified during the analysis sequence by analysis of a Continuing Calibration Verification (CCV) standard and a Continuing Calibration Blank (CCB). The response of the CCV must be within the SOP-specified criteria (e.g., $\pm 10\%$ recovery of the true value). The CCB must be free of target analytes at or above the value to be reported or appropriate corrective action must be taken. If any ICVs/CCVs or blanks exceed their acceptance criteria, appropriate corrective action must be taken.</p>
Atomic Absorption (GFAA/ CVAA)	Initial Calibration	<p>Initial calibration will include analysis of a calibration blank and a minimum of four (4) calibration standards covering the anticipated range of measurement. Duplicate injections are made for each concentration. Response readings, e.g., absorbance, are recorded and the resultant standard calibration curve calculated. If the SOP or program-specified criteria are not met, appropriate corrective action must be taken.</p> <p>An ICV standard will be analyzed immediately after standardization. The ICV must be within SOP-specified criteria (e.g., $\pm 5\%$ of the true value for drinking water, and $\pm 10\%$ in most other cases), or the initial calibration must be repeated. The ICV must be from a source other than that used for initial calibration.</p> <p>An ICB will be analyzed after the ICV. The ICB must be free of target analytes at and above a concentration in which sample results are reported, or corrective action must be taken.</p>
	Continuing Calibration	<p>The initial calibration is verified during the analysis sequence by evaluation of a CCV standard and a CCB, as described above. The CCV value must be within SOP-specified criteria (e.g., $\pm 10\%$ recovery of the true value except for mercury within $\pm 20\%$ of the true value). The CCB must be free of target analytes at and above the concentration reported in samples.</p> <p>If any ICVs/CCVs or blanks exceed their acceptance criteria, corrective action must be taken.</p>

Table 10. Minimum Instrument Calibration Procedures

Technique	Activity	Minimum Requirements
Inorganic Colorimetric Methods	Initial Calibration	<p>A full initial standard calibration curve will be prepared for all colorimetric analyses on a daily basis. Working standards to define this curve will include a minimum of five (5) concentrations which cover the anticipated range of measurement, plus a calibration blank. At least one of the calibration standards will be at a concentration which will enable verification of instrument response near the reporting limit as defined in Section 8.6 or a level suitable for meeting specific program requirements. The requirement for an acceptable initial calibration is described in the analytical SOP. If the criteria are not met, appropriate corrective action must be taken. Calibration data, e.g., correlation coefficient, is entered into the laboratory notebook, or associated instrument printouts, and retained with the sample data.</p> <p>In lieu of a full initial curve, a daily calibration verification may be analyzed. This daily calibration will at a minimum consist of a blank and a mid-range standard. Results must be within SOP-specified criteria. If not, reanalysis of the standards may be done once to verify the readings; otherwise, a new curve will be developed.</p> <p>For procedures that require pretreatment steps, a minimum of one standard shall be prepared with the pretreatment. If the pre-treated standard is within SOP-specified criteria, the curve will be used. If the pre-treated sample is not within the criteria, the reason will be determined. If it is determined that the difference between the curves is inherent in the procedure, the curve will be based on the standards prepared and carried through the pretreatment.</p> <p>An ICV will be analyzed immediately after the standardization, followed by an ICB. The ICV must be from a source other than that used for initial calibration. The ICV must be within SOP-specified criteria and the ICB must be free of target analytes or appropriate corrective action must be taken.</p>
	Continuing Calibration	<p>The initial calibration is verified during the analysis sequence by analysis of a CCB and a CCV. If any ICVs/CCVs or blanks exceed their acceptance criteria, analysis is terminated, and the instrument is recalibrated. All samples since the last valid calibration verification are reanalyzed.</p>
Ion Chromatography	Initial Calibration	<p>The ion chromatograph will be calibrated prior to each day of use. Calibration standards will be prepared from appropriate reference materials and will include a blank and a minimum of three concentrations to cover the anticipated range of measurements. At least one of the calibration standards will be at a concentration which will enable verification of instrument response near the reporting limit. If SOP-specified calibration criteria cannot be achieved, appropriate corrective action must be taken. Calibration data, e.g., correlation coefficient, will be archived with sample raw data.</p>
	Continuing Calibration	<p>A continuing calibration standard and blank will be analyzed at a frequency of 10% and at the end of the analysis shift. The response calculated as a percent recovery of the standard must meet SOP or program-specific criteria. The response of the blank must be less than the concentration to be reported for samples analyzed.</p>

Table 10. Minimum Instrument Calibration Procedures

Technique	Activity	Minimum Requirements
GC/MS		All GC/MS instrumentation is calibrated to set specifications prior to sample analysis. These specifications vary depending on the requirements of the analytical program and the designated analytical method.
	Tuning and Mass Calibration	<p>Mass spectrometers are calibrated with perfluorotributylamine (FC-43) or perfluorophenanthrene (FC- 5311) as required to ensure correct mass assignment. In addition, at the beginning of the daily work shift, the GC/MS system must be tuned with decafluorotriphenylphosphine (DFTPP) for semivolatiles analysis and 4-bromofluorobenzene (BFB) for volatiles analysis, and calibrated to target compounds.</p> <p>The majority of the laboratory work utilizes U.S. EPA-CLP or SW-846 protocols, which define the work shift as a 12-hour period initiated by the injection of DFTPP, BFB, or the dioxin/furan window mix. For drinking water programs (500 series methods), a 12-hour work shift is specified in the method for calibration frequency. For wastewater programs (600 series methods), the tune expires when the day's analytical sequence is complete; however, no time limit is given for the length of the daily GC/MS work shift. Ion abundances will be within the windows dictated by the specific program requirements.</p>
	Initial Calibration	<p>After an instrument has been tuned, initial calibration curves (minimum of 3-5 points) are generated for the compounds of interest. The low level standard must be at a concentration which will enable verification of instrument response near the reporting limit or at a concentration acceptable to meet program requirements. The other standards must extend through the linear working range of the detector. The parameters requiring quantitation must meet SOP or program-specified criteria prior to initiation of sample analysis. Any sample extracts containing parameters of interest which exceed the concentration of the high level standard, must be diluted to bring the parameters within the range of the standards. Instrument response to these target compounds are evaluated against SOP-specified criteria. Linearity is verified by evaluating the response factors (RF) for the initial calibration standards against SOP-specified criteria.</p> <p>Once an acceptable calibration is obtained, samples may be analyzed up until the expiration of the tune. At that time, the instrument must be re-tuned prior to further analysis. After acceptable tuning, a continuing calibration standard may be analyzed in lieu of a full multi-point calibration if the SOP-specified criteria are met.</p> <p>The majority of compounds analyzed for GC/MS comprise EPA's Target Compound List (TCL) or Priority Pollutant List (PPL). For add-on compounds not on the current TCL or PPL, initial calibration may be performed using a single point calibration of the additional compound(s), unless prior arrangements are made for a full three-to-five point calibration. Calibration data, to include linearity verification, will be maintained in the laboratory's records of instrument calibrations.</p>
	Continuing Calibration	During each operating shift, a single calibration standard may be analyzed to verify that the instrument responses are still within the initial calibration determinations, as defined in the specific SOPs. If criteria cannot be met, appropriate corrective action must be taken.

Table 10. Minimum Instrument Calibration Procedures

Technique	Activity	Minimum Requirements
GC and HPLC		Gas chromatographs and high performance liquid chromatographs will be calibrated prior to use as described in analytical SOP or program requirements. Calibration standard mixtures will be prepared from appropriate reference materials and will contain analytes appropriate for the method of analysis or program requirements.
	Initial Calibration	Initial calibration will include a minimum of 3 to 5 calibration standards covering the anticipated range of measurement. The low level standard must be at a concentration which will enable verification of instrument response near the reporting limit or at a concentration acceptable to meet program requirements. The other standards must extend through the linear working range of the detector. The parameters requiring quantitation must meet SOP or program-specified criteria prior to initiation of sample analysis. Any sample extracts containing parameters of interest which exceed the concentration of the high level standard, must be diluted to bring the parameters within the range of the standards.
	Continuing Calibration	<p>The response of the instrument will be verified for each analysis sequence by evaluation of a daily calibration verification standard at a mid-range concentration. In order to demonstrate that the initial calibration curve is still valid, the calibration check standard must be within SOP or program-specified acceptance criteria for the compounds of interest or the instrument must be recalibrated. For multi-analyte methods, this check standard may contain a representative number of target analytes rather than the full list of target compounds. Optionally, initial calibration (e.g., the full range of concentration levels) can be performed at the beginning of the analysis sequence.</p> <p>Within the analysis sequence, instrument drift will be monitored by analysis of a mid-range calibration standard every ten samples or 12 hour sequence (depending on the method protocol), including external QC. If the SOP or program-specified calibration criteria are not met for the compounds of interest, appropriate corrective action must be taken.</p>

5.5 Measurement Traceability

5.5.1 General

Traceability of measurements is 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 is subject to ongoing certifications of accuracy.

At a minimum, these include procedures for checking specifications for balances, thermometers, temperature, De-ionized (DI) and Reverse Osmosis (RO) water systems, automatic/ependorf pipettes and other volumetric measuring devices. Wherever possible, subsidiary or peripheral equipment is checked against standard equipment or standards that are traceable to national or international standards [with the exception of class A glassware (including glass microliter syringes that have a certificate of accuracy)].

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. Balances are calibrated on each day of use (*Balance Calibration, Care and Use*; UQA-003). All thermometers and temperature monitoring devices are calibrated annually against a traceable reference thermometer. Temperature readings of ovens, refrigerators, and incubators are checked on each day of use (*Thermometer Calibrations*; UQA-034).

Laboratory DI and RO water systems have documented preventative maintenance schedules and the conductivity of the water is recorded on each day of use (*Water Quality*; UQA-035).

5.5.2 Reference Standards

The receipt of all reference standards is documented in LabNet. Standards are obtained from commercial vendors and sources may vary depending upon the availability of mixes and solutions from vendors. Each production unit is responsible to ensure, when available, that all standards are traceable to EPA, NIST, A2LA, SARMS and are accompanied by a Certificate of Analysis that documents the standard purity. If a standard cannot be purchased from a vendor that supplies a Certificate of Analysis, the purity of the standard is documented by analysis.

The receipt of each dry chemical, purchased stock solution or reference material to be used as a standard is assigned a unique ID number. The chemical name, manufacturer, lot number, date received, expiration date, date opened and initials of the analyst who opened the chemical are documented. The expiration dates for ampulated solutions shall not exceed the manufacturer's expiration date. Expiration dates for laboratory-prepared stock and diluted standards shall be no later than the expiration date of the stock solution or material or the date calculated from the holding time allowed by the applicable analytical method, whichever comes first. Expiration dates for pure chemicals shall be established by the laboratory and be based on chemical stability, possibility of contamination, and environmental and storage conditions. Expired standard materials shall be either revalidated prior to use or discarded. Revalidation may be performed through assignment of a true value and error window statistically derived from replicate analyses of the material as compared to an unexpired standard. The laboratory labels all standard and QC materials with expiration dates.

The preparation of all daughter solutions, whether a single or multiple-component stock, intermediate, or working standard solution, is documented in a standard solution preparation logbook, in a designated section of the analytical logbook or in the LabNet systems reagent program. This documentation references the Standard ID of the respective parent solution(s) used in its preparation, providing a solid trail back to the solution or chemical received from the vendor. These records include the standard name, final volume, matrix, final concentration, analyst initials, prep date and expiration date. A daughter solution should not have an expiration date which post-dates any of the parent solutions used in its preparation.

Reference standards are labeled with a unique Standard Identification Number, date received, and the expiration date. All documentation received with the reference standard or documentation of standard purity is retained as a QC record and references the Standard Identification Number. All efforts are made to purchase standards that are $\geq 97.0\%$ purity. If this is not possible, the purity is used in performing standards calculations.

The accuracy of calibration standards is checked by comparison with a standard from a second source. In cases where a second standard manufacturer is not available, a different lot is acceptable for use as a second source. The appropriate QC criteria for specific standards are defined in laboratory SOPs. In most cases, the analysis of an ICV or LCS is used as the second source confirmation.

Storage conditions, such as shelf life, ambient or chilled, controlled or restricted access, wet or desiccated, etc., are in conformance with the specifications set in the associated method, the program requirements, or the manufacturer's recommendation, as appropriate.

5.5.3 Reagents

Reagents are, in general, required to be analytical reagent grade unless otherwise specified in method SOPs. Reagents must be, at a minimum, the purity required in the test method. The date of reagent receipt, date the reagent was opened, and the date of reagent preparation (where applicable) are documented in LabNet for reagent traceability.

5.6 Sampling

Sample representativeness and integrity are the foundations upon which meaningful analytical results rely. Where documented and approved SAPs and/or QAPPs are in place, they must be made available to the laboratory before sample receipt, and approved by laboratory management before sample receipt.

5.7 Sample Handling, Transport, and Storage

5.7.1 General

COC can be established either when bottles are sent to the field, or at the time of sampling. STL can provide all of the necessary coolers, reagent water, sample containers, preservatives, sample labels, custody seals, COC forms, ice, and packing materials required to properly preserve, pack,

and ship samples to the laboratory. Complete details for sample container preparation are contained within UCM-001. A summary of sample receipt is as follows with complete details available within the *Sample Receipt and Handling SOP* (USR-001).

Samples are received at the laboratory by the designated sample custodians and a unique LabNet job (batch) number and unique bottle ID is assigned. The following information is recorded for each sample shipment:

- ◆ Client/Project Name.
- ◆ Date and Time of Laboratory Receipt.
- ◆ Laboratory Job Number
- ◆ Signature or initials of the personnel receiving the cooler and making the entries.

Upon inspection of the cooler and custody seals, the sample custodian opens and inspects the contents of the cooler, and records the cooler temperature. If the cooler arrival temperature exceeds the required or method specified temperature range by $\pm 2^{\circ}\text{C}$ (for samples with a temperature requirement of 4°C , a cooler temperature of just above the water freezing temperature to 6°C is acceptable); sample receipt is considered "compromised" and the procedure described in Section 4.7.1 is followed. All documents are immediately inspected to assure agreement between the test samples received and the COC.

Any non-conformance, irregularity, or compromised sample receipt as described in Section 4.7.1 is documented in an SDR and Sample Receipt Checklist and brought to the immediate attention of the Project Manager for resolution with the client. The COC, shipping documents, documentation of any non-conformance, irregularity, or compromised sample receipt, record of client contact, and resulting instructions become part of the permanent project record.

Samples that are being tested at another STL facility or by an external subcontractor are repackaged, iced, and sent out under COC.

Following sample labeling as described in Section 5.7.2, the sample is placed in storage. Refrigerated storage coolers are maintained at $4 \pm 2^{\circ}\text{C}$. The temperature is continually being monitored by an electronic monitoring software program. (*Thermometer Calibrations and Electronic Monitoring: UQA-034*) All samples are stored according to the requirements outlined in the test method, and in a manner such that they are not subject to cross contamination or contamination from their environment.

Access to the laboratory is restricted to laboratory personnel or escorted guests as described in Section 5.2. Therefore, once sample possession is relinquished to the laboratory, the sample is in a designated secure area (e.g., the laboratory facility) accessible only to authorized personnel. Locked storage coolers are available for protocol (e.g., AFCEE and CLP) that require internal COC procedures.

5.7.2 Sample Identification and Traceability

The sample custodian organizes the sample containers, COCs, and all pertinent information associated with the samples. The sample identity is verified against all associated sample information. Any inconsistencies are documented via an SDR and forwarded to the Project Manager for resolution with the client prior to identifying the sample(s) into LabNet.

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.

All unused portions of samples, including empty sample containers, are returned to the secure sample control area.

5.7.3 Sub-Sampling

Taking a representative sub-sample from a container containing a soil or solid matrix 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.

After thoroughly mixing the sample within the sample container or transfer to a wip bag (or other suitable plastic bag), a sub-sample from various quadrants and depths of the sample are taken to acquire the required sample weight. Any non-homogenous looking material is avoided and noted as such within the sample preparation record.

5.7.4 Sample Preparation

Sample preparation procedures vary for each matrix and analytical method are as referenced in the laboratory SOPs.

5.7.5 Sample Disposal

Samples are retained in STL storage facilities for 30 days after the project report is sent unless prior written arrangements have been made with the client. Samples may be held longer or returned to the client per written request. Unused portions of samples are disposed of in accordance with federal, state and local regulations. The laboratory removes or defaces sample labels prior to disposal unless this is accomplished through the disposal method (e.g., samples are incinerated). Complete details on the disposal of samples, digestates, and extracts is available within the *Laboratory Waste Disposal Procedures* SOP (UWM-001).

5.8 Assuring the Quality of Test Results

5.8.1 Proficiency Testing

The laboratory analyzes Proficiency Test (PT) samples as required for accreditation and as outlined in NELAC. The laboratory participates in the PT program semi-annually for each PT field of testing for which it is accredited, according to the NELAC PT field of testing published guidelines. This includes drinking water, wastewater and solid/soil matrices.

The laboratory also participate various client PT programs, when submitted.

PT samples are handled and tested in the same manner (procedural, equipment, staff) as environmental samples. Results of PT samples are distributed to the laboratory section managers for review and corrective action, if required. Any required corrective action response to deficiencies is submitted to the QA department for review and are filed with the PT study records. PT test sample data is archived using the requirements for project and raw data record retention. Refer to the SOP: *PT Sample Tracking/Analysis (UQA-018)* for further details.

5.8.1.1 Double Blind Performance Evaluation

The laboratory participates in an annual double blind performance evaluation study. An external vendor is contracted to submit double blind samples to the laboratory. Both the level of customer service and the accuracy of the test results are assessed objectively by the external contractor, who provides a detailed report to the Corporate Quality Director and to the laboratory. This is administered as a double blind program in order to assess all facets of the laboratory's operations.

5.8.2 Control Samples

Control samples (e.g., QC indicators) are analyzed with each batch of samples to monitor laboratory performance in terms of accuracy, precision, sensitivity, selectivity, and interferences. Control samples must be uniquely identified and correlated to unique batches. Control samples further evaluate data based upon (1) Method Performance, which entails both the preparation and measurement steps; and (2) Matrix Effects, 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.

Control sample types and typical frequency of their application are outlined Sections 5.8.2.1 through 5.8.2.5 and Tables 11 through 15. Note that frequency of control samples vary with specific regulatory, methodology and project specific criteria. Complete details on method and regulatory program control samples are as listed in Sections 7 and 8 of each method SOP.

5.8.2.1 Method Performance Control Samples: Preparation Batch

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 (Table 11) and are processed through the entire analytical procedure with investigative/field samples.

Field blanks, equipment blank and trip blanks, when received, are analyzed in the same manner as other field samples. However, a field blank 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".

5.8.2.2 Method Performance Control Samples: Matrix

Matrix control samples include sample duplicates (MD), sample matrix spikes (MS), and sample surrogate spikes. These control samples help monitor for potential physical and chemical effects which may interfere with the precision and/or accuracy of the selected analytical method. Since interferences can enhance or mask the presence of target analytes, matrix control samples measure the degree of interference and are used to assist in the interpretation of the analytical results. The laboratory avoids performing matrix QC on known field blank samples, such as trip blanks and rinsates, since these samples are not indicative of the sample matrix.

Table 11. Preparation Batch Control Samples

Control Type	Details	
Method Blank (MB)	Use	Monitors for potential contamination introduced during the sample preparation and analytical processes.
	Typical Frequency ¹	1 per batch of ≤ 20 samples per matrix type per sample extraction or preparation method.
	Description	<p><u>Organics:</u> Laboratory pure water for water samples or a purified solid matrix for soil or solid samples (when available or when requested); solid matrices commonly include sodium sulfate, vendor or agency supplied soil or solid, or purchased sand; these solids may require purification at the laboratory prior to use.</p> <p><u>Inorganics:</u> Laboratory pure water for both water and soil or sediment samples. Volume/weights are selected to approximately equal the typical sample volume/weight used in sample preparation; and final results in a soil/solid batch 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 to actual field samples.</p>
Laboratory Control	Use	Measures the accuracy of the method in a blank matrix and assesses method performance independent of potential field sample matrix affects.
Sample (LCS)	Typical Frequency ¹	1 per batch of ≤ 20 samples per matrix type per sample extraction or preparation method. For multi-analyte methods, the LCS may consist of surrogates in the blank matrix, and or a representative selection of target analytes/internal standards.
	Description	Prepared from a reference source of known concentration and processed through the preparation and analysis steps concurrently with the field samples. Aqueous LCS's may be processed for solid matrices unless a solid LCS is requested; 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.
Known QC Sample	Use	Comply with regulatory requirements; check the accuracy of an analytical procedure; troubleshoot method performance problems; verify an analyst in training's ability to accurately perform a method; to verify the return-to-control after method performance problems; and may also be used as an LCS.
	Typical Frequency ¹	As defined by the client or QAPP.
	Description	Obtained from outside suppliers or agencies; generally require preparation from concentrated materials by dilution into a standard matrix; contain known analytes or compounds; acceptance limits are provided by the vendor.

¹ Denotes an STL required frequency.

Table 12. Matrix Control Samples

Control Type	Details	
Matrix Duplicate (MD)	Use	Monitors the effect of site matrix on the precision of the method; and of the reproducibility of laboratory preparation and measurement techniques. Note: Precision may also be affected by the degree of homogeneity of the sample, particularly in the case of non-aqueous samples or aqueous samples with particulates. Sample homogeneity and matrix effect should be considered when field samples are used to assess reproducibility. Note: A field duplicate, when received, measures Representativeness of sampling and the effect of the site matrix upon precision.
	Typical Frequency ¹	1 per 20 samples per matrix or per SAP/QAPP ² .
	Description	Performed by analyzing two aliquots of the same field sample independently; analyzed for each associated sample matrix (e.g., when requested by the client or the analytical method).
Matrix Spike (MS)	Use	Measures the effect of site sample matrix on the accuracy of the method.
	Typical Frequency ¹	1 per 20 samples per matrix or per SAP/QAPP.
	Description	Aliquot of a field sample which is spiked with the analytes or compounds of interest; analyzed for each associated sample matrix (when requested by the client or analytical method). The determination of MS percent recovery (% R) requires an analysis of a fortified sample and a non-fortified sample under the same procedural conditions (e.g., sample volumes, dilutions, procedural conditions, etc.). The concentration determined in the non-fortified sample is subtracted from the fortified sample concentration before determining the %R. The degree of homogeneity of the sample, particularly in the case on non-aqueous samples or samples with particulates, may affect the ability to obtain representative recoveries.
Matrix Spike Duplicate (MSD)	Use	Measures effect of site sample matrix on precision of method.
	Typical Frequency ¹	1 per 20 samples per matrix, when requested by the client or the analytical method, or per SAP/QAPP ² .
	Description	Alternative to sample duplicate. Generally, inorganic protocols specify an MD/MS and organic protocols specify an MS/MSD.
Surrogate Spike	Use	Measures method performance to sample matrix (organics only).
	Typical Frequency ¹	Every QC and analytical sample.
	Description	Compounds similar to the target analytes in structure, composition and chromatography, but not typically found in the environment, are added to each QC and analytical sample, prior to preparation (e.g., extraction). If the surrogates in an analytical batch do not all conform to established control limits, the pattern of conformance in investigative and control samples is examined to determine the presence of matrix interference or the need for corrective action.
Internal Standards	Use	Monitor the qualitative aspect of organic and 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 Denotes an STL required frequency.

² Either an MSD or an MD is required per 20 samples per matrix or per SAP/QAPP.

5.8.2.3 Matrix QC Frequencies

The frequency of matrix QC indicators depends on regulatory program compliance, a project's data quality objectives, or a client's requirements. The following frequency will be applied to samples when the regulatory programs are known and it does not conflict with project or client requirements.

Table 13. EPA Program Requirements

Program	Description ¹
SDWA	MD performed at a 10% frequency or 1 per preparation batch of ≤10 samples, whichever is more frequent.
CWA	MS (GC methods) and MD is performed at a 10% frequency or 1 per preparation batch of ≤10 samples, whichever is more frequent. For GC/MS Methods, MS is performed at a 5% frequency or 1 per preparation batch of ≤20 samples, whichever is more frequent.
RCRA	MS/MSD or MS/MD is performed at a rate of 5% per client (independent of the preparation batch). For clients submitting less than 10 samples, the method matrix QC requirement may be satisfied by another clients sample within the same prep batch unless the paperwork indicates a client requirement for matrix QC. Matrix QC will only be reported to the client who owns the data.
U.S. EPA CLP	MS/MSD or MS/MD is performed at a rate of 5% or 1 set per Sample Delivery Group (SDG) per matrix, independent of the prep batch. For NFESC samples, samples are processed in simultaneous or continuous batches.

¹ MS, MSD and MD may not be applicable to some analytical protocols because of the nature of the sample or protocol.

5.8.2.4 Method Performance Control Samples: Instrument Measurement

Control samples are used to ensure that optimum instrument performance is achieved. These samples help ensure that the proper identification and quantitation of target compounds or analytes are achieved. The instrument control samples appropriate to each analytical technique are described in laboratory SOPs for each respective method. A brief description of these checks is included in Table 14.

Table 14. Instrument Performance Control Samples

Control Type	Description	
<i>Inorganics</i>		
ICV	Use	Calibration standard of known concentration prepared from a source other than that used for the calibration standards.
	Sequence	Analyzed after the standard curve to confirm calibration.
ICB	Use	Blank water or solvent; confirms the calibration and assures that any potential contamination is less than the reporting limit.
	Sequence	Analyzed immediately after the ICV.
ICP Interference Check Samples (ICSA/ICSB)	Use	Verifies the absence of spectral interferences.
	Sequence	Analyzed consecutively at the beginning of each eight hour analytical sequence, after the ICV/ICB, and again at an eight hour frequency following a CCV/CCB. When CLP protocols are followed, the ICSA/B will be analyzed with the analytical sequence, before the final CCV/CCB.
Reporting Limit Verification Standard (CRA & CRI)	Use	Verifies linearity near the reporting limit for CLP metals analyses. (Note: CRI is at a level 2X the CRDL; CRA is near the CRDL).
	Sequence	Analyzed after the ICB. The CRI is also analyzed at the end of the eight hour analytical sequence, prior to analysis of the final CCV/CCB.
CCV	Use	Confirm that the instrument performance has not significantly changed during the analytical sequence; to verify stable calibration throughout the sequence; and/or to demonstrate that instrument response did not drift over a period of non-use. Made from a source other than that used for the standard curve.
	Sequence	Analyzed at 10% or every two hours, whichever is more frequent; also analyzed at the end of the analytical sequence.
CCB	Use	Water blank used to confirm that the baseline has not drifted and to monitor for contamination at the reporting limit.
	Sequence	Analyzed at a rate of 10% for inorganics and at a rate of 1 per 10 readings/injections or every two hours, whichever is more frequent, for CLP metals; also analyzed at the end of the analytical sequence.
ICP Metals Linear Range	Use	Verify linearity and document the upper limit of the calibration range for each element.
Analysis Standard (LRS)	Sequence	Performed quarterly with a blank and a minimum of five standard concentrations to cover the anticipated range of measurement; one of the calibration standards will be at or near the reporting limit. The calibration curve generated must have a correlation coefficient ≥ 0.995 in order to consider the responses linear over that range.
ICP Inter-Element Correction (IEC)	Use	Correction factors for spectral interference (particularly due to Al, Ca, Fe, and Mg).
	Sequence	Determined at least annually for all wavelengths used for each analyte reported by ICP; or any time the ICP is adjusted in any way that may affect the IECs.

Table 14. Instrument Performance Control Samples

Control Type	Description	
<i>Organics</i>		
GC/MS Tuning & Performance	Use	Ensures correct mass assignment and is monitored through response to target compounds during initial and continuing calibration, with minimum response criteria for specified system performance check compounds (SPCCs), and linearity is verified by evaluating the response factors (RF) for calibration check compounds (CCCs).
	Sequence	Tuned at the beginning of the daily work shift. Throughout the analysis, blanks, internal standard areas, surrogates, chromatographic baseline, resolution of peaks, and overall quality of the chromatography are used collectively to monitor instrument performance.
GC & HPLC Instrument Performance	Use	Monitored through retention time shift evaluation, linearity checks, and degradation checks of selected target compounds (e.g., for Endrin or DDT as appropriate).
	Sequence	Continuing calibration verification (e.g., blanks, shifts in chromatographic baseline or retention times, resolution of peaks, and overall quality of the chromatography) throughout the analytical sequence is accomplished through analysis of calibration check standards.

5.8.2.5 Method Performance Control Samples: Analysis Batch

Matrix specific control samples are used to assess the precision and accuracy of the method as applied to the specific sample matrix. These indicators provide information on sample matrix effects that is independent of the efficiency of the preparatory technique. The method performance control samples appropriate to each analytical technique are identified in the respective method. A brief description of these checks is included in Table 15.

These control samples are performed to provide a tool for evaluating how well the method performed for the respective matrix. These values are used by the client to assess the validity of a reported result within the context of the project's data quality objectives. For matrix specific QC results falling outside laboratory control limits which are attributed to matrix affects, no systematic corrective action is taken.

Table 15. Analysis Batch Performance Control Samples

Control Sample Type	Description	
ICP Serial Dilution	Use	5X Dilution of a field sample (performed at the instrument) to check for possible physical and/or chemical interferences.
	Sequence	5% of field samples or 1 per ≤20 samples per batch.
GFAA Analytical Bench Spike	Use	Required by the method; prepared at the instrument by fortifying the digestate with a known quantity of the analyte of interest.
	Sequence	Performed on each sample immediately following the unspiked original analysis.
Method of Standard Addition (MSA)	Use	When specified by the analytical protocol or by client request.
	Sequence	When specified by the analytical protocol or by client request.

5.8.3 Statistical Control Limits and Charts

Statistical control limits and control charts are used to establish method performance of a given analysis and to monitor trends of QC results graphically over time. Once a data base of the laboratory results for a method/matrix/QC analyte combination is established, the acceptability of a given analysis of that QC parameter (and of the analytical batch to which it belongs) can be evaluated in light of the laboratory's normal performance. This is intended to help identify problems before they might affect data. Often, patterns of response that are not at all evident in sets of numbers are very distinct when the same values are viewed as a chronological graph.

Establishment of Limits

The purpose of using statistical control limits is to define, for each analyte in a given method/matrix/QC type combination, a range of expected values. This range encompasses the random variation that occurs normally in the laboratory and allows one to evaluate control samples in that context, rather than according to an arbitrary or external set of values. Limits for accuracy and precision are defined below:

Accuracy

As recoveries of a QC analyte in a given matrix are tabulated over time, a mean value for recovery is established, as is the standard deviation (s) of those recoveries. If the analysis is in statistical control (e.g., if the set of QC recoveries over time show random variation about the mean) approximately 99.7% of all recoveries for that QC will fall within three standard deviations (3s) of the mean. Thus, assuming that the mean itself is an acceptable level of recovery, the values corresponding to 3s above and 3s below the mean are defined as the Control Limits. Any single recovery outside these values is assumed to have resulted from some circumstance other than normal variation and shall be investigated.

Roughly 95% of points should fall within 2s of the mean. The values +2s and -2s are the Warning Limits. Any normal result has approximately a 1/20 chance of being between 2s and 3s from the

mean, so a result in this region doesn't necessarily warrant corrective action, but attention should be paid to such points.

Precision

Precision is used to indicate matrix variability so that appropriate decisions can be made by the client when repeated analyses vary significantly. The coefficient of variation, expressed as a percentage (e.g., the %RSD) for the data set used to calculate accuracy control limits defines the control limit for precision. Duplicate analyses of the QC samples, such as duplicates or MS/MSD, should have an RPD less than or equal to this established precision control limit to be considered free of matrix interferences.

The laboratory calculates statistical control limits on an annual basis. Such limits are available on a project or QAPP-specific basis.

5.8.4 Calibration

Calibration protocols are method-specific, are briefly described in Table 10 and are defined in the Sections 6 & 7 of the method SOPs.

5.8.5 Glassware Cleaning

All glassware is thoroughly cleaned prior to use to ensure that sample integrity is not affected from artifacts caused by contaminated glassware.

A summary of general cleaning procedures follows with details provided in the *Laboratory Glassware Cleaning* SOP (UQA-009):

General laboratory glassware is cleaned with a low- or non-phosphate detergent, followed by thorough rinsing with tap water and deionized water.

Volumetric flasks and pipettes used for inorganics (method dependent), test tubes and caps used for micro-COD procedures, phosphate glassware, and metals-related glassware include an acid-washing step.

BOD glassware cleaning includes a nitric or sulfuric acid and/or a NOCHROMIX-washing step.

Organic glassware includes a solvent-wash.

Non-volumetric organic glassware may optionally be kiln dried at 400°C.

5.8.6 Permitting Departures from Documented Procedure

Where a departure from a documented SOP, test method, or policy is determined to be necessary, or unavoidable, the departure is documented in a CAR or SDR and reported in the case narrative. In most cases, these departures can be made with the approval of the section manager, project manager and the client. Issues of serious concern, as determined by the Section Manager or Project Manager, will be brought to the attention of the Laboratory Director and/or QA Manager. In some

instances, it is appropriate to inform the client before permitting a departure. The Project Manager will make the determination as to the degree of notification required by the client.

On rare occasions, special analytical techniques will be requested for research, project specific requirements, or client needs. In these instances, SOPs may not be available, however, the analyst will thoroughly record the analytical steps and observations within a bound preformatted logbook.

5.8.7 Development of QC Criteria, Non-Specified in Method/Regulation

Where a method or regulation does not specify acceptance and/or rejection criteria, the laboratory must examine the data user's needs and the demonstrated sensitivity, accuracy and precision of the available test methods in determining appropriate QC criteria.

Data users often need the laboratory's best possible sensitivity, accuracy, and precision using a routinely offered test method, or are unsure of their objectives for the data. For routine test methods that are offered as part of STL's standard services, the laboratory bases the QC criteria on statistical information such as determination of sensitivity, historical accuracy and precision data, and method verification data. The method SOP includes QC criteria for ongoing demonstration that the established criteria are met (e.g., acceptable LCS accuracy ranges, precision requirements, method blank requirements, initial and continuing calibration criteria, etc.).

In some cases, a routine test method may be far more stringent than a specific data user's needs for a project. The laboratory may either use the routinely offered test method, or may opt to develop an alternate test method based on the data user's objectives for sensitivity, accuracy, and precision. In this case, it can be appropriate to base the QC criteria on the data user's objectives, and demonstrate through method verification and ongoing QC samples that these objectives are met.

For example, a client may require that the laboratory to test for a single analyte with specific DQOs for sensitivity, accuracy, and precision as follows: Reporting Limit of 10 ppm, Accuracy $\pm 25\%$, and RSD of $<30\%$. The laboratory may opt to develop a method that meets these criteria and document through the Method Blank results, MDL study, and LCS results that the method satisfies those objectives. In this case, both the method and the embedded QC criteria have been based on the client's DQOs.

In some cases, the data user needs more stringent sensitivity, accuracy, and/or precision than the laboratory can provide using a routine test method. In this case, it is appropriate that the laboratory provide documentation of the sensitivity, accuracy, and precision obtainable to the data user and let the data user determine whether to use the best available method offered by the laboratory, or determine whether method development or further research is required.

5.9 Project Reports

The SOP for data package assembly and reporting formats is defined in the *Data Management, Process Operation SOP* (UDM-001) and a summary of this procedure follows.

Analytical reports comprise final results (uncorrected for blanks and recoveries unless specified), methods of analysis, levels of reporting, surrogate recovery data, and method blank data. In addition, special analytical problems will be noted in the case narratives. The number of significant figures reported are consistent with the limits of uncertainty inherent in the analytical method. Consequently, most analytical results will be reported to no more than two (2) or three (3) significant figures. Data are normally reported in units commonly used for the analyses performed.

Concentrations in liquids are expressed in terms of weight per unit volume (e.g., milligrams per liter, mg/L). Concentrations in solid or semi-solid matrices are expressed in terms of weight per unit weight of sample (e.g., micrograms per kilograms, ug/kg). Reporting limits take into account all appropriate concentration, dilution, and/or extraction factors, unless otherwise specified by program requirements (e.g., IRPMS reports).

A client report is generated with various steps of approval prior to printing of the final version. If any analytical anomalies were encountered during the analyses, e.g., an out-of-control matrix duplicate, it is documented in a case narrative. The case narrative is prepared by the respective operating unit and submitted to the data management section to insert in the final report.

The final report forms are printed, data packages are organized, a glossary of flags and acronyms is added, and reports are paginated.

5.9.1 General

The criteria described in Section 5.9.2 apply to all Project Reports that are generated under NELAC requirements. The criteria described in Section 5.9.3 and 5.9.4 apply to all Project Reports.

5.9.2 Project Report Content

- ◆ Title
- ◆ Laboratory name, address, telephone number, contact person
- ◆ Unique Laboratory Project Number
- ◆ Name and Address of Client
- ◆ Client Project Name (if applicable)
- ◆ Laboratory Sample Identification
- ◆ Client Sample Identification
- ◆ Matrix and/or Description of Sample
- ◆ Dates: Sample Receipt, Collection, Preparation and/or Analysis Date
- ◆ Definition of Data Qualifiers
- ◆ Reporting Units
- ◆ Test Methods
- ◆ Report Paginated

The following are required where applicable to the specific test method or matrix:

- ◆ Solid Samples: Indicate Dry or Wet Weight
- ◆ Whole Effluent Toxicity: Statistical package used
- ◆ If holding time \leq 48 hours, Sample Collection, Preparation and/or Analysis Time
- ◆ Indication by flagging where results are reported below the quantitation limit.

5.9.3 Project Narrative

A Project Narrative and/or Cover Letter is included with each project report and, at a minimum, includes an explanation of any and all of the following occurrences:

- ◆ Non-conformances
- ◆ "Compromised" sample receipt (see Section 4.7.1)
- ◆ Method Deviations
- ◆ QC criteria failures

Project Release

The Project Manager or his designee authorizes the release of the project report with a signature.

Where amendments to project reports are required after issue, these are documented in the form of an RDR (refer to Section 4.8) and can be in the form of a separate document and/or electronic data deliverable resubmittal. The revised report is clearly identified as revised with the date of revision and the initials of the person making the revision. Specific pages of a project report may be revised using the above procedure with an accompanying cover letter indicating the page numbers of the project revised. The original version of the project report will be kept intact and the revisions and cover letter included in the project files.

5.9.4 Subcontractor Test Results

Subcontracted data is clearly identified as such, and the name, address, and telephone number for the laboratory performing the test is included in the project report. Subcontracted results from laboratories external to STL are not reported on STL report forms or STL letterhead. Test results from more than one STL facility are clearly identified with the name of the STL facility that performed the testing, address, and telephone number for that facility. Data from subcontractors' reports may be added to an STL electronic deliverable.

Data subcontracted within STL may be reported on the originating laboratory's report forms provided the following mandatory requirements are met:

- ◆ The name, address, and telephone number of the facility are provided.
- ◆ Analytical results produced by the STL intra-company subcontractor are clearly identified as being produced by the subcontractor facility.
- ◆ The intra-company subcontractor's original report, including the chain of custody is retained by the originating laboratory.
- ◆ Proof of certification is retained by the originating laboratory.
- ◆ All information as outlined in Section 5.9.2 is included in the final report where the report is required to be compliant with NELAC, for both the originating and subcontracting laboratory.

5.9.5 Electronic Data Deliverables

Electronic Data Deliverables (EDD) are routinely offered as part of STL's services. STL 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 EDD development staff by the PM for review and undergo the contract review process in Section 4.4.1. Once the laboratory has committed to providing diskettes in a specific format, the coding of the format may need to be performed. This coding is documented and validated. The validation of the code is retained as a QC record.

EDDs are subject to a secondary review to ensure their accuracy and completeness. If EDD generation is automated, review may be reduced to periodic screening if the laboratory demonstrates that it can routinely generate that EDD without errors. Any revisions to the EDD format are reviewed until it is demonstrated that it can routinely be generated without errors. (EDD SOP: UIS-001)

5.9.6 Project Report Format

STL offers a wide range of project reporting formats, including EDDs, short report formats, and complete data deliverable packages modeled on the Contract Laboratory Protocol (CLP) guidelines. More information on the range of project reports available in the Data Management SOP (UDM-001). Regardless of the level of reporting, all projects undergo the levels of review as described in Section 5.3.6.

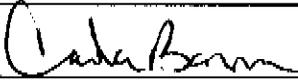
Appendix. List of Cited SOPs and Work Instructions

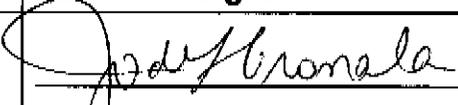
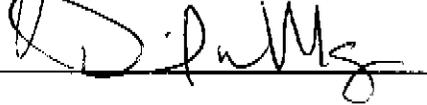
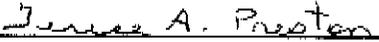
Cited Sec. No(s)	Description	Document No.
1.6; 5.7.1	Container Management: Process Operation	UCM-001
1.6; 4.4.2	Project Management: Project Planning Process	UPM-003
4.1	Signature Authority	UQA-030
4.1.1	Work Instruction: Equipment & Instrumentation Listing	CHI-22-09-103
4.1.2.9	Internet Use Policy Electronic Mail Use Computer System Account and Naming Policy Computer System Password Policy Software Licensing Policy Virus Protection Policy	P-I-001 P-I-002 P-I-003 P-I-004 P-I-005 P-I-006
4.3.1	Document Control	UQA-006
4.3.1.1; 5.3.2	Approved SOP Listing	CHI-22-09-SOP
4.3.2; 4.12.3	Data Management: Record Retention & Purging	UDM-002
4.4.2	Project Kick-Off Meetings	UPM-002
4.4.2	Production Meetings	UPM-004
4.4.3.6	IDL's for CLP Metals and Cyanide	UQA-010
4.4.3.6; 5.3.5	Method Detection Limits (MDLs)	UQA-017
4.5	Work Sharing Process - Policy	S-C-001
4.6	Procurement Quality Assurance Process	UQA-020
4.6.1	Testing Solvents and Acids	S-T-001
4.7.2	Client Confidentiality	UQA-004
4.8; 4.11	Sample Discrepancy Reports (SDRs) / Resubmitted Data Reports (RDRs) / Corrective Action Reports (CARs)	UQA-029
4.8; 4.11	Quality Systems Management Review	UQA-002
4.11	Preventive Action Measures	UQA-019
4.12.2	Work Instruction: Records Management Form	CHI-22-05-032
4.13	Internal Audits	UQA-013
5.1.2	Training Program: Mechanisms and Documentation Processes Defined by Operational Assessment	UQA-014
5.1.2	STL Chicago Demonstration of Capability Certification Statement	CHI-22-09-271
5.1.2	STL Chicago Yearly Method Capability Review Work Instruction: WC	CHI-22-09-279
5.1.3	Ethics Policy	P-L-006
5.3.1	Work Instruction: Methods Capabilities	CHI-22-09-255
5.3.2	SOP Change Protocol	UQA-032
5.3.5	MDL Policy	S-Q-003
5.3.6.1	Acceptable Manual Integration Practices	S-Q-004
5.3.6.2	Data Review Checklists GC Extractables / HPLC GC Volatiles GC/MS: Volatiles and Semivolatiles Metals Wet Chemistry	CHI-22-17-034 CHI-22-19-003 CHI-22-20-038 CHI-22-14-004; 5; 6 CHI-22-12-014
5.4.1	Work Instruction: Equipment Tracking Form	CHI-22-09-068
5.4.2	Instrument and Equipment Out-of-Service Tagging.	UQA-012

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**TITLE: SAMPLE PREPARATION
 Metals Digestion by SW-846 3000 Series**

Updated by: Carla Bonner Analyst, Metals Department	Signature: 	Date: 2-15-05
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Approved by: Jodi L. Gromala Supervisor, Metals Dept.	Signature: 	Date: 2-15-05
David W. Mazur Env. Health & Safety Coor.		2/16/05
Terese A. Preston Quality Manager		2/16/05

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1.0 SCOPE / APPLICATION

This Standard Operating Procedure (SOP) outlines the guidelines for the preparation of wastewaters, extracts, wastes and soil samples for metals analysis by Trace Inductively Coupled Argon Plasma (ICP) and Graphite Furnace AA (GFAA). This SOP was written using the following methods of SW-846, Third Edition:

Method	Description
3005A	Surface and ground waters for analysis by Trace ICP.
3010A	Waters and extracts for analysis by Trace ICP.
3020A	Waters and extracts for analysis by GFAA (excluding As and Se).
3020A Modified	Waters and extracts for analysis by GFAA (including As and Se w/ H ₂ O ₂).
3050B	Soil and waste samples for analysis by Trace ICP or GFAA.
7060A	Waters for As by GFAA.
7740	Waters for Se by GFAA.

On occasion, clients request slight modifications to this SOP. These modifications are addressed on a case-by-case basis with the range of accuracy (i.e., MDLs, linearity check or PT sample) verified prior to implementation. Any modifications would be written into a Quality Assurance Plan (QAP), authorized via laboratory signature approval, and mentioned in the data package's case narrative.

1.1 Method Sensitivity

1.1.1 Method Detection Limits

Not Applicable. Refer to the analytical SOPs.

1.1.2 Reporting Limits

Not Applicable. Refer to the analytical SOPs.

1.1.3 Definitions

Refer to Section 3.0 of the Laboratory's Quality Manual (LQM).

1.2 Summary of Method

Water and soil samples are digested with nitric acid, hydrochloric acid and/or hydrogen peroxide to produce digestates that are in the correct acid media for analysis by the Trace ICP or GFAA.

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2.0 INTERFERENCES

Matrix interferences are usually not present for the digestion process. Analytical matrix interferences may be apparent during the instrumental analysis of the digestates. The type of interferences for the instruments are discussed in the appropriate SOPs.

3.0 SAFETY

Employees must abide by the policies and procedures in the Corporate Safety Manual, Radiation Safety Manual and this document.

3.1 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.
- Acid vapor can be dangerous. Work in a well ventilated area (i.e., fume hood).
- Hydrogen peroxide (H₂O₂) is a strong oxidizer and is corrosive. The digestion must be cooled sufficiently before the addition of H₂O₂ to avoid a reaction and possible violent effervescence, or boiling over of the digestion. A splash/splatter hazard is possible and a face shield should be worn

3.2 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.
Hydrogen Peroxide	Oxidizer Corrosive	1 ppm-TWA	Vapors are corrosive and irritating to the respiratory tract. Vapors are very corrosive and irritating to the eyes and skin.

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Material (1)	Hazards	Exposure Limit (2)	Signs and symptoms of exposure
Nitric Acid	Corrosive Oxidizer Poison	2 ppm-TWA 4 ppm-STEL	Nitric acid is extremely hazardous; it is corrosive, reactive, an oxidizer, and a poison. Inhalation of vapors can cause breathing difficulties and lead to pneumonia and pulmonary edema, which may be fatal. Other symptoms may include coughing, choking, and irritation of the nose, throat, and respiratory tract. Can cause redness, pain, and severe skin burns. Concentrated solutions cause deep ulcers and stain skin a yellow or yellow-brown color. Vapors are irritating and may cause damage to the eyes. Contact may cause severe burns and permanent eye damage.
1 – Always add acid to water to prevent violent reactions. 2 – Exposure limit refers to the OSHA regulatory exposure limit.			

4.0 EQUIPMENT AND SUPPLIES

- Top loading balance
- Hot plate (w/ thermometer)
- Hot Block w/ digestion vessels (w/ thermometer)
- 250 mL beakers
- 100 mL graduated cylinders
- Whatman No. 541 filter paper
- Funnels
- 100 & 50 mL Class A volumetric flasks
- Fume hood(s)
- Eppendorf Pipettes
- Watch glasses (ribbed & non-ribbed)
- Filters and plunger apparatus
- 100 & 50 mL digestate vessels (which are checked to ensure volume markings are within 2.5% Tolerance).
- 100 mL Snap-Cap containers for digestates (which are checked to ensure volume markings are within 2.5% Tolerance).

5.0 REAGENTS AND STANDARDS

5.1 Reagents

- Concentrated Nitric Acid (Instra Pure)
- Concentrated Hydrochloric Acid (Instra Pure)
- 30% Hydrogen Peroxide Solution

Purchased from a chemical vendor.

- Life of Reagent: Specified by the Manufacturer, usually 1 year.
- Storage Requirements: Acid Cabinet

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5.2 Standards

5.2.1 Trace ICP and GFAA Intermediate Standards

These standards are prepared from multi-element solutions purchased from vendors. Single element spikes may be used if needed. These solutions expire 1-year from the date of receipt.

Standard	Preparation
Trace ICP Spike Solution	<ul style="list-style-type: none"> • Add ~400 mLs of Milli-Q water to a 1-L Class A volumetric flask. • Add 100 mLs each of HP1381-A-500, HP1381-B-500 and HP1381-C-500; • Add 9 mLs of 1,000 ppm Se; • Add 8 mLs of 1,000 ppm Pb; • Add 6 mLs of 1,000 ppm As; • Add 5 mLs of 1,000 ppm Tl; and • Add 40 mLs of InstraPure nitric acid. • Swirl to mix; Dilute to volume with Milli-Q water. <p><u>Life of Standard:</u> Expiration date of the earliest expiring standard. <u>Storage Requirements:</u> None.</p>
GFAA Spike Solution	<ul style="list-style-type: none"> • Add ~500 mLs of Milli-Q water to a 1-L Class A volumetric flask. • Add 20 mLs of nitric acid; • Add 10 mLs of STL-CLP-60R • Swirl to mix. Dilute to volume with Milli-Q water. <p><u>Life of Standard:</u> Expiration date of the earliest expiring standard. <u>Storage Requirements:</u> None.</p>
GFAA Ag Spike Solution	<ul style="list-style-type: none"> • Add ~500 mLs of Milli-Q water to a 1-L Class A volumetric flask. • Add 20 mLs each of nitric acid and hydrogen peroxide. • Add 2 mLs of 1000 ppm Ag. • Swirl to mix. Dilute to volume with Milli-Q water. <p><u>Life of Standard:</u> As defined by the manufacturer. <u>Storage Requirements:</u> None.</p>

Refer to Appendix A for the individual element concentrations within the spiking solutions. Matrix spikes for TCLP extracts are added after filtration of the TCLP extract and before preservation. Refer to SOP No. USP-1311.

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6.0 CALIBRATION (NON-DAILY)

Not Applicable.

7.0 PROCEDURE

7.1 Quality Control Checks

QC Indicator	Preparation	Frequency
Method Blank (MB)	For soil sample batches, use 100 mLs of Milli-Q water.	1 per 20 or fewer samples.
	For water sample batches, use 50 mLs of Milli-Q water.	1 per 20 or fewer samples.
Matrix Duplicate (MD) ¹	Aliquot of the same field sample that is digested independently.	1 per 20 or fewer samples.
Laboratory Control Sample (LCS) ²	For soil sample batches, use 100 mLs of Milli-Q water and spike as listed below. ³	1 per 20 or fewer samples.
	For water sample batches, use 50 mLs of Milli-Q water and spike as listed below. ³	1 per 20 or fewer samples.
Matrix Spike (MS); MS Duplicate (MSD) ¹	Aliquot of the same field sample that is spiked as listed below ³ and digested independently.	1 per 20 or fewer samples.

¹ The sample selection for MS/MSD/MD is rotated among client samples so that various matrix problems may be noted and/or addressed.

² LCS Duplicate (LCD) is performed when requested by the client, contract or QAP.

³ The LCS and MS/MSD are spiked with a known amount of analyte and processed through the digestion procedure. The spiking procedure is as follows:

Instrument	Waters Spike Volume	Soils Spike Volume
Trace ICP	0.5 mL of Trace ICP Intermediate Spiking Solution.	1 mL of Trace ICP Intermediate Spiking Solution.
GFAA	0.5 mL of GFAA Intermediate Spiking Solution.	1 mL of GFAA Intermediate Spiking Solution.

Refer to Appendix A for the individual element concentrations within the spiking solutions. Matrix spikes for TCLP extracts are added after filtration of the TCLP extract and before preservation. Refer to USP-1311.

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7.2 Sample Preservation and Storage

Matrix	Holding Time	Preservation
Waters	180 days	HNO ₃ , pH <2; Cool 4 + 2°C
Soils	180 days	Cool 4 + 2°C

7.2.1 Sample Handling Procedures (Other than Soils / Waters)

Matrix	Description
Wipes	The entire wipe is digested with results reported as ug/wipe.
Paint Chips	Care is taken to remove the paint from the substrate. The chips are then cut and ground into a fine powder. Sample size is 0.1 to 0.5 grams.
Solids *	Dried and ground with a mechanical crusher.

*Bricks, wood, etc..

7.3 Sample Preparation

- Since the pH is checked by the sample custodian at sample receipt, the digestion analysis will check the pH at random and/or if the analyst has a reason to suspect that the sample may not be preserved.
- The start and end temperature of the hot plate or hot block digestion is documented within LabNet.

NOTE: The LCS and MB must be filtered when analyzed with dissolved metals that are filtered in the laboratory (unpreserved samples).

7.4 Calibration / Standardization

Not Applicable.

7.5 Preventive Maintenance

- To minimize contamination during sample preparation, the fume hoods and counter areas must be kept clean and free of dust.
- The digestion hoods are cleaned on a regular basis (a minimum of once a month) and documented within the hood maintenance log.

7.6 Sample Digestion

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7.6.1 Method 3005A

- Transfer 50 mLs of the well-mixed (homogenized) sample into a 50 mL digestion vessel.
- Add 1 mLs of InstraPure nitric acid and 2.5 mLs of InstraPure hydrochloric acid.
- Cover the vessel with a ribbed watch glass and heat on a preheated hot block at 90-95°C until the volume has been reduced to 10-15 mLs.
- Remove the vessels from the hot block and allow to cool.
- Fill to a 50 mL final volume in the digestion vessel with Milli-Q water and filter using the plunger apparatus.
- The sample is now ready for analysis.

NOTE: When using the Hot Plates, all volumes remain the same in the 250 mL beaker. When filtering, wash down the sides of the beaker with Milli-Q water and filter into a 50 mL volumetric flask through Whatman 541 filter paper. Dilute the sample to a final 50 mL volume using Milli-Q water.

7.6.2 Method 3010A

- Transfer 50 mLs of the well-mixed (homogenized) sample into a 50 mL digestion vessel.
- Add 1.5 mLs of InstraPure nitric acid.
- Cover the vessel with a ribbed watch glass and place on a preheated hot block set at 90-95°C.
- Evaporate the sample down to a low volume – just enough to cover the bottom of the vessel. **The sample should not boil or any portion of the vessel bottom allowed to go dry.**
- Remove the vessel from the hot block and allow to cool.
- Add another 1.5 mL portion of InstraPure nitric acid.
- Cover the vessel with a non-ribbed watch glass and return to the hot block to allow a gentle reflux to occur.
- Continue to add InstraPure nitric acid as necessary, until the digestion is complete (no change in appearance with continued refluxing).
- Uncover and evaporate to a low volume, not allowing any part of the vessel to go dry.
- Remove the vessels from the hot block and allow to cool.
- Add 2.5 mLs of InstraPure hydrochloric acid and 2.5 mLs of Milli-Q water.
- Warm the vessel for another 15 minutes to dissolve any precipitate.
- Remove from hot block and allow to cool.
- Fill to a 50 mL final volume in the digestion vessel with Milli-Q water and filter using plunger apparatus.
- The sample is now ready for analysis.

NOTE: When using the Hot Plate, the volume remains the same in a 250 mL beaker. When filtering wash down the sides of the beaker with Milli-Q water and filter the sample into a 50 mL volumetric flask through Whatman 541 filter paper. Dilute to the 50 mL final volume with Milli-Q water.

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7.6.3 Method 3020A

- Transfer 50 mLs of the well-mixed (homogenized) sample into a 50 mL digestion vessel.
- Add 1.5 mLs of InstraPure nitric acid.
- Cover the vessel with a ribbed watch glass and place on a preheated hot block set at 90-95°C.
- Evaporate the sample down to a low volume – just enough to cover the bottom of the vessel. **The sample should not boil or any portion of the vessel bottom allowed to go dry.**
- Remove the vessels from the hot block and allow to cool.
- Add another 1.5 mL portion of InstraPure nitric acid.
- Cover the vessel with a non-ribbed watch glass and return to the hot block to allow a gentle reflux to occur.
- Continue to add InstraPure nitric acid as necessary, until the digestion is complete (no change in appearance with continued refluxing).
- Uncover and evaporate to a low volume, not allowing any part of the vessel to go dry.
- Remove the vessel from the hot block and allow to cool.
- Add 5 mLs of Milli-Q water and continue warming for 10-15 minutes to dissolve any precipitates.
- Remove from the hot block and allow to cool.
- Fill to a final 50 mL volume in the digestion vessel with Milli-Q water and filter using plunger apparatus.
- The sample is now ready for analysis.

NOTE: When using the Hot Plate, all volumes remain the same in 250 mL beaker. When filtering wash down the sides of the beaker with Milli-Q water and filter the sample into a 50 mL volumetric flask through Whatman 541 filter paper. Dilute to the 50 mL final volume with Milli-Q water.

7.6.4 Method 3020A Modified

This method is equivalent to Method 3020A, however, 1 mL of hydrogen peroxide is added to the sample with the initial 1.5 mLs of nitric acid.

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7.6.5 Method 3050B

- Weigh out 1.00 – 2.00 grams of the well-mixed sample into a 250 mL beaker. The exact weight is recorded in the LabNet digestion spreadsheet.
- For samples with a high liquid content, more sample may be used as long as the digestion is complete.

NOTE: When using the hot blocks, soils are generally weighed to 1.00-1.20 grams due to the size of the digestion vessels. All other volumes are the same as for the hot plate/beaker digestions.

Add 5 mLs of InstraPure nitric acid and 5 mLs of Milli-Q water.

- Cover the beaker with a non-ribbed watch glass and place on a preheated hotplate set at 90-95°C for 15 minutes without boiling.
- Remove the beaker from the hot plate and allow to cool.
- Add 5 mLs of InstraPure nitric acid and reflux for 30 minutes.
- If brown fumes are generated, repeat this last step until no brown fumes are generated indicating complete reaction with the nitric acid.
- Allow the solution to evaporate to a low volume – just enough to cover the bottom of the beaker. **Do not allow the sample to boil.**
- Remove the beaker from the hot plate and allow to cool.
- Add 2 mLs of Milli-Q water and 3 mLs of 30% hydrogen peroxide.
- Cover the beaker and heat until the reaction is complete.
- Remove the beaker from the hot plate and allow to cool.
- Continue to add 30% hydrogen peroxide in 1 mL aliquots with warming until the effervescence is minimal or until the general sample appearance is unchanged. **Do not add more than a total of 10 mLs of hydrogen peroxide.**
- Cover the sample with a ribbed watch glass and heat until the volume has been reduced to ~5mLs or heat at 90-95°C for 2-hours without boiling.
- Maintain a covering of solution on the bottom of the beaker at all times.

If the sample is being analyzed by the Trace ICP:

- Allow the sample to cool.
- Add 10 mLs of InstraPure hydrochloric acid.
- Place the beaker on the hot plate and heat for 15 minutes without boiling.
- Remove the beaker from the hot plate and allow to cool.
- Wash down the sides of the beaker with Milli-Q water and filter into a 100 mL snap-cap container through Whatman 541 filter paper.
- Dilute the sample to the 100 mL mark in a snap-cap container.
- The sample is now ready for analysis.

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If the sample is being analyzed by GFAA:

- Allow the sample to cool.
- Wash down the sides of the beaker with Milli-Q water and filter into a 100 mL Class A volumetric flask through Whatman 541 filter paper.
- Dilute the sample to the 100 mL snap-cap container through Whatman 541 filter paper.
- The sample is now ready for analysis.

7.6.6 Methods 7060A / 7740

- Transfer 50 mLs of the well-mixed (homogenized) sample into a 50 mL digestion vessel.
- Add 1 mL of Hydrogen Peroxide and 0.5 mL of InstraPure nitric acid.
- Place the vessel on a preheated hot block set at 90-95°C.
- Evaporate the sample down to a volume slightly less than 25 mLs.
- Remove the samples and allow to cool.
- Fill to a final 50 mL volume in the digestion vessel with Milli-Q water and filter using the plunger apparatus.
- The sample is now ready for analysis.

NOTE: When using the Hot Plate, all volume remains the same in a 250 mL beaker. When filtering, wash down the sides of the beaker with Milli-Q water. Filter the sample through Whatman 541 filter paper into a 50 mL volumetric flask. Dilute to volume with Milli-Q water.

7.7 Documentation

7.7.1 LabNet Digestion Spreadsheets

Sample digestion and standard traceability are documented within the LabNet spreadsheets. The spreadsheets must be completed for each days work. The time of digestion and temperature of the hot plate/block must be recorded. Refer to Appendix B for an examples of the GFAA and Trace ICP digestion spreadsheets.

7.7.2 Traceability of Standards

Custom made and single element stock standard solutions which are traceable to NIST or EPA are purchased. Upon receipt, each standard is entered into the LabNet database and is issued a unique source ID#. The manufacturer, lot #, date received, expiration date, and the initials of the analyst are also entered.

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8.0 QUALITY CONTROL

8.1 QC Summary

QC Standard	Indicator
Method Blank (MB)	Examined to determine if there was any contamination introduced during the digestion process.
Laboratory Control Sample (LCS)	Used to determine the completeness of the digestion process. The accuracy is measured by the percent recovery (%R) of each standard.
Matrix Duplicate (MD)	Demonstrate analytical precision and is reported as Relative Percent Difference (RPD).
Matrix Spike (MS) / MS Duplicate (MSD)	Used to demonstrate analytical accuracy and is reported as % recovery.

8.2 Corrective Action

Since this is a preparation procedure, out-of-control situations will not be identified until the filtrates are analyzed. Refer to the analytical SOPs for corrective actions.

9.0 DATA ANALYSIS AND CALCULATIONS

Not Applicable.

10.0 WASTE MANAGEMENT AND POLLUTION PREVENTION

All waste will be disposed of in accordance with Federal, State and Local regulations. Where reasonably feasible, technological changes have been implemented to minimize the potential for pollution of the environment. Employees will abide by this method and the policies in section 13 of the Corporate Safety Manual for "Waste Management and Pollution Prevention."

10.1 Waste Streams Produced by the Method

- Waste from this procedure will enter the "Corrosive Wastewater" wastestream.

11.0 METHOD PERFORMANCE CRITERIA

Refer to sections 1, 6, 7 and 8.

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12.0 REFERENCES

Refer to Section 1.0.

13.0 ATTACHMENTS

Appendix A. Metals Digestion Standard Spike Concentrations

Appendix B. Example: GFAA and Trace ICP LabNet Digestion Spreadsheets

<u>Historical File:</u>	Revision 00:	08/15/91	Revision 07:	10/16/97
	Revision 01:	03/16/93	Revision 08:	03/31/99
	Revision 02:	08/20/93	Revision 09:	05/05/00
	Revision 03:	01/20/94	Revision 10:	07/06/01
	Revision 04:	11/22/95	Revision 11:	01/09/03
	Revision 05:	02/18/97	Revision 12:	01/07/04
	Revision 06:	10/07/97	Revision 13:	02/11/05

Reasons for Change, Revision 13:

- Annual Review – No Changes

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**STL CHICAGO
LABORATORY STANDARD OPERATING PROCEDURE**

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Appendix A.

Metals Digestion Standard Spike Concentration

Trace-ICP

Vendor	Stock Name	Elements	Conc. (mg/L)
Environmental Express	HP1381-A-500	Al, Ba	2,000
		Ca, Mg, K, Na	10,000
	HP1381-B-500	Se	10
		Pb	20
		As	40
		Tl, Be, Cd	50
		Cr	200
		Cu	250
		Co, Ni, Li, V, Bi, Mn, Zn	500
		B, Fe, Sr	1,000
	HP1381-C-500	Ag	50
		Sb, P	500
		Mo, Sn, Ti	1,000
		Si	5,000
Inorganic Ventures	Single Element Standard	As	1,000
		Pb	1,000
		Se	1,000
		Tl	1,000

GFAA

Vendor	Stock Name	Elements	Conc. (mg/L)
Inorganic Ventures	STL-CLP-80R	Sb, Tl	500
		As	400
		Cr, Cu, Pb	200
		Se	100
		Cd	50
	Single Element Standard	Ag	1,000

TCLP (MS)

Vendor	Stock Name	Elements	Conc. (mg/L)
Inorganic Ventures	STL-TCLP-1A	Hg	25
		Cu	25
		Zn, Ni	50
		Cd, Se, Ag	100
		Cr, As, Pb	500
	Single Element Standard	Ba	10,000

**STL CHICAGO
LABORATORY STANDARD OPERATING PROCEDURE**

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Appendix B.

Example: LabNet Digestion Spreadsheets

Acid Digestion (ICAP)

Report Date: 2/15/05 9:39

Method Code...: 3005	Batch Date...: 01/21/05	QC Code.....:	Equipment Code.:
Batch Code...: 139935	Batch Time...: 1013	Calc Code.....: PFACW	Import Code....:
Status.....: RVWD	User Name....: crb	Location Code..: 57222	

BATCH:	Item	Description	Description Information
	1	Analyst:	crb
	2	Reviewer:	lmr
	3	Prep Time Start:	9:20
	4	Hot Plate #	1154
	5	Temperature oC Initial:	95
	6	Temperature oC Final:	95
	7	Repipettor Volume Check:	ok
	8	HN03 Preservative Lot #	n/a
	9	HN03 (Conc.) Lot #	a37042
	10	H2O2 (Conc.) Lot #	n/a
	11	HCL (Conc.) Lot #	a33046
	12	Comment:	622-sb,ba,be,cd,cr,co,pb,ni,
	13	Comment:	se,v-total
	14	Comment:	soluble the same plus-fe,mn

SAMPLE:	Grp	Pos	Sample ID	Dilution	DIGTR Text	MLI mL	MLF mL	PREPF N/A	DLFAC N/A
	1	1	__MB_139935__		Complete	50	50	1.0000	1.000
	1	2	__LCS_M04LSPK003__		Complete	50	50	1.0000	1.000
	1	3	233622_1__		Complete	50	50	1.0000	1.000
	1	4	233622_1_D__		Complete	50	50	1.0000	1.000
	1	5	233622_2__		Complete	50	50	1.0000	1.000
	1	6	233622_2_D__		Complete	50	50	1.0000	1.000
	1	7	233622_3__		Complete	50	50	1.0000	1.000
	1	8	233622_3_D__		Complete	50	50	1.0000	1.000
	1	9	233622_4__		Complete	50	50	1.0000	1.000
	1	10	233622_4_MD_9		Complete	50	50	1.0000	1.000
	1	11	233622_4_MS_M04LSPK003_9		Complete	50	50	1.0000	1.000
	1	12	233622_4_MSD_M04LSPK003_9		Complete	50	50	1.0000	1.000
	1	13	233622_4_D__		Complete	50	50	1.0000	1.000
	1	14	233622_4_D_MD_13		Complete	50	50	1.0000	1.000
	1	15	233622_4_D_MS_M04LSPK003_13		Complete	50	50	1.0000	1.000
	1	16	233622_4_D_MSD_M04LSPK003_13		Complete	50	50	1.0000	1.000
	1	17	233622_5__		Complete	50	50	1.0000	1.000

Acid Digestion (ICAP)

Report Date: 2/15/05 9:39

Method Code...: 3005	Batch Date...: 01/21/05	QC Code.....:	Equipment Code..:
Batch Code...: 139935	Batch Time...: 1013	Calc Code.....: PFACW	Import Code.....:
Status.....: RVWD	User Name....: crb	Location Code..: 57222	

SAMPLE:	Grp Pos	Sample ID	Dilution	DIGTR Text	MLI mL	MLF mL	PREPF N/A	DLFAC N/A
1	18	233622_5_D__		Complete	50	50	1.0000	1.000
1	19	233622_6__		Complete	50	50	1.0000	1.000
1	20	233622_6_D__		Complete	50	50	1.0000	1.000
1	21	233622_7__		Complete	50	50	1.0000	1.000
1	22	233622_7_D__		Complete	50	50	1.0000	1.000
1	23	233622_8__		Complete	50	50	1.0000	1.000
1	24	233622_8_D__		Complete	50	50	1.0000	1.000
1	25	233622_9__		Complete	50	50	1.0000	1.000
1	26	233622_9_D__		Complete	50	50	1.0000	1.000
1	27	_____						

SAMPLE:	Grp Pos	Sample ID	Dilution	VOL mL	COLORB Text	COLORF Text	CLARIB Text	CLARIF Text
1	1	__MB_139935__		50				
1	2	__LCS_M04LSPK003__		50				
1	3	233622_1__		50				
1	4	233622_1_D__		50				
1	5	233622_2__		50				
1	6	233622_2_D__		50				
1	7	233622_3__		50				
1	8	233622_3_D__		50				
1	9	233622_4__		50				
1	10	233622_4_MD_9		50				
1	11	233622_4_MS_M04LSPK003_9		50				
1	12	233622_4_MSD_M04LSPK003_9		50				
1	13	233622_4_D__		50				
1	14	233622_4_D_MD_13		50				
1	15	233622_4_D_MS_M04LSPK003_13		50				
1	16	233622_4_D_MSD_M04LSPK003_13		50				
1	17	233622_5__		50				
1	18	233622_5_D__		50				
1	19	233622_6__		50				
1	20	233622_6_D__		50				
1	21	233622_7__		50				

Acid Digestion (ICAP)

Report Date: 2/15/05 9:39

Method Code...: 3005	Batch Date...: 01/21/05	QC Code.....:	Equipment Code.:
Batch Code...: 139935	Batch Time...: 1013	Calc Code.....: PFACW	Import Code.....:
Status.....: RVWD	User Name....: crb	Location Code..: 57222	

SAMPLE:	Grp	Pos	Sample ID	Dilution	VOL mL	COLORB Text	COLORF Text	CLARIB Text	CLARIF Text
	1	22	233622_7_D		50				
	1	23	233622_8		50				
	1	24	233622_8_D		50				
	1	25	233622_9		50				
	1	26	233622_9_D		50				
	1	27							

SAMPLE:	Grp	Pos	Sample ID	dilution	ARTIFA Text				
	1	1	MB_139935						
	1	2	LCS_M04LSPK003						
	1	3	233622_1						
	1	4	233622_1_D						
	1	5	233622_2						
	1	6	233622_2_D						
	1	7	233622_3						
	1	8	233622_3_D						
	1	9	233622_4						
	1	10	233622_4_MD_9						
	1	11	233622_4_MS_M04LSPK003_9						
	1	12	233622_4_MSD_M04LSPK003_9						
	1	13	233622_4_D						
	1	14	233622_4_D_MD_13						
	1	15	233622_4_D_MS_M04LSPK003_13						
	1	16	233622_4_D_MSD_M04LSPK003_13						
	1	17	233622_5						
	1	18	233622_5_D						
	1	19	233622_6						
	1	20	233622_6_D						
	1	21	233622_7						
	1	22	233622_7_D						
	1	23	233622_8						
	1	24	233622_8_D						
	1	25	233622_9						

Acid Digestion (ICAP)

Report Date: 2/15/05 9:39

Method Code...: 3005	Batch Date...: 01/21/05	QC Code.....:	Equipment Code..:
Batch Code...: 139935	Batch Time...: 1013	Calc Code.....: PFACW	Import Code.....:
Status.....: RVWD	User Name.....: crb	Location Code...: 57222	

SAMPLE:	Grp Pos	Sample ID	Dilution	ARTIFA Text				
	1 26	233622_9_D__						
	1 27	_____						

2/15/05 9:42

Acid Digestion (ICAP)	Status.....: RVWD	User Name.....: crb	Location Code...: 57222
Method Code...: 3010	Batch Date...: 02/11/05	QC Code.....:	Equipment Code..:
Batch Code...: 141789	Batch Time...: 1140	Calc Code.....: PFACW	Import Code.....:

SAMPLE: Grp Pos		Sample ID	Dilution	TEST CODE	TEST POS																	
					Date / Time	D I G I C P																
1	1	___MB_141789_			2/11/05 0950	0																
1	2	___LCS_M05BSPK001_			2/11/05 0950	0																
1	3	234141_1___			2/11/05 0950	0																
1	4	234141_2___			2/11/05 0950	0																
1	5	234173_1___			2/11/05 0950	0																
1	6	234173_1_MD_5			2/11/05 0950	0																
1	7	234173_1_MS_M05BSPK001_5			2/11/05 0950	0																
1	8	234173_1_MSD_M05BSPK001_5			2/11/05 0950	0																
1	9	234173_2___			2/11/05 0950	0																
1	10	234173_3___			2/11/05 0950	0																
1	11	234173_4___			2/11/05 0950	0																
1	12	234173_5___			2/11/05 0950	0																
1	13	234173_6___			2/11/05 0950	0																
1	14	234173_7___			2/11/05 0950	0																
1	15	234192_1___			2/11/05 0950	0																
1	16	234192_2___			2/11/05 0950	0																
1	17	_____																				
1	18	_____																				

Acid Digestion (ICAP)

Report Date: 2/15/05 9:42

Method Code...: 3010	Batch Date...: 02/11/05	QC Code.....:	Equipment Code..:
Batch Code...: 141789	Batch Time...: 1140	Calc Code.....: PFACH	Import Code.....:
Status.....: RVMD	User Name....: crb	Location Code...: 57222	

BATCH:	Item	Description	Description Information
	1	Analyst:	crb
	2	Reviewer:	lmr
	3	Prep Time Start:	9:50
	4	Hot Plate #	1154
	5	Temperature oC Initial:	95
	6	Temperature oC Final:	95
	7	Repipettor Volume Check:	ok
	8	HNO3 Preservative Lot #	n/a
	9	HNO3 (Conc.) Lot #	a45036
	10	H2O2 (Conc.) Lot #	n/a
	11	HCL (Conc.) Lot #	a48034
	12	Comment:	141-#1,2-ba,be,b,cd,cr,co,fe,
	13	Comment:	pb,mn,ni,ag,zn,cu
	14	Comment:	173-ba 192-cr,fe,mn,ni

SAMPLE:	Grp	Pos	Sample ID	Dilution	DIGICP Text	MLI mL	MLF mL	PREPF N/A	DLFAC N/A
	1	1	___MB_141789_		Complete	50	50	1.0000	1.000
	1	2	___LCS_M05BSPK001_		Complete	50	50	1.0000	1.000
	1	3	234141_1___		Complete	50	50	1.0000	1.000
	1	4	234141_2___		Complete	50	50	1.0000	1.000
	1	5	234173_1___		Complete	50	50	1.0000	1.000
	1	6	234173_1_MD_5		Complete	50	50	1.0000	1.000
	1	7	234173_1_MS_M05BSPK001_5		Complete	50	50	1.0000	1.000
	1	8	234173_1_MSD_M05BSPK001_5		Complete	50	50	1.0000	1.000
	1	9	234173_2___		Complete	50	50	1.0000	1.000
	1	10	234173_3___		Complete	50	50	1.0000	1.000
	1	11	234173_4___		Complete	50	50	1.0000	1.000
	1	12	234173_5___		Complete	50	50	1.0000	1.000
	1	13	234173_6___		Complete	50	50	1.0000	1.000
	1	14	234173_7___		Complete	50	50	1.0000	1.000
	1	15	234192_1___		Complete	50	50	1.0000	1.000
	1	16	234192_2___		Complete	50	50	1.0000	1.000
	1	17	___						

Acid Digestion (ICAP)

Report Date: 2/15/05 9:42

Method Code...: 3010	Batch Date...: 02/11/05	QC Code.....:	Equipment Code..:
Batch Code...: 141789	Batch Time...: 1140	Calc Code.....: PFACW	Import Code.....:
Status.....: RVWD	User Name.....: crb	Location Code...: 57222	

SAMPLE:	Grp	Pos	Sample ID	Dilution	DIGICP Text	MLI mL	MLF mL	PREPF N/A	DLFAC N/A
	1	18	_____						
SAMPLE:	Grp	Pos	Sample ID	Dilution	VOL mL	COLORB Text	COLORF Text	CLARIB Text	CLARIF Text
	1	1	MB_141789_		50				
	1	2	LCS_M05BSPK001_		50				
	1	3	234141_1_		50				
	1	4	234141_2_		50				
	1	5	234173_1_		50				
	1	6	234173_1_MD_5		50				
	1	7	234173_1_MS_M05BSPK001_5		50				
	1	8	234173_1_MSD_M05BSPK001_5		50				
	1	9	234173_2_		50				
	1	10	234173_3_		50				
	1	11	234173_4_		50				
	1	12	234173_5_		50				
	1	13	234173_6_		50				
	1	14	234173_7_		50				
	1	15	234192_1_		50				
	1	16	234192_2_		50				
	1	17	_____						
	1	18	_____						
SAMPLE:	Grp	Pos	Sample ID	Dilution	ARTIFA Text				
	1	1	MB_141789_						
	1	2	LCS_M05BSPK001_						
	1	3	234141_1_						
	1	4	234141_2_						
	1	5	234173_1_						
	1	6	234173_1_MD_5						
	1	7	234173_1_MS_M05BSPK001_5						
	1	8	234173_1_MSD_M05BSPK001_5						
	1	9	234173_2_						
	1	10	234173_3_						

Acid Digestion (ICAP)

Report Date: 2/15/05 9:42

Method Code...: 3010	Batch Date...: 02/11/05	QC Code.....:	Equipment Code..:
Batch Code...: 141789	Batch Time...: 1140	Calc Code.....: PFACW	Import Code.....:
Status.....: RVD	User Name.....: crb	Location Code...: 57222	

SAMPLE:	Grp	Pos	Sample ID	Dilution	ARTIFA Text				
	1	11	234173_4__						
	1	12	234173_5__						
	1	13	234173_6__						
	1	14	234173_7__						
	1	15	234192_1__						
	1	16	234192_2__						
	1	17	_____						
	1	18	_____						

Acid Digestion with H2O2 (GFAA)

Report Date: 2/15/05 9:45

Method Code...: 3020M	Batch Date...: 02/10/05	QC Code.....:	Equipment Code.:
Batch Code...: 141731	Batch Time...: 2055	Calc Code.....: PFACW	Import Code....:
Status.....: RVWD	User Name...: rlc	Location Code..: 57222	

BATCH:	Item	Description	Description Information
	1	Analyst:	rlc
	2	Reviewer:	lmr
	3	Prep Time Start:	1900
	4	Hot Plate #	1565
	5	Temperature oC Initial:	95
	6	Temperature oC Final:	95
	7	Repipettor Volume Check:	ok
	8	HNO3 Preservative Lot #	n/a
	9	HNO3 (Conc.) Lot #	a45036
	10	H2O2 (Conc.) Lot #	a24a02
	11	HCL (Conc.) Lot #	n/a
	12	Comment:	GFAA + GFAA Ag 234106-Sb,Tl
	13	Comment:	234122-Tl (CLP-like) 234141-
	14	Comment:	Sb,As,Se,Tl 234153-Se

SAMPLE:	Grp	Pos	Sample ID	Dilution	DIGGFA Text	MLI mL	MLF mL	PREPF N/A	DLFAC N/A
1	1		__MB_141731__		Complete	50	50	1.0000	1.000
1	2		__LCS_M04LSPK002__		Complete	50	50	1.0000	1.000
1	3		234106_1__		Complete	50	50	1.0000	1.000
1	4		234106_2__		Complete	50	50	1.0000	1.000
1	5		234106_3__		Complete	50	50	1.0000	1.000
1	6		234106_4__		Complete	50	50	1.0000	1.000
1	7		234106_5__		Complete	50	50	1.0000	1.000
1	8		234106_6__		Complete	50	50	1.0000	1.000
1	9		234106_7__		Complete	50	50	1.0000	1.000
1	10		234106_8__		Complete	50	50	1.0000	1.000
1	11		234106_9__		Complete	50	50	1.0000	1.000
1	12		234106_10__		Complete	50	50	1.0000	1.000
1	13		234106_11__		Complete	50	50	1.0000	1.000
1	14		234106_12__		Complete	50	50	1.0000	1.000
1	15		234122_7__		Complete	50	50	1.0000	1.000
1	16		234122_8__		Complete	50	50	1.0000	1.000
1	17		234122_17__		Complete	50	50	1.0000	1.000

Acid Digestion with H2O2 (GFAA)

Report Date: 2/15/05 9:45

Method Code...: 3020M		Batch Date...: 02/10/05		QC Code.....:		Equipment Code..:		
Batch Code...: 141731		Batch Time...: 2055		Calc Code.....: PFACW		Import Code.....:		
Status.....: RVWD		User Name.....: rlc		Location Code...: 57222				
SAMPLE:	Grp Pos	Sample ID	Dilution	DIGGFA Text	MLI mL	MLF mL	PREPF N/A	DLFAC N/A
	1 18	234141_1		Complete	50	50	1.0000	1.000
	1 19	234141_2		Complete	50	50	1.0000	1.000
	1 20	234141_2_MD_19		Complete	50	50	1.0000	1.000
	1 21	234141_2_MS_M04LSPK002_19		Complete	50	50	1.0000	1.000
	1 22	234141_2_MSD_M04LSPK002_19		Complete	50	50	1.0000	1.000
	1 23	234153_3		Complete	50	50	1.0000	1.000
SAMPLE:	Grp Pos	Sample ID	Dilution	VOL mL	COLORB Text	COLORF Text	CLARIB Text	CLARIF Text
	1 1	__MB_141731_		50				
	1 2	__LCS_M04LSPK002_		50				
	1 3	234106_1		50				
	1 4	234106_2		50				
	1 5	234106_3		50				
	1 6	234106_4		50				
	1 7	234106_5		50				
	1 8	234106_6		50				
	1 9	234106_7		50				
	1 10	234106_8		50				
	1 11	234106_9		50				
	1 12	234106_10		50				
	1 13	234106_11		50				
	1 14	234106_12		50				
	1 15	234122_7		50	colorless	colorless	clear	clear
	1 16	234122_8		50	colorless	colorless	clear	clear
	1 17	234122_17		50	colorless	colorless	clear	clear
	1 18	234141_1		50				
	1 19	234141_2		50				
	1 20	234141_2_MD_19		50				
	1 21	234141_2_MS_M04LSPK002_19		50				
	1 22	234141_2_MSD_M04LSPK002_19		50				
	1 23	234153_3		50				
SAMPLE:	Grp Pos	Sample ID	Dilution	ARTIFA Text				

Acid Digestion with H2O2 (GFAA)

Report Date: 2/15/05 9:45

Method Code...: 3020M	Batch Date...: 02/10/05	QC Code.....:	Equipment Code.:
Batch Code...: 141731	Batch Time...: 2055	Calc Code.....: PFACW	Import Code.....:
Status.....: RVWD	User Name.....: rlc	Location Code..: 57222	

SAMPLE:	Grp	Pos	Sample ID	Dilution	ARTIFA Text				
	1	1	MB_141731_						
	1	2	LCS_M04LSPK002_						
	1	3	234106_1_						
	1	4	234106_2_						
	1	5	234106_3_						
	1	6	234106_4_						
	1	7	234106_5_						
	1	8	234106_6_						
	1	9	234106_7_						
	1	10	234106_8_						
	1	11	234106_9_						
	1	12	234106_10_						
	1	13	234106_11_						
	1	14	234106_12_						
	1	15	234122_7_						
	1	16	234122_8_						
	1	17	234122_17_						
	1	18	234141_1_						
	1	19	234141_2_						
	1	20	234141_2_MD_19						
	1	21	234141_2_MS_M04LSPK002_19						
	1	22	234141_2_MSD_M04LSPK002_19						
	1	23	234153_3_						

2/15/05 9:47

Acid Digestion: Solids (ICAP)		Status.....: RVWD	User Name.....: crb	Location Code...: 57222
Method Code...: 3050		Batch Date...: 02/10/05	QC Code.....:	Equipment Code.:
Batch Code...: 141667		Batch Time...: 1142	Calc Code.....: PFACS	Import Code.....:
SAMPLE: Grp Pos	Sample ID	Dilution	TEST POS Date / Time	D I G S O L
1 1	__s_MB_141667__		2/10/05 1210	0
1 2	__s_LCS_M05BSPK001__		2/10/05 1210	0
1 3	234125_1_s__		2/10/05 1210	0
1 4	234126_2_s__		2/10/05 1210	0
1 5	234126_2_s_MD_4		2/10/05 1210	0
1 6	234126_2_s_MS_M05BSPK001_4		2/10/05 1210	0
1 7	234126_2_s_MSD_M05BSPK001_4		2/10/05 1210	0
1 8	234126_4_s__		2/10/05 1210	0
1 9	234126_6_s__		2/10/05 1210	0
1 10	234126_8_s__		2/10/05 1210	0

Acid Digestion: Solids (ICAP)

Report Date: 2/15/05 9:47

Method Code...: 3050	Batch Date...: 02/10/05	QC Code.....:	Equipment Code..:
Batch Code...: 141667	Batch Time...: 1142	Calc Code.....: PFACS	Import Code.....:
Status.....: RVWD	User Name....: crb	Location Code...: 57222	

BATCH:	Item	Description	Description Information
	1	Analyst:	crb
	2	Reviewer:	lmr
	3	Prep Time Start:	12:10
	4	Hot Plate #	1740
	5	Temperature oC Initial:	95
	6	Temperature oC Final:	95
	7	Repipettor Volume Check:	ok
	8	HNO3 Preservative Lot #	n/a
	9	HNO3 (Conc.) Lot #	a45036
	10	H2O2 (Conc.) Lot #	a45a09
	11	HCL (Conc.) Lot #	a48034
	12	Comment:	125-k
	13	Comment:	126-hsl
	14	Comment:	

SAMPLE:	Grp	Pos	Sample ID	Dilution	DIGSOL Text	WEIGHT g	MLF mL	PREPF N/A	DLFAC N/A
	1	1	_s_MB_141667_		Complete	1.000	100	100.0000	1.0000
	1	2	_s_LCS_M05BSPK001_		Complete	1.000	100	100.0000	1.0000
	1	3	234125_1_s__		Complete	1.041	100	96.0615	0.9606
	1	4	234126_2_s__		Complete	1.082	100	92.4214	0.9242
	1	5	234126_2_s_MD_4		Complete	1.086	100	92.0810	0.9208
	1	6	234126_2_s_MS_M05BSPK001_4		Complete	1.082	100	92.4214	0.9242
	1	7	234126_2_s_MSD_M05BSPK001_4		Complete	1.062	100	94.1620	0.9416
	1	8	234126_4_s__		Complete	1.106	100	90.4159	0.9042
	1	9	234126_6_s__		Complete	1.067	100	93.7207	0.9372
	1	10	234126_8_s__		Complete	1.091	100	91.6590	0.9166

SAMPLE:	Grp	Pos	Sample ID	Dilution	VOL mL	COLORB Text	COLORF Text	TEXTUR Text	ARTIFA Text
	1	1	_s_MB_141667_		100				
	1	2	_s_LCS_M05BSPK001_		100				
	1	3	234125_1_s__		100				
	1	4	234126_2_s__		100				
	1	5	234126_2_s_MD_4		100				

Acid Digestion: Solids (ICAP)

Report Date: 2/15/05 9:47

Method Code...: 3050	Batch Date...: 02/10/05	QC Code.....:	Equipment Code.:
Batch Code...: 141667	Batch Time...: 1142	Calc Code.....: PFACS	Import Code.....:
Status.....: RVWD	User Name....: crb	Location Code...: 57222	

SAMPLE:	Grp	Pos	Sample ID	Dilution	VOL mL	COLORB Text	COLORF Text	TEXTUR Text	ARTIFA Text
	1	6	234126_2_s_MS_M05BSPK001_4		100				
	1	7	234126_2_s_MSD_M05BSPK001_4		100				
	1	8	234126_4_s_		100				
	1	9	234126_6_s_		100				
	1	10	234126_8_s_		100				

Appendix. List of Cited SOPs and Work Instructions

Cited Sec. No(s)	Description	Document No.
5.4.3	Selection of Calibration Points	P-T-001
5.5.1	Balance Calibration, Care and Use	UQA-003
5.5.1; 5.7.1	Thermometer Calibrations and Electronic Monitoring	UQA-034
5.5.1	Water Quality	UQA-035
5.7.1	Sample Receipt: Handling and Processing	USR-001
5.7.5	Laboratory Waste Disposal Procedures	UWM-001
5.8.1	PT Sample Tracking/Analysis	UQA-018
5.8.5	Glassware Cleaning Procedures	UQA-009
5.9.5	EDD SOP	UIS-001
5.9; 5.9.6	Data Management: Process Operation	UDM-001

ATTACHMENT G

Reporting Limits and Method Detection Limits

STL Reference Data Summary

Structured Analysis Code: A-82-1U-01-07

Target Analyte List: All Analytes

Matrix: SOLID

Extraction: LEACHATE, DI (Routine)

Method: Perchlorate (314.0)

QC Program: STANDARD TEST SET

Location: STL Sacramento

Analyte List		RL	Detection Limits			Run Date	Check List 20006						Spike List 20007							
Syn	Compound		Units	MDL	Units		T	A	Amt	Units	LCL	UCL	RPD	T	A	Amt	Units	LCL	UCL	RPD
5200	Perchlorate	40	ug/kg	3.39	ug/kg	20040126	C	Y	500	ug/kg	75	125	20	C	Y	500	ug/kg	75	125	20

Structured Analysis Code: I-88-1U-01-07

Target Analyte List: All Analytes

Matrix: WATER

Extraction: NO SAMPLE PREPARATION PERFORMED / DIRECT INJI

Method: Perchlorate (314.0)

QC Program: STANDARD TEST SET

Location: STL Sacramento

Analyte List		RL	Detection Limits			Run Date	Check List 20006						Spike List 20007							
Syn	Compound		Units	MDL	Units		T	A	Amt	Units	LCL	UCL	RPD	T	A	Amt	Units	LCL	UCL	RPD
5200	Perchlorate	4.0	ug/L	0.339	ug/L	20040126	C	Y	50	ug/L	85	115	15	C	Y	50	ug/L	80	120	20

Method Limit Report for Project 20005335

Test Long Description	TMX	Units	Limits				
			RL	MDL	LCSLL	LCSUL	LCSRPD
Method: Perchlorates (314)							
Perchlorate	Solid	mg/Kg					
Method: Metals Analysis (ICAP Trace) (6010TR)							
Aluminum	Solid	mg/Kg	20.	5.05	80	120	20
Antimony	Solid	mg/Kg	2.0	0.43	80	120	20
Arsenic	Solid	mg/Kg	1.0	0.37	80	120	20
Barium	Solid	mg/Kg	1.0	0.072	80	120	20
Beryllium	Solid	mg/Kg	0.40	0.018	80	120	20
Cadmium	Solid	mg/Kg	0.20	0.058	80	120	20
Calcium	Solid	mg/Kg	10.	1.84	80	120	20
Chromium	Solid	mg/Kg	1.0	0.10	80	120	20
Cobalt	Solid	mg/Kg	0.50	0.12	80	120	20
Copper	Solid	mg/Kg	1.0	0.22	80	120	20
Iron	Solid	mg/Kg	10.	2.20	80	120	20
Lead	Solid	mg/Kg	0.50	0.25	80	120	20
Magnesium	Solid	mg/Kg	10.	1.01	80	120	20
Manganese	Solid	mg/Kg	1.0	0.053	80	120	20
Nickel	Solid	mg/Kg	1.0	0.48	80	120	20
Potassium	Solid	mg/Kg	50.	6.0	80	120	20
Selenium	Solid	mg/Kg	1.0	0.45	80	120	20
Silver	Solid	mg/Kg	0.50	0.10	80	120	20
Sodium	Solid	mg/Kg	100.	78.6	80	120	20
Thallium	Solid	mg/Kg	1.0	0.57	80	120	20
Vanadium	Solid	mg/Kg	0.50	0.15	80	120	20
Zinc	Solid	mg/Kg	2.0	1.37	80	120	20
Strontium	Solid	mg/Kg	0.50	0.39	80	120	20
Method: Mercury (CVAA) Solids (7471)							
Mercury	Solid	ug/Kg	16.7	6.1	80	120	20
Method: Explosives by 8330 (HPLC) (8330)							
HMX	Solid	ug/Kg	200.	30.2	86	117	30
RDX	Solid	ug/Kg	200.	33.3	90	115	30
1,3,5-Trinitrobenzene	Solid	ug/Kg	100.	7.0	82	125	30
1,3-Dinitrobenzene	Solid	ug/Kg	100.	5.0	86	112	30
Nitrobenzene	Solid	ug/Kg	100.	7.7	90	109	30
2,4,6-TNT	Solid	ug/Kg	100.	14.7	67	152	30
Tetryl	Solid	ug/Kg	250.	117.	60	130	30
2,4-Dinitrotoluene	Solid	ug/Kg	100.	7.9	87	114	30
2,6-Dinitrotoluene	Solid	ug/Kg	200.	10.0	90	112	30
2-Amino-4,6-Dinitrotoluene	Solid	ug/Kg	200.	6.4	90	112	30
4-Amino-2,6-Dinitrotoluene	Solid	ug/Kg	200.	85.6	88	119	30

Method Limit Report for Project 20005335

Test Long Description	TMX	Units	Limits				
			RL	MDL	LCSLL	LCSUL	LCSRPD
Method: Explosives by 8330 (HPLC) (8330)							
2-Nitrotoluene	Solid	ug/Kg	200.	17.0	88	114	30
4-Nitrotoluene	Solid	ug/Kg	200.	30.2	86	114	30
3-Nitrotoluene	Solid	ug/Kg	200.	11.6	89	115	30
Method: NG/PETN by 8332M (HPLC) (8332)							
Nitroglycerine	Solid	ug/Kg	200.	122.	74	142	30
PETN	Solid	ug/Kg	400.	142.	50	150	30
Method: % Solids Determination (SOLIDS)							
% Solids	Solid	%					
% Moisture	Solid	%					

ATTACHMENT H

Data Quality Objectives

Table I-8
Summary of measurement quality objectives for Method 8330 Explosives

Initial Calibration (I.9.2.2.8)	<u>Primary Evaluation:</u> r • 0.995, RSD • 20%, r ² • 0.990 <u>Alternative Evaluation:</u> Mean %RSD for all target analytes • 20%, with maximum allowable restriction noted at right for individual analytes.	No allowance <u>Alternative Evaluation:</u> Maximum allowable %RSD for each individual target analyte • 40%
ICV (I.9.3)	%Rec = 85% - 115%	No allowance
CCV (I.9.5 / I.9.5.2)	<u>Primary Evaluation:</u> %Drift • 15%, %D • 15% <u>Alternative Evaluation:</u> Mean %Drift/%D for all target analytes • 15%, with maximum allowable restriction noted at right for individual analytes.	No allowance <u>Alternative Evaluation:</u> Maximum allowable %Drift/%D for each individual target analyte • 30%
MB (I.10.2.1 / I.11.4.1)	<u>Target Analytes:</u> Analytes < one-half MRL	Not applicable
LCS (I.10.2.2 / I.11.4.2)	<u>Water:</u> %Rec = 60% - 120% ² <u>Solids:</u> %Rec = 60% - 120% ²	<u>Sporadic Marginal Failures</u> ¹ : %Rec = 40% - 150%
MS (I.10.2.3 / I.11.4.3 / I.11.4.3.2)	%Rec = 50% - 140% ²	<u>Sporadic Marginal Failures</u> ¹ : %Rec = 40% - 150%
MSD/MD (I.10.2.4 / I.11.4.4)	RPD • 50%	RPD • 60%
Surrogates (I.10.2.5 / I.11.4.5)	<u>Interference-Free Matrix:</u> <u>Water:</u> %Rec = 60% - 140% <u>Solids:</u> %Rec = 50% - 150% <u>Project Sample Matrix:</u> %Rec = 50% - 150%	Not applicable
Target Analyte Confirmation (I.12.3)	RPD • 40%	RPD • 40%

¹ The number of sporadic marginal failure (SMF) allowances depends upon the number of target analytes reported from the analysis. For instance, if between 7 to 15 explosives are reported from the high-performance liquid chromatography analysis, 1 SMF is allowed to the expanded criteria presented for the LCS. If greater than 15 explosives are reported, 2 SMFs are allowed for the LCS. If the MS includes only a subset of compounds, allow only 1 SMF for this QC element.

² Due to the tendency for Tetryl to decompose, an expanded criteria may be applied at 45% - 140% for both water and soil matrices.

Table I-1
Summary of Measurement quality objectives for Method 6010 Inductively Coupled Plasma (ICP) Metals

Quality Control Element	Description of Element	Frequency of Implementation	Acceptance Criteria
Initial Calibration (I.9.2.1.1)	<u>Option 1</u> - 1 std and blank, and a low-level check standard at MQL <u>Option 2</u> - 3 stds and blank	Daily	<u>Option 1</u> - Low-level check standard \pm 20% <u>Option 2</u> - $r \geq 0.995$
Instrumental Precision (I.9.2.1.1)	%RSD 3 integrations (exposures)	Each calibration and calibration verification standards (ICV/CCV)	%RSD < 5%
Initial Calibration Verification (ICV) (I.9.3)	Midlevel (2nd source) verification	After initial calibration	%Recovery \pm 10%
Initial Calibration Blank (ICB) (I.9.4)	Interference-free matrix to assess analysis contamination	After initial calibration	Analytes < MDL
Interelement Check Standards (ICS) (I.8.1)	ICS-A - interferences only ICS-B - interferences and target analytes	Beginning of analytical sequence	%Recovery \pm 20% for target analytes
Continuing Calibration Blank (CCB) (I.9.4)	Interference-free matrix to assess analysis contamination	Every 10 samples and at end of analytical sequence	Analytes < MDL
Continuing Calibration Verification (CCV) (I.9.5 / I.9.5.1)	Midlevel verification	Every 10 samples and at end of analytical sequence	%Recovery \pm 10%
Method Blank (MB) (I.10.2.1 / I.11.4.1)	Interference-free matrix to assess overall method contamination	1 per sample batch	Analytes < one-half MRL
Laboratory Control Sample (LCS) (I.10.2.2 / I.11.4.2)	Interference-free matrix containing all target analytes	1 per sample batch	%Rec = 80% - 120% <u>Sporadic marginal failures</u> ¹ : %Rec = 60% - 140%
Matrix Spike (MS) (I.10.2.3 / I.11.4.3 / I.11.4.3.1)	Sample matrix spiked with all/subset of target analytes prior to digestion	1 per sample batch	%Rec = 75% - 125%
Matrix Duplicate (MD) or Matrix Spike Duplicate (MSD) (I.10.2.4 / I.11.4.4)	Refer to text for MD or MS.	1 per sample batch	RPD \leq 25%
Post Digestion Spike (PDS) (I.10.3.1 / I.11.4.6)	Sample digestate spiked with all/subset of target analytes	1 per sample batch on MS sample	%Rec = 75% - 125%
Serial Dilution (SD) (I.10.3.2)	1:4 dilution analyzed to assess matrix effects	As needed to assess new and unusual matrices	Agreement between undiluted and diluted results \pm 10%
Method of Standard Additions (MSA) (I.12.2.1)	Method of quantitation	As needed for samples with suspected or confirmed matrix effects	$r \geq 0.995$

¹ The number of Sporadic Marginal Failure (SMF) allowances depends upon the number of target analytes reported from the analysis. For instance, if between 7 to 15 metals are reported from the ICP analysis, one (1) SMF is allowed to the expanded criteria presented. If greater than 15 metals are reported from the ICP analysis, two (2) SMFs are allowed.

Table I-2
Summary of Measurement quality objectives for Method 7010/7470/7471 Series GFAA/CVAA Metals

Quality Control Element	Description of Element	Frequency of Implementation	Acceptance Criteria
Initial Calibration (I.9.2.1.2)	3 stds and blank(GFAA) 5 stds and blank(CVAA)	Daily	$r \geq 0.995$
Instrumental Precision (I.9.2.1.2)	RPD of 2 injections	All standards, and ICV/CCV	RPD \pm 10%
Initial Calibration Verification (ICV) (I.9.3)	Midlevel (2nd source) verification	After initial calibration	%Rec \pm 10%
Initial Calibration Blank (ICB) (I.9.4)	Interference-free matrix to assess analysis contamination	After initial calibration	Analytes < MDL
Continuing Calibration Blank (CCB) (I.9.4)	Interference-free matrix to assess analysis contamination	Every 10 samples and at end of analytical sequence	Analytes < MDL
Continuing Calibration Verification (CCV) (I.9.5 / I.9.5.1)	Midlevel verification	Every 10 samples and at end of analytical sequence	%Rec \pm 20%
Method Blank (MB) (I.10.2.1 / I.11.4.1)	Interference-free matrix to assess overall method contamination	1 per sample batch	Analytes < one-half MRL
Laboratory Control Sample (LCS) (I.10.2.2 / I.11.4.2)	Interference-free matrix containing target analytes	1 per sample batch	%Rec = 80% - 120%
Matrix Spike (MS) (I.10.2.3 / I.11.4.3 / I.11.4.3.1)	Sample matrix spiked with target analytes prior to digestion	1 per sample batch	%Rec = 80% - 120%
Matrix Duplicate (MD) or Matrix Spike Duplicate (MSD) (I.10.2.4 / I.11.4.4)	Refer to text for MD or MS.	1 per sample batch	RPD \leq 20%
Post Digestion Spike (PDS) (I.10.3.1 / I.11.4.6)	Sample digestate spiked with target analytes	Every sample	%Rec = 85% - 115%
Serial Dilution (SD) (I.10.3.2)	1:4 dilution analyzed to assess matrix effects	As needed to assess new and unusual matrices	Agreement between undiluted and diluted results \pm 10%
Method of Standard Additions (MSA) (I.12.2.1)	Method of quantitation	As needed for samples with suspected or confirmed matrix effects	$r \geq 0.995$

Note: GFAA = Graphite furnace - atomic absorption spectroscopy.
CVAA = Cold vapor - atomic absorption.

Summary of Calibration and QC Procedures for Method 314.0

Method	Applicable Parameter	QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action ^a	Flagging Criteria ^b
314.0	Perchlorate	Multipoint calibration for all analytes (minimum 5 standards are recommended)	Initial calibration prior to sample analysis	option 1 linear- RSD $\leq 15\%$	Correct problem then repeat initial calibration	Apply R to all results for all samples associated with the calibration
				option 2 linear – least squares regression $r > 0.995$		
				option 3 non-linear – COD ≥ 0.990 (6 points will be used for second order, 7 points will be used for third order)		
		Second-source calibration verification – quality control sample	Once per multipoint calibration, upon reestablishing calibration, quarterly	Instrument response within $\pm 10\%$ of expected value	Correct problem then repeat initial calibration	Apply R to all results for all samples associated with the calibration
		Instrument Performance Check (IPC)	Daily, before sample analysis	Conductance within 10% of original value (original value within $\pm 10\%$ of MCT)	Prepare fresh IPC solution	Apply R to all results for the sample
				$\% D_{\text{MCT}} < 25\%$, instrument response within $\pm 20\%$ of expected response	Redetermine MCT or correct problem and reanalyze IPC	
				Retention time shifts $< 5\%$, or overall retention time $< 80\%$ of original recorded value	Correct problem, clean or replace column	
Initial calibration verification	Daily, before sample analysis or when eluent is changed	Instrument response within $\pm 25\%$ of expected value using a standard at or below the MQL	Correct problem then repeat initial calibration	Apply R to all results for all samples associated with the calibration		

Summary of Calibration and QC Procedures for Method 314.0

Method	Applicable Parameter	QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action*	Flagging Criteria*
314.0 (cont.)	Perchlorate (cont.)	Continuing calibration verification	After every 10 samples and at the end of the analysis sequence	Instrument response within $\pm 15\%$ of expected response, alternately using separate mid and high level standards	Correct problem then repeat initial calibration verification and reanalyze all samples since last successful calibration verification	Flagging conventions, Table 8.4
		Method blank	One per analytical batch	Perchlorate not detected > MQL	Correct problem then reprep and analyze method blank and all samples processed with the contaminated blank	Flagging conventions, Table 8.4
				Perchlorate detected, but < MQL	None; data qualification may be required	
		Pretreated laboratory reagent blank	Required in any analytical batch which includes samples that have been pretreated to reduce the common anion levels	Perchlorate not detected > MQL	Correct problem then reprep and analyze method blank and all samples processed with the contaminated blank	Flagging conventions, Table 8.4
				Perchlorate detected, but < MQL	None; data qualification may be required	
		LCS	One LCS per analytical batch	QC acceptance criteria Table 7.63	Correct problem then reanalyze the LCS; if the LCS is still out, reprepare and reanalyze the LCS and all samples in the affected AFCEE batch	Flagging conventions, Table 8.4
		MS/MSD	One MS/MSD per matrix per site; or as required by the project-specific FSP	QC acceptance criteria, Table 7.63	none	Flagging conventions, Table 8.4
Demonstrate ability to generate acceptable accuracy and precision using four replicate analyzes of a QC check sample	Once per analyst	QC acceptance criteria, Table 7.63	Recalculate results; locate and fix problem with system and then rerun demonstration for those analytes that did not meet criteria	Apply R to all results for all samples analyzed by the analyst		

Summary of Calibration and QC Procedures for Method 314.0

Method	Applicable Parameter	QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action^a	Flagging Criteria^b
314.0 (cont.)	Perchlorate (cont.)	MCT determination	At initial set-up, once per 12 month period	Calculate %D _{A/H} for the perchlorate peak at increasing concentrations of mixed common anion solution The MCT is the matrix conductance where the %D _{A/H} exceeds 20%	option 1 -least squares regression: plot %D _{A/H} versus matrix conductance, ($r^2 > 0.95$) option 2 - Use the conductance level of the highest mixed anion solution which yielded a %D _{A/H} value < 20%	Samples cannot be analyzed without a valid MCT
		MQL verification	At initial set-up, once per 12 month period	Instrument response within $\pm 30\%$ of expected response for a mixed common anion solution containing perchlorate at the RL and conductance within $\pm 10\%$ of the MCT	Lower the MCT by 10% and repeat the MQL verification	Samples cannot be analyzed without a valid MQL verification
		MDL study	At initial set-up, once per 12 month period	Detection limits established will be < the MQLs in Table 7.62	none	Apply R to all results for in all samples analyzed
		Results reported between MDL and MQL	None	none	none	Apply F to all results between MDL and MQL