Quality Assurance Project Plan

Lake Shore Foundry
653 Market Street
Waukegan, Illinois
Revision: 0

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TITLE/SIGNATURE PAGE

QUALITY ASSURANCE PROJECT PLAN
FOR THE RCRA INTERIM MEASURES AT
LAKE SHORE FOUNDRY, INC., WAUKEGAN, IL
U.S. EPA ID NUMBER
REVISION 0

27 APRIL 2007

Prepared by: Deigan and Associates, LLC

Prepared for: Lake Shore Foundry

John Garland, President, Lake Shore Foundry, Inc.	Date
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Richard Wright, STL Project Manager	Date
Terese Preston, STL QA Manager	Date
Jill Groboski U.S. EPA Project Manager & QA Reviewer	Date

QUALITY ASSURANCE PROJECT PLAN DISTRIBUTION LIST

The following have received a copy of this Quality Assurance Project Plan:

Jill Groboski, USEPA Project Manager and QA Reviewer
John Garland, Lake Shore Foundry
Gary Deigan, Deigan & Associates, LLC Project Manager
Richard Wright, Severn Trent Laboratories Project Manager

LIST OF ACRONYMS/ABBREVIATIONS

ADL	Acceptable Detection Limit
ASTM	American Standards for Testing Materials
CFR	Code of Federal Regulations
COC	Chain of Custody
DQA	Data Quality Assessment
DQO	Data Quality Objective
FSP	Field Sampling Plan
HASP	Health and Safety Plan
IEPA	Illinois Environmental Protection Agency
IMWP	Interim Measures Work Plan
MDLs	Method Detection Limits
MS/MSD	Matrix Spike/Matrix Spike Duplicate
OSHA	Occupational Safety and Health Administration
QA/QC	Quality Assurance/Quality Control
QAPP	Quality Assurance Project Plan
QLs	Quantitation Limits
PARCC	Precision, Accuracy, Representativeness, Completeness, and Comparability
PE	Performance Evaluation Sample
PID	Photoionization Detector
PPE	Personal Protective Equipment
RPD	Relative Percent Difference
RSD	Relative Standard Deviation
SOP	Standard Operating Procedure
STL	Severn Trent Laboratories
SW-846	Test Methods for Evaluating Solid Waste
TCLP	Toxicity Characteristic Leaching Procedure
USEPA	United States Environmental Protection Agency

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Section 1.0 Project Description

This Quality Assurance Project Plan (QAPP) presents the organization, objectives, planned activities, and specific Quality Assurance/Quality Control (QA/QC) procedures associated with the Interim Measures (IM) for the Lake Shore Foundry, Inc. (LSF) in Waukegan, Illinois in response to the Agreed Consent Order, effective November 17, 2006. Specific protocols for sampling, sample handling and storage, chain-of-custody, and laboratory and field analyses will be described. All QA/QC procedures will be structured in accordance with applicable technical standards, U.S. EPA's requirements, regulations, guidance, and technical standards. This QAPP has been prepared in accordance with the U.S. EPA Region 5 QAPP policy as presented in *U.S. EPA RCRA QAPP Instructions*, dated April 1998.

1.1 Introduction and Overall Project Objectives

This QAPP has been prepared on behalf of LSF by Deigan & Associates, LLC. This QAPP and a Health and Safety Plan (HASP) have been appended to the Interim Measures Work Plan (IMWP), dated 16 January 2007 and revised 27 April 2007. A Field Sampling Plan (FSP) has been directly incorporated into the QAPP, and is primarily presented in Section 4.

The purposes of the Agreed Order for the LSF facility are to ensure that the risks from the previous releases of hazardous wastes at or near the Facility are known and understood, and to mitigate any potential threats to human health or the environment. The objective of this environmental investigation is to obtain the environmental data needed to determine the nature and extent of hazardous levels of lead contamination at the Facility. This information will be used to perform interim measures on the facility.

The Decision Statement for this investigation is as follows: What is the nature, risk and extent of select total lead and TCLP lead in onsite soil that presents unacceptable risks, which would therefore warrant remedial action?

1.2 Site/Facility Description

The Site is at 653 Market Street in Waukegan, Lake County, Illinois. The dimensions of the property are approximately 270 feet north-south and 135 feet east-west. The 0.77 acre LSF property contains a single corrugated metal building. The Facility is located on the western shoreline of Lake Michigan. The Elgin, Joliet, and Eastern railroad borders the facility on the west and north sides. Lake Michigan borders the facility on the east side. A City ROW is south of the facility. The ground surface is relatively flat with fill soil covering much of the ground throughout the facility property. **Figure 1** shows the location of this property imposed on a 2002 aerial photo.

The LSF property and adjoining properties have a 100+ year history of heavy industrial uses, including Moen, US Steel, Fansteel/VR Wesson, Waukegan Paint & Lacquer, Diamond Scrap Yard and numerous other factories and warehouses.

The foundry was established in 1900 and produces prototype, short run and high production non-ferrous alloys. Previous sampling was conducted by the United States Environmental Protection Agency (USEPA) in February 2003 and in September 2004 [Booz Allen Hamilton (BAH), *Trip Report for Soil Sampling Activities, Lake Shore Foundry*, 24 November 2004]. In February 2003, the USEPA and the Illinois Environmental Protection Agency (IEPA) conducted a Compliance Sampling Inspection to determine if any site contamination had occurred which would indicate the release of lead that would render soils or other residues and characteristic hazardous waste under 40CFR 261.24. During the CSI, six samples were collected from areas outside the facility building/structure from the ground surface. Samples were analyzed for Toxicity Characteristic Leaching Procedure (TCLP) metals. TCLP lead concentrations up to 440 mg/L were detected, exceeding the regulatory limit set forth in 40 CFR 261.24 of 5 mg/L. On September 21, 2004, USEPA, IEPA, and USEPA's contractors performed sampling on LSF property to determine whether the soil was a characteristic hazardous waste based on TCLP metals. The results from ten of the twelve soil samples collected from depths of 3 inches to 2 feet below ground surface (bgs) were above the regulatory limit for lead (5 mg/L), ranging from 1.23 mg/L to 43.2 mg/L (BAH, 2004). This sampling and analysis, however, was not sufficient to design a removal plan or adequately quantify the vertical or horizontal extent of soils having elevated TCLP and total lead levels.

1.3 Project Objectives and Intended Data Usages

For this project, it will be necessary to gather sufficient information to evaluate the nature and extent of releases of lead contamination in soil to conduct interim measures to remediate this threat.

Overall objectives of the data collection will be as follows:

- Test surface and subsurface soil on the facility to determine the extent lead-contaminated soil above the TCLP regulatory limit of 5 mg/L set forth in 40 CFR 261.24;

- Evaluate the levels of total lead measured in surface and subsurface soil by comparing the average surface (0 – 6 inch) soil lead concentration and subsurface soil (> 6 inches to the water table) lead concentration to the USEPA Region 9 preliminary remediation goal of 800 mg/kg for a commercial/industrial exposure scenario; and

- Develop an appropriate interim measures removal or treatment plan for the facility to address characteristically hazardous sources of lead contamination.

1.4 Project Target Parameters

LSF currently manufactures brass, bronze and aluminum sand & permanent mold castings. The facility previously manufactured red brass and tin bronze, products which contained lead. Previous investigations by USEPA in September 2004 also tested for TCLP arsenic, barium, cadmium, chromium, selenium, and silver. TCLP lead exceeded regulatory limits while the other metals were either not detected or did not exceed regulatory limits. In addition, Interim Measures addressing lead contamination will result in the removal of other residual contaminants that may be present. Thus, the list of target parameters for this project is limited to analysis of total and TCLP lead.

1.5 Sampling Locations

Figure 2 in the IMWP shows the intended soil sampling locations, which is fully incorporated into this QAPP through reference. It is possible, however, that depending on the nature of encountered field conditions, sampling locations may be changed. The person who shall be responsible for making such decisions will be the Site Field Manager whose responsibilities are described in Section 2 of this QAPP.

The rationale for the selected sampling locations (and depths) is fully described in Section 2 of the Interim Measures Work Plan (IMWP).

1.6 Project Schedule

In accordance with the schedule set forth in the Agreed Consent Order, Effective November 17, 2006, interim measures work is to be completed no later than 120 days after USEPA's approval of a work plan for soil removal. A report of the removal and associated analysis shall be submitted to U.S. EPA no later than 45 days after completion of the removal.

Section 2.0 Project Organization and Responsibility

Figure 2 presents the organizational structure for the LSF Interim Measures Investigation. All lines of communication, management activities, and technical direction within this project team will follow this organization arrangement. Any directions or communication from the USEPA will be given to LSF. Lake Shore Foundry will subsequently communicate directions to Deigan & Associates, LLC project manager. The USEPA project manager will be notified of all proposed changes in personnel.

Responsibilities of key project personnel are outlined below.

USEPA Project Manager

- Direct, review, and approve QAPP and Interim Measures Work Plan (IMWP)
- Provide technical consultation services to Lake Shore Foundry and the Deigan & Associates, LLC Project Manager.
- Review progress reports detailing work accomplished.
- Review final reports.

USEPA Quality Assurance Reviewer

- Review and approve the QAPP.
- Assist in review of the IMWP.

Deigan & Associates, LLC Project Manager

- Responsible for planning, coordinating, monitoring, and evaluating all project field activities.
- Before sampling, meet with quality assurance (QA) manager and field staff to discuss and establish sampling purposes, sampling methodology, number of samples, size of samples, sample preservation methods, chain-of-custody (COC) requirements, analyses required, and which samples will be duplicated in the field.
- Resolve all technical problems.
- Meet with team members to discuss and review analytical results prior to completion of reports.
- Responsible for environmental reports and documents.

Deigan & Associates, LLC Quality Assurance Manager

- Oversee assessment activities to ensure that sampling methodology, sample preservation methods, and COC procedures are being followed.
- Assist in any QA issues with field or laboratory questions, as needed.
- Coordinate data validation requests through USEPA.
- Maintain a record of all samples submitted to the laboratory, the analyses being performed on each sample, the final analytical results, and data validation reports.

Deigan & Associates, LLC Data Manager

- Maintain a record of all samples collected and the sample identification information on each sample.
- Manage data acquired from field assessments and laboratory analyses.
- Assemble data into computer format.

Deigan & Associates, LLC Field Team Leader

- Complete on-site Health and Safety Plan (HASP).
- Responsible for oversight of all field activities and ensure that all procedures for the field activities related to the QAPP are executed and documented properly.
- Submit all data generated during field assessment to the data manager.

Deigan & Associates, LLC Field Technical Staff & Subcontractors

- Before sampling, meet with Deigan & Associates, LLC project manager to discuss and establish sampling purposes, sampling methodology, number of samples, size of samples, sample preservation methods, COC requirements, analyses required, and which samples will be duplicated in the field.
- Be responsible for collection of equipment needed for property assessment work, which would include personal protective equipment (PPE), sampling equipment, sample containers and coolers, water level meters, monitoring devices, and any other equipment deemed necessary.
- Oversee drilling and soil boring work to ensure that proper procedures are following during soil sample collection from borings.
- Monitor hazardous conditions while conducting field operations.
- Submit all COC records and field paperwork to the field team leader.

Severn Trent Laboratories (STL) Project Manager

- Responsible for all samples submitted to STL, including those released to other STL locations.
- Responsible for summarizing quality assurance/quality control (QA/QC) requirements for the project.
- Maintain laboratory schedule and ensure that technical requirements are understood by laboratory personnel.
- Provide technical guidance to Deigan & Associates, LLC project manager.
- Ensure accuracy of the laboratory data.

STL QA Manager

- Responsible for evaluating adherence to policies and ensuring that systems are in plan to provide QA/QC as defined in the QAPP.
- Initiate and oversee audits of corrective action procedure.
- Perform data reviews.
- Maintain documentation of training.

Ms. Jill Groboski will serve as the USEPA Project Manager and QAPP reviewer.
Mr. Gary Deigan will serve as the Deigan & Associates, LLC project manager. Ms. Terry Bosko will serve as the Deigan & Associates, LLC QA manager and data manager.

Mr. Kerry VanAllen will serve as the Deigan & Associates, LLC field team leader. Resumes for key personnel are included in **Appendix A.**

All Deigan & Associates, LLC site personnel will be trained as mandated by the Occupational Safety and Health Administration (OSHA) Act regulations (29 Code of Federal Regulations [CFR] 1910.120). Additionally, all site personnel will be properly trained in procedures for collecting, labeling, packaging, and shipping of liquid and solid environmental samples. The Deigan & Associates, LLC project manager will maintain personnel training records.

Severn Trent Laboratory (STL) of University Park, Illinois will be used for laboratory analysis. STL is an IEPA-accredited laboratory. Mr. Richard Wright will serve as the STL project manager. He will be ultimately responsible for ensuring the quality of the laboratory data. The STL QA manager will be Terese Preston.

Section 3.0 Quality Assurance Objectives for Measurement Data

The overall QA objective for the interim measures is to develop and implement procedures for field sampling, COC, laboratory analysis, and reporting using U.S. EPA and IEPA protocols. Specific procedures for sampling, COC, laboratory instrument calibration, laboratory analysis, reporting of data, internal quality controls, audits, preventative maintenance of field equipment, and corrective actions are described in other sections of this QAPP.

Data quality objectives (DQOs) for measurements during this project will be addressed in terms of precision, accuracy, representativeness, completeness, and comparability (PARCC). The numerical PARCC parameters will be determined from the project DQOs to ensure that they are met. The DQOs and resulting PARCC parameters will require that the sampling be performed using standard methods, with properly operated and calibrated equipment, and conducted by trained personnel.

3.1 Precision

Precision is the degree of agreement among repeated measurements of the same parameter under the same or similar conditions. Precision is reported as either relative percent difference (RPD) or relative standard deviation (RSD), depending on the end use of the data.

3.1.1 Field Precision Objectives

Field precision will be assessed through the collection and analysis of duplicate samples. Water matrix samples can be readily duplicated due to their homogeneous nature; conversely, the duplication of soil samples is much more difficult due to their nonhomogenous nature. Due to this difficulty, soil duplicate recovery should be ± 35 percent of the original sample. A summary of duplicates samples to be collected in presented in **Table 1**, along with the other quality control samples. One duplicate sample will be collected for every 10 analytical samples for soil. At least one duplicate soil sample will be collected for each round of sampling performed.

3.1.2 Laboratory Precision Objectives

The precision of laboratory analyses will also be based on collection and analysis of duplicate samples. Precision is reported as RPD or RSD. Duplicate samples will be analyzed at a rate of 1 per 10 samples or in accordance with laboratory Standard Operating Procedures (SOPs).

3.2 Accuracy

Accuracy is the extent of agreement between a measured value and the accepted, or true, value of the parameter being measured.

3.2.1 Field Accuracy Objectives

Sampling accuracy is assessed by evaluating the results of trip and field blank samples for contamination. Field and/or trip blanks are prepared for analysis of organic compounds. Trip blanks are required only when VOCs will be analyzed. Trip blanks are submitted at the rate of one trip blank per shipping container containing field samples for laboratory VOC analysis. No field or trip blanks are collected for metals analysis.

3.2.2 Laboratory Accuracy Objectives

For inorganics analyses, reference standard samples, laboratory control samples, and percent recoveries are utilized for laboratory accuracy determination. The laboratory QA objectives are controls are summarized in **Table 1** and presented in the STL laboratory QAPP provided in **Appendix B**. For organics, the analyses of MS/MSD samples are also utilized to determine laboratory accuracy.

3.3 Representativeness

Representativeness is a qualitative term that describes the extent to which a sampling design adequately reflects the environmental conditions of the site. It also reflects the ability of the sample team to collect samples and laboratory personnel to analyze those samples in such manners that the data generated accurately and precisely reflect the conditions at the site.

3.3.1 Measures to Ensure Representativeness of Field Data

Representativeness will be achieved by establishing the level of allowable uncertainty in the data and then statistically determining the number of samples needed to characterize the population through the DQO process. It will also be achieved by ensuring that sampling locations are properly selected. Representativeness is dependent upon the proper design of the sampling program will be accomplished by ensuring that this QAPP, IMWP, and standard procedures are followed. The QA goal will be to have all samples and measurements representative of the media sampled. Soil intervals will be homogenized for all analyses except VOCs to help ensure that representative soil samples are collected.

9.2.2 Measures to Ensure Representativeness of Laboratory Data

Representativeness of laboratory data cannot be quantified. However, adherence to the prescribed analytical methods and procedures, including holding times, blanks, and duplicates, will ensure that the laboratory data is representative.

3.4 Completeness

Completeness is defined as the measure of the quantity of valid data obtained from a measurement system compared to the quantity that was expected under normal

conditions. While a completeness goal of 100 percent is desirable, an overall completeness goal of 90 percent may be realistically achieved under normal field sampling and laboratory analysis conditions.

3.4.1 Field Completeness Objective

The field sampling team will take measures to have data generated in the field be valid data. However, some samples may be lost or broken during handling and transit. Therefore, field completeness goals for this project will be to have 90 percent of all samples to be valid data.

3.4.2 Laboratory Completeness Objectives

Laboratory completeness will be a measure of the quantity of valid data measurements and analyses obtained from all the measurements and analyses completed for this project. The laboratory completeness goal is for 90 percent of the samples analyzed to be valid data.

3.5 Comparability

The confidence with which one data set can be compared to another is a measure of comparability. The ability to compare data sets is particularly critical when a set of data for a specific parameter is compared to historical data for determining trends.

9.2.2 Measures to Ensure Comparability of Field Data

Ensuring that this QAPP and the IMWP are adhered to and that all samples are properly handled and analyzed will satisfy the comparability of field data. Additionally, efforts will be made to have sampling completed in a consistent manner by the same sampling team.

3.5.2 Measures to Ensure Comparability of Laboratory Data

Analytical data are comparable when the data are collected and preserved in the same manner followed by analysis with the same standard method and reporting limits. Data comparability is limited to data from the same environmental media. Analytical method quality specifications have been established to help ensure that the data will produce comparable results. **Table 2** summarizes the laboratory reporting limits.

The purpose of the QAPP is to produce reliable data that will be generated through out the Interim Measures on the LSF facility. This will be accomplished by:

- Ensuring the validity and integrity of the data;
- Providing ongoing control of data quality;
- Evaluating data quality; and

- Providing usable quantitative data for analysis interpretation and decision-making.

3.6 Decision Rules

A Decision Rule is a statement which allows for a course of action or non-action to be taken, based on assumptions made to draw out and test its logical or empirical consequences. All available information will be used to determine the nature and extent of lead contamination on the LSF facility. To aid in determining areas to remediated as part of Interim Measures, LSF will ask the following questions:

- What is the extent of TCLP lead in soil above the regulatory threshold of 5 mg/L?
- What is the extent of lead in soil above the USEPA Region 9 preliminary remediation goal of 800 mg/kg for a commercial/industrial exposure scenario?
- What areas on the facility require active remediation?
- Can remaining contaminants be managed by excluding exposure pathways through engineered barriers and environmental land use controls (ELUCs)?

Samples of surface and subsurface soil will be collected for analysis as described in the IMWP in order to assess the level of contamination. A site map showing the assessment boundaries is provided in Figure 2 in the IMWP. Detailed information on the sample locations and sample depths are provided in the IMWP.

The decision rules for the LSF facility can be stated as follows.

- Where TCLP lead levels exceed the regulatory threshold for characteristically hazardous sources of lead contamination, an appropriate interim measures removal or treatment plan will be prepared for the facility.

- Where total lead levels exceed the USEPA Region 9 preliminary remediation goal of 800 mg/kg for a commercial/industrial exposure scenario, then LSF may opt to resample the specific locations associated with elevated contaminant levels. If any of the resample results confirm the original data, LSF will consider the two options listed below. If all the resample results are below USEPA Region 9 PRG, no further remedial action will be pursued at the property;

- If soil lead levels exceed the USEPA Region 9 PRG, LSF may pursue development of a site-specific lead cleanup level using USEPA's Adult Lead Model, or pursue an exposure route exclusion through the use of engineered barriers or ELUCs; or

- If an exposure route cannot be eliminated through exposure route exclusion or ELUCs, then LSF may develop an Interim Measures to address total lead contamination.

Section 4.0 Sampling Procedures

The following sections detail the procedures that will be followed during the IM at the LSF facility. This section presents the FSP for the facility.

4.1 Sample Network Design and Rationale

Sample locations, analytical parameters, and frequency of sampling are discussed in the IMWP. Figure 2 in the IMWP shows the intended soil sampling locations, which is fully incorporated into this QAPP through reference. It is possible, however, that depending on the nature of encountered field conditions, sampling locations may be changed. The person who shall be responsible for making such decisions will be the Site Field Manager whose responsibilities are described in Section 2 of this QAPP.

4.2 Sampling Procedures

The field sampling procedures are discussed in the IMWP and the field sampling SOPs are presented in **Appendix D**. A site-wide grid pattern of twenty surface and subsurface borings for soil (see Figure 2 in the IMWP) will be established using a base grid and a supplemental grid. Discrete soil samples will be collected at each boring in the 0- to 6-inch interval and at every two feet in depth, beginning at 6 inches below ground surface (bgs) and continuing to the above the interface of the groundwater/vadose zone. Groundwater on lakefront parcels near the facility has been encountered as shallow as 4 feet below ground surface (bgs), with most encountered at approximately 11 to 14 ft bgs. No soil samples will be obtained at or below the water table. Up to 8 samples will be collected from each boring. Geoprobe direct push sampling techniques will be utilized using a Bob-cat or truck mounted rig.

The twenty surface (0-6” bgs) samples will be analyzed for total lead. TCLP lead analysis will be performed on surface soil samples if total lead concentrations exceed 100 mg/kg. All subsurface samples (> 6” bgs) will be analyzed for TCLP lead. Field QA/QC requirements for each environmental medium is shown in **Table 2**. The laboratory SOPs for these analytical parameters are presented in **Appendix C**. All sample container preservation and volume requirements are outlined in Section 7 and summarized in **Table 3**.

4.3 Sample Handling and Analysis

All soil samples will be shipped to STL for laboratory analysis. Laboratory test parameters for the sampling program will include analysis for the following parameters:

- Total lead;
- Toxicity characteristic leaching procedure (TCLP) lead; and

- pH

Total lead will be analyzed using SW846-Method 6010; TCLP lead will be analyzed using SW-846 Methods 1310/6010. Quality assurance/quality control (QA/QC) samples will be submitted in accordance with the QAPP protocols presented in the following sections. Requirements for QA/QC samples are presented in **Table 2.**

4.4 Decontamination Procedures

All sampling equipment will be decontaminated before being used to collect a sample. The decontamination protocol for sampling equipment is presented in **Table 4**. Whenever possible, disposable sampling supplies will be used (e.g., plastic scoops, aluminum trays, disposable bailers) to minimize the quantity of decontamination fluids. The management of water generated during decontamination will be in accordance with the requirements outlined in Section 4.5. All decontamination wastewater will be containerized.

4.5 Management of Investigation Derived Wastes

For purposes of this IM, investigative-derived wastes (IDW) are defined as any by-product of the field activities that is suspected or known to be contaminated with any hazardous substances. The performance of field activities may produce waste products such as decontamination wastewater and expendable personnel protective equipment. In order to collect the decontamination wastewater, DOT-approved containers will be set up in a central area where sampling teams can empty 5-gallon buckets from decontamination procedures performed during sampling activities.

Each type of waste will be segregated during the field activity and containerized separately. All storage containers will be labeled appropriately. Deigan and Associates, LL will refer to the U.S. EPA's *Management of Investigation-Derived Wastes During Site Inspections* (U.S. EPA, 1991) for guidance on off-site disposal policy, if this action is deemed necessary.

Section 5.0 Custody Procedures

Proper sample handling and custody procedures are important to ensuring the quality and validity of data obtained through field and laboratory analyses. Custody procedures will be used to document the authenticity of data collected during the LSF Interim Measures. The data requiring custody procedures includes field samples and data files that can include field books, logs, and laboratory reports. An item is considered in custody if it is in a person's possession, in view of the person after being in their possession, sealed in a manner that it cannot be tampered with after having been in possession, or in a secure area restricted to authorize personnel.

Sample handling procedures include field documentation, chain of custody documentation, sample shipment, and laboratory sample tracking. Various aspects of sample handling and shipment as well as the proposed sample identification system and documentation are discussed in the following sections.

5.1 Field Custody Procedures

5.1.1 Field Books

Detailed records of the field activities will be maintained in field books dedicated to the LSF facility site. Entries will be dated and signed by personnel recording the data. The entries will be made in ink. Each field book will have a unique numerical identifier permanently attached and each page will be numbered permitting indexing of key data. At a minimum, information recorded in the field books will include documentation of sample locations, sampling times, types of samples collected, weather conditions, and any other information pertinent to the assessment.

5.1.2 Field Identification System

Each sample collected during the Interim Measures will be given a unique identification code. Each unique sample identification will consist of the following:

- ***Project identification code***. A three letter designation identifying the property from which the sample was collected. Examples of this include the following:

  LII—Lake Shore Foundry facility, Interim Measures Investigation Sample
  LIC—Lake Shore Foundry facility, Interim Measures Confirmation Sample

- ***Sample matrix code***. Each sample will be further identified by a code corresponding to the sample matrix:
  SS – surface soil sample
  SB – subsurface soil sample
  FD – field duplicate sample.
  EB – excavation bottom sample.
  EW–excavation sidewall sample.

Location Code & Sample Depth Interval or Compass Location. Each sample will be identified by a location code and depth interval as follows (note that surface soil samples will be numbered consecutively and not given an additional location identifier). Sidewall confirmation samples will be given a compass direction code (N, S, E, W). In addition, the depth interval (expressed in feet below ground surface) for subsurface soil samples will be recorded for below ground surface soil samples.

- ***Example. LII-SB-GP-02 (5.5 ft.)*** – This example illustrates a subsurface soil sample collected from the LSF facility interim measures investigation at Geoprobe location 02 at a depth of 5 ½ feet below ground surface.

Sample bottle labels will be placed on the sample bottles by STL prior to their shipment to the site. Sample labels will then be scribed in the field prior to their being filled. The sample collector will place the following information on the sample bottle label:

- Sample identification
- Date and time of sample collection
- Samplers initials
- Required analyses (if not pre- labeled by the laboratory)
- Type of preservative (if not pre-labeled by the laboratory).

5.1.3 Field Sample Handling

The possession and handling of samples will be documented in the time of collection to delivery to the laboratory. Field personnel are responsible for ensuring that chain-of – custody procedures are followed. Field personnel will maintain custody of all samples until they are relinquished to another custodian, a laboratory worker, or to the freight shipper. The chain of custody standard operating procedure is located in **Appendix D**.

All samples must be catalogued on a COC form using sample identification codes. A copy of the COC form is included in **Appendix E**. The date and time of collection will be recorded on the form as well as the number of each type of sample, the method of preservation, and the type of analysis.

5.1.4 Field Sample Packaging and Shipping

Samples will be packaged and transported in a manner that maintains the integrity of the sample and permits the analysis to be performed within the prescribed holding time. Prior to shipment each sample container will be inspected for label with the proper sample identification code.

Samples will be either hand-delivered or shipped via overnight (FED EX) courier to the STL University Park Illinois laboratory. The laboratory will be contacted in advance to

expect shipment so that holding times of the samples will be conserved. The chain of custody forms will be sealed in a plastic bag and transported inside the sample cooler. In addition, any shipping receipts will be incorporated into the chain of custody documentation. Samples will be packed in the cooler using bubble wrap packing materials, or other similar protective shipping supplies. Any samples suspected of being highly contaminated will additionally be sealed in a re-sealable bag. The cooler will be taped closed using custody seals provided by STL to prevent tampering during transport. Upon relinquishing the sample cooler to STL, field personnel will sign custody of the samples over to the laboratory by signing and dating the bottom of the COC form. One copy of the COC documentation will be retained by the field manager and a second copy will be retained by the laboratory. The integrity of the custody seals shall be noted by STL on the COC form upon arrival.

5.1.5 Field Documentation

Field COC procedures will ensure the proper documentation of each sample from collection in the field until delivery at the laboratory. Custody of samples shall be maintained and documented at all times. This documentation for each sample will include the following information:

- COC form.
- Sample label with sample identification code.
- Shipping documents.

This field documentation will allow for proper identification and verification of all samples on arrival at STL Labs.

5.2 Laboratory Chain of Custody

STL will perform laboratory custody procedures for sample receiving and logging, sample storage, tracking during sample preparation and analysis, and storage of data in accordance with their standard operating procedures. The STL project manager will be responsible for ensuring that laboratory custody protocol is maintained. The laboratories standard operating procedure for sample custody is presented in Section 5.7 of the laboratory QA manual. The laboratory QA manual is presented on a CD-ROM in **Appendix B**.

5.3 Final Evidence Files Custody Procedure

Deigan & Associates, LLC will be responsible for the custody of the evidence files and maintain and update the contents of the files during the project. The evidence files will include all records relevant to sampling and analysis activities such as boring logs, Field logs, photographs, subcontractor reports, laboratory data deliverables, COC forms, and data reviews. Deigan & Associates, LLC will turn over these files to LSF. Lake Shore Foundry shall preserve during the pendency of the Agreed Order and for a minimum of

six (6) years after its termination, all data, records, and documents in its possession or in the possession of its divisions, officers, employees, agents, contractors, successors, and assigns which relate in any way to this Order or to hazardous waste management and/or disposal at the Facility. After six (6) years, Lake Shore Foundry shall make such records available to USEPA for inspection or shall provide copies of any such records to USEPA. Lake Shore Foundry shall notify USEPA, in writing, at least thirty (30) days prior to the destruction of any such records, and shall provide USEPA with the opportunity to take possession of any such records.

Section 6.0 Calibration Procedures and Frequency

The calibration procedures to be employed for both the field and laboratory instruments used during the LSF Interim Measures are referenced in this section. Measuring and test equipment used in the field and laboratory will be subjected to a formal calibration program. The program will require equipment of the proper type, range, accuracy, and precision to provide data compatible with the specified requirements and the desired results. Calibration of measuring and test equipment may be performed internally using in-house reference standards were externally by agencies or manufacturers. The responsibility for the calibration of lab equipment rests with STL. The responsibility for calibration of field equipment rests with Deigan & Associates, LLC and its suppliers and vendors. Documented and approved procedures will be used for calibrating measuring and testing equipment. Widely accepted procedures, such as those published by USEPA and ASTM, or procedures provided by manufacturer's equipment manuals will be adopted.

Calibrated equipment will be uniquely identified by the manufacturer's serial number, a unique equipment identification number, or other means. This identification, along with a label indicating when the next calibration is due will be attached to the equipment. If this is not possible, the records traceable to the equipment will be readily available for reference. It will be the responsibility of all equipment operators to check the calibration status from the due date labels or records prior to using the equipment.

Measuring and testing equipment will be calibrated at prescribed intervals and as part of operational use. Frequency will be based in the type of equipment, its inherent stability, manufacturer's recommendations, values given in national standards, intended use, and experience. Equipment will be calibrated whenever possible using reference standards having known relationships to nationally recognized standards or accepted values of physical constants. If national standards do not exist, the basis for calibration will be documented.

Physical and chemical reference standards will be used only for calibration. Equipment that fails calibration or becomes inoperable during use will be removed from service, segregated to prevent inadvertent use, and tagged to indicate the fault. Such equipment will be recalibrated and repaired to the satisfaction of the laboratory personnel or Deigan & Associates, LLC field personnel as applicable. Equipment that cannot be repaired will be replaced. Records will be prepared and maintained for each piece of calibrated measuring and test equipment to document that established calibration procedures have been followed. Records for subcontractor field equipment and Deigan & Associates, LLC equipment used only for this specific project will be kept in the project files. STL will maintain laboratory calibration records.

6.1 Field Instrument Calibration

Instruments used to gather, generate, or measure field data will be calibrated in sufficient frequency and in such a manner that accuracy and reproducibility of results are consistent with the manufacturer's recommendations. Field measurement instruments may include pH meters, PID, and particulate meters. As applicable, field instruments will be calibrated daily prior to use the calibration will be consistent with the standard procedure. The field calibration procedures are presented in the field SOPs located in **Appendix D.**

Calibration procedures will be documented in the field logbook and field sampling sheets. Documentation will include the following:

- Date and time of calibration
- Identity of the person performing the calibration
- Reference standard used if applicable
- Reading taken and adjustments to attain proper reading
- Any corrective action.

Trained personnel will operate the field measurement equipment in accordance with the appropriate standard procedures and manufacturers specifications. Deigan & Associates, LLC field staff will examine field measurement equipment used during field sampling to verify that they are in proper operating condition. The field team leader will periodically audit the calibration and field performance of the field equipment to ensure that the system of field calibration meet the manufacturer's specifications.

6.2 Laboratory Instrument Calibration

Proper calibration of laboratory equipment is a key element in the quality of the analysis done by the laboratory. Each type of instrumentation and each USEPA approved method have specific requirements for the calibration procedures, depending on the analytes of interest in the sample matrix. The calibration procedures and frequencies of the equipment used to perform the analyses will be in accordance with requirements established by the USEPA. The laboratory QA Manager will be responsible for ensuring that the laboratory instrumentation is maintained in accordance with specifications. Individual laboratory standard operating procedures will be followed for corrective actions and preventative maintenance frequencies. Laboratory quality control, calibration procedures, and corrective action procedures are discussed in Section 5 of the STL QA Manual provided in **Appendix B**. Instruments and preventative maintenance is discussed in Section 5.4 of the STL quality assurance manual provided in **Appendix B**.

Section 7.0 Analytical Procedures

Soil samples collected during field sampling activities for the LSF Interim Measures investigation will be analyzed by the STL of University Park, IL. STL has been accredited by the IEPA for Drinking Water, Wastewater and Hazardous Waste Analyses (**Accreditation located in Appendix C)**. In order to preserve the integrity of samples both before and during analyses, specific analytical methods and requirements for those methods will be followed. Samples will be collected, prepared, and analyzed in accordance with the analytical methods outlined in STL SOPs presented in **Appendix B**. STL will coordinate all analytical services for these LSF Interim Measures. The specific analytical method and reporting limits for each parameter are presented in **Table 2.** Preparatory methods for analytical parameters are discussed in the laboratory SOPs included in **Appendix B.**

Proper sample containers, preservation, holding times, and volumes for each analytical parameter are outlined in **Table 3.** STL will provide all sample containers and preservatives for this project. All sample containers supplied by STL will have been cleaned according to USEPA standards, according to the procedures specified in U.S. EPA's *Specifications and Guidance for Obtaining Contaminant-Free Sample Containers* (U.S. EPA, 1992) or the most current revision. It will be ensured that the bottles used for the sampling activity do not contain target organic contaminants exceeding the level specified in the above-mentioned document. Quality control documentation will be supplied with the sample containers and preservatives in order to verify their purity, so that containers and preservatives can be traced back to their certificate of analysis from their lot number. The QC documentation and certificate of analysis shall be maintained on file with STL. Additionally, laboratory-grade deionized water for rinsing field equipment and instruments will be provided.

Section 8.0 Internal Quality Control Checks

8.1 Field Quality Control Requirements

Where applicable, quality control checks will be strictly followed during the assessment through the use of replicate measurements, equipment calibration checks, and data verification by field personnel. Field sampling precision and data quality will be evaluated through the use of sample duplicates, equipment blanks, and trip blanks. Sample duplicates will provide decision information regarding homogeneity, handling, transportation, storage and analysis. If there is any discrepancy in the sample data the Deigan & Associates, LLC Project Manager will be notified and, if deemed necessary, resampling of the questionable point will be scheduled. Requirements for field QA/QC samples are listed in **Table 2.** QA/QC sample quantities are also identified in the site-specific sampling plans. Collection of the samples will be in accordance with the applicable SOPs in **Appendix D.**

8.2 Laboratory Quality Control Requirements

The laboratory identified in Section 7 of this QAPP has a QC program in place to ensure the reliability and validity of the analysis performed at the laboratory. The laboratory QA manager will be responsible for ensuring that the laboratory's data precision and accuracy are maintained in accordance with specifications. Internal laboratory duplicates and calibration checks are performed on one of every 10 samples submitted for analysis. Other internal laboratory quality assurance/quality control is performed according to laboratory SOPs provided in **Appendix B**. Precision and accuracy laboratory controls by parameter and matrix for STL are presented in **Table 5.**

All data obtained will be properly recorded. The data package will include a full deliverable package capable of allowing the recipient to reconstruct QC information and compare it to QC criteria. Any samples analyzed in nonconformance with the QC criteria will be reanalyzed by the laboratory, if sufficient volume is available. It is expected that sufficient volumes/weights of samples will be collected to allow for reanalysis when necessary.

Section 9.0 Data Reduction, Validation and Reporting

All data generated through field activities, or by the laboratory operation shall be reduced and validated prior to reporting. No data shall be disseminated by the laboratory until it has been subjected to these procedures which are summarized in subsections below. In order to perform the data evaluation steps, the reported laboratory data will be supported by data packages which include sample receipt and tracking information, COC records, data summary forms, and raw analytical data for all field samples, standards, QC checks, QC samples, and all other project specific documents that are generated.

9.1 Data Reduction

9.1.1 Field Data Reduction Procedures

Deigan & Associates, LLC field personnel will manage raw data during field activities. Data such as geologic profiles and pH readings will be recorded on the appropriate field forms provided in **Appendix E** or in field log books. The Deigan & Associates, LLC data manager will periodically collect data gathered during investigation activities in order to maintain results. As appropriate, the data manager will coordinate transfer of raw data or computer formats such as Microsoft Excel to better organize and track incoming data. This will enable the data manager to identify potential data gaps. Any flaws in field QA/QC will be brought to the attention of the QA manager.

9.1.2 Laboratory Data Reduction Procedures

The STL Project Manager will be responsible for laboratory data management. STL procedures for data review and data reporting are discussed in Section 5.3 and 5.9 of the STL QA manual, located in **Appendix B.** Analytical data reports generated by STL will present sample results including all QA/QC samples. All data including QA/QC results will become part of the project files and will be maintained by the data manager. Upon report delivery, Deigan & Associates, LLC personnel will analyze laboratory data in accordance with accepted methodologies and will supervise the data management. Additionally, data preparation is presented in Section 5.9 of STL's QA manual, provided in **Appendix B.**

9.2 Data Validation

Data validation procedures shall be performed for both field and laboratory operations as described below.

9.2.1 Procedures Used to Validate Field Data

The procedures to evaluate field data for this investigation include checking for transcription errors and review of field log books, on the part of field crew members. This task will be the responsibility of the Field Manager, who will otherwise not participate in

making any of the field measurements, or in adding notes, data or other information to the log book.

9.2.2 Procedures Used to Validate Laboratory Data

Procedures to validate laboratory data will be derived from the U.S. EPA's Contract Laboratory Program, National Functional Guidelines For Organic Data Review, and Contract Laboratory Program, National Functional Guidelines for Inorganic Data Review. Essentially, all technical holding times shall be reviewed, instrument performance check sample results shall be evaluated, and results of initial and continuing calibration will be reviewed and evaluated. Also, results of all blanks, surrogate spikes, MS/MSDs, laboratory control samples, and target compound identification and quantitation will be reviewed/evaluated. The laboratory that generates the data will perform data validation. Data validation results in accepted, qualified, or rejected data. All fixed laboratory data will be validated by the laboratory. The validation procedure specifies the verification process of every quality-control measure used in the field and laboratory. Data validation procedures followed by STL are discussed in Section 5.3 of the STL QA Manual provided in **Appendix C**. Each analytical report will be reviewed for compliance with the applicable methods and for the quality of the data reported.

The overall completeness of the data package will also be evaluated by the Deigan & Associates, LLC Data Manager. Completeness checks will be administered to determine whether deliverables specified in the QAPP are present. At a minimum, deliverables will include sample chain-of-custody forms, analytical results, QC summaries, and supporting raw data from instrument printouts. The reviewer will determine whether all required items are present and request copies of missing deliverables.

9.3 Data Reporting

Data reporting procedures shall be carried out for field and laboratory operations as indicated below.

9.3.1 Field Data Reporting

Field data reporting shall be conducted principally through the transmission of report sheets containing tabulated results of all measurements made in the field, and documentation of all field calibration activities.

9.3.2 Laboratory Data Reporting

The Deigan and Associates QA Manager must perform a final review of the report summaries and case narratives to determine whether the report meets project requirements. The data package submitted will be a "CLP-like" data package consisting of all the information presented in a CLP data package (but without the CLP forms).

9.4 Data Acquisition Requirements and Data Quality Management

The Deigan & Associates, LLC data manager will periodically collect data gathered during investigation activities in order to maintain results. As appropriate, the data manager will coordinate transfer of raw data or computer formats such as Microsoft Excel to better organize and track incoming data. This will enable the data manager to identify potential data gaps.

10.0 Performance and Systems Audits and Frequency

Performance and system audits will be completed to ensure that the field sampling activities and laboratory analyses are performed following the procedures established in this QAPP, including the attached standard operating procedures and site-specific sampling plans. The audits may be either internally- or externally-lead as described below.

10.1 Technical Systems Audits

Generally, system audits are qualitative measure of adherence to sampling quality assurance measures overall, including sample collection handling, decontamination procedures, COC, and recording requirements in the field. They may also include sample receiving, sample log-in, and instrument operating records review in the laboratory.

10.1.1 Field Data

Deigan & Associates, LLC field geologist will be present at the site during sampling activities. The geologist will be in contact with the Deigan & Associates, LLC Project Manager, who will then review compliance with the project objectives and sampling protocol outlined in this QAPP. Any anticipated modifications to the QAPP sampling or measuring procedures will be reported to the LSF's and USEPA's Project Manager. Deigan & Associates, LLC field staff will report modifications to the Deigan & Associates. LLC Project Manager and document such modifications in the field logbook.

Sample data precision will be determined by the collection and subsequent analysis of sample duplicates, equipment blanks, and trip blanks to verify reproducibility.

10.1.2 Field Screening Instruments

Deigan & Associates, LLC field technical staff will audit and maintain the performance of field screening instruments. Instruments will be calibrated according to the standard procedures located in **Appendix D** and regular preventative maintenance will be performed as described in **Table 4.**

10.1.3 Report Preparation

The reports generated by this Interim Measures work will be submitted to LSF and USEPA. All reports will undergo a technical peer review conducted by the Deigan & Associates, LLC technical team. Deigan & Associates, LLC team will sign off on the report indicating such review.

10.1.4 Laboratory Data

Laboratory results will be reviewed for compliance against the project reporting limits. This review will be done by Deigan & Associates, LLC data management specialist.

10.2 Performance Evaluation Audits

Generally, performance audits are a quantitative measure of field sample collection and laboratory analyses quality.

10.2.1 Field Audits

Deigan & Associates, LLC QA Manager will conduct audits of field activities. USEPA may also conduct an independent field audit. At least one field audit will be completed near the beginning of sample collection activities. A second audit will occur during implementation of the interim action. The field audit will include the following checklist:

- Review of field sampling records
- Review of field measurement procedures
- Examination of sample identification
- Review of field instrument calibration records and procedures
- Recalibration of the field the instruments
- Review of the sample handling and packaging procedures
- Review of chain of custody procedures.

If deficiencies are observed during the audit, the deficiency shall be noted in writing and a follow-up audit may be completed if deemed necessary by the Deigan & Associates, LLC QA Manager. Corrective action procedures may need to be implemented depending on the findings of the audit. Such actions will be documented in the field logbook.

10.2.2 Laboratory Audits

STL will perform the analytical services required during these assessments. STL's laboratory certifications are presented in **Appendix C**. The STL QA manager will be responsible for ensuring that the laboratory data precision and accuracy are maintained in accordance with specifications and laboratory SOPs. STL may also be audited by USEPA or IEPA, at the agencies discretion.

11.0 Preventative Maintenance

11.1 Field Instrument Preventative Maintenance

The field equipment for this project includes routine sampling equipment and a photo-ionization detector. Specific preventative maintenance procedures to be followed for field equipment are based on those recommended by the manufacturer. Field instruments will be checked and calibrated daily before use. Calibration checks will be documented on the Field Calibration log sheets. The maintenance schedule and trouble-shooting procedures for field instruments are indicated in **Table 4** as well. Critical spare parts such as tape and batteries will be kept on-site to reduce potential downtime. Backup instruments and equipment will be available on-site or within 1-day shipment to avoid delays in the field schedule.

11.2 Laboratory Instrument Preventative Maintenance

As part of the QA Program Plan, a routine preventative maintenance program is conducted by STL to minimize the occurrence of instrument failure and other system malfunctions. Designated laboratory employees regularly perform routine scheduled maintenance and repair of [or coordinate with the vendor for the repair of] all instruments. All maintenance that is performed is documented in the laboratory's operating record. All laboratory instruments are maintained in accordance with manufacturer's specifications. Instruments and preventative maintenance is discussed in Section 5.4 of the STL quality assurance manual provided in **Appendix B**.

12.0 Specific Routine Procedures Used to Evaluated Data Precision, Accuracy and Completeness

The purpose of this section is to indicate the methods by which it will be ensured that the data collected for this investigation falls in line with the data quality objectives (DQOs) for the site. Factors considered in this assessment include, but are not limited to:

- The risk assessment parameters chosen based on conditions and possible receptors involved in a project (i.e. ecological data quality levels, human health data quality levels, soil screening guidance, and the like).

- The contaminants known and/or suspected to be of concern on a project, as they relate to the data quality level parameters chosen.

- The choice of analytical and sample preparation methods for contaminants of concern, whose method detection limits will meet or exceed the data quality level concentrations for those contaminants.

Once these goals and objectives are evaluated and chosen, analytical data quality will be assessed to determine if the objectives have been met. In addition, the data will be reviewed for indications of interferences to results caused by sample matrices, cross contamination during sampling, cross contamination in the laboratory, and sample preservation and storage anomalies (i.e., samples holding time or analytical instrument problems). Data verification may result in accepted, qualified, or rejected data.

12.1 Accuracy Assessment

Accuracy for the metals analysis will be assessed through determination of percent recoveries for laboratory control samples, (as well as MS samples). Percent recovery for MS/MSD results is determined according to the following equation:

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

Percent recovery for LCS and surrogate compound results is determined according to the following equation:

$$\%\ R = \frac{\text{Experimental Concentration}}{\text{Known amount added}} \times 100$$

12.2 Precision Assessment

The relative percent difference (RPD) between the spike and matrix spike, or matrix spike and sample duplicate in the case of metals, and field duplicate pair or laboratory

duplicate pair is calculated to compare to precision DQOs and plotted. The RPD is calculated according to the following formula.

$$RPD = \frac{(\text{Amount in Sample 1 - Amount in Sample 2})}{0.5(\text{Amount is Sample 1 + Amount in Sample 2})} \times 100$$

12.3 Completeness Assessment

Completeness is the ratio of the number of valid sample results to the total number of samples analyzed with a specific matrix and/or analysis. Following completion of the analytical testing, the percent completeness will be calculated by the following equation:

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

12.4 Assessment of Data

The assessment of the data obtained from the investigation is a critical part of determining what the next step in the RCRA Corrective Action process should be. It must be determined if the data are of the appropriate quality, quantity and representativeness to support the project objectives. The affect of the loss of data deemed unacceptable for use, for whatever reason, on the project objectives must be discussed.

The field and laboratory data collected during this investigation will be used to evaluate the nature and extent of contamination at the site. The QC results associated with each analytical parameter for each matrix will be compared to the objectives presented in Section 3 of this QAPP. Only data generated in association with QC results meeting these objectives will be considered useable for decision making purposes. In addition, the data obtained will be both qualitatively and quantitatively assessed on a projectwide, matrix-specific, parameter-specific and unit-specific basis. This assessment will be performed by the Deigan and Associates. LLC QA Manager and the results presented and discussed in the final investigation report. Factors to be considered in this assessment of field and laboratory data will include, but not necessarily be limited to, the following.

- Were all samples obtained using the methodologies and SOPs proposed in the QAPP?
- Were all proposed analyses performed according to the SOPs provided in the QAPP?
- Were samples obtained from all proposed sampling locations and depths?
- Do any analytical results exhibit elevated detection limits due to matrix interferences or contaminants present at high concentrations?
- Were any analytes not expected to be present at the facility, or a given unit, identified as either target parameters or Tentatively Identified Compounds (TICs)?

- Were all field and laboratory data validated according to the validation protocols, including project-specific QC objectives, proposed in the QAPP?
- Which data sets were found to be unusable (qualified as "R") based on the data validation results?
- Which data sets were found to be usable for limited purposes (qualified as "J") based on the data validation results?
- What affect do qualifiers applied as a result of data validation have on the ability to implement the project decision rules?
- Has sufficient data of appropriate quality been generated to support a human health and/or ecological screening risk assessment?
- Were the human health and/or ecological screening risk assessments conducted properly?
- Can valid conclusions be drawn for all matrices at each unit and/or area under investigation?
- Were all issues requiring corrective action fully resolved?
- Were the project-specific decision rules used as proposed during the actual investigation?
- For any cases where the proposed procedures and/or requirements have not been met, has the affect of these issues on the project objectives been evaluated?
- Have any remaining data gaps been identified and summarized in the final investigation report?
- Based on the overall findings of the investigation and this assessment, were the original project objectives appropriately defined? If not, have revised project objectives been developed?

13.0 Corrective Action

Corrective action is the process of identifying, recommending, approving and implementing measures to counter unacceptable procedures or out of QC performance which can affect data quality. Corrective action can occur during field activities, laboratory analyses, data validation and data assessment. All corrective action proposed and implemented should be documented in the QA section of the deliverable. Corrective action should only be implemented after approval by the Deigan & Associates, LLC project Manager, or his designee. If immediate corrective action is required, approvals secured by telephone from the Deigan & Associates, LLC project manager should be documented in an additional memorandum. For noncompliance problems, a formal corrective action program will be determined and implemented at the time the problem is identified. The person who identifies the problem is responsible for notifying the Deigan & Associates, LLC project manager, who in turn will notify the U.S. EPA RCRA Project Manager. If the problem is analytical in nature, information on these problems will be promptly communicated to the U.S. EPA RCRA Project Manager. Implementation of corrective action will be confirmed in writing through the same channels. Any nonconformance with the established QC procedures in the QAPP/FSP or IMWP will be identified and corrected in accordance with the QAPP. The Deigan & Associates, LLC Project Manager, or his designee, will issue a nonconformance report for each nonconformance condition.

13.1 Field Corrective Action

Corrective action in the field may be needed when the sample network is changed (e.g., more/less samples, sampling locations other than those specified in the QAPP, etc.), sampling procedures and/or field analytical procedures require modification, etc. due to unexpected conditions. In general, the field team (technician, Deigan & Associates, LLC Project Manager, and Deigan & Associates, LLC QA Officer) may identify the need for corrective action. The field staff in consultation with the field team leader will recommend a corrective action. The Deigan & Associates, LLC Project Manager will approve the corrective measure which will be implemented by the field team. It will be the responsibility of the Deigan & Associates, LLC Project Manager to ensure the corrective action has been implemented.

If the corrective action will supplement the existing sampling plan (i.e., additional soil borings) using existing and approved procedures in the QAPP, corrective action approved by the Deigan & Associates, LLC Project Manager will be documented. If corrective actions result in less samples (or analytical fractions), alternate locations, etc., which may cause project QA objectives not to be achieved, it will be necessary that all levels of project management, including the U.S. EPA RCRA Project Manager, concur with the proposed action.

Corrective action resulting from internal field audits will be implemented immediately if data may be adversely affected due to unapproved or improper use of approved methods.

The Deigan & Associates, LLC QA officer will identify deficiencies and recommend corrective action to the Deigan & Associates, LLC Project Manager. Implementation of corrective actions will be performed by the Deigan & Associates, LLC field operations manager and field team. Corrective action will be documented in QA section of the deliverables.

Corrective actions will be implemented and documented in the field record book. No staff member will initiate corrective action without prior communication of findings through the proper channels. If corrective actions are insufficient, work may be stopped by the U.S. EPA RCRA Project Manager.

If at any time a corrective action issue is identified which directly impacts project DQOs, the U.S. EPA RCRA Project Manager and/or the U.S. EPA RCRA Enforcement QA Coordinator will be notified immediately.

13.2 Laboratory Corrective Action

Corrective action in the laboratory may occur prior to, during and after initial analyses. A number of conditions such as broken sample containers, multiple phases, low/high pH readings, potentially high concentration samples may be identified during sample log-in or just prior to analysis. Following consultation with lab analysts and section leaders, it may be necessary for the STL QC manager to approve the implementation of corrective action. The SOPs included in Appendix C of this QAPP specify some conditions during or after analysis that may automatically trigger corrective action or optional procedures. These conditions may include dilution of samples, additional sample extract cleanup, and automatic reinjection/reanalysis when certain QC criteria are not met, etc. A summary of method-specific corrective actions are found in the SOPs in **Appendix C.** The bench chemist will identify the need for corrective action. The STL manager, in consultation with the staff, will approve the required corrective action to be implemented by the laboratory staff. The STL QA manager will ensure implementation and documentation of the corrective action. If the nonconformance causes project objectives not to be achieved, it will be necessary to inform all levels of project management, including the U.S. EPA RCRA Project Manager, to concur with the corrective action. These corrective actions are performed prior to release of the data from the laboratory. The corrective action will be documented in both the STL's corrective action log (signed by analyst, section leader and QC coordinator), and the narrative data report sent from the laboratory to the Deigan & Associates, LLC data validator. If corrective action does not rectify the situation, the laboratory will contact the Deigan & Associates, LLC Project Manager.

13.3 Corrective Action During Data Validation and Data Assessment

The facility may identify the need for corrective action during either the data validation or data assessment. Potential types of corrective action may include resampling by the field team or reinjection/reanalysis of samples by the laboratory. These actions are dependent upon the ability to mobilize the field team, whether the data to be collected is

necessary to meet the required QA objectives (e.g., the holding time for samples is not exceeded, etc.). If the Deigan & Associates, LLC data assessor identifies a corrective action situation, it is the Deigan & Associates, LLC Project Manager who will be responsible for approving the implementation of corrective action, including resampling, during data assessment. All corrective actions of this type will be documented by the Deigan & Associates, LLC QA manager.

14.0 Quality Assurance Reports to Management

The deliverable associated with the tasks identified in the IMWP will contain a separate QA section in which data quality information collected during the task is summarized. The section will include the QA officer report on the accuracy, precision, and completeness of the data, as well as the results of the performance and system audits, and any corrective action needed or taken during the project.

Figure 1
Project Organization Chart
Lake Shore Foundry Interim Measures Work

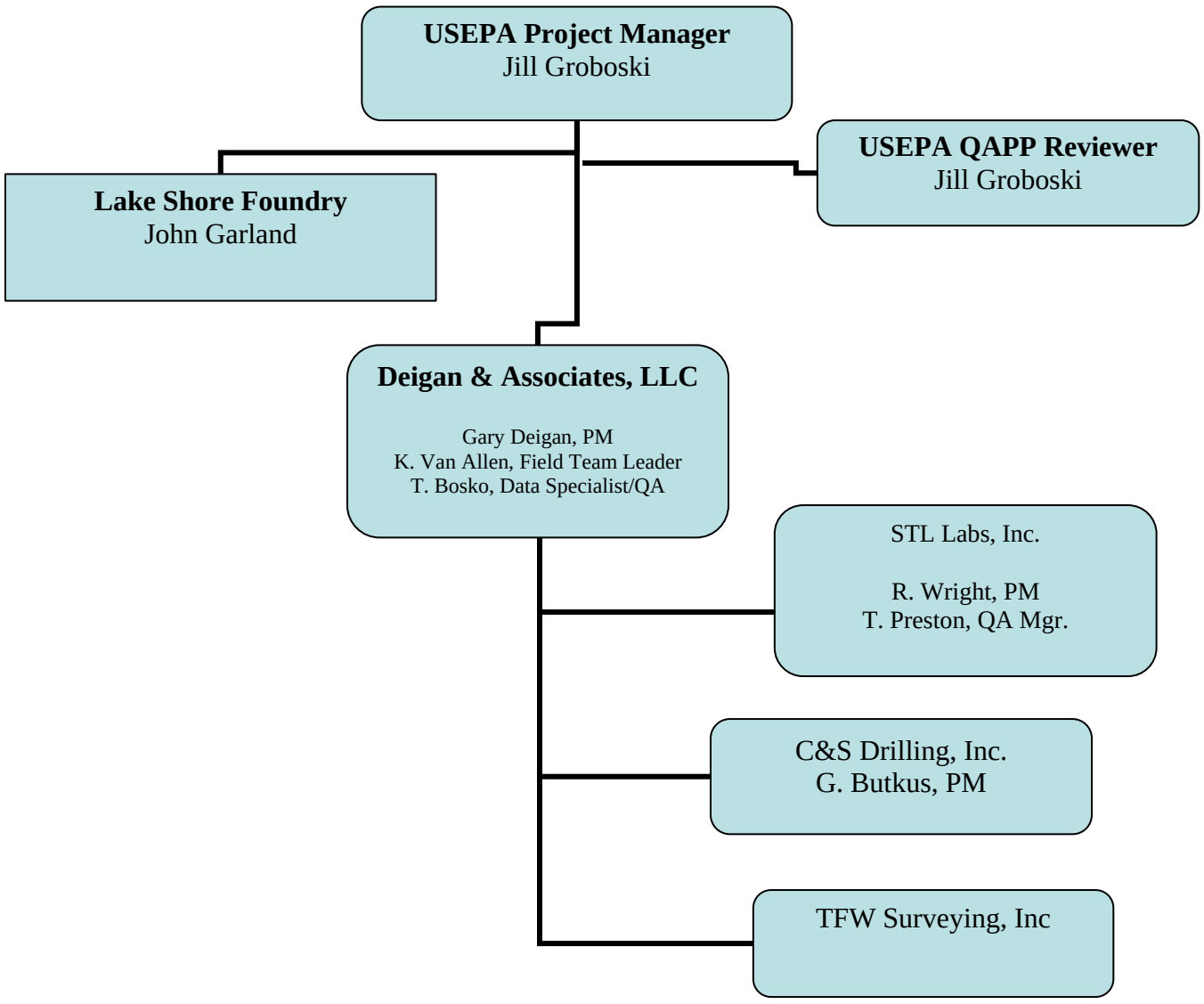

Figure 1
Site Location Map
Lake Shore Foundry, Inc.
653 Market St., Waukegan, Lake County, IL. 60085

TABLES

Table 1
QA/QC Sample Requirements
Waukegan Brownfields Hazardous Substance Assessment Grant

	QC Sample Type	Frequency of Samples/Analysis	Details
Field Samples	Duplicate Samples	1 duplicate per 10 samples	Duplicate sample to be colleced by the same methods at the same time as the original sampled. Used to verify sample and analytical reproducibility
Laboratory Samples	Matrix Spike/Matrix Spike Duplicate (MS/MSD	Every 20 samples or in accordance with laboratory SOP	Laboratory spiked sample to evaluate matrix and measurement methodology.
	Method Blanks (MB)	Every 20 samples or in accordance with laboratory SOP	Laboratory blank sample to assess potential for contamination from laboratory instruments or procedures.
	Laboratory Control and Duplicates (LCS)	Every 20 samples or in accordance with laboratory SOP	Evaluates laboratory reproducibility.

Table 2
Analytical Parameters, Reporting Limits, and TACO Tier 1 Remediation Objectives for Total Lead, TCLP Lead, and pH
Lake Shore Foundry Interim Measures

Analytical Parameter			Soil						Water		
CAS No.	Chemical Name	EPA Analytical Method	Laboratory Reporting Limit[1] (mg/kg)	Method Detection Limit[2] (mg/kg)	Ingestion Exposure Route[2] (mg/kg)	Inhalation Exposure Route[2] (mg/kg)	Soil Component of the Groundwater Ingestion Exposure Route (Class I)[3]* (mg/kg)	TCLP Regulatory Threshold[4] (mg/L)	Laboratory Reporting Limit[1] (mg/L)	Method Detection Limit[2] (mg/L)	Class I Groundwater Remediation Objective[3,5] (mg/L)
--	pH	9045C	0.2 pH units	--	--	--	--	--	0.2 pH units	--	--
7439-92-1	Lead	6010B	0.5	0.25	800	---	107	5	0.005	0.0026	0.0075

[1] These are routine reporting limits, which will vary depending on sample size/volume, dilution factors, dry weight reporting for soil, and changes in method detection limits (Source: STL SOPs, presented in Appendix C).

[2] Determined on an annual basis and are subject to change (Source: STL SOPs, presented in Appendix C).

[3] 35 IAC Part 742, Amended at 26 Ill. Reg. 2683, effective February 5, 2002); industrial/commerical scenario.

[4] 40 CFR 261.24

[5] Reporting limit (RL) for TCLP metals is equivalent to the water RL.

* most stringent of pH-based value presented; objective for TCLP metals analysis is equivalent to Class I groundwater remediation objective.

-- = Not available.

Table 3
Sample Container, Preservation, and Holding Time Requirements
Lake Shore Foundry Interim Measures

Matrix	Analysis	Container	Preservation	Holding Time
Soil	Metals	1- 8 oz glass jar	Cool to 4±2° C	6 months; 24 hours - hexavelent chromium; 28 days - mercury)
	TCLP Metals	1- 8 oz glass jar	Cool to 4±2° C	6 months; 28 days - mercury)
	pH	1 - 8 oz glass jar	Cool to 4±2° C	Not specified

Table 4
Field Equipment Maintenance Procedures and QA Objectives
Lake Shore Foundry Interim Measures

Instrument	Maintenance Procedures/Schedule	Precision	Accuracy	Completeness
Photoionization Detector	1. Calibrate at the beginning and end of each day, and as necessary during use. 2. Recharge battery at the end of each day. 3. Clean lamp and dust filter as necessary. 4. Replace water traps if they become wet.	--	--	--

Table 5
Precision and Accuracy Laboratory Controls by Parameter and Matrix for STL
Lake Shore Foundry Interim Measures

Parameter	Compound	Water			Soil		
		Accuracy		Precision	Accuracy		Precision
		LCSLL	LCSUL	LCS RPD	LCSLL	LCSUL	LCS RPD
Metals	Antimony	80	120	20	80	120	20
	Arsenic	80	120	20	80	120	20
	Barium	80	120	20	80	120	20
	Beryllium	80	120	20	80	120	20
	Cadmium	80	120	20	80	120	20
	Chromium	80	120	20	80	120	20
	Cobalt	80	120	20	80	120	20
	Copper	80	120	20	80	120	20
	Lead	80	120	20	80	120	20
	Mercury	80	120	20	80	120	20
	Nickel	80	120	20	80	120	20
	Selenium	80	120	20	80	120	20
	Silver	80	120	20	80	120	20
	Thallium	80	120	20	80	120	20
	Tin	80	120	20	80	120	20
	Vanadium	80	120	20	80	120	20
	Zinc	80	120	20	80	120	20
	Calcium	80	120	20	80	120	20
	Magnesium	80	120	20	80	120	20
	Potassium	80	120	20	80	120	20
	Sodium	80	120	20	80	120	20
	Iron	80	120	20	80	120	20
	Manganese	80	120	20	80	120	20
	Cyanide	85	115	20	85	115	20

Notes:

Accuracy & Precision limits are based on the laboratory's internal statistical control limits and are subject to change based upond updates to the database. The limits listed above for metals are default limits for LCS recoveries. Recoveries for the MS/MSD are 80-120% for method SW846(6010) and 75-125% for Method SW846(6020).

When sufficient sample for MS/MSD analyses are not provided by client, laboratory performs LCS/LCD to satisfy this QC requirement.

Not all compounds will be used to provide corrective action for out-of control LCS recoveries; laboratory will determine these compounds.

LCSLL - laboratory control sample lower limit (or surrogate lower limit for surrogates)

LCSUL - laboratory control sample upper limit (or surrogate upper limit for surrogates)

LCSRPD - laboratory control sample relative percent difference.

Source: STL SOPs (Appenidix B)

Appendix A

Resumes for Key Contractor Staff

Professional Qualifications

Gary J. Deigan
Principal

Credentials

Southern Illinois University,
B.S. Civil Engineering Technology, 1983

Member, Solid Waste Association of North America (SWANA), Air & Waste Management Assn., National Brownfields Assn.

Professional History

Deigan & Associates, LLC
1998 to Present
Entrix, Inc., 1998
Roy F. Weston, Inc., 1984-1998

Qualifications & Experience Overview

Mr. Deigan has over 20 years experience in a wide variety of civil/environmental engineering projects from baseline data collection, feasibility studies, and conceptual planning through facility design and remedial/construction management. Primary applications have been in the areas of environmental liability management for former industrial and redeveloped municipal properties. He has significant project experience in solid waste consulting, RCRA permitting, CERCLA project planning and implementation; environmental due diligence reviews, strategic environmental planning, impaired and brownfield property redevelopment, technical project management, and marketing of professional services.

Mr. Deigan has consulted on numerous Siting of Regional Pollution Control facilities throughout Illinois, including landfill and waste transfer station and compost facilities. He has also been called upon extensively by municipalities to review environmental impact of independent power plant development projects. He has focused expertise in investigation, design, and remedial planning and risk-based closure/redevelopment at active and former wood preserving sites, smelting and foundry sites, chlorinated solvent sites, LUST sites, and a variety of multi-contaminant Brownfield sites.

His clients include small businesses, property buyers and sellers municipal and county government, law firms, and Fortune 500 Firms. He has presented technical papers at a variety of seminars and technical conferences. He has served as an expert witness and given testimony on numerous environmental cases and public hearings.

References are available upon request.

Professional Qualifications

Mary Therese Bosko, CPSS

Education

B.S., Forest Science, University of Illinois
M.S., Forest Soils, Virginia Tech

Key Projects/Experience Overview

Site Remediation Work Plan, Several Sites, Illinois, Confidential Client, Risk Assessment Specialist. Developed IEPA TACO (35 IAC Part 742) Tier 2 and Tier 3 remediation objectives for metals and PAHs in surface and subsurface soil as part of Site Remediation Program (35 IAC Part 740) activities at six operating compressor stations. Tier 3 objectives were reviewed and approved by the IEPA Pollution Control Board.

Remediation Objective Report, MGP Site, Illinois, Confidential Client, Risk Assessment Specialist. Proposed Tier 1, Tier 2, and Tier 3 remediation objectives for a historical MGP site in Illinois following IEPA TACO (35 IAC Part 742) methodology. Site contaminants included PAHs and heavy metals, found in both soil and groundwater.

Environmental Assessment, Job Corps Training Facility Site, Chicago, IL, City of Chicago, Department of Environment, Risk Assessment Specialist. Member of interdisciplinary team of scientists and engineers that prepared a site management plan for this brownfield site converted from industrial property to an educational center. This 17.5-acre site was filled with dredge material from the adjacent Chicago Sanitary and Ship Canal. The risk assessment identified PCB- and lead-contaminated soils that required remediation. The Bowers model was used to develop lead cleanup levels for adult receptors with approval of the methodology from the IEPA.

Environmental Assessment, Former Burnside Steel Foundry, Chicago, IL, City of Chicago, Department of Environment, Risk Assessment Specialist. Member of an interdisciplinary team that prepared a site management plan for this brownfield site under the IEPA Site Remediation Program. Responsible for the risk assessment, which indicated a limited area of PAH-contaminated soil that needed remediation prior to anticipated redevelopment as an industrial property.

Environmental Assessments, Various Sites, Chicago, IL, Public Building Commission of Chicago (PBCC), Risk Assessment Specialist. Prepared risk assessments for five properties in Chicago located by the PBCC as sites for new schools. The properties were prior industrial sites or adjacent to existing industrial areas. The risk assessments evaluated whether the sites posed any significant risks to those students and staff who would occupy schools at these locations, as well as to those persons involved in developing and constructing the sites. All risk assessments were part of the site management plans for the properties and were reviewed by IEPA under the Site Remediation Program.

Human Health and Ecological Risk Assessments, Alternative Remedial Contracting Strategy (ARCS) Program, EPA, Region IV, V, VI, IX, Risk Assessment Specialist. Prepared numerous multiple-pathway HHRAs and ERAs for CERCLA National Priority Listing (NPL) sites in Illinois, Indiana, Ohio, North Carolina, South Carolina, Florida, Texas, Wisconsin, Washington and Michigan. Work included review, organization, and summarization of data and calculation of risk estimates according to EPA risk assessment guidance criteria.

Professional Qualifications

Kerry W. Van Allen
Sr. Geologist/Hydrogeologist

Education
Bachelor of Sciences in Geology, 1982; University of Illinois, Champaign-Urbana, IL.

Professional Affiliations
Member of American Institute of Architects, National Water Well, and Association of Engineering Geologists

Representative Experience
Over 20 years of technical and managerial experience in environmental and geotechnical consulting related to geologic and hydrogeologic investigations. Investigations are performed for determining the extent and magnitude of contaminant impact of commercial and/or industrial sites. Assessments have been conducted on numerous sites covering the entire Midwest region for municipal, federal and state government, and private sector clientele. Over 16 years of experience in conducting Phase I environmental site assessments for financial institutions, real estate developers and lawyers. Assessments were conducted using procedures recognized under regulatory and/or ASTM guidelines.

***Brownfield* Projects**
City of Chicago *Brownfields* Pilot Program. Worked closely with the Chicago Department of Environment and Department of Planning & Development on numerous projects to assess and remediate abandoned industrial or waste sites. Projects involved cooperation with various governmental agencies and the IEPA under the Illinois Site Remediation Program.
Currently working with the Village of Bartlett on investigation, remediating a former industrial facility and several commercial facilities. Worked closely with the Village's developer to ensure IEPA concurrence on the remediation goals and timing to meet critical deadlines for the project development. This work is being done through an U.S. EPA Brownfields Grant. The end use of the site will be the "Town Center", which includes both upscale commercial and residential land use, located in downtown Bartlett. For the Village of Bartlett project, we are currently working on obtaining four NFR status letters through the IEPA Site Remediation Program (SRP) and the Leaking Underground Storage Tank (LUST) Program.

IEPA SRP Projects
Over 16 years of experience as a principal investigator and geologist for several industrial facilities. Projects were implemented through the former Illinois Volunteer Cleanup Program and the current Site Remediation Program. Prepared technical work plans for assessing soil and groundwater impact at chemical manufacturing, paint manufacturing, plating and other various industrial sites throughout the Midwest. Negotiated with IEPA Project Managers to obtain site specific cleanup objectives, and ensure adequate investigation to meet the IEPA regulatory guidelines set forth under IAC Part 742. Most projects included preparation of Remedial Action Plans, specifying technical approach and

Professional Qualifications

costs. Overseen the installation of soil and groundwater remediation systems (i.e., vapor extraction, pump and treat, bioremediation, chemical injection and excavation). Most recent SRP projects include the work being done for the Village of Bartlett "Town Center" project, as described above.

IEPA LUST Projects
Over 16 years of experience in conducting Site Characterizations, Corrective Actions and UST removal at northeastern Illinois gasoline station facilities. Work was conducted in accordance to applicable IEPA LUST Program Part 732 regulatory guidelines. Principal investigator and geologist for field efforts, oversight of UST removal and remediation contractors. Site characterizations included installation of soil borings and monitoring wells to assess soil and groundwater conditions on the properties. Information was used to determine classification of the sites. Prepared technical work plans, reports and necessary budget forms for UST owner reimbursement through the IEPA LUST Program funds. Most projects were successful for obtaining the IEPA No Further Remediation Letter (NFR), therefore allowing site redevelopment as either residential or commercial properties.

Solid Waste Landfills
Consulting and expert testimony for several Illinois Counties, pertaining to technical review of solid waste landfill siting and/or expansion permit applications. Projects include detailed review of all aspects of geologic, hydrogeologic and geotechnical investigations as it pertains to Section 39.2 of the Illinois Environmental Protection Act, and IAC Parts 811 and 812 regulatory guidelines. Two of the most recent projects included technical review of landfill expansions for the Streator Landfill and the Livingston Landfill, both located in Livingston County.

Background also includes experience as a Principal field geologist/hydrogeologist for several Illinois and Indiana solid waste landfill siting applications and expansion of existing facilities. Responsibilities included oversight of all drilling and monitor well installations, documenting soil, bedrock and groundwater conditions. Conducted field permeability, and groundwater quality testing for determining background levels of select parameters. Responsibilities also included preparation of detailed geologic and hydrogeologic maps, interpretation of field data, and development of technical documents summarizing findings.

Agrichemical Distribution Facilities
As part of the Illinois Department of Agriculture study program, several projects have been conducted to evaluate agrichemical distribution facilities over numerous state counties in Illinois. Background information was obtained from the facility owner or operator. Using this information, the facilities were assessed for pesticide, herbicide, nitrate/nitrite concentrations.

Qualifications Summary

Mr. Wright holds a M.S. in Environmental Science from Governors State University. He has been with the Chicago laboratory for 19 years and has considerable environmental laboratory experience. Mr. Wright oversees analytical projects for a variety of industrial, engineering and government clients. His experience includes laboratory project management; project pricing and proposal preparation; project methodology review; QAPP preparation; and sampling of groundwaters, soils, leachates and industrial waste streams. He has expertise in sampling requirements and the design of sampling plans, and previously managed the field sampling department.

Professional Experience

Project Manager STL Chicago 1992 to Present

Mr. Wright's experience includes managing Project Management section, laboratory projects, and maintaining over-sight of Field Sampling operations. Responsible for laboratory project management staff activity, for both internal and external clients, including project assignment, forecasting revenues and workloads, planning, and continual improvement projects. Project Manager duties include providing technical expertise and quality assurance support relevant to analytical methods/procedures, quality assurance policy and objectives, coordination of client and/or technical meetings, representing clients in regulatory meetings, project prioritization and utilization of resources to ensure project execution, service evaluations data deliverable and turnaround time requirements, report deadlines, QAPP input, certifications, and overall project review. Responsible for providing the laboratory staff with the project profiles, background data and documents necessary to implement analytical activities. Monitor the progress of analytical activities for quality, timeliness, changes in scope of work, and cost. Utilize and integrate managerial, technical and support personnel to ensure project execution.

Field Manager 1987 - 1992

Responsible for preparing proposals, planning, coordinating and monitoring field project activities. Administer project changes, provide technical and quality assurance support, Sampling Plan and QAPP input, audits, prepare and review billing. Supervise and staff field operations personnel. Review field documentation, work plans, operating practices, safety plans and project reports.

Field Technician 1986 - 1987

Sampling groundwater monitoring wells, surface and industrial waters, leachates and soils. Experienced in landfill gas migration, groundwater levels, NPDES compliance monitoring, leachate levels, groundwater recharge data and monitoring well assessments. Responsible for documentation of field sampling procedures and recording of any deviations which may have occurred in the sampling process. Coordinated sampling efforts with the laboratory log-in and management staff.

Statistics Instructor 1985 - 1986

Experience as a statistics instructor in self designed course which integrated theory with the use of computerized analysis packages.

Education

- MS Environmental Science – Governors State University (1985)
- BA Environmental Science – Governors State University (1983)

Professional Training

- Participated in over thirty professional training seminars, conferences and expositions pertaining to project management and field monitoring, analytical chemistry, supervision, health and safety and proposal administration.
- Ethics Training
- Customer Service Training

Appendix B

STL Labs, Inc.
STL Quality Assurance, Laboratory SOPs, and QA Manual on CD-ROM

STL Chicago
STANDARD OPERATING PRACTICE (SOP) CHANGE FORM

Original SOP Number/Revision #: ME-6010B | Rev No. 06 Last Mod ID (circle): NA / (A)
SOP Title: Metals Analysis: Trace Inductively Coupled Argon Plasma by SW846 6010B
Affected SOP Section Number(s): 1.0 Scope / Application
Effective Date: 09/09/05

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Revision Number with Mod ID:

The following SOP change is in effect as of the stated date. This form will remain attached to the referenced SOP until such a time that the SOP is updated, approved, and redistributed, at which time it will become part of the historical SOP record. **Append this form to the front of the SOP copy.**

1. Reason for SOP Change: NFESC Audit for DOD Compliance - Corrective Action Response

2. Summary of Procedure Change (circle to indicate if there are attachments to this form: No / (Yes): # pages attached = 3)

"Specific requirements pertaining to the DOD QSM Version 3.0 are located in Appendix A. These requirements are additionally applicable to all NFESC projects. Any deviations from these procedures and/or variances from, must be addressed appropriately in accordance with standard operating protocol and pre-approved on a project by project basis."

The Tables will be added to the SOP proper upon the next revision. This form serves to document changes in the SOP until that time, as stipulated above.

Maureen Kenny 08/31/05
Initiated/Reviewed By: Name/Date

Initiated/Reviewed By: Name/Date

Approval Signature/Date: Section Manager 9-1-05

Approval Signature/Date: QA Manager or Designee Teresa A. Preston 9/6/05

STL Chicago

DoD QSM Version 3: Appendix DOD-B Quality Control Requirements Summary

Table B-1 Summary of QC Check Definitions, Purpose, and Evaluation – Inorganics (Metals)

QC Check	Definition	Purpose	Evaluation
Calibration Blank (Metals only)	Reagent water containing no analytes of interest, but acidified to the same pH as all samples	To determine the zero point of the calibration curve for all initial and continuing calibrations	Continued calibration blank responses above 2x the MDL require corrective action
CCV	This verification of the initial calibration that is required during the course of analysis at periodic intervals. Continuing calibration applies to both external standard and internal standard calibration techniques, as well as to linear and non-linear calibration models	To verify that instrument response is reliable, and has not changed significantly from the current ICAL	If the values for the analytes are outside the acceptance criteria, the initial calibration may not be stable. Results associated with out-of-control CCV results require reanalysis or flagging
Demonstrate Acceptable Analyst Capability	Analyst runs QC samples in series to establish his/her ability to produce data of acceptable accuracy and precision	To establish the analysts' ability to produce data of acceptable accuracy and precision	The average recovery and standard deviation of the replicate must be within designated acceptance criteria.
Dilution test (Metals only)	Analysis of a positive sample, which has been diluted to a concentration 1/5th of the original, to confirm that there is no interference in the original sample analysis.	To assess matrix interference	Agreement within 10% between the concentration for the undiluted sample and 5x the concentration for the diluted sample indicates the absenxe of interferences, and such samples may be analyzed without using MSA. Results outside acceptance limits indicate a possible matrix effect. ICP: must run a post-digestion spike GFAA: must run a recovery test
Duplicate Sample	Two identical portions of material collected for chemical analysis, and identified by unique alphanumeric codes. The duplicate may be portioned from the same sample, or may be two identical samples taken from the same site. The two portions are taken and prepared and analyzed identically.	To provide information on the heterogeneity of the sample matrix or to determine the precision of the intralaboratory analytical process for a specific sample matrix	To provide information on the heterogeneity of the sample matrix. The greater the heterogeneity of the matrix, the greater the RPD between the sample and the duplicate
ICAL	Analysis of analytical standards at different concentrations that are used to determine and calibrate the quantitation range of the response of the analytical detector or method	To establish a calibration curve for the quantification of the analytes of interest	Statistical procedures are used to determine the relationship between the signal response and the known concentration of analytes of interest. The ICAL must be successful before any samples or other QC check samples can be analyzed.
IDL (6010 and 6020 only)	The process to determine the minimum concentration of a substance (analyte) that an instrument can differentiate from noise. The procedure for calculating varies by method.	To provide a quarterly evaluation of instrument sensitivity	IDLs must be established before samples can be analyzed.
Interference check solutions (ICP only)	A pair of solutions containing interfering elements that are used to verify the correction factors of analytes of concern	To verify the established correction factors by analyzing the interference check solution at the beginning of the analytical sequence	No samples can be run if this check does not pass acceptance criteria
LCS containing all analytes required to be reported	A QC standard of known composition prepared using reagent free water or an inert solid that is spiked with analytes of interest at the midpoint of the calibration curve or at the level of concern.	To evaluate method performance by assessing the ability of the lab/analyst to successfully recover the target analytes from a control (clean) matrix.	This is a required QC Check. The inability to achieve acceptable recoveries in the LCS indicate problems with the accuracy/bias of the measurement system.
Linear dynamic range or high-level check standards (ICP only)	High-level check standard periodically analyzed to verify the linearity of the calibration curve at the upper end	To verify quantitative accuracy of data up to the high-level standard	The QC check establishes the upper linear range of the calibration
Low-Level calibration check standard (ICP only)	A reference standard that contains a quantity of analyte (greater than or equal to 3x the LOD (MDL))	To confirm the accuracy of measurements at or near the LOQ (RL), It establishes the LOQ of the calibration curve for those ICP methods that rely on single point calibration. It also may be used to validate a client's project RL	This QC check must be within acceptance criteria before any samples are analyzed.

Table B-1 cont.			
QC Check	**Definition**	**Purpose**	**Evaluation**
MS	A sample prepared by adding a know amount of targeted analyte(s) to an aliquot of a specific environmental sample	To assess the performance of the method as applied to a particular matrix	The lack of acceptable recoveries in the matrix spike often points to problems with the sample matrix. One test of this is a comparison to the LCS recoveries. If the corresponding LCS recoveries are within acceptable limits, a matrix effect is likely. The lab should not correct for recovery; only report the results of the analyses and the associated MS results and indicate that the results from these analyses have increased uncertainty
MSD	A 2nd replicate MS prepared in the lab, spiked with an identical, known amount of targeted analyte(s), and analyzed to obtain a measure of the precision of the recovery for each analyte	To assess the performance of the method as applied to a particular matrix and provide information of the homogeneity of the matrix.	When compared to the MS, the MSD will provide information on the heterogeneity of the sample matrix.
MDL Verification Check	A low-level spike taken through the prep and analytical steps at approximately 2x the MDL used to verify that the laboratory can detect analytes at the calculated MDL	To validate the MDL on an ongoing basis	If the MDL verification check fails, reprep/reanalyze at a higher level to set a higher MDL or the MDL study must be repeated.
MB	A sample of a matrix similar to the batch of associated samples in which no target analytes or interferences are present at concentrations that impact the analytical results.	To assess background interferences or contamination in the analytical system that might lead to high bias or false positive data.	This QC is used to measure lab accuracy/bias. The MB could indicate whether contamination is occurring during sample prep and analysis. If analytes are detected > ½ RL, reanalyze or B-Flag results for all samples in prep batch. For common lab contaminants, no analytes detected > RL. See DoD Box D-5; & Sec. D.1.1.1
MDL Study	The process to determine the minimum concentration of an analyte that can be measured and reported with 99% confidence that the analyte concentration is greater than zero and is determined from analysis of a sample in a given matrix containing the analyte.	To determine the lowest concentration of an analyte that can be measured and reported with 99% confidence that the analyte concentration is greater than zero.	MDLs must be established prior to sample analysis. The RL or LOQ is at least 3x the MDL. Used in combination with the MDL verification check to validate the MDL on an ongoing basis.
MSA (ICP/GFAA only)	Adding known amounts of standard to one or more aliquots of the processed sample solution.	To compensate for a sample constituent that enhances or depresses the analyte signal, thus producing a different slope from that of the calibration standards. It will not correct for additive interferences that cause a baseline shift.	This is the method used when matrix interferences are present and do not allow determination of accurate sample results
Post digestion spike addition (ICP and ICP/MS only)	An analyte spike added to a portion of prepared sample to verify absence or presence of matrix effects	To confirm the presence of a matrix interference. Assess matrix effects based on: 1. the occurrence of new and unusual matrices included within the batch, or 2. contingency analysis based on SD or MS failures	To verify the absence of an interference, the spike recovery criteria is 75%-125% Results outside this criteria require MSA for all samples within the batch
Recovery Test (GFAA only)	An analyte spike added to a portion of prepared sample to verify absence or presence of matrix effects	To confirm the presence of a matrix interference. Assess matrix effects based on: 1. the occurrence of new and unusual matrices included within the batch, or 2. contingency analysis based on SD or MS failures	To verify the absence of an interference, the spike recovery criteria is 85%-115% Results outside this criteria require MSA for all samples within the batch

Table B-1 cont.			
QC Check	**Definition**	**Purpose**	**Evaluation**
Second source calibration verification	A standard obtained or prepared from a source independent of the source of standards for the initial calibration. Its concentration should be at or near the middle of the calibration range. It is done after the initial calibration.	To verify the accuracy of the initial calibration	The concentration of the 2nd source calibration verification, determined from the analysis, is compared to the known value of the standard to determine the accuracy of the ICAL. This independent verification of the ICAL must be acceptable before sample analysis can begin.

Notes:
1. Project-specific requirements identified by the client supersede any requirements listed. The requirements are meant to be default, to be used when project-specific direction based on DQOs is not available.
2. If there is a contradiction between the method and the DoD tables, the requirements specified in the tables shall be followed.
3. If the requirements in the DoD tables do not yet correspond with the most recent version of the SW-846 method, or a new method that analyzes for the same group of analytes becomes available, the requirements in the method shall be followed where appropriate.

STL Chicago

DoD QSM Version 3: Appendix DOD-B Quality Control Requirements Summary

Table B-6: Inorganic Analysis by ICP and AA - Methods 6010 and 7000 Series

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria
IDOC	Per Instrument/Analyst	DoD acceptance criteria if available; otherwise method specific criteria	Correct / Repeat for those analytes which failed criteria	NA
MDL	Annually or quarterly MDL Checks performed	40 CFR 136B; MDL verification checks must produce a signal at least 3x the instrument's noise level.	Run MDL check at higher level and set MDL higher or reconduct MDL study	NA
IDL (ICP only)	Every 3 months	Detection limits established shall be ≤ MDL	NA	NA
Linear dynamic range (LRS) or high-level check (ICP only)	Every 6 months	Within ± 10% of expected value	NA	NA
ICAL ICP: min 1 high std and a calibration blank GFAA: min 3 stds and a calibration blank CVAA: min 5 stds and a calibration blank	Daily initial calibration prior to sample analysis	ICP: No acceptance criteria unless more than 1 standard is used, in which case $r \geq 0.995$ GFAA: $r \geq 0.995$ CVAA: $r \geq 0.995$	Correct problem then repeat initial calibration	NA
2nd Source calibration verification (ICV)	Once after each initial calibration	Value of 2nd source for all analytes within ± 10% of expected value	Correct problem and verify 2nd source standard. Rerun, if that fails, correct problem and repeat initial calibration.	NA
Continuing Calibration verification (CCV)	CCV – After every 10 field samples and at end of analysis sequence. (DoD Box 58: CCV standards shall be at or below the middle of the calibration range)	ICP: within ± 10% of expected value GFAA: within ± 20% of expected value (Data associated with an unacceptable CCV may be fully usable under the following conditions: 1. CCV (high bias) and samples ND, then raw data may be reported with appropriate flag 2. 2. CCV (low bias) and samples exceed maximum regulatory limit/decision level (DoD Box 60: Project specific permission from appropriate DoD personnel is required to report data generated from a run with noncompliant CCV.)	Correct problem, rerun CCV. If that fails, repeat ICAL and reanalyze all samples since the last good CCV (DoD Box 59...if the lab chooses to demonstrate the success of routine corrective action through the use of 2 consecutive CCVs, then the concentrations of the two CCVs must be a two different levels within the original calibration curve with at least one falling below the middle of the calibration range.)	NA
Low-level calibration check standard (ICP only)	Daily, after one-point initial calibration	Within ± 20% of expected value	Correct problem, then reanalyze. No sample may be analyzed without a valid low-level calibration check standard. Low-level calibration check standard should be ≤ RL	NA
MB	One per prep batch	No analytes detected > ½ RL For common lab contaminants, no analytes > RL	Correct problem, then see criteria in box D-5; if required, reprep/reanalyze MB and all associated samples	Apply B-flag to all results for the contaminated analyte for all samples in the associated prep batch
Calibration Blank (ICB / CCB)	Before beginning a sample run, after every 10 samples, and at the end of the analysis sequence	No analytes detected > 2x MDL	Correct problem, then reprep and reanalyze calibration blank and previous 10 samples	Apply B-flag to all results for the contaminated analyte for all samples in the associated prep batch
Interference check solution (ICS) (ICP only)	At the beginning of analytical run	ICS-A: Absolute value of concentration for all non-spiked analytes < 2x MDL (unless they are verified trace impurity from one of the spiked analytes) ICS-AB: ± 20% of expected value	Terminate analysis; locate and correct problem; reanalyze ICS	NA
LCS (containing all analytes to be reported)	One LCS per prep batch	DoD specified QC criteria, if available	Correct problem, reprep/reanalyze the LCS and all samples in the associated prep batch for all failed analytes, if sufficient sample is available	Apply Q-flag to specific analyte(s) in all samples in the prep batch.

Table B-6 cont.				
QC Check	**Minimum Frequency**	**Acceptance Criteria**	**Corrective Action**	**Flagging Criteria**
Dilution Test	Each prep batch or when a new or unusual matrix is encountered	5x dilution must agree within ± 10% of the original determination Note: Only applicable for samples with concentrations > 50x MDL (ICP) or > 25x MDL (GFAA and CVAA)	ICP: Perform post-digestion spike (PDS) GFAA: Perform recovery test CVAA: Perform matrix spike	NA
Post-digestion spike (PDS) (ICP only)	When dilution test fails or analyte concentration in all samples < 50 x MDL	Spike addition must produce a level between 10-100 x MDL. Recovery limit: 75-125% of expected value	Run samples by MSA or see flagging criteria	Apply J-flag to all associated sample results (for same matrix) for specific analyte(s)
Recovery Test (GFAA only)	When dilution test fails or analyte concentration in all samples < 25x MDL	Recovery limit: 85-115% of expected value	Run samples by MSA or see flagging criteria	Apply J-flag to all associated sample results (for same matrix) for which MSA was not run
MSA or Internal Standard Calibration	When matrix interference is suspected	NA	NA	NA Document in Case Narrative
MS	One per prep batch per matrix	For matrix evaluation, use DoD specified QC criteria for LCS	Examine the project-specific DQOs. Contact client for additional corrective action measures.	Apply J-flag to specific analyte(s) in the parent sample
MSD or Sample Duplicate	One per prep batch per matrix	RPD ≤ 20% (between MS and MSD or sample and sample duplicate)	Examine the project-specific DQOs. Contact client for additional corrective action measures.	Apply J-flag to specific analyte(s) in the parent sample
Results reported between LOD and LOQ			Apply J-flag to all results between LOD (MDL) and LOQ (RL)	

Notes:
1. Project-specific requirements identified by the client supersede any requirements listed. The requirements are meant to be default, to be used when project-specific direction based on DQOs is not available.
2. If there is a contradiction between the method and the DoD tables, the requirements specified in the tables shall be followed.
3. If the requirements in the DoD tables do not yet correspond with the most recent version of the SW-846 method, or a new method that analyzes for the same group of analytes becomes available, the requirements in the method shall be followed where appropriate.

SOP No.	Revision No.	Date	Page
UME-6010B	06	01/05/05	1 of 25

TITLE: **Metals Analysis**
Trace Inductively Coupled Argon Plasma by SW-846 6010B
(Simultaneous Operation)

Updated by:	Signature:	Date:
Todd D. Smith Senior Analyst		1-4-05

Approved by:	Signature:	Date:
Jodi L. Gromala Supervisor, Metals Dept.		1-4-05
David W. Mazur Env. Health & Safety Coor.		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.

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.

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.

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 (HNO_3) - 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).

5.2.1 Calibration Standards

Prepared with Milli-Q water that has been acidified with 1% HNO_3 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. HNO_3 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	• Add ~50 mLs of Milli-Q water to a 200 mL Class A volumetric flask. • Re-pipette 2 mLs conc. HNO_3 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	• Add ~50 mLs of Milli-Q water to a 200 mL Class A volumetric flask. • Re-pipette 2 mLs conc. HNO_3 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	• Add ~50 mLs of Milli-Q water to a 200 mL Class A volumetric flask. • Re-pipette 2 mLs conc. HNO_3 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	• Add ~50 mLs of Milli-Q water to a 200 mL Class A volumetric flask. • Re-pipette 2 mLs conc. HNO_3 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.

Standard	Preparation
S2A	• Add ~50 mLs of Milli-Q water to a 200 mL Class A volumetric flask. • Re-pipette 2 mLs conc. HNO_3 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	• Add ~50 mLs of Milli-Q water to a 200 mL Class A volumetric flask. • Re-pipette 2 mLs conc. HNO_3 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% HNO_3 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)	• 10 mLs conc. HNO_3 • 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)	• 10 mLs conc. HNO_3 • 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.

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]	• 10 mLs conc. HNO_3 • 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 Milli-Q water and mix thoroughly. * Cd Intermediate = 1:10 dilution of 1,000 ppm Cd. * Be Intermediate = 1:10 dilution of 1,000 ppm Be
Interferent Check Standard (ICSA)	• 10 mLs conc. HNO_3 • 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)	• 10 mLs conc. HNO_3 • 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 $\geq$ 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.

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 ≤ 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 ≥ 5X reporting limit, 20 RPD; if < 5X reporting limit ± reporting limit; if < reporting limit no control range.
[5] If the analyte concentration is >10X the MDL, results should agree within ±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.

Matrix	Holding Time[1]	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. ≥ 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	± 10% of the Known Conc.

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)

- 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.

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 $\geq$ 0.995.

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	• reanalyze the standard curve; • prepare a new stock and/or working standards; • check the reagents/solutions and prepare fresh if necessary.
ICV	• 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	• 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.

QC Indicator	Suggested Corrective Actions
LCS	<u>If the LCS is low</u>: • 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. <u>If the LCS is high:</u> • 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	• 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	• a CAR will be written and approved by the supervisor or section manager.
MS / MSD	• a CAR will be written and approved by the supervisor or section manager.
Serial Dilution (L)	• prepare a new serial dilution to verify proper preparation; • a CAR will be written and approved by the supervisor or section manager.
CCV	• 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	• 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.

QC Indicator	Suggested Corrective Actions
Additional CAs	• 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 ≥ 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 = $\frac{\text{(spiked sample) - (unspiked sample)}}{\text{spiked concentration}} \times 100$

9.2 Precision (RPD)

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

9.3 Concentration

$$\text{mg/kg or L} = \frac{C \times V \times D}{W}$$

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.

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 01: 01/29/99
Revision 02: 03/20/00
Revision 03: 06/29/01
Revision 04: 09/13/02
Revision 05: 10/30/03
Revision 06: 01/03/05

Reasons for Revision; Revision 06:

- Annual Review – Maintenance Log added as attachment.

Table 1.

Element and Reporting Limits

Element	ICAP 61E (ICP3) Wavelength (nm)	ICAP 61E (ICP4) Wavelength (nm)	ICAP 61E (ICP5) Wavelength (nm)	Reporting Limits[1] 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

[1]These 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.
[2]Y 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.

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

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

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

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

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 =			
				As = Y =			
				As = Y =			

Reviewed by:_____ Date:_____ CHI-22-14-038/E-04/03

STL Chicago
TJA Trace ICAP (61E) - ICP3
Instrument Maintenance Log

Page No._____

	Date/Initials	Date/Initials	Date/Initials	Date/Iniitals	Date/Initials	Date/Initials	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:_____

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			

Comments on Reverse

TASK : CAR's ___ ___ ___	PRI REV	SEC REV	COMMENTS
LabNet Batch Status Report Displays Data At RVWD Status			
Incomplete JOB Status Report reveals no Outstanding Data			
NARRATIVE: 1) Holding Times			
2) Method References			Method: a) CLP b) SW846 c) Other
3) % Recoveries / RPD's			
4) Analytical Difficulties/Typos			

COMMENTS:

QC CYCLE	REASON
1	
2	

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	
☐	☐	1. Verification of standard traceability and expiration (daily).
		2. Calibration is clearly documented:
☐	☐	a. Instrument is calibrated using a Blank and three Calibration Standards. The correlation coefficient must be ≥0.995.
☐	☐	b. Reanalysis of the top calibration standard as a sample. Control limits are 95 - 105%. (Run once daily prior to sample analysis).
		3. Calibration Verification: (10% Frequency):
☐	☐	a. ICV/CCV: **Std./CLP** – Recovery 90-110% **EPA 200.7 (ICV)** – Recovery 95-105%
☐	☐	b. ICB/CCB: **Std. QC:** < RL; **CLP QC:** < CRDL; **SW-846 QC:** < 3x MDL. (custom criteria code)
		4. **CLP QC:** An Initial & Final for each sample analysis run:
☐	☐	a. CRI - 2x RL; No Limit Set
☐	☐	b. ISA/ISAB - 80-120% Recovery
		5. **Std. QC:** Analyzed at the beginning of the day and every 8 hours thereafter:
☐	☐	a. CRI: 2x CRDL; No Limit Set
☐	☐	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	
		1. Each Prep Batch consists of a maximum of 20 samples of a similar matrix:
☐	☐	a. Prep Batches must be clearly identified
☐	☐	b. 1 Prep Blank **CLP** - < CRDL; **Std. QC** - < RL **TCLP** - < TCLP Reporting Limit
☐	☐	c. 1 LCS **Std./CLP** - 80-120% Rec.; **EPA 200.7** - 85-115% Rec.
☐	☐	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
☐	☐	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
☐	☐	f. Analytical MS **TCLP** - >50% (MSA performed if <50% recovery)
☐	☐	g. Serial Dilution 1 per 20 samples; 10% Difference Limit
☐	☐	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).
☐	☐	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:

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%

Title: LABORATORY QUALITY MANUAL

STL Chicago
2417 Bond Street
University Park, Illinois 60466-3182
(708) 534-5200

Approved by:	Signature:	Date
Michael J. Healy Laboratory Director	Michael J. Healy	7/6/05
Terese A. Preston Quality Manager	Terese A. Preston	7/08/05

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COPY # : 020

ISSUED TO : Gary Deigan / Deigan & Associates

Full Signature Approvals Are Kept on File
with STL's QA Standard Practice Records

STL Chicago
STANDARD OPERATING PRACTICE (SOP) CHANGE FORM

Original SOP Number/Revision #: UQA-LQM /Rev No. 04 Last Mod ID (circle): NA / _A
SOP Title: STL Chicago Quality Manual
Affected SOP Section Number(s): 1.1
Effective Date: 09/26/05

CONTROLLED DISTRIBUTION

COPY # :
ISSUED TO :

Uncontrolled

Full Signature Approvals Are Kept on File with Severn Trent Laboratories Standard Practice Records

Revision Number with Mod ID:

The following SOP change is in effect as of the stated date. This form will remain attached to the referenced SOP until such a time that the SOP is updated, approved, and redistributed, at which time it will become part of the historical SOP record. **Append this form to the front of the SOP copy.**

1. Reason for SOP Change: Self-Declaration for DOD QSM Version 3.0

2. Summary of Procedure Change (circle to indicate if there are attachments to this form: No / Yes: # pages attached = ___)

The following shall be added to Section 1.1 of the LQM: "STL Chicago has reviewed the Department of Defense Quality Systems Manual (DoD QSM) and compared this document to the Laboratory Quality Manual and associated Standard Operating Procedures. STL Chicago has documented its level of compliance with the DoD QSM Standard and identified areas of specific variances between the laboratory's LQM, QSM and associated SOP's. The laboratory will work with all DoD clients to negotiate specific project requirements and data quality objectives, on a project-by-project basis. These requirements will reflect both the laboratory's Quality System and the requirements of the DoD QSM. Project-specific QAPP's will clearly identify and outline all negotiations and ultimate expectations for the specific project. "

Marcella D. Kinney 09/26/05
Initiated/Reviewed By: Name/Date

Initiated/Reviewed By: Name/Date

Jesse A. Preston 9/26/05
Approval Signature/Date: Section Manager

Michael J. Healy 9/26/05
Approval Signature/Date: QA Manager or Designee

TABLE OF CONTENTS

Tables

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Appendix

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, soil, solid, waste and drinking water.

Associated with this activity are services to ensure client requirements are known, communicated and satisfactorily addressed, and a deliverables package presenting the analytical results meets client needs. 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)
- Department of Defense (DoD)
- Navy Facilities Engineering Service Center (NFESC)
- Clean Water Act (CWA)
- Comprehensive Environmental Response, Compensation, and Liability Act (CERCLA)
- 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 at www.stl-inc.com or available from the laboratory.

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 operations. 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 4.0, February 2005.

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, Final Version 3, March 2005

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
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w. Table of contents, listing reference, glossaries and appendices	TOC Table of Contents Appendix A List of Cited SOPs and Work Instructions Appendix B STL Chicago's Method Capability Listing Appendix C STL Chicago's Description and Floor Space for Analytical Facilities

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. 9601et 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 the method reporting limit or lowest calibration standard. Used in conjunction with the MRL Check standard in LCG 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 ±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 ±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 ½ the spike level used for the MDL Study or ½ the method reporting limit or ½ the lowest calibraton standard.

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 Organizational Chart

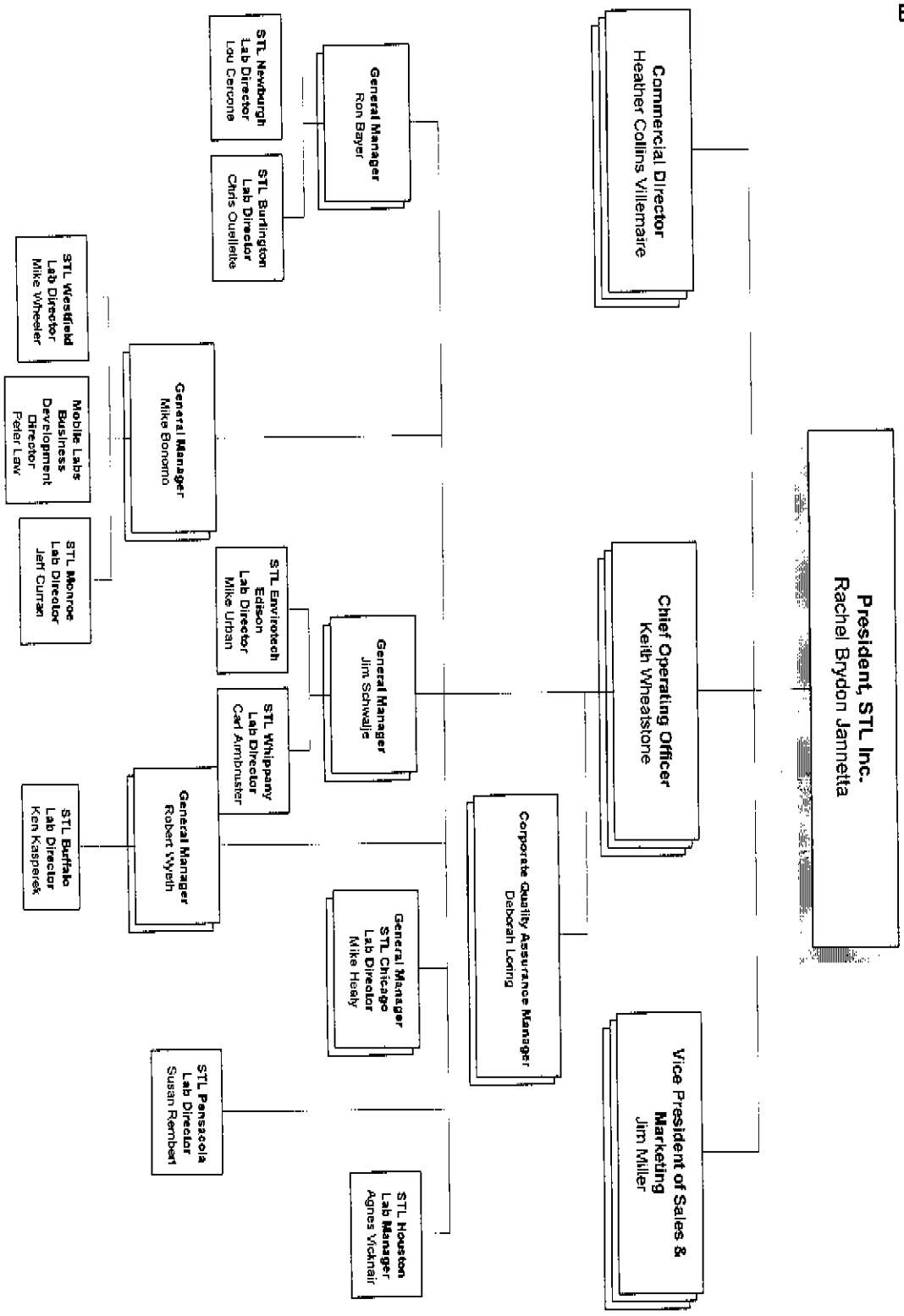

Figure 1. STL Organization Chart

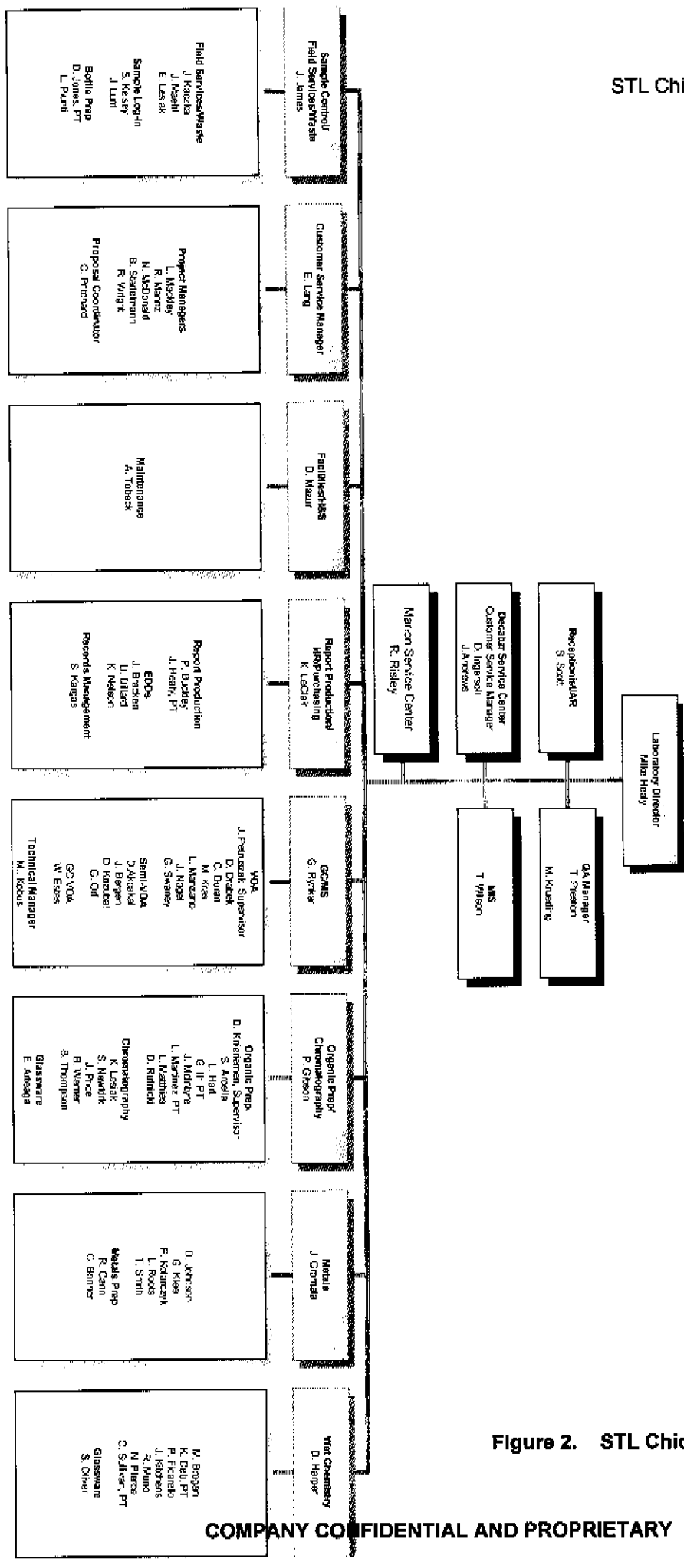

Figure 2. STL Chicago Organizational Chart

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 83 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/MS	GC	HPLC	ICP	ICPMS	AA	CVAA	AutoAnalyzer	IC	TOC	TOX
14	14	6	2	1	3	2	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 ensure 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 ensure 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, verification 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,
- 23 years laboratory experience.
- Terese A. Preston, Quality Assurance Manager, BA Biology,
- 21 years laboratory experience.
- Diane L. Harper, Inorganics Section Manager, MA Biology,
- 25 years laboratory experience.
- Jodi L. Gromala, Metals Section Manager, BS Biology,
- 18 years laboratory experience.
- Patti J. Gibson, Chromatography/Organic Extractions Section Manager, BS Biology,
- 16 years laboratory experience.
- Gary L. Rynkar, GC/MS Section Manager, BS Environmental Biology,
- 17 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 Coordinator

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 ensure 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 contain 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 parenthetic 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
- Ensure 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 yearly basis, which is tracked by an established review schedule (*Approved SOP Listing*; CHI-22-09-SOP). These reviews are conducted by the creator of the SOP and/or Department Manager, QA Specialist and/or QA Manager, Health and Safety Coordinator, and Lab Manager all of whom provide the approval signature for each SOP where appropriate to the subject of the 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 are 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". The laboratory also takes guidance from the STL Corporate MDL SOP (S-Q-003). 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 and/or low level check standard. 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 2-3x the respective MDL to ensure confidence in the value reported. Special project (i.e., ILEPA TACO limits; MIDEQ limits) or program (i.e., AFCEE; LCG; DoD) specific reporting limit requirements are routinely evaluated by the QA and project management staff. Every effort is made to meet project goals or objectives if it is within the laboratory's capability to do so within minimal risk to the quality of the data. Data evaluated below the RL 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. Refer to the laboratories MDL SOP (UQA-017) for additional tools that are used in the MDL evaluation process. If the laboratory continually demonstrates that the method reporting limits are not achieved, equipment, technique, and the method are reviewed to ensure 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, Lab Manager or Project 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 STL's corporate *Testing Solvents and Acids* procedure (S-T-001) for all of the STL laboratories. Notification of approval of specific lot numbers are sent via e-mail to the QA Manager, who subsequently forwards it to the facility manager. A listing of approved lot numbers is also available electronically on the STL Website under Corporate Information / Technology / Approved Solvent Spreadsheet.

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 (for those items indicated on the COC), the LabNet Sample Receipt Checklist and on a Sample Discrepancy Report (SDR) normally as a directed Job Note to the appropriate Project Manager; 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 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), a Customer Complaint Form (CHI-22-09-340) or in a format specifically designed for that purpose (e.g., phone conversation record or e-mail). The Laboratory Director, Project Manager, QA Manager, and Section 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 or electronic 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	- LQM - QMP (Corporate) - QAPPs - SOPs - Work Instructions	- Audits – Internal - Audits - External - Audit Responses - Certifications - PTs - SDR/RDRs - CARs - Review Checklists - Logbooks* - Standard Certificates - Method & Software Validation/Verification - MDL/IDL/IDOC Studies - Statistical Evaluations - Training Records - CDOC Evaluations - QA Reports - Electronic QA Files	- COC - Contracts & Amendments - Correspondence - QAPP - SAP - Telephone Logs - E-mails - Electronic Data - Data Report	- Accounting - Corporate Safety Manual - Permits - Disposal Records - Employee Handbook - Personnel files - Employee Signature & Initials Form -Technical & Administrative Policies

*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		Retention Requirement *
Raw Data	All*	10 Years from completion (Electronic Data Reports - .pdf & EDD) 5 Years from completion for Hardcopy when not available in electronic form 5 Years from archival for electronic raw data
Controlled Documents	All*	5 Years from document retirement date
QC Records	All*	5 Years from archival
Project Records	All*	5 Years from project completion
Administrative Records	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
Specific Client Program / Project	Per contractual requirement

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	QA Department or Designee	**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 approximately the 3rd day of the month, the QA manager prepares a monthly QA report. The report is sent to the Laboratory Director, Project Managers, Section Managers and Department Supervisors. At a minimum, the report content contains the items listed below (Figure 3). A separate report is prepared for and submitted to the Corporate Quality Control Director and the Lab Director. This report contains a narrative summary which includes audit details; revised report details; client complaints; certifications/approvals; preventive actions; QA Highlights and QA Lowlights. Also included are the monthly metrics spreadsheet, PT summary and External Audit Summary. 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. STL Chicago Monthly QA Report Format

1	Audits
	A. External System Audits B. Internal System Audits C. Internal Data Audits
2	Revised Reports / Client Complaints / Client Compliments
	A. Revised Reports (RDR) B. Customer Complaints C. Customer Compliments
3	Certification Changes
	A. Certification Status B. Certified Parameter List
4	Proficiency Testing
	A. PES Results/Scores B. PES Failure Summary C. PES History of Non-Acceptable Scored Analyte/Compound
5	Miscellaneous QA and Operational Issues
	A. Current SOP Status (with 'on-time' percentages calculated for SOPs < 1 year) B. Listing of SOPs > 1 Yr C. Listing of SOPs in Progress
6	QAPP/Project Review Status
7	Holding Time Violations
8	Monthly QA Report Metrics

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, H&S officer, and Section Manager/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. Each administrative/non-technical section also maintains training records for each employee. All Training Records are also kept on file in the QA Department for periodic review with the appropriate Section Manager/Supervisor. 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 Health & Safety officer maintains training documentation related to H&S issues.

The QA Department maintains the following evidence items on file for each employee:

- An Ethics Agreement signed by each staff member (renewed each year). (Figure 6)
- A Confidentiality Agreement signed by each staff member (renewed each year).
- Representative Signature and Initials by each staff member (renewed each year).
- The employee has read and understood the latest, approved version of all test methods and/or SOPs for which the employee is responsible (annual review/sign-off of SOP revisions).
- A training record specific to the job functions performed.
- Copy of external Training seminars or class completion certificates.

The following evidence items are on file (in addition to those listed above) for each technical employee:

- Initial Demonstration of Capability (IDOC) for each method. (CHI-22-09-271) (Figure 4)
- Annual evidence of Continued Demonstration of Capability (CDOC) for each method (CHI-22-09-243) (Figure 5)

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 (Figure 4) is recorded and maintained in the analyst'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 analyst's 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). When an analyst has not yet completed the IDOC requirement, they can perform a task under the supervision of a qualified analyst, 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.

On an annual basis, the analyst's method capabilities must be evaluated, which may include, but is not limited to, successful analysis of a blind sample on the specific test method (PT) or a similar test method; an annual DOC of four successive and acceptable LCSs; Control Chart Evaluations over a given time period. The QA Department in conjunction with the appropriate Section Manager will accumulate specific required information to satisfy the CDOC (Continued Demonstration of Capability) requirement. Documentation will be filed within the analyst training file. *Figure 5* shows an example of a *Continued Demonstration of Method Proficiency* statement (CHI-22-09-243).

Although training is a continual process, initial training is considered complete once the trainee has attended the initial general orientation(includes specific forms to be reviewed and signed, Timesheet Training, Employee Handbook, Drug Policy Form, Ethics/Confidentiality forms, Internet and E-Mail Usage, IT Policy Form, Benefit Info), presentations (ex. Ethics Orientation and Comprehensive Training, QA Orientation-including Manual Integration and Selection of Calibration Points for technical staff, Health & Safety Orientation), and review of those SOP's applicable to the employee's responsibilities. Documentation is appropriate to the training item. Specific training related to the department is assessed and documented within the employee's training record, which is updated over the course of the employee's training progress. This process is applicable to both Technical and Non-Technical employees.

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
Employee Orientation / HR	Week 1	All
Ethics – Corporate Overview	Week 1	All
Environmental Health & Safety	Month 1	All
Ethics Data Integrity	Month 1 Month 1	All Technical and PMs
Ethics Refresher	Annually	All
Quality Assurance	Quarter 1	All
Initial Demonstration of Capability (IDOC)	Prior to unsupervised method Performance	Technical
Continued Demonstration of Capability (CDOC)	Annually	Technical

[1] From the date of initial employment unless otherwise indicated.

The Ethics, Data Integrity and 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.

Further details of the laboratory's training program are described in the Laboratory Training SOP (UQA-014).

Figure 4. Initial Demonstration of Capability Certification Statement

STL Chicago
Initial Demonstration of Method Capability
Certification Statement

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.

_____ _____
Supervisor/Manager Signature Date

_____ _____
QA Signature Date

Figure 5. Continued Demonstration of Method Proficiency

STL Chicago
Continued Demonstration of Method Proficiency

Analyst Name:_____
SOP No.:_____
Analytical Method:_____
Similar Test Methods:_____
Analyte(s):_____

Documentation of Continued Proficiency

Continued Proficiency has been demonstrated by one of the following:

_____ 1. Successful analysis of a blind performance sample (blind to the analyst) on a similar test method using the same technology (Documentation required for one of the test methods).
PT-IS(s): _____
(See attached PT Summary)
_____ 2. Another demonstration of capability.
Description: _____
_____
_____ 3. Successful analysis of a blind performance sample (double-blind to the analyst/QA) on a similar test method using the same technology (Documentation required for one of the test methods).
PT Description: _____ Job#: _____
_____ 4. At least 4 consecutive laboratory control samples with acceptable levels of precision and accuracy.
(See Attached Control Chart)
_____ 5. If Items 1-4 cannot be performed, analysis of authentic samples with results statistically indistinguishable from those obtained by another analyst.

The analyst identified above, using the cited method(s) which is in use at this laboratory and defined within the laboratory's document control system, has read, understood and agrees to perform this most recent version of the test method.

_____ _____
Analyst Signature Date

_____ _____
QA 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 6). 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 6. 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 contact 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 locked at all times with controlled and documented access. Access is controlled by various measures including locked doors, electronic access cards, security codes, and a staffed reception area 8:00 a.m. to 5:00 p.m. Monday through Friday. All visitors sign in and are escorted by STL personnel while at the facility.

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.. Refer to the description of floor space in Appendix C for additional details.

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.

STL Chicago maintains an updated list of all current primary and secondary accreditations. This information is available through the STL web-site (http://stlnet.stl-inc.com). The web-site contains links to all certifications and methods for which the laboratory is currently accredited. In addition, a listing of STL Chicago's Method Capabilities appears in Appendix B *(Methods Capabilities Work Instruction (CHI-22-09-255)*. The table also identifies those methods for which NELAP accreditation is offered and for which the laboratory holds NELAP certification. Certifications are subject to change, and may do so based on laboratory needs and performance. All certifications must be confirmed with appropriate laboratory personnel.

5.3.1 Method Selection

Since numerous methods and analytical techniques are available, continued communication between the client and laboratory is imperative to ensure 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 7).

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
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 7).

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. Where an SOP is based on a published method, the laboratory maintains a copy of the reference method.

Figure 7. Proprietary Information Statement

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 ensure 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).

Validation

Validation is the process of establishing evidence that provides a high degree of assurance that a specific process will consistently produce a product meeting pre-determined specifications and user needs. Software validation involves documentation of original specifications, identity of code, printout of code, software name, software version and any other specific procedures outline in the manufacturers Validation Process. Most often, this documentation exists as a Software Validation Certificate, obtainable from the appropriate manufacturer. Per STL *Policy S-ITQ-007 Software Testing, Verification and Validation*, purchased software that has not been modified at the source code level is not required to be internally validated. As specified in STL Chicago's SOP *UIS-006 Procedures and Processes Related to Entry, Storage, Back-up/Retrieval and Management of Bench Level Electronic Data*, all software related to instrument data gathering was installed in its entirety, with no changes made to base codes or algorithms. Where possible, Software Validation Certificates have been obtained, and are filed within the QA Department.

The Validation of the LabNet system, STL's proprietary LIM's and STL Chicago's end-processing and reporting system, was completed both as a corporate initiative and at STL Chicago during the implementation of the system. The system 'methods' applicable to STL Chicago, containing the algorithms and formulas were tested and documented by the QA Department. Results of this initiative are documented and kept on file in the QA Department.

Verification

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 initial verification of LabNet software programs were conducted by the QA Department with the assistance of the section managers and appropriate personnel. The QA Department also documents the approval of, and verifies, any program modifications. All records of the verification are retained in the QA Department.

Verification of instrumental software was also completed at the time of implementation, either by way of manual comparison to computer generated data or comparison to data generated by the previous system being replaced. Documentation of the most recent systems of all verification procedures is on file in the QA Department. Additionally, an Instrument Validation Checklist (Figure 8, CHI-22-09-286) is provided to each department, which includes a section outlining Software Verification Requirements and both the process and location of such documentation for newly installed systems.

EDD validation and verification is discussed in STL Chicago's SOP *UIS-001 EDD Specifications, Development Generation and Review.*

The above procedures do not apply to general purpose software, except where those applications are used to perform calculations in support of client data. In those cases, verification will be required.

Figure 8: Instrument Validation Checklist

STL-CHICAGO
INSTRUMENT VALIDATON CHECKLIST

Instrument Type: _____
Model#:_____
Serial #:_____
Lab Equip Code: _____ LIMS Equip Code: _____

Installation Date: _____
Installed By: _____

Instrument installed per specifications. Operational and functional per install guidelines.
Signature/Date of installer: _____

Outstanding items yet to be completed (If applicable):_____
_____
_____
_____

Completion Date: _____
Signature/Date Lab Representative: _____

Instrument passes all initial required lab checks and calibrations as appropriate
for method of analysis: _____

Appropriate MDL's as applicable per method complete: _____
*MDL's must be forwarded to the QA Dept. for Validation to be considered complete.

Methods to be analyzed (may change over time): _____
_____

In-Service Date: _____

Signature/Date of Lab Representative: _____
Signature/Date of QA Representative: _____
****

QA Only
Software Verification Required (Y/N)
Verification Completed as Described: _____
_____
_____
_____
Software Verification Documentation Location:_____
Signature/Date of QA Representative: _____

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 (Maintenance Logs may be hard-copy bound or electronic).

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 (hard-copy bound or electronic CHI-22-09-341) 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.

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	Daily
	Replace windows	As required
	Check or change cuvette	Daily
	Check & drain compressor drain	Daily
	Clean atomizer cell/furnace hood	Daily
	Nebulizer cleaned/dried	Weekly or as required
	Check/change marble stones	Weekly
	Clean filters	Weekly
	Change graphite tube/platform	As required
	Empty waste container	Daily
	Remove carbon tube and check wear	Daily
	Check sample introduction probe	Daily
Leeman Mercury Analyzer	Check tubing for wear	Daily
	Fill rinse tank with 10% HCl	Daily
	Insert clean drying tube filled with Magnesium Perchlorate	Daily
	Fill reductant bottle with 10% Stannous Chloride	Daily
ICP	Check pump tubing	Daily
	Check liquid argon supply	Daily
	Check fluid level in waste container	Daily
	Check filters	Weekly
	Clean or replace filters	As required
	Check torch	Daily
	Check sample spray chamber for debris	Monthly
	Clean and align nebulizer	Monthly
	Check entrance slit for debris	Monthly
	Change printer ribbon	As required
	Replace pump tubing	As required
ICP MS	Change pump tubing	Weekly
	Clean torch	Weekly
	Check / clean nebulizer	Weekly
	Clean cones	Daily
	Check air filters	Weekly
	Check multiplier voltages & do cross calibration	Weekly
	Replace sample uptake tubing	Monthly
	Check rotary pump oil	Monthly
	Check oil mist filters	Monthly
	Check chiller water level	Monthly
UV-Vis Spectrophotometer	Clean ambient flow cell	As required
	Precision check/alignment of flow cell	As required
	Wavelength verification check	Semi-annually

Table 9. Major Equipment Maintenance

Instrument	Procedure	Frequency
Auto Analyzers	Clean sampler	Daily
	Check all tubing	Daily
	Clean inside of colorimeter	Daily
	Clean pump well and pump rollers	Quarterly
	Clean wash fluid receptacle	Weekly
	Oil rollers/chains/side rails	Weekly
	Clean optics and cells	Quarterly
Hewlett Packard GC/MS	Ion gauge tube degassing	As required
	Pump oil-level check	Monthly
	Pump oil changing	Annually
	Analyzer bake-out	As required
	Analyzer cleaning	As required
	Resolution adjustment	As required
	COMPUTER SYSTEM AND PRINTER:	
	Air filter cleaning	As required
	Change data system air filter	As required
	Printer head carriage lubrication	As required
	Paper sprocket cleaning	As required
	Drive belt lubrication	As required
Gas Chromatograph	Compare standard response to previous day or since last initial calibration	Daily
	Check carrier gas flow rate in column	Daily via use of known compound retention
	Check temp. of detector, inlet, column oven	Daily
	Septum replacement	As required
	Check system for gas leaks with SNOOP	W/cylinder change as required
		Monthly
	Check for loose/frayed wires and insulation	As Required
	½"Bake injector/column	As Required
	Change/remove sections of guard column	As Required
	Replace connectors/liners	As Required
	Change/replace column(s)	
Electron Capture Detector (ECD)	Detector wipe test (Ni-63)	Semi-annually
	Detector cleaning	As required
Flame Ionization Detector (FID)	Detector cleaning	As required
Flame Photoionization Detector (FPD)	Clean and/or Replace Lamp	As required
Photoionization Detector (PID)	Change O-rings	As required
	Clean lamp window	As required
HPLC	Change guard columns	As required
	Change lamps	As required
	Change pump seals	Semi-annually or as required
	Replace tubing	As required
	Change fuses in power supply	As required
	Filter all samples	Daily
	Change autosampler rotor/stator	As required

Table 9. Major Equipment Maintenance

Instrument	Procedure	Frequency
Balances	Class "S" traceable weight check Clean pan and check if level Field service	Daily, when used Daily At least Annually
Conductivity Meter	0.01M KCl calibration Conductivity cell cleaning	Daily As required
Turbidimeter	Check light bulb	Daily, when used
Deionized/Distilled Water	Conductivity Point Sources Daily conductivity check Check deionizer light Monitor for VOA's System cleaning Replace cartridge & large mixed bed resins	Water Quality SOP UQA-035 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, DoD, 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	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. 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.
	Continuing Calibration	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., ± 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.
Atomic Absorption (GFAA/ CVAA)	Initial Calibration	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 (GFAA) 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. An ICV standard will be analyzed immediately after standardization. The ICV must be within SOP-specified criteria (e.g., ±5% of the true value for drinking water, and ±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. 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.

Table 10. Minimum Instrument Calibration Procedures

Technique	Activity	Minimum Requirements
	Continuing Calibration	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., ±10% recovery of the true value except for mercury within ±20 % of the true value). The CCB must be free of target analytes at or above the concentration reported in samples. If any CCV or CCB exceed their acceptance criteria, corrective action must be taken.
Inorganic Colorimetric Methods	Initial Calibration	An 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 linear range of the method, plus a calibration blank. At least one of the calibration standards will be at a concentration which will enable verification of instrument response at 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. In lieu of an initial curve, a daily calibration verification check may be analyzed. This calibration check 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. 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. 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.
	Continuing Calibration	The initial calibration is verified after every 10 readings and at the end of the analytical shift, with the analysis of a continuing calibration verification standard (CCV) and a blank (CCB). If any CCV or CCB exceed SOP-specified acceptance criteria, appropriate corrective action is taken per SOP. All samples since the last valid calibration verification check are reanalyzed.
Ion Chromato-graphy	Initial Calibration	The ion chromatograph will be calibrated approximately monthly or when any significant change is made to the system. Calibration standards will be prepared from appropriate reference materials and will include a blank and a minimum of three concentrations to cover the linear range of the instrument. At least one of the calibration standards will be at a concentration which will enable verification of instrument response at 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. An ICV will be analyzed on a daily basis, prior to sample analysis and 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.

Table 10. Minimum Instrument Calibration Procedures

Technique	Activity	Minimum Requirements
	Continuing Calibration	The initial calibration is verified after every 10 readings and at the end of the analytical shift, with the analysis of a continuing calibration verification standard (CCV) and a blank (CCB). If any CCV or CCB exceed SOP-specified acceptance criteria, appropriate corrective action is taken per SOP. All samples since the last valid calibration verification check are reanalyzed.
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	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. 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 or BFB. For wastewater programs (600 series methods), the tune expires after 24 hours. Ion abundances will be within the windows dictated by the specific program requirements.
	Initial Calibration	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. 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. 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.
	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.

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	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. Within the analysis sequence, instrument drift will be monitored by analysis of a mid-range calibration standard of varying concentrations 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.

5.5 Measurement Traceability

5.5.1 General

Traceability of measurements is ensured 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/eppendorf 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).

The main DI/RO units are located in a separate area. These include both a DI and RO system. The DI/RO System is connected by modem to the company who maintains the system for STL Chicago (Crossbow Industrial Water). Additionally, there are several Milli-Q Units, described below, which draw directly from this system, in several of the laboratory areas. There are also DI water feeds in each laboratory. The following checks and maintenance are followed.

Daily

Main System

Daily Conductivity Check- check set point
This is recorded in a log kept near the system
Deionizer Light
General Inspection for leaks etc..
Work Instruction for Alarm Re-Setting Posted Near System

Crossbow conducts a thorough monthly check of both systems and provides a full report to STL Chicago. This includes flow and pressure checks, DI Low Alarm Check, RO low pressure switch, pumps and valves, Total Hardness and Cl2 level checks, Control Circuits and finally an entire system check for damage and corrosion. These reports are filed.

Milli-Q Units

Milli-Q Units are located in the GC/MS VOA, Metals, Wet Chem and Extraction Laboratories. These units also contain check point settings. These settings are checked daily, prior to use, by the appropriate laboratory personnel, prior to using the unit to provide water for method blanks or other uses. If the units are not operating at the appropriate set point level, the Facility Manager is called and the appropriate corrective action is taken (change filter on unit, check main DI/RO unit etc...). In this sense, all units are being checked on a daily basis for proper operation and samples of the water being analyzed.

Point Source Checks for Specific Conductivity and pH

The following water point sources are checked on a weekly basis, as they are the first outlet from the water source. This has proven to be an adequate representation of the water used by the lab:

TCLP Laboratory-DI
Metals Digestion- Milli-Q

The following are checked on a quarterly basis:

TCLP Laboratory-DI
Wet Chemistry: General Lab-DI
We Chemistry: Instrumentation Laboratory-DI
Organic Extractions: Dishroom-DI
Organic Extractions-Milli-Q
Metals Digestion-Milli-Q
Wet Chemistry-Milli-Q
GC/MS Volatiles Laboratory-Milli-Q

These procedures and documentation are described in STL Chicago Water Quality SOP UQA-035. The description, as above, will be added to the next revision of the Laboratory Quality Manual LQM.

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.

References 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 $\pm 2^{\circ}C$ (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 ensure 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 or Job Note 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 $4 \pm 2^{\circ}C$. 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. If sub-sampling is required at the login stage it is done with guidance and instruction from the project manager.

General sub-sampling procedure in the laboratory calls for a thorough mixing of the sample within the sample container or to transfer the sample to another suitable container from which a representative sub-sample can be taken to achieve the required sample weight. Any non-homogenous looking material is avoided and noted as such within the sample preparation record. Refer to individual preparation SOPs for additional details.

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 participates in 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	Organics: 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. Inorganics: 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.**
Laboratory Control Sample (LCS)	Use	Measures the accuracy of the method in a blank matrix and assesses method performance independent of potential field sample matrix affects.
	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.

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
		Inorganics
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 ensures that any potential contamination is less than the reporting limit.
ICP Interference Check Samples (ICSA/ICSB)	Sequence Use	Analyzed immediately after the ICV. 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 $\geq$ 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
		Organics
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 ≤ 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), Automated Data Review (ADR), Enviro Data, EQUIS, GISKEY, Excel, Access 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 electronic data 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 initialed and dated by the programmer and kept on file.

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.

6.0 ATTACHMENTS

Appendix A: List of Cited SOPs and Work Instructions
Appendix B: Methods Capabilities Work Instruction
Appendix C: Description and Floor Space for Analytical Facilities

Historical File:		Revision 00: 01/29/99		
		Revision 01: 12/15/00		
		Revision 02: 09/10/02		
		Revision 03: 06/07/04		
		Revision 04: 07/05/05		

Reason for Change: Revision 04:

- Update section 3.0 – Terms & Definitions: Clarification on DLCK and MDLCK and MRL definition
- Update section 4.1 – Organization Chart
- Update section 4.1.1 – Table 3. Major Equipment List
- Update section 4.1.2.4 – Technical Manager's experience level
- Update section 4.4.3.6 – Additonal DQOs: MDL-Reference to Corporate MDL SOP added; RL – Reference to Lab MDL SOP added
- Update section 4.6.1 – Solvent Lot Testing notification / posting location added
- Update section 4.12.2 – Table 4 and 5 – STL Record Types and Record Retention
- Update section 4.15.3 – Monthly QA Report
- Update section 4.7.1 – clarification on documentation practices for compromised samples
- Update section 5.1.2 – Training text updated to include non-technical training, defines what constitutes 'initial training/orientation' and defines who is considered to be a 'qualified analyst'. Basic re-organization of this section and addition of the Continued Demonstration of Method Proficiency form.
- Update Section 5.3 – Test Methods to include discussion and a link to the STL web-site regarding STL Chicago's Methods Capability Listing which was added as Appendix B
- Update/Clarify section 5.3.7 – Verification and Validation discussion; Addition of Instrument Validation Checklist.
- Update section 5.4.2 – Major Equipment maintenance updated to incorporate current electronic maintenance documentation practices also updated to include Conductivity Point Sources and Daily Conductivity Check – referencing the Lab's Water Quality SOP
- Update Table 9 to include ICP MS and DI/RO system text
- Update section 5.5.1 – Measurement Traceability: Discussion added regarding DI/RO systems, Milli-Q Units; Point Source Checks for Specific Conductivity and pH. Added reference to the lab's Water Quality SOP
- Update section 5.9.5 – EDD Discussion
- Addition of Section 6 – Attachments A, B and C
- General Text Clarifications

Appendix A. List of Cited SOPs and Work Instructions

Cited Sec. No(s)	Description	Document No.
1.5	Corporate Quality Management Plan (QMP)	QMP
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
4.4.3.6; 5.3.5	MDL Policy	S-Q-003
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 Report (SDR) / Resubmitted Data Request (RDR) / Corrective Action Report (CAR)	UQA-029
4.8; 4.11	Quality Systems Management Review	UQA-002
4.8	Customer Complaint Form	CHI-22-09-340
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	Initial Demonstration of Capability Certification Statement	CHI-22-09-271
5.1.2	Continued Demonstration of Method Performance	CHI-22-09-243
5.1.2	Training Program: Mechanisms and Documentation Processes Defined by Operational Assessment	UQA-014
5.1.3	Ethics Policy	P-L-006
5.3; 5.3.1	Work Instruction: Methods Capabilities	CHI-22-09-255
5.3.2	SOP Change Protocol	UQA-032
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
5.3.7	Work Instruction: Instrument Validation Checklist	CHI-22-09-286

Appendix A. List of Cited SOPs and Work Instructions

Cited Sec. No(s)	Description	Document No.
5.3.7	Software Testing, Verification & Validation	S-ITQ-007
5.3.7	Procedures & Processes Related to Entry, Storage, Back-up/Retrieval and Management of Bench Level Electronic Data	UIS-006
5.3.7; 5.9.5	EDD Specifications, Development, Generation & Review	UIS-001
5.4.1	Work Instruction: Equipment Tracking Form	CHI-22-09-068
5.4.2	Instrument Tracking Spreadsheet / Maintenance Log	CHI-22-09-341
5.4.2	Instrument and Equipment Out-of-Service Tagging.	UQA-012
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
Table 9; 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; 5.9.6	Data Management: Process Operation	UDM-001

Appendix B. STL Chicago's Methods Capability Listing

STL Chicago
Methods Capability Listing

SEVERN TRENT STL

Unit	Parameter	Method No.	NELAP	Matrix
GCE	Pesticides/PCBs	OLC02.1		W
GCE	Pesticides/PCBs	OLM04.2		W/S
GCE	Pesticides/PCBs	EPA 608	X	W
GCE	Organochlorine Pesticides	SW 8081A	X	W/S
GCE	PCBs	SW 8082	X	W/S
GCE	Organophosphorus Pesticides by GC	SW 8141A	X	W/S
GCE	Chlorinated Herbicides by GC	SW 8151A	X	W/S
GCV	Petroleum Hydrocarbons (DRO)	SW 8015B	X	W/S
GCV	Petroleum Hydrocarbons (GRO)	SW 8015B	X	W/S
HPLC	PAHs by HPLC	E 610	X	W
HPLC	PAHs by HPLC	SW 8310	X	W/S
HPLC	Explosives	SW 8330	X	W/S
M	GFAA Silver	SW 7761	X	W
M	GFAA Arsenic	SW 7060A	X	W/S
M	GFAA Cadmium	SW 7131A	X	W/S
M	GFAA Chromium	SW 7191	X	W/S
M	GFAA Lead	SW 7421	X	W/S
M	GFAA Antimony	SW 7041	X	W/S
M	GFAA Selenium	SW 7740	X	W/S
M	GFAA Thallium	SW 7841	X	W/S
M	GFAA Silver	E 272.2	X	W
M	GFAA Arsenic	E 206.2	X	W
M	GFAA Cadmium	E 213.2	X	W
M	GFAA Chromium	E 218.2	X	W
M	GFAA Lead	E 239.2	X	W
M	GFAA Antimony	E 204.2	X	W
M	GFAA Selenium	E 270.2	X	W
M	GFAA Thallium	E 279.2	X	W
M	GFAA Metals As, Cd, Pb, Sb, Se, Tl, Cr, Ag	E 200.9	X	W
M	Hardness	E 200.7	X	W
M	ICP Metals	E 200.7	X	W

M	CVAA Mercury	SW 7470A	X	W
M	CVAA Mercury	SW 7471A	X	S
M	CVAA Mercury	E 245.1	X	W
M	ICP Metals	SW 6010B	X	W/S
M	Metals-GFAA (As, Pb, Se, Tl)	ILM04.0		W/S
M	Metals-ICP	ILM04.0		W/S
M	Metals-Mercury	ILM04.0		W/S
MSB	GC/MS Semi-Volatiles	E 625	X	W
MSB	GC/MS Semi-Volatiles	SW 8270C	X	W/S
MSB	GC/MS Semi-Volatiles	SW 8270C (SIM)		W
MSB	GC/MS Semi-Volatiles	OLM04.2		W/S
MSB	GC/MS Semi-Volatiles	OLC02.1		W
MSV	VOAs by GC/MS	E 624	X	W
MSV	GC/MS Volatiles	SW 8260B	X	W/S
MSV	GC/MS Volatiles	OLM04.2		W/S
MSV	GC/MS Volatiles	OLC02.1		W
P	GC/MS Soil VOAs in EnCore Samples	SW 5035		S
P	California W.E.T. Test	CA Title 22		S
P	TCLP	SW 1311	X	S
P	SPLP	SW 1312	X	S
P	Extractable Organics; Accel. Liq.-Liq. Waters	SW 3520C		W
P	Extractable Organics; Separatory Funnel	SW 3510C		W
P	Extractable Organics; Accel. Soxhlet	SW 3541A		S
P	Extractable Organics; Sonication	SW 3550B		S
P	Acid Cleanup	SW 3665A		W/S
P	Alumina Cleanup	SW 3610B		W/S
P	Florisil Clean-up	SW 3620B		W/S
P	Gel Permeation Column Clean-up	SW 3640B		S
P	Sulfur Clean-up	SW 3660B		W/S
P	Waste Dilution	SW 3580A		S
P	Metals Digestions; Surface/Ground Water for ICP	SW 3005A		W
P	Metals Digestions; Waters/Extracts for ICP	SW 3010A		W
P	Metals Digestion; Waters/Extracts for GFAA (except As & Se)	SW 3020A		W
P	Metals Digestion; Waters/Extracts for GFAA	SW 3020A (M)		W
P	Metals Digestions; Soils/Wastes for ICP/GFAA	SW 3050B		S

P	Metals Digestions; Waters for As by GFAA	SW 7060		W
P	Metals Digestions; Waters for Se by GFAA	SW 7740		W
W	Alkalinity	EPA 310.1	X	W/S
W	Alkalinity	SM 2320B	X	W/S
W	Ammonia - Nessl.	EPA 350.2	X	W/S
W	Ammonia - Nessl.	SM 4500NH3C		W/S
W	BOD - 5 Day	EPA 405.1	X	W
W	BOD - 5 Day	SM 5210B	X	W
W	Bromide, IC	EPA 300.0	X	W
W	Bromide, IC	SW-846 9056	X	W/S
W	Bromide, IC	SM 4110B		W
W	Carbonaceous BOD	SM 5210B	X	W
W	Chloride, Lachat	EPA 325.2	X	W/S
W	Chloride, Lachat	SM 4500ClE		W/S
W	Chloride, Lachat	SW-846 9251	X	W/S
W	Chloride, IC	EPA 300.0	X	W
W	Chloride, IC	SW-846 9056	X	W/S
W	Chloride, IC	SM 4110B		W
W	Chlorine, Residual	EPA 330.4	X	W
W	Chlorine, Residual	SM 4500 Cl F	X	W
W	COD - High Level	HACH 8000	X	W/S
W	COD - Low Level	HACH 8000	X	W/S
W	Chromium, Hexavalent	SM 3500-CrD	X	W/S
W	Chromium, Hexavalent	SW-846 3060A/7196A	X	W/S
W	Cyanide, Amenable	EPA 335.1	X	W/S
W	Cyanide, Amenable	SM 4500CN G		W/S
W	Cyanide	EPA 335.2	X	W/S
W	Cyanide	SW-846 9010B/9014	X	W/S
W	Cyanide	SM 4500CN C, E	X	W/S
W	Cyanide	ILM04.0		W/S
W	Ferrous Iron	SM 3500 Fe D		W/S
W	Flashpoint	SW-846 1010	X	W/S
W	Fluoride / Fluorine	EPA 340.2	X	W/S
W	Fluoride / Fluorine	SM 4500F C	X	W/S
W	Fluoride, IC	EPA 300.0	X	W
W	Fluoride, IC	SW-846 9056	X	W

W	Langlier Index	SM 2330A+B	X	W/S
W	Nitrate-NO2 (LACHAT)	EPA 353.2	X	W/S
W	Nitrate-NO2 (LACHAT)	SM 4500NO3F	X	W/S
W	Nitrate, IC	EPA 300.0	X	W
W	Nitrate, IC	SW-846 9056	X	W
W	Nitrate, IC	SM 4110B		W
W	Nitrite	EPA 354.1	X	W/S
W	Nitrite	SM 4500NO2B	X	W/S
W	Nitrite, IC	EPA 300.0	X	W
W	Nitrite, IC	SW-846 9056	X	W
W	Nitrite, IC	SM 4110B		W
W	Oil & Grease	E 1664	X	W
W	Oil & Grease (Soil-Soxhlet)	SW-846 9071B	X	W/S
W	Oxygen, Dissolved	EPA 360.1	X	W
W	Oxygen, Dissolved	SM 4500 O C, G		W
W	pH - Low/High	EPA 150.1	X	W
W	pH - Low/High	SM 4500H+B	X	W
W	pH - Low/High	SW-846 9045C / 9040B	X	W/S
W	Paint Filter	SW-846 9095	X	W
W	Phenol (LACHAT)	EPA 420.2	X	W/S
W	Phenol (LACHAT)	SW-846 9066	X	W/S
W	Phosphate, Ortho	EPA 365.2	X	W/S
W	Phosphate, Ortho	SM 4500 PE	X	W/S
W	Phosphate, Ortho , IC	EPA 300.0		W
W	Phosphate, Ortho , IC	SW-846 9056	X	W
W	Phosphate, Ortho , IC	SM 4110B		W
W	Phosphorus	EPA 365.2	X	W/S
W	Phosphorus	SM 4500 PE		W/S
W	Specific Conductance	EPA 120.1	X	W
W	Specific Conductance	SM 2510B	X	W
W	Specific Conductance	SW-846 9050A	X	W/S
W	Specific Gravity	ASTM D2710F		W/S
W	Sulfate / Sulfur - Turbidimetric	EPA 375.4M	X	W/S
W	Sulfate - Turbidimetric	SM 4500SO4E		W/S
W	Sulfate - Turbidimetric	SW-846 9038M	X	W/S
W	Sulfate, IC	EPA 300.0	X	W

W	Sulfate, IC	SW-846 9056	X	W
W	Sulfate, IC	SM 4110B		W
W	Sulfide	EPA 376.1	X	W/S
W	Sulfide	SM 4500SE		W/S
W	Sulfide	SW-846 9030B/9034	X	W/S
W	Sulfide, Reactive	SW 7.3.4.2	X	W/S
W	TDS (Total Dissolved Solids)	EPA 160.1	X	W
W	TDS (Total Dissolved Solids)	SM 2540C	X	W
W	TKN - Nesslerization	EPA 351.3	X	W/S
W	TKN - Nesslerization	SM 4500NorgC		W/S
W	TOC (TIC/DIC) [Organic Carbon; Inorganic & Dissolved]	EPA 415.1	X	W
W	TOC (TIC/DIC) [Organic Carbon; Inorganic & Dissolved]	SM 5310C	X	W
W	TOC (TIC/DIC) [Organic Carbon; Inorganic & Dissolved]	SW-846 9060	X	W
W	TOC (TIC/DIC) [Organic Carbon; Inorganic & Dissolved]	Lloyd Kahn		S
W	TOX (Total Organic Halogens)	SM 5320B		W
W	TOX (Total Organic Halogens)	SW-846 9020B	X	W/S
W	TS - Water (Total Solids)	EPA 160.3	X	W
W	TS - Water (Total Solids)	SM 2540B		W
W	TSS (Total Suspended Solids)	EPA 160.2	X	W
W	TSS (Total Suspended Solids)	SM 2540D		W
W	TDS (Total Dissolved Solids)	EPA 160.1	X	W
W	TDS (Total Dissolved Solids)	SM 2540C	X	W
W	TVS (Total Volatile Solids)	160.4	X	W
W	TVDS (Total Volatile Dissolved Solids)	160.4	X	W
W	TVSS (Total Volatile Suspended Solids)	160.4	X	W

Matrix: W (Water) S (Soil/Solid) O (Other)
Note: NELAP accreditation may be matrix and program specific. Refer to STL Chicago's IL NELAP Certificate No.: 001027
available on the STL web-site.

Appendix C. Severn Trent Laboratories Chicago
Description and Floor Space for Analytical Facilities

Lab Areas	Description	Approx. Feet^2
Organic Extractions	The extraction area has the capacity for performing 36 continuous liquid-liquid extractions, 60 sonification extractions, and 50 separatory funnel extractions each day. The configuration of the extractors and the fume exhausts were designed to facilitate rapid, efficient sample preparation. Separate areas are used for sample cleanups. Contains separate refrigerated sample storage.	2240 (lab) 166 (coolers)
Organic Glassware Cleaning	Dedicated to eliminating cross contamination, this isolated area is equipped with sinks, ample counter space, and pass through shelves for storing clean glassware. Water in this area is supplied by the RO/DI system.	520
GC Extractables and HPLC	The GC Extractables and HPLC area has independent and segregated HVAC systems and a specially designed compressed gas generation and distribution system. One GC is dedicated to Pest/PCB screening. Shares refrigerated storage area with GC/MS BNA.	1080
GC/MS BNA	The GC/MS BNA area is specially designed with independent and segregated HVAC systems to minimize cross contamination. Shares refrigerated storage area with GC Extractables and HPLC.	1050
GC/MS VOA and GC Purge & Trap	The GC/MS VOA and GC Purge & Trap area is specially designed with independent and segregated HVAC systems to minimize cross contamination. One GC and one GC/MS are dedicated to screening. Contains separate refrigerated sample storage area.	GC/MS VOA - 1200 GC P&T - 700
Metals Prep	This isolated room is equipped with sinks, benches and hoods required for performing metals digestion. This area also houses the TCLP extraction apparatus, which can accommodate 52 samples at a time.	590
Inorganic Glassware Cleaning	Dedicated to eliminating cross contamination, this isolated area is equipped with sinks, ample counter space, and pass through shelves for storing clean glassware. Water in this area is supplied by the RO/DI system.	340
Metals - ICP and AA	The Metals Instrumentation area is specially designed with independent and segregated HVAC systems to minimize cross contamination.	2075
Mercury Lab	The Mercury preparation and analysis area has independent and segregated HVAC systems to minimize cross contamination. This area contains a CVAA instrument and a hood for ventilation and sample preparation.	260
Wet Chem Lab	The Wet Chem Lab is specially designed with independent and segregated HVAC systems to minimize cross contamination. Includes a draft free, temperature controlled weigh room. Cyanides, phenols, anions, solids, and other traditional "wet chemistry" analyses are performed here. All distillation procedures are conducted in ventilated hoods. Water in this area is supplied by the RO/DI system.	1500

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TITLE: SAMPLE PREPARATION

Toxicity Characteristic Leaching Procedure (TCLP)

Updated by:	Signature:	Date:
Paul F. Kolarczyk Senior Analyst, Metals Dept.	Paul F. Kolarczyk	07-28-05

Approved by:	Signature:	Date:
Jodi L. Gromala Section Manager, Metals Dept.	Jodi L. Gromala	7-28-05
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with STLs QA Standard Practice Records

1.0 SCOPE / APPLICATION

This Standard Operating Procedure (SOP) outlines the guidelines for the Toxicity Characteristic Leaching Procedure (TCLP). This SOP was written using 40 CFR 261 (Appendix II) and SW-846, 3rd Edition, Method 1311 as 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

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

TCLP is designed to determine the mobility of both organic and inorganic contaminants present in liquid, solids and multi-phasic wastes.

Two distinct methods are utilized depending on whether volatile organics or other organic and metal constituents will be analyzed. A special zero-headspace extractor (ZHE) is used for volatile sample preparation and 2.0-Liter HDPE plastic or Teflon bottles are used for the other constituents.

- For solid wastes or wastes that contain significant amounts of solid material, the particle size is reduced and the liquid phase (if any) is separated from the solid phase and stored for later analysis. The solid phase is extracted with an amount of extraction fluid that is equal to 20 times the weight of the solid material.

- A portion of the extract for metals analysis only is spiked by the TCLP analyst with the analytes of concern (at the regulatory level) and acidified with nitric acid to a pH < 2 (refer to Attachment 1).

The TCLP sample is then analyzed by the appropriate method for organic and inorganic constituents. Refer to Figure 1 for the TCLP Flowchart; Table 1 for a listing of the Toxicity Characteristic Constituents and Regulatory Levels; and Table 2 for the maximum sample holding times.

2.0 INTERFERENCES

- Since this is a preparation procedure, interferences will only become apparent at the spiking and analysis stage. Interferences for spiking and for instrumentation are discussed in the analytical SOPs.
- A physical interference may occur for pH readings if the waste material is high in organic material (such as an oil). The waste may coat the pH probe, which affects the ability to obtain an accurate reading. When this type of interference occurs, pH paper is used instead of a meter for the final pH measurement. The use of pH paper is documented in the laboratory logbook.

3.0 SAFETY

- Employees will adhere to the practices and policies in the STL Corporate Safety Manual (CSM) and will read the MSDSs for the materials used in this method before handling or using the material.

3.1 Specific Safety Concerns or Requirements

- The standards contain potentially harmful elements. Care should be taken to avoid contact with the stock solutions. In case of contact, rinse with cold water for 15 minutes.
- If contact occurs with a standard containing Hydrofluoric Acid, flush with water and apply Calcium Gluconate Gel (located in standards cabinet) immediately. Seek medical attention.

4.0 EQUIPMENT AND SUPPLIES

- The extractor is a custom made rotary-type design that meets the specifications of tumbling the samples at a rate of 30±2 RPMs, which is checked monthly and documented in the extraction logbook.
- 2-Liter plastic bottles (HDPE for inorganics).
- 2-liter Teflon bottles [For organics (BNA, Herb/Pest)].
- pH meter and paper - pH meter accurate to ±0.05 pH units at 25°C. Refer to the pH SOP (UWC-150.1) for details on meter calibration.
- Filtering apparatus - pressure filter using compressed Nitrogen as the purge gas.
- Zero Headspace extraction vessel (ZHE) - purchased unit for volatiles.
- 9.5 mm Sieve.
- Filter paper - glass fiber, 0.7 um pore size.

NOTE: Filters shall be made of borosilicate glass fiber. When evaluating the mobility of metals, filters shall be acid-washed prior to use by rinsing with 1 N nitric acid followed by 3 consecutive rinses with deionized distilled water (a minimum of 1 L per rinse is recommended).

- Lab balance capable of reading ± 0.01 g
- *Tedlar Bags *Registered Trademark
- ZHE Extraction Fluid Transfer Device - any device capable of transferring the extraction fluid to the ZHE without changing the nature of the extraction fluid is acceptable (e.g., a positive displacement or a peristaltic pump, a gas tight syringe, pressure filtration unit).

5.0 REAGENTS AND STANDARDS

5.1 Hydrochloric Acid (HCl), 1.0 N

To a 1-L Class A volumetric flask containing ~500 mLs of Milli-Q water, carefully add 83 mLs of concentrated hydrochloric acid. Swirl the flask to mix. Dilute to volume with Milli-Q water.

- Life of Reagent: 1 year
- Storage Requirements: None

5.2 Nitric Acid (HNO_3), 1.0 N

To a 1-L Class A volumetric flask containing ~500 mLs of Milli-Q water, carefully add 64 mLs of concentrated nitric acid. Swirl the flask to mix. Dilute to volume with Milli-Q water.

- Life of Reagent: 1 year
- Storage Requirements: None

5.3 Sodium Hydroxide (NaOH), 1.0 N

To a 1-L Class A volumetric flask containing ~500 mLs of Milli-Q water, add 40.0 g of sodium hydroxide pellets. Swirl the flask to mix. This is an **EXOTHERMIC** reaction. The flask should be placed in a cool water bath when mixing. Dilute to volume with Milli-Q water.

- Life of Reagent: 1 year
- Storage Requirements: None

5.4 Glacial Acetic Acid, Reagent Grade

Purchased.

- Life of Reagent: 1 year
- Storage Requirements: None

5.5 Extraction Fluid #1

To a 1-L Class A volumetric flask containing ~500 mLs of Milli-Q water, carefully add 5.7 mLs of glacial acetic acid. Swirl the flask to mix. Then add 64.3 mLs of 1.0 N sodium hydroxide solution (Rgt. 5.3) and swirl to mix once again. Dilute to volume with Milli-Q water. The pH of this extraction fluid should be 4.93 ± 0.05.

- Life of Reagent: 1 day
- Storage Requirements: None

5.6 Extraction Fluid #2

To a 1-L Class A volumetric flask containing ~500 mLs of Milli-Q water, carefully add 5.7 mL of glacial acetic acid. Swirl the flask to mix. Dilute to volume with Milli-Q water. The pH of this Extraction Fluid should be 2.88 ± 0.05.

- Life of Reagent: 1 day
- Storage Requirements: None

6.0 CALIBRATION (NON-DAILY)

Not Applicable.

7.0 PROCEDURE

7.1 Quality Control Checks

Refer to Section 8.1.

7.2 Sample Preservation and Storage

Parameter	From: Field Collection To: TCLP Extraction	From: TCLP Extraction To: Preparative Extraction	From: Preparative Extraction To: Determinative Analysis	Total Elapsed Time
Volatiles	14 days	NA	14 days	28 days
Semi-Volatiles [1]	14 days	7 days	40 days	61 days
Mercury	28 days	NA	28 days	56 days
Metals (except Hg)	180 days	NA	180 days	360 days

[1] BNAs, Pesticides and Herbicides
NA = Not Applicable

7.3 Sample Preparation / Size

7.3.1 Inorganics & Semi-Volatiles (BNAs, Pesticides and Herbicides)

Type of Sample	Sample Size
Samples containing 100% solids	100g solid
Samples containing 0.5% - 99.9% solids	100 g solid ideally, 75.0 g solid minimum
Samples containing < 0.5% solids	Refer to Section 7.6.1.14

7.3.2 Volatiles (ZHE)

Type of Sample	Sample Size
Samples containing 100% solids	25 g solid
Samples containing 0.5% - 99.9% solids	25 g solid
Samples containing <0.5% solids	Refer to Section 7.6.2.7

7.4 Calibration / Standardization

Refer to SOP No. UWC-150.1 for instructions on calibrating the pH meter.

7.5 Preventive Maintenance

- The main preventive maintenance required is keeping the area and all equipment clean and free of contaminants.
- The pH probe should be checked periodically for bubbles. The probes are replaced when needed.
- The ZHE's shall be checked for leaks after every use.

7.6 Sample Extraction

7.6.1 Procedure when Volatiles are Not Involved

Although a minimum sample size of 100 grams is required, a larger sample size may be necessary, depending on the percent solids of the waste sample. Enough waste sample should be collected such that at least 75 grams of the solid phase of the waste (as determined using glass fiber filter filtration) is extracted. This will ensure that there is adequate extract for the required analyses (semivolatiles, metals, pesticides and herbicides).

The determination of which extraction fluid to use (Sec. 7.6.1.12) may also be conducted at the start of this procedure. This determination shall be on the solid phase of the waste (as obtained using glass fiber filter filtration).

7.6.1.1 If the waste will obviously yield no free liquid when subjected to pressure filtration, weigh out a representative 100.0 g portion of the sample and proceed to 7.6.1.11.

7.6.1.2 If the sample is liquid or multi-phasic, liquid/solid separation is required. This involves the filtration device outlined in Secs. 7.6.1.3 through 7.6.1.9.

7.6.1.3 Pre-weigh the filter and the container which will receive the filtrate.

7.6.1.4 Assemble the filter holder and filter.

7.6.1.5 Weigh out a representative 100 g sub-sample of the waste and record the weight.

7.6.1.6 Allow slurries to stand to permit the solid phase to settle. Wastes that settle slowly may be centrifuged prior to filtration.

7.6.1.7 Transfer the waste sample to the filter holder.

NOTE: If waste material has obviously adhered to the container used to transfer the sample to the filtration apparatus, determine the weight of this residue and subtract it from the sample weight determined in Sec. 7.6.1.5 to determine the weight of the waste sample which will be filtered.

Gradually apply pressure of 10 psi, until gas moves through the filter. If this point is not reached under 10 psi, and if no additional liquid has passed through the filter in any two minute interval, slowly increase the pressure in 10 psi increments to a maximum of 50 psi. After each incremental increase of 10 psi, if the pressurizing gas has not moved through the filter in any two minute interval, proceed to the next 10 psi increment. When the

pressurizing gas begins to move through the filter, or when liquid flow has ceased at 50 psi, filtration is stopped.

7.6.1.8 The material in the filter holder is defined as the solid phase of the waste, and the filtrate is defined as the liquid phase.

NOTE: Some wastes, such as oily wastes and some paint wastes will obviously contain some material that appears to be a liquid - but even after applying pressure filtration this material may not filter. In this case, the material within the filtration device is defined as a solid and is carried through the extraction as a solid.

7.6.1.9 Determine the weight of the liquid phase by subtracting the total weight of the filtrate container (Sec. 7.6.1.3) from the total weight of the filtrate-filled container. The liquid phase may now be either analyzed (Sec. 7.6.1.15) or stored at 4 $\pm$ 2°C until it is checked for compatibility with the rotated extract (Sec. 7.6.1.15).

The weight of the solid phase of the waste sample is determined by subtracting the weight of the liquid phase from the weight of the total waste sample, as determined in Sec. 7.6.1.5 or 7.6.1.7. Record the weight of the liquid and solid phases.

NOTE: If the weight of the solid phase of the waste is < 75 g. Review the beginning of Section 7.3 about sample sizes.

7.6.1.10 The sample will be handled differently from this point, depending on whether it contains more or less than 0.5% solids. If the sample obviously has >0.5% solids, go to Sec. 7.6.1.11. If it appears that the solid may comprise less than 0.5% of the total waste, the percent solids will be determined as follows:

- Remove the solid phase and filter from the filtration apparatus.
- Dry the filter and solid phase at 100 $\pm$ 20°C until two successive weighings yield the same value. Record the final weight.
- Calculate the percent solids as follows:

$$\frac{\text{(weight of waste \& filters) - (tared weight of filters)}}{\text{initial weight of waste}} \times 100 = \%\ \text{solids}$$

- If the solid phase comprises <0.5% of the waste, it is discarded and the liquid phase is defined as the TCLP extract. Proceed to Sec. 7.6.1.14.
- If the solid is $\geq$0.5% of the waste, return to Sec. 7.6.1.1 and begin the procedure with a new sample of waste. Do not extract the solid that has been dried.

7.6.1.11 If the sample has more than 0.5% solids, it is now evaluated for particle size. If the solid material is capable of passing through a 9.5 mm sieve, proceed to sec. 7.6.1.12. If the particle size is larger than 9.5 mm, the solid material is prepared for extraction by crushing until it is < 9.5 mm.

7.6.1.12 This step describes the determination of the appropriate extracting fluid to use.

- Weigh out a small sub-sample of the solid phase of the waste, reduce the solid (if necessary) to a particle size of approximately 1 mm in diameter or less, and transfer a 5.0 g portion to a 250 mL beaker.
- Add 96.5 mL DI water, cover with watch glass, and stir vigorously for five minutes using a magnetic stirrer. Measure and record the pH. If the pH is $\leq$ 5.0, extraction fluid # 1 is used. Proceed to sec. 7.6.1.13.
- If the pH is >5.0, add 3.5 mL 1.0 N hydrochloric acid, stir for 30 seconds and heat to 50°C. Continue heating at 50°C for ten minutes.
- Let the solution cool to room temperature and record the pH. If pH is $\leq$ 5.0, use extraction fluid #1. If the pH is > 5.0, use extraction fluid #2.

7.6.1.13 Transfer the solid material into the extractor vessel, including the filter used to separate the initial liquid from the solid phase.

NOTE: If any of the solid phase remains adhered to the walls of the filter holder, or the container used to transfer the waste, its weight shall be determined, subtracted from the weight of the solid phase of the waste, as determined above, and this weight is used in calculating the amount of extraction fluid to add into the extractor bottle.

Slowly add an amount of the appropriate extraction fluid into the extractor bottle equal to 20 times the weight of the solid phase that has been placed into the extractor bottle. Close the extractor bottle tightly, and place in the rotary extractor and rotate for 18 $\pm$ 2 hours. The ambient room temperature shall be maintained at 23 $\pm$ 2°C during the extraction period.

7.6.1.14 Following the 18 hour extraction, the material in the extractor vessel is separated into its component liquid and solid phases by filtering through a new glass fiber filter as outlined in Sec. 7.6.1.7.

7.6.1.15 The TCLP extract is now prepared as follows:

- If the waste contained no initial liquid phase, the filtered liquid material obtained from Sec. 7.6.1.14 is defined as the TCLP extract. Proceed to Sec. 7.6.1.16.

- If compatible (e.g., will not form a precipitate or has multiple phases), the filtered liquid is combined with the initial liquid phase of the waste. This combined liquid is defined as the TCLP extract.

- If the initial liquid phase of the waste, as obtained from Sec. 7.6.1.9 is not compatible with the filtered liquid resulting from Sec. 7.6.1.14, the liquids are not combined. The liquids are collectively defined as the TCLP extract and are analyzed separately.

7.6.1.16 The TCLP extracts are transferred to metals digestion and/or organic extractions for further preparation according to the procedures for the particular analysis (organics or metals) before being analyzed. Internal Chain of Custody (ICOC) procedures will be initiated for those extracts which require it. Following the collection of the TCLP extract, the pH of the extract should be recorded. Immediately remove an aliquot and reserve for analysis (metals only). Metals must be acidified with Nitric Acid to pH <2. Refrigerate the aliquots at 4 ± 2°C.

7.6.2 Procedure for Volatiles by ZHE

The ZHE device has approximately a 500 mL internal capacity. Although a minimum sample size of 100 grams is required in Section 7.6.1, the ZHE can only accommodate a maximum 100% solids sample of 25 grams. This is due to the need to add an amount of extraction fluid equal to 20 times the weight of the solid phase. Sec. 7.6.2.4 provides the means by which to determine the approximate sample size for the ZHE device. Although the following procedure allows for particle size reduction during the procedure, this could result in the loss of volatile compounds. If possible, any particle size reduction (see Sec. 7.6.2.5) should be conducted on the sample as it is being taken. Particle size reduction should only be conducted during the procedure if there is no other choice.

In carrying out the following steps, do not allow the waste to be exposed to the atmosphere for any more time than is absolutely necessary.

7.6.2.1 Pre-weigh the (evacuated) container which will receive the filtrate, and set it aside.

7.6.2.2 Place the ZHE piston within the body of the ZHE (it may be helpful to first moisten the piston o-rings slightly with extraction fluid). Secure the gas inlet/outlet flange (bottom flange) onto the ZHE body in accordance with the manufacturer's instructions. Secure the glass fiber filter between the support screens and set it aside. Set liquid inlet/outlet flange (top flange) aside.

7.6.2.3 If the waste will obviously yield no free liquid when subjected to pressure filtration, weigh out a representative 25 g sample of the waste, record the weight, and proceed to Sec. 7.6.2.5.

7.6.2.4 This sec. provides the means by which to determine the approximate sample size for the ZHE device. If the waste is liquid or multi-phasic, follow the procedure outlined in Steps 7.6.1.2 to 7.6.1.9 (using the Section 7.6.1 filtration apparatus), and obtain the percent solids by dividing the weight of the solid phase of the waste by the original sample size used. If it appears that the solid may comprise <0.5% of the waste, see below.

- Determine the percent solids by using the procedure outlined in Sec. 7.6.1.10. If the waste contains <0.5% solids, proceed to Sec. 7.6.2.7 and follow until the liquid phase of the waste is filtered using the ZHE device (Sec. 7.6.2.8). This liquid filtrate is defined as the TCLP extract and is analyzed directly.

- If the sample is $\geq$ 0.5% solids, the maximum amount of sample the ZHE can accommodate is determined by dividing 25 grams by the percent solids obtained from Sec. 7.6.2.4. Weigh out a new representative sample of the determined size by the following calculation:

$$\text{weight of waste to change ZHE} = \frac{25}{\text{percent solids}} \times 100$$

7.6.2.5 After a representative sample of the waste has been weighed out and recorded, the sample is now evaluated for the particle size (see beginning of Procedure 7.6.2). If the solid material within the waste will obviously pass through a 9.5 mm sieve, proceed immediately to Sec. 7.6.2.6. If the particle size is larger than that described above, the solid material which does not meet the above criteria is separated from the liquid phase by sieving, and the solid is prepared for extraction by crushing until the particle size is < 9.5 mm.

NOTE: Wastes and appropriate equipment should be refrigerated, if possible, to 4±2°C prior to particle size reduction. If reduction of the solid phase of the waste is necessary, exposure of the waste to the atmosphere should be avoided to the furthest extent possible.

When particle size has been appropriately altered, the solid is re-combined with the rest of the waste.

7.6.2.6 Waste slurries should not be allowed to stand to permit the solid phase to settle. Wastes that settle slowly shall not be centrifuged prior to filtration. Again, this is to minimize the loss of volatile compounds to the atmosphere.

7.6.2.7 Transfer the entire sample (liquid and solid phases) quickly to the ZHE. If there is no solid/liquid separation, proceed to sec. 7.6.2.11.
Secure the filter and support screens into the top flange of the device and secure the top flange to the ZHE body in accordance with the manufacturer's instructions. Tighten all ZHE fittings and place the device in the vertical position (gas inlet/outlet flange on the bottom). Do not attach the extract collection device to the top plate.

NOTE: If waste material has obviously adhered to the container used to transfer the sample to the ZHE, determine the weight of this residue and subtract it from the sample weight determined in Sec. 7.6.2.4, to determine the weight of the waste sample which will be filtered.

Attach a gas line to the gas inlet/outlet valve (bottom flange), and with the liquid inlet/outlet valve (top flange) open, begin applying gentle pressure of 1-10 psi (more if necessary) to slowly force all headspace out of the ZHE device.

At the first appearance of liquid from the liquid inlet/outlet valve, quickly close the valve and discontinue pressure.

7.6.2.8 Attach the evacuated pre-weighed filtrate collection container to the liquid inlet/outlet valve and open valve. Begin applying gentle pressure of 1 - 10 psi to force the liquid phase into the filtrate collection container. If no additional liquid has passed through the filter in any two-minute interval, slowly increase the pressure in 10 psi increments to a maximum of 50 psi.

After each incremental increase of 10 psi, if no additional liquid has passed through the filter in any two-minute interval, proceed to the next 10 psi increment. When liquid flow has ceased, such that continued pressure filtration at 50 psi does not result in any additional filtrate within any two-minute period, filtration is stopped. Close the liquid inlet/outlet valve, discontinue pressure to the piston, and disconnect the filtrate collection container.

NOTE: Instantaneous application of high pressure can degrade the glass fiber filter and may cause premature plugging.

7.6.2.9 The material in the ZHE is defined as the solid phase of the waste, and the filtrate is defined as the liquid phase.

NOTE: Some wastes, such as oily wastes and some paint wastes, will obviously contain some material which appears to be a liquid - but even after applying pressure filtration this material will not filter. If this is the case, the material within the filtration device is defined as a solid, and is carried through the TCLP extraction as a solid.

If the original waste contained <0.5% solids (see Sec. 7.6.2.4) this filtrate is defined as the TCLP extract, and is analyzed directly - proceed to Sec. 7.6.2.13.

7.6.2.10 Determine the weight of the liquid phase by subtracting the weight of the filtrate container (see Sec. 7.6.2.1) from the total weight of the filtrate-filled container. The liquid phase may now be either analyzed or stored at 4 $\pm$ 2°C until time of analysis. The weight of the solid phase of the waste sample is determined by subtracting the weight of the liquid phase from the weight of the total waste sample (see Sec. 7.6.2.4). Record the final weight of the liquid and solid phases.

7.6.2.11 The following details how to add the appropriate amount of extraction fluid to the solid material within the ZHE and agitation of the ZHE vessel.

Extraction fluid #1 is used in all cases.

- With the ZHE in the vertical position, attach a connector for the extraction fluid syringe to the liquid inlet/outlet valve. Release gas pressure on the ZHE piston (from the gas inlet/outlet valve), open the liquid inlet/outlet valve, and begin transferring extraction fluid into the ZHE. Apply pressure to the plunger to add extraction fluid into the ZHE until the amount of fluid introduced into the device equals 20 times the weight of the solid phase of the waste that is in the ZHE.

- After the extraction fluid has been added, immediately close the liquid inlet/outlet valve and disconnect the syringe & connector. Check the ZHE to make sure that all valves are in their closed positions. Pick up the ZHE and physically rotate the device in an end-over-end fashion two or three times. Reposition the ZHE in the vertical position with the liquid inlet/outlet valve on top.

  Put 5-10 psi behind the piston and slowly open the liquid inlet/outlet valve to bleed out any headspace (into a hood) that may have been introduced due to the addition of extraction fluid. This is a check to show that the piston moves under 15 psi and that the o-rings are ok. This bleeding shall be done quickly and shall be stopped at the first appearance of liquid from the valve. Re-pressurize the ZHE with 5-10 psi and check all ZHE fittings to ensure that they are closed. Document the pressure in the TCLP logbook.

- Place the ZHE in the rotary extractor apparatus and rotate the ZHE for 18 $\pm$ 2 hours. The temperature of the room shall be maintained at 23 $\pm$ 2°C during agitation.

7.6.2.12 Following the 18 hour extraction, check the pressure behind the ZHE piston by looking at the gas pressure gauge. If the pressure has not been maintained (i.e., no gas release is observed) the device is leaking. Replace ZHE o-rings or other fittings, as necessary, and re-do the extraction with a new sample of waste. The original extract can not be used. If the pressure within the device has been maintained, the material in the extractor vessel is once again separated into its component liquid and solid phases. If the waste contained an initial liquid phase, the liquid may be filtered directly into the same filtrate collection container holding the initial liquid phase of the waste, unless doing so would create multiple phases, or unless there is not enough volume left within the filtrate collection container. A separate filtrate collection container must be used in these cases. Filter through the glass fiber filter, using the ZHE device as discussed in Sec. 7.6.2.8.

7.6.2.13 If the waste contained no initial liquid phase, the filtered liquid material obtained from Sec. 7.6.2.12 is defined as the TCLP extract. If the waste contained an initial liquid phase the filtered liquid material obtained from Sec. 7.6.2.12 and the initial liquid phase (Sec. 7.6.2.8) are collectively defined as the TCLP extract.

7.6.2.14 Extracts are then transferred to GC/MS Volatiles and stored in the GC/MS Volatiles cooler until analysis. Internal Chain of Custody (ICOC) procedures will be initiated for those extracts which require it.

7.7 Documentation

7.7.1 Analysis Logbook

The extraction of samples is documented within the extraction logbook and supported by the LabNet print-out. The logbook must be completed for each day's analysis (Attachment 2).

8.0 QUALITY CONTROL

NOTE: All quality control measures described in the appropriate analytical methods shall be followed.

8.1 QC Summary

8.1.1 A minimum of one blank (using the same extraction fluid as used for the samples) must be analyzed for every 20 extractions that have been conducted in an extraction vessel. The extraction fluid is to be made up daily and the pH determined and recorded within the acceptable limits.

8.1.2 A blank extraction fluid must be prepared for each type of fluid used per batch. If both extraction fluids are used, two blanks must be analyzed. The blank for the volatile analysis is the ZHE vessel filled with the extraction fluid and run through the procedure.

8.1.3 A matrix spike shall be performed for each waste type (e.g. wastewater treatment sludge, contaminated soil, etc..) unless the result exceeds the regulatory level and the data is being used solely to demonstrate that the waste property exceeds the regulatory level. A minimum of one matrix spike must be analyzed for each analytical batch. At a minimum, follow the matrix spike addition guidance provided in each analytical method.

8.1.4 Matrix spikes are to be added after filtration of the TCLP extract and before preservation. Matrix spikes should not be added prior to TCLP extraction of the sample.

8.1.5 In most cases, matrix spikes should be added at a concentration equivalent to the corresponding regulatory level. If the analyte concentration is less than one half the regulatory level, the spike concentration may be as low as one half of the analyte concentration, but may not be less than 5x the method detection limit. In order to avoid differences in matrix effects, the matrix spikes must be added to the same nominal volume of TCLP extract as that which was analyzed for the unspiked sample.

8.1.6 The purpose of the matrix spike is to monitor the performance of the analytical methods used, and to determine whether matrix interferences exist. Use of other internal calibration methods, modification of the analytical methods, or use of alternate analytical methods may be needed to accurately measure the analyte concentration of the TCLP extract when the recovery of the matrix spike is below the expected analytical method performance.

8.2 Corrective Action

Since this is a preparation step, problems will not be known until the filtrates are analyzed. Corrective action for poor blank results will require all samples in the set to be re-prepared. Refer to the analytical SOPs for corrective actions.

9.0 Data Analysis and Calculations

Since this is a preparatory procedure, refer to the analytical SOPs for matrix and method QC calculations.

9.1 Multiphasic Wastes with Non-compatible Liquid Phases

Determine the volume of the individual phases, analyze as appropriate, and combine the results mathematically by using a volume weighted average:

$$\text{Final Analyte Conc.} = \frac{(V_1)(C_1) + (V_2)(C_2)}{V_1 + V_2}$$

Where:
V_1 = Volume in first phase (L)
V_2 = Volume in second phase (L)
C_1 = Conc. in first phase (mg/L)
C_2 = Conc. in second phase (mg/L)

10.0 Waste Management and Pollution Control

Waste from this procedure will enter the "Waste Water" wastestream.

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.

10.1 Waste Streams Produced by the Method

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

- Aqueous waste from the extraction step will be turned into the waste technician after the analysis has been completed on digestate. The concentration of, if present, heavy metals will dictate the disposal procedure.
- Aqueous waste that has heavy metal levels below regulatory levels will be turned into the waste technician for disposal in the "Waste Water" wastestream.
- Aqueous waste that has heavy metal levels above regulatory limits should be marked appropriately and turned into the waste technician for disposal into the "Heavy Metal Corrosive Waste Water" wastestream.

11.0 METHOD PERFORMANCE CRITERIA

Refer to section 1, 6, 7 and 8.

12.0 REFERENCES

Refer to Section 1.0

13.0 ATTACHMENTS

Figure 1: TCLP Flowchart
Table 1: TCLP Constituents and Regulatory Levels
Attachment 1: TCLP Metals Spiking
Attachment 2: TCLP Extraction Log

Historical File:

Revision 00: 03/21/91
Revision 01: 06/19/92
Revision 02: 08/17/93
Revision 03: 11/03/94
Revision 04: 10/22/96
Revision 05: 03/30/99
Revision 06: 05/05/00
Revision 07: 05/25/01
Revision 08: 06/02/03
Revision 09: 06/25/04
Revision 10: 07/27/05

Reasons for Change, Revision 10:

- Annual Review – Updated Sections 7.6.1.16 and 7.6.2.13 to include where the TCLP extracts were to be located after extraction and to include ICOC language.

U:\QC\SOP\SP\USP-1311.DOC

Figure 1.

TCLP Flowchart

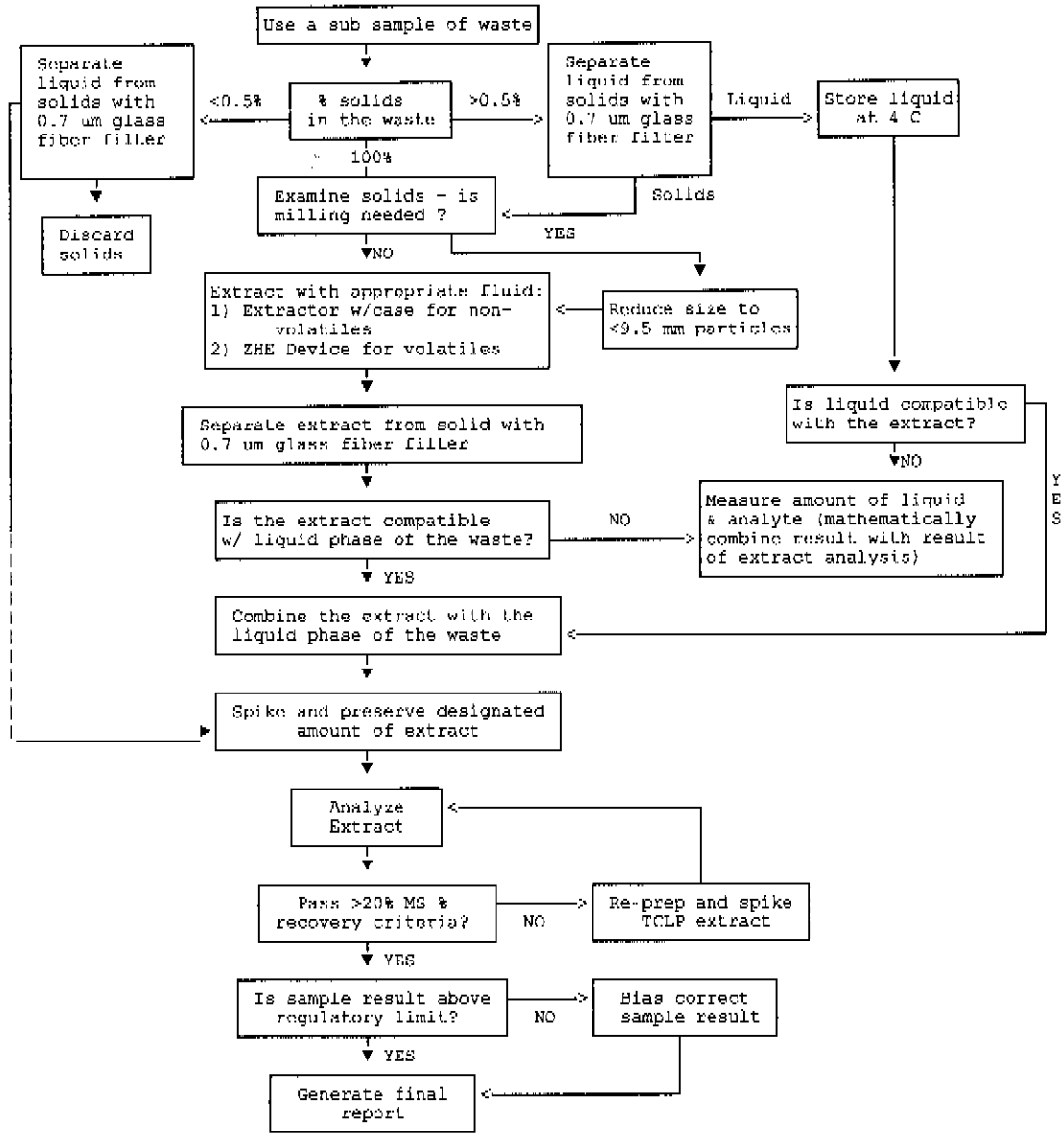

Table 1.

TCLP Constituents and Regulatory Levels

EPA HW Number	Constituent	CAS No.	Regulatory Level (ug/L)
D004	Arsenic	7740-38-2	5,000
D005	Barium	7440-39-3	100,000
D018	Benzene	71-43-2	500
D006	Cadmium	7440-43-9	1,000
D019	Carbon Tetrachloride	56-23-5	500
D020	Chlordane	57-74-9	30
D021	Chlorobenzene	108-90-7	100,000
D022	Chloroform	67-66-3	6,000
D007	Chromium	7440-47-3	5,000
D023	o-Cresol	95-48-7	[*1]200,000
D024	m-Cresol	108-39-4	[*1]200,000
D025	p-Cresol	108-44-5	[*1]200,000
D026	Cresol		[*1]200,000
D016	2,4-D	94-75-7	10,000
D027	1,4-Dichlorobenzene	106-46-7	7,500
D028	1,2-Dichloroethane	107-06-2	500
D029	1,1-Dichloroethylene	75-35-4	700
D030	2,4-Dinitrotoluene	121-14-2	130
D012	Endrin	72-20-8	20
D013	Heptachlor (& its epoxides)	76-44-8	8
D032	Hexachlorobenzene	118-74-1	130
D033	Hexachloro-1,3-butadiene	87-68-3	500
D034	Hexachloroethane	67-72-1	3,000
D008	Lead	7439-92-1	5,000
D013	Lindane	58-89-9	400
D004	Mercury	7439-97-6	200
D014	Methoxychlor	72-43-5	10,000
D035	Methyl Ethyl Ketone (2-Butanone)	78-93-3	200,000
D036	Nitrobenzene	98-95-3	2,000
D037	Pentachlorophenol	87-86-5	100,000
D038	Pyridine	110-86-1	5,000
D010	Selenium	7782-49-2	1,000
D011	Silver	7740-22-4	5,000
D039	Tetrachloroethylene	127-18-4	700
D015	Toxaphene	9001-35-2	500
D040	Trichloroethylene	79-01-6	500
D041	2,4,5-Trichlorophenol	95-95-4	400,000
D042	2,4,6-Trichlorophenol	88-06-2	2,000
D017	2,4,5-TP (Silvex)	93-72-1	1,000
D043	Vinyl Chloride	75-01-4	200

[1] If o-, m-, p-cresol concentration cannot be differentiated, the total cresol (D026) concentration is used. The regulatory level for total cresol is 200, 000 ug/L.

Attachment 1.

TCLP Metals Spiking

The purpose of the matrix spike is to monitor the performance of the analytical methods used and to determine whether matrix interferences exist.

Matrix spikes are to be added after filtration of the TCLP extract and before preservation. Matrix spikes should not be added prior to the TCLP extraction of the sample.

In order to avoid differences in matrix effects, the matrix spikes must be added to the same nominal volume of the TCLP extract as that which was analyzed for the unspiked sample.

The following steps detail the TCLP metals spiking procedure:

- Measure out 100 mLs of TCLP extract and transfer it into a small container.
- Using an eppendorf pipet, dispense 1 mL of each standard, STL-TCLP-1A and TCLP-2 (a.k.a., CGBA 10-A), into the TCLP extract.
- Preserve the TCLP spiked extract with 2 mLs of concentrated nitric acid.
- Store at 4 ± 2°C.

NOTE:

TCLP Stock Spike Solution Concentration:
Ba = 1000 ppm; As, Cr, Pb = 500 ppm; Cd, Se, Ag = 100 ppm.

Element Concentrations in Spiked Samples:
Ba = 100 ppm; As, Cr, Pb = 5 ppm; Cd, Se, Ag = 1 ppm

Attachment 2.

Example: TCLP Extraction Logbook

STL Chicago
TCLP Extraction Logbook

Page Number:_____

Rotator RPM Checked:_____ (Limits: 30 ± 2 RPMs)
Group Number:_____
LabNet Batch No.:_____
Sample Size Specifications:____<9.5 mm_____

Extraction Start Date / Time: _____
Extraction Start Temperature:_____ °C
Extraction End Date / Time: _____
Extraction End Temperature: _____ °C

Filtration Start Time:_____
Filtration End Time:_____
Thermometer ID: _____
Min./Max.Temp: _____/_____ °C
Control Limits: 23 ± 2 °C

Sample Number					
Sample Description					
Sample Weight (g)					
Liquid-Solid Separation (Yes/No)					
Volume of Mother Liquid (mLs)					
Solid Extraction Material (g)					
Extraction Fluid Selection pH of Initial Solution: If <5.0, use Extraction Fluid #1					
pH of Acid/Heat Treated Solution: If <5.0, use Extraction Fluid #1 If >5.0, use Extraction Fluid #2					
Extraction Fluid Type (1 or 2)					
Extraction Vessel Type / Pressure Check					
Extraction Fluid Volume (mLs)					
Extract Filtered (Yes or No)					
Mother Liquid Added (mLs)					
Combined Filtrate Volume (mLs)					
Final pH Reading					
Spike Source ID # / Volume Added (mLs)					
Filtrate Preserved					
ZHE: Initial psi / Final psi					

Comments:_____

Extraction Vessel Codes: T = Teflon Organics/Metals; ZHE = Zero Headspace VOA's; HDPE = High Density Polyethylene Metals

Analyst: _____ Date:_____

Reviewer:_____ Date:_____

STL Chicago
STANDARD OPERATING PRACTICE (SOP) CHANGE FORM

Original SOP Number/Revision #: USP-3000 Rev #13
SOP Title: Sample Preparation (Metals Digestion by SW-846 3000 Series)
Affected SOP Section Number(s): 8.2 Corrective Action
Effective Date: 6-1-05

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Full Signature Approvals Are Kept on File with Severn Trent Laboratories Standard Practice Records

Revision Number with Mod ID:

The following SOP change is in effect as of the stated date. This form will remain attached to the referenced SOP until such a time that the SOP is updated, approved, and redistributed, at which time it will become part of the historical SOP record. **Append this form to the front of the SOP copy.**

1. Reason for SOP Change: Corporate Internal Audit Response, Jan 24-25, 2005

2. Summary of Procedure Change (circle to indicate if there are attachments to this form: No / Yes: # pages attached = _)

Delete current text and replace with the following:

- A Corrective Action Report (CAR) will be filled out by the prep analyst any time there is a deviation from the routine prep procedures. This includes but is not limited to: limited sample volume, sample spills, extremely vigorous reactions, spiking issues or any other issues that may occur during the preparation procedure. The Section Manager will review CAR and document any corrective actions needed.
- All analysis "out-of-control" situations are identified in the Analytical Soft.

Initiated/Reviewed By: Name/Date — 6/6/05

Initiated/Reviewed By: Name/Date

Approval Signature/Date: Section Manager

Approval Signature/Date: QA Manager or Designee — 06/07/05

STL Chicago
STANDARD OPERATING PRACTICE (SOP) CHANGE FORM

Original SOP Number/Revision #: USP-3000 | Rev No. 13 Last Mod ID (circle): NA / (B)
SOP Title: Sample Preparation : Metals Digestion by SW-846 3000 Series
Affected SOP Section Number(s): 7.1 Quality Control Checks
Effective Date: 09/09/05

CONTROLLED DISTRIBUTION

COPY # :
ISSUED TO : **Uncontrolled**

Full Signature Approvals Are Kept on File with Severn Trent Laboratories Standard Practice Records

Revision Number with Mod ID:

The following SOP change is in effect as of the stated date. This form will remain attached to the referenced SOP until such a time that the SOP is updated, approved, and redistributed, at which time it will become part of the historical SOP record. **Append this form to the front of the SOP copy.**

1. Reason for SOP Change: NFESC Audit for DOD Compliance - Corrective Action Response

2. Summary of Procedure Change (circle to indicate if there are attachments to this form: No / (Yes) # pages attached = 1)

Table 7.1 will be amended to reflect the addition Teflon stands for both the Method Blank and laboratory Control Sample Soil Preparation sections. This amendment will be specific to those programs/clients/projects mandating the use of a 'soil' matrix for the preparation of soil MB and LCS. This includes all DOD/NFESC projects. The change will be incorporated in the SOP proper upon the next revision. This form serves to document changes to the SOP until that time as stipulated above.

Maureen Kenny 08/30/05
Initiated/Reviewed By: Name/Date

Initiated/Reviewed By: Name/Date

Approval Signature/Date: Section Manager

Approval Signature/Date: QA Manager or Designee

6.0 CALIBRATION (NON-DAILY)

Not Applicable.

7.0 PROCEDURE

7.1 Quality Control Checks

See SOP Change Form

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.

SOP No.	Revision No.	Date	Page
USP-3000	13	02/18/05	1 of 15

TITLE: SAMPLE PREPARATION
Metals Digestion by SW-846 3000 Series

Updated by:	Signature:	Date:
Carla Bonner Analyst, Metals Department	Carla Bonner	2-15-05

Approved by:	Signature:	Date:
Jodi L. Gromala Supervisor, Metals Dept.	Jodi L. Gromala	2-15-05
David W. Mazur Env. Health & Safety Coor.	D. W. Mazur	2/16/05
Terese A. Preston Quality Manager	Terese A. Preston	2/16/05

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with STLs QA Standard Practice Records

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 Couples 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_2O_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.

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 (H_2O_2) is a strong oxidizer and is corrosive. The digestion must be cooled sufficiently before the addition of H_2O_2 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.

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

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	• 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. Life of Standard: Expiration date of the earliest expiring standard. Storage Requirements: None.
GFAA Spike Solution	• 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. Life of Standard: Expiration date of the earliest expiring standard. Storage Requirements: None.
GFAA Ag Spike Solution	• 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. Life of Standard: As defined by the manufacturer. Storage Requirements: None.

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.

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.

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

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.

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.

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 in 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.

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.

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.

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

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Historical File:

Revision 00: 08/15/91
Revision 01: 03/16/93
Revision 02: 08/20/93
Revision 03: 01/20/94
Revision 04: 11/22/95
Revision 05: 02/18/97
Revision 06: 10/07/97
Revision 07: 10/16/97
Revision 08: 03/31/99
Revision 09: 05/05/00
Revision 10: 07/06/01
Revision 11: 01/09/03
Revision 12: 01/07/04
Revision 13: 02/11/05

Reasons for Change, Revision 13:

- Annual Review – No Changes

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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-60R	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

Appendix B.

Example: LabNet Digestion Spreadsheets

Acid Digestion (ICAP)
Method Code..: 3005
Batch Code...: 139935

Status.....: RVWD
Batch Date...: 01/21/05
Batch Time...: 1013

User Name.....: crb
QC Code.....:
Calc Code.....: PFACW

Location Code..: 57222
Equipment Code.:
Import Code....:

SAMPLE: Grp	Pos	Sample ID	Dilution	TEST CODE / TEST POS Date / Time	DIGTR 1
1	1	___MB_139935_		1/21/05 0920	0
1	2	___LCS_M04LSPK003_		1/21/05 0920	0
1	3	233622_1____		1/21/05 0920	0
1	4	233622_1_D___		1/21/05 0920	0
1	5	233622_2____		1/21/05 0920	0
1	6	233622_2_D___		1/21/05 0920	0
1	7	233622_3____		1/21/05 0920	0
1	8	233622_3_D___		1/21/05 0920	0
1	9	233622_4____		1/21/05 0920	0
1	10	233622_4__MD__9		1/21/05 0920	0
1	11	233622_4__MS_M04LSPK003_9		1/21/05 0920	0
1	12	233622_4__MSD_M04LSPK003_9		1/21/05 0920	0
1	13	233622_4_D___		1/21/05 0920	0
1	14	233622_4_D_MD__13		1/21/05 0920	0
1	15	233622_4_D_MS_M04LSPK003_13		1/21/05 0920	0
1	16	233622_4_D_MSD_M04LSPK003_13		1/21/05 0920	0
1	17	233622_5____		1/21/05 0920	0
1	18	233622_5_D___		1/21/05 0920	0
1	19	233622_6____		1/21/05 0920	0
1	20	233622_6_D___		1/21/05 0920	0
1	21	233622_7____		1/21/05 0920	0
1	22	233622_7_D___		1/21/05 0920	0
1	23	233622_8____		1/21/05 0920	0
1	24	233622_8_D___		1/21/05 0920	0
1	25	233622_9____		1/21/05 0920	0
1	26	233622_9_D___		1/21/05 0920	0
1	27	_____			

*** BATCH WORKSHEET *** (V2)

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	HNO3 Preservative Lot #	n/a
	9	HNO3 (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 Code...: 139935
Status.....: RVWD

Batch Date...: 01/21/05
Batch Time...: 1013
User Name....: crb

QC Code.....:
Calc Code.....: PFACW
Location Code..: 57222

Equipment Code.:
Import Code....:

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	____						

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	DIGICP 1
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	_____			

*** BATCH WORKSHEET *** (V2)

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	

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 Code...: 141789
Status.....: RVWD

Batch Date...: 02/11/05
Batch Time...: 1140
User Name....: crb

QC Code.....:
Calc Code.....: PFACW
Location Code..: 57222

Equipment Code.:
Import Code....:

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)
Method Code..: 3020M
Batch Code...: 141731

Status.....: RVWD
Batch Date...: 02/10/05
Batch Time...: 2055

User Name.....: rlc
QC Code.....:
Calc Code.....: PFACW

Location Code..: 57222
Equipment Code.:
Import Code....:

SAMPLE: Grp Pos	Sample ID	Dilution	TEST CODE / TEST POS / Date / Time	DIGFA / 1
1 1	___MB_141731_		2/10/05 1900	0
1 2	___LCS_M04LSPK002_		2/10/05 1900	0
1 3	234106_1___		2/10/05 1900	0
1 4	234106_2___		2/10/05 1900	0
1 5	234106_3___		2/10/05 1900	0
1 6	234106_4___		2/10/05 1900	0
1 7	234106_5___		2/10/05 1900	0
1 8	234106_6___		2/10/05 1900	0
1 9	234106_7___		2/10/05 1900	0
1 10	234106_8___		2/10/05 1900	0
1 11	234106_9___		2/10/05 1900	0
1 12	234106_10___		2/10/05 1900	0
1 13	234106_11___		2/10/05 1900	0
1 14	234106_12___		2/10/05 1900	0
1 15	234122_7___		2/10/05 1900	0
1 16	234122_8___		2/10/05 1900	0
1 17	234122_17___		2/10/05 1900	0
1 18	234141_1___		2/10/05 1900	0
1 19	234141_2___		2/10/05 1900	0
1 20	234141_2__MD__19		2/10/05 1900	0
1 21	234141_2__MS_M04LSPK002_19		2/10/05 1900	0
1 22	234141_2__MSD_M04LSPK002_19		2/10/05 1900	0
1 23	234153_3___		2/10/05 1900	0

*** BATCH WORKSHEET *** (V2)

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 H202 (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____							

Acid Digestion: Solids (ICAP)
Method Code..: 3050
Batch Code...: 141667

Status.....: RVWD
Batch Date...: 02/10/05
Batch Time...: 1142

User Name.....: crb
QC Code.....:
Calc Code.....: PFACS

Location Code..: 57222
Equipment Code.:
Import Code....:

SAMPLE: Grp	Pos	Sample ID	Dilution	TEST CODE / TEST POS Date / Time	DIGSOL / 1
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

*** BATCH WORKSHEET *** (V2)

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_MO5BSPK001_4		100				
	1	7	234126_2_S_MSD_MO5BSPK001_4		100				
	1	8	234126_4_S___		100				
	1	9	234126_6_S___		100				
	1	10	234126_8_S___		100				

STL Chicago
STANDARD OPERATING PRACTICE (SOP) CHANGE FORM

Original SOP Number/Revision #: USR-001 Rev. 17
SOP Title: Sample Receipt: Handling & Processing Procedures
Affected SOP Section Number(s): 5.10
Effective Date: 06-17-05

CONTROLLED DISTRIBUTION

COPY # :
ISSUED TO :

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Full Signature Approvals Are Kept on File with Severn Trent Laboratories Standard Practice Records

Revision Number with Mod ID:

The following SOP change is in effect as of the stated date. This form will remain attached to the referenced SOP until such a time that the SOP is updated, approved, and redistributed, at which time it will become part of the historical SOP record. **Append this form to the front of the SOP copy.**

1. Reason for SOP Change: External Audit Response for WDNR Audit April 19-22, 2005

2. Summary of Procedure Change (circle to indicate if there are attachments to this form: No / Yes: # pages attached = 0)

A Note will added below the table in Section 5.10, indicating that when samples to be reported to the WDNR are received that the temperature at the time of receipt will be verified to be <= 4.0 deg C. If the temperature is above the limit specified, a Directed Job Note will be sent to the appropriate Project Manager and the client contacted. The Directed Job Note will be included in the final report.

Maureen D. Kemp 06/17/05
Initiated/Reviewed By: Name/Date

Initiated/Reviewed By: Name/Date

Approval Signature/Date: Section Manager

Approval Signature/Date: QA Manager or Designee

CHI-22-09-039/D-1/99

SOP No.	Revision No.	Date	Page
USR-001	17	04/29/05	1 of 22

TITLE: SAMPLE RECEIPT
Handling and Processing Procedures

Updated by:	Signature:	Date:
Marilyn G. Krueding Quality Assurance Specialist	Marilyn G. Krueding	04/28/05
Jeffrey A. James Section Manager, Sample Receipt	Jeffrey A. James	4/28/05

Approved by:	Signature:	Date:
Jeffrey A. James Section Manager, Sample Receipt	Jeffrey A. James	4/28/05
Terese A. Preston Quality Manager	Terese A. Preston	4/28/05

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1.0 SCOPE / APPLICATION

This Standard Operating Procedure (SOP) describes the documentation and handling processes required for the receipt, tracking and communication of environmental samples at STL Chicago (STL).

2.0 TEMPERATURE MONITORING

2.1 Sample Storage

The temperatures of the refrigerated areas are maintained at 2-6°C; with freezers at <-10°C. All samples are maintained in refrigerated or freezer storage (where appropriate) prior to and after sample analysis. All sample storage temperatures are monitored via an Electronic Monitoring System 7-days a week. The QA department reviews and saves (.pdf format) the pictorial printout (Attachment 1) of all the monitored areas (standards and samples/extracts) twice daily (with at least four hours between).

Any out-of-control refrigerator storage temperatures are recorded within a Corrective Action Log (electronic) within the QA Department. Primary Corrective Action is recorded (thermostat adjustment or defrosting) will be recorded. The temperatures are rechecked for the out-of-control units later the same day. If the Primary Corrective Action does resolve the issue, the lead Sample Custodian (or appropriate personnel) and Facility Manager are contacted directly and/or via e-mail. This is also recorded in the CAR Log in the QA Department. Further Secondary Corrective Action (required maintenance by outside contractor) is pursued at that time. Resolutions of the problem and return to control are ultimately recorded in the CAR Log in the QA Department.

If an equipment failure (compressor failure, door left open, etc...) results in the storage refrigerator temperature exceeding the upper or lower control limits or the temperature cannot be stabilized, the samples will be moved to suitably controlled storage until the equipment failure is corrected or the refrigerator temperature is stabilized.

2.2 Sample Receipt

All samples that are not hand-delivered directly from the sample site, will have temperatures taken. Sample custodians will document sample receipt temperature readings on the designated client chain-of-custody (COC-Attachment 2) and within LabNet (LIMS) Sample Receipt Checklist (Attachment 7). These readings will be reported to the client in the final data report.

"Samples collected in the field must be transported to the laboratory as expeditiously as possible. When a 4°C requirement for preserving the sample is indicated, the samples must be packed on ice or chemical refrigerant to keep them cool during collection and transportation. It is acknowledged that during transit it is not always possible to rigorously control the temperature of the samples.

As a general rule, storage at low temperature is the best way to preserve most samples. It is impossible to set acceptance temperature limits for the cooler temperature because of the complexity of this issue." (Reference: AFCEE IRP Manual, Section 2.2.1).

When, in the judgment of the laboratory, the temperature of the samples upon receipt may have affected the stability of the analytes of interest, the problem will be discussed by the Project Manager (PM) with the client."

In situations where a definitive sample temperature criteria is required by a QAPP, contract, etc.., the client will be notified and an SDR will be written. The samples in question will be logged into the LabNet, but the sample status will be put on "HOLD".

Refer to STL Chicago's Sample Acceptance Policy (Attachment 10) for further clarification of the policy utilized when samples are received at the laboratory. This policy is forwarded to applicable clients and is posted in the sample receipt area of the laboratory. The Section Managers of both the Log-in and Project Management areas will assume the responsibility of insuring this policy is made available to all applicable clients.

3.0 SAMPLE RECEIPT

Protective over garments, gloves and safety glasses will be worn. Samples suspected of having a very strong odor, are known to be hazardous, or appear to be unstable must be placed in the available hood for processing. The samples must be labeled with special handling instructions for the analysts.

3.1 When samples are received from couriers, the air bills are signed, dated, and timed by the sample custodians. If the client name is not present on the air bill, it is written in by the sample custodians. Copies of the air bills are maintained with the original paperwork. Original airbills are relinquished to the Accounts Payable Department for processing.

3.2 When sample coolers are received with COC seals on them, they are cut and saved until they are checked against the seal numbers on the COC. The cooler is then opened, the COC is removed from the cooler to determine what samples were received and to match up the seal numbers. If there is a certain batch of samples that require a quick turnaround time or short hold-times, that batch is processed first. Be aware of the collection dates in reference to holding times (Attachment 3).

NOTES:

- STL Chicago's Policy requires the use of crushed ice as a coolant and no longer provides blue ice.
- All samples coolers are reviewed at time of receipt for proper required presence of field QC (example: NFESC samples will be checked for the presence of required field blanks and rinsates). Any discrepancies will be noted at time of log-in as a directed note/SDR to the appropriate Project Manager.

3.3 If the COC seals do not match, the difference(s) are noted in an SDR (Attachment 8).

3.4 Empty one cooler at a time to ensure that there is no confusion of samples or paper work. A COC should be enclosed with each batch of samples. Group the sample bottles according to the client ID on the COC. If no COC is present, the samples are arranged in an alpha-numeric order. Once all the samples are out of the cooler, compare the samples with the COC to ensure that everything is present.

3.5 Enter all sampling information including Client ID's, sample dates and times into LabNet from the COC. If a sampling time is not present on the COC, check the sample container for this documentation and enter it into LabNet. Note this in the Job Notes. If neither the COC nor the sample container document the 'sampling time', contact the PM via an SDR. Once resolved or if there are no discrepancies, review the sampling time entries entered into LabNet to ensure that they match the COC. If a sampling time is not documented for a Trip Blank, on the COC, document the time as that of the first VOA sampled.

3.6 All water samples that require pH preservation and Method 608 samples will be checked for preservation by using pH paper and a disposable transfer pipette (Attachment 3). The sample custodians will document this verification by signing their initials and date in the "Notes" section under "Properly Preserved" on the COC and within the sample receipt checklist in the LabNet job (a.k.a., batch number).

3.7 If the samples are not preserved, an SDR is initiated and submitted to the PM. If the PM determines by discussion with the client that the sample(s) will be preserved in the laboratory by the login personnel, this information will be noted in the SDR.

3.8 If the samples are to be preserved by the login personnel, the information is recorded in the preservative logbook. This information includes: date; client name; sample number; preservative; preservative lot number; and the name of the person preserving the sample.

3.9 If the metals water samples are not preserved, they will be preserved in login and a sticker will be placed over the lid of the sample bottle. This pre-printed sticker will document "Sample preserved in login, do not analyze for 18 hours".

3.10 The date and time of the preservation will be documented by the sample custodian on the sticker.

3.11 STL Chicago Residual Chlorine Procedure. STL Chicago has discussed possible sources of chlorinated samples and identified at-risk-parameters. The following describes the laboratory's policy concerning the testing of Residual Chlorine at time of sample receipt. Project Managers have contacted applicable clients in the attempt to identify those samples possibly coming from chlorinated waste streams. The result of client contacts has indicated that the possibility of such an event is unlikely. However, STL Chicago has implemented a procedure, outlined below, to be followed if such an event occurs. The laboratory has identified the following at-risk-parameters.

NPDES WW Effluents Inorganic:
Phenol .. BOD, TOX tested at bench
Cyanide
Ammonia

NPDES WW Effluents Organics:
GCMS VOA (THM's) Method 624

Drinking Water Inorganic:
Phenol ... BOD, TOX tested at bench
Cyanide
Ammonia

In the event the Project Manager has identified a possible sample from a chlorinated waste stream, a RESCHLCK review will be entered into the LabNet system, flagging the samples for the Log-In personnel. The Log-In personnel will test the applicable parameters using Potassium-Iodide Paper for the presence of residual chlorine at the time of sample receipt. Additionally, a line item is present on the Sample Receipt Checklist "Residual Chlorine Check Required" to document if such a check is required. If such samples show positive for Residual Chlorine, the appropriate personnel (Wet Chemistry Manager or applicable analyst) will be notified appropriately, and the samples treated as per method requirements. For those samples received past hours or over the week-ends, notification will take place at the next possible time and the samples treated.

In the case of GCMA VOA samples for Method 624, due to interferences from the required preservative for possible chlorinated samples, the samples must be verified to be free from residual chlorine. The Project Managers will contact the clients to verify that samples are being collected prior to chlorination. This verification will be used as a basis for not requiring a RESCHLCK, unless the Project Manager suspects otherwise. Upon which the above procedure will be required.

3.12 All water VOA vials will be checked for air bubbles and excessive head space. If observed, these items will be documented on the sample receipt checklist or on an SDR if more than 1 vial of a single sample is affected, in which case the PM will be contacted. Due to the nature of the sample/analysis, all water VOAs are checked for pH preservation at the time of analysis. Documentation is preformatted into each instrument run log.

3.13 Document any discrepancies on the SDR, i.e., missing samples, broken bottles, sample spillage, insufficient sample volume, incorrect preservative or discrepancies in sample ID, etc., and distribute it to the appropriate personnel.

3.14 If there are any problems, call the appropriate PM by referring to the LabNet Project Number. If the PM for the project is unavailable, the designated alternate PM should be notified.

3.15 All the COC's must be signed and dated by sample custodians.

NOTE: Login personnel will obtain a second temperature of samples during the login process when the samples have been outside of cold storage for an inordinate length of time. If, at that time, it is observed that the temperature is approaching 6°C, the samples will be stored temporarily in walk-in Cooler 8. The login process will be completed electronically. The samples will be returned to the login area to complete the process.

4.0 SHIPMENT RECEIPT CUSTODY RECORD (AFTER-HOUR RECEIPTS)

The laboratory's routine working hours, which includes Saturday coverage, are defined to our clients so that trained sample custodians are available to process sample receipts. When samples are received at a time when the sample custodians are not available or when samples cannot be logged in at the time of receipt, the samples are placed in a walk-in cooler and a Shipment Receipt Custody Record (Attachment 4) is completed. These samples are promptly logged in on the next working day.

Cooler temperatures of unscheduled sample receipts are not taken by non-sample custodians. By these personnel opening and measuring the temperatures of unscheduled and after hours cooler shipments, the laboratory "accepts" custody for the samples without verification of the COC (i.e., complete sample integrity). Clients are encouraged to notify their PM of late sample deliveries so that sample custodians are available on-site to process the receipts.

Samples dropped off after hours are subject to non-compliance due to the short amount of time that the samples are actually on ice. It will be noted that the samples were "chilled" and a temperature will be taken during the login process, the next day. This information is documented on the Shipment Receipt Custody Record.

5.0 SAMPLE LOGIN

5.1 Confirm the information on the COC against the sample labels (for example: date due, work order number. Enter all analyses requested by the client, from the COC. Login personnel will document discrepancies between the COC and bottles received via an SDR (i.e., VOA vials are received, but an 8260 analysis does not appear on the COC). However, all analyses will be logged into LabNet from the COC, unless otherwise directed by the client. While the bottle labels have analysis on them, they are

only to be used as a guide, to aid the samplers in decision making in the field. The COC is a legal document for logging in samples based on field identification and requested suite of analysis.

5.2 The sample custodians pull up each clients Project from LabNet to process the samples. All the information necessary to process the samples has already been entered into the designated Project Number by the PMs to ensure smooth & efficient sample login.

5.3 The COC is reviewed to ensure comparability between the samples and the documentation on this form. The custodians complete the form by answering several questions concerning the sample condition. (These questions are also synonymous with LabNet's sample receipt checklist). (Attachment 7) Any problems with sample condition will be noted on the COC and an SDR/LabNet Job Note will be initiated and submitted to the PM. The PM's will forward the original Log-In Job Note with documentation that the client was informed and any additional comments to the Report Generation Department. The Job Note will then be included in the final report. If required by the client, a cooler receipt form will be filled out containing the same information. All initial sample receipts and sample subcontracting transfers will be documented on this internal COC.

5.4 A sample number consists of a sequential number that is assigned to a sequentially assigned Job Number. For example, an assigned Job Number of 223102 for samples 1 through 10 are noted as 223102-1; 223102-2, 223102-3, etc.. Each container within a sampling point is also given a unique sample number to provide for a container-numbering system that uniquely identifies each sample container.

NOTE: All samples on "HOLD" will be assigned a sample number and logged into LabNet.

5.5 Samples received that cannot be logged in at the time of receipt, will be checked for short hold times and quick TAT, and the temperature taken. A LabNet sample receipt checklist and a copy of the COC will be completed and placed in the login pending file. The original COC will be returned to the cooler, and the cooler will be resealed with COC tags. The assigned sample numbers will be documented on the top of the cooler.

5.6 The PM will designate the QC or Deliverables within their project (i.e., P1, P2, L2QFMDL, L3QFND, L4QFRLU, L4QFAFCE; L4QFCLP, etc..). [Refer to the Data Management SOP (UDM-001) for a description of the data deliverables.] Additionally, the Project Managers will identify via an ICOCREVIEW flag, if the samples require Internal Chain of Custody tracking and storage.

5.7 Matrix Spike (MS) and MS Duplicate (MSDs) samples all have one sample number and are designated within LabNet.

5.8 For soluble metals analysis, the samples are either field filtered or filtered by the metals and/or TCLP preparation personnel. Login personnel transport filtered samples to the lab for filtration.

CLP metal samples are logged-in in batches of 20 samples. (Total and soluble metals may be logged in different batches.) Different matrices will be in separate batches.

If a sample requires a leachate procedure, the original material is assigned the leaching procedure. A new sample number is created for the leachate and the appropriate tests are assigned. The maximum batch size will be 20 original samples. The PM and client will determine what samples are to be spiked.

5.9 After all the COC and sample information is reviewed to be in agreement, each sample bottle is labeled with their printed LabNet sample number. In the event that sample login receives only one (1) sample bottle to be used for all analyses including Volatiles, a label will be placed over the lid of the bottle indicating that it is the only sample bottle. Volatiles must be analyzed first. The volatiles analyst will check the label after their analysis is complete, indicating that the sample can be used for the remaining analyses. The sample will be stored in the Volatiles walk-in cooler.

5.10 If a sample requires a quick turnaround time or has a short hold time, the sample is given directly to the analyst. The Wet Chemistry parameters with short hold times (48 hr or less) are written on the sample bottle and brought directly to the WC lab where they are placed on a designated cart. Above the cart is a board where the sample Job Number, Sample ID, number of samples and which parameters are required is recorded. If the sample does not receive any special treatment, it is placed in the appropriate cooler.

Cooler No.	Contents
1 (ICOC)	Consecutive sequence of samples requiring internal COC (ICOC) (i.e., CLP or Special Project samples requiring ICOC as designated by the PM). This cooler is kept locked at all times. When samples are needed from this Cooler, the analyst comes to the sample custodians to sign out the samples on an Internal Sample Transfer Custody Record (Attachment 5). The samples are then retrieved from the cooler for analysis. Upon returning the samples, the analyst will relinquish them back to the sample custodians by signing the Internal Sample Transfer Custody Record. The samples are then returned to the locked Cooler. NOTE: Metals digestates that require ICOC will be kept locked in Room 1502B (Located in the instrument laboratory).
2 (ICOC)	Consecutive sequence of Metals and Leachate samples; and samples that are a waste or solid are also stored in this cooler. Refer to the description listed for Cooler 1.
3 (ICOC)	Refer to the description listed for Cooler 1.
4	Organic extraction samples that are in process (ICOC N/A).
5	Consecutive sequence of GC and GC/MS Volatiles. This cooler is kept locked at all times. All GC/MS and GC Volatile samples (including ICOCs) samples are

Cooler No.	Contents
	relinquished directly over to this cooler / department.
6	Organic extracts. This cooler is kept locked as may contain ICOC extracts.
7	Consecutive sequence of all Wet Chemistry samples. This cooler is kept locked as it may contain ICOC samples in process.
8	Consecutive sequence of Metals, Cyanide, Sulfide, TOC, non-CLP soils, and Leachate parameters. Samples that are a waste or solid are also stored in this cooler. (ICOC N/A)
NOTE: NPDES samples requiring North Carolina DEHNR/DEM certification must be maintained at a temperature between 1-4°C. To accomplish this, these samples will be stored in transferable coolers, along with wet ice, and its own thermometer in each cooler. These coolers will then be stored within our walk-in coolers. While these samples are in house, until the analysis has been completed and the final report submitted to the client, the thermometer with these samples will be monitored two times daily, with at least 4 hours in between readings. The readings will be recorded on a Cooler Temperature Control Log sheet (Attachment 9).	

6.0 SAMPLE LOGIN

With LabNet, the PMs create a Project that has the pre-selected test methods. The sample custodians pull up the project file in LabNet and use the information to assign the sample numbers within the Job (set of project samples). Refer to Attachment 6 for instructions of logging samples into LabNet.

7.0 SAMPLE TRACKING

7.1 All samples will remain in the appropriate coolers prior to and after analysis. For Cooler 8, the analyst will list the samples they are taking out of the cooler on the wet/dry board posted next to this cooler.

7.2 CLP/ICOC samples must be relinquished to the analyst. The analyst and sample custodian must sign the original COC or an Internal COC tracking form (by Job Number) for relinquishing custody of the samples from the sample custodian to the analyst. When the samples are returned to the appropriate cooler, the analyst will sign the custody of the returning sample(s) back to the sample custodian.

7.3 Any change in the sample during the time of custody will be noted on the COC, i.e., sample breakage or depletion. This information should be passed on to the appropriate laboratory personnel and the PM.

8.0 "SUB-OUT ANALYSES" FORMAT

When it becomes necessary to sub-contract samples, the following procedure should be followed.

8.1 The PM in charge of the samples that need to be subbed out will contact the sample custodians and supply information and paperwork regarding samples/ analyses to be subbed, location of contract laboratory, and any special instructions.

8.2 If any other laboratory personnel contact sample custodians in regards to sub-contracting samples, they will contact the PM to secure the proper paperwork.

8.3 The samples to be subbed will be removed from the cooler and inventoried to assure that all the samples are present. If only a portion of the requested analyses are to be subbed, the samples may have to be split (will be defined by the PM).

8.4 STL's practice is to log subcontract work on a separate LabNet job. This enables the laboratory to better track the subcontracted parameters. In order to link the 'parent' job to the subcontract job the following procedure will be followed:

1. Parent Job: In the 'Additional Analysis/Remarks' field, login personnel will note the Subcontracted Job Number.

2. Subcontracted Job: In the 'Additional Analysis/Remarks' field, login personnel will note the corresponding Parent Job Number.

3. LabNet: Using the job notes feature, login personnel will indicate the Parent and Subcontract Job Numbers.

8.5 Along with the paperwork supplied by the PM, the sample custodians will include a COC with the samples.

8.6 If the sub-contracting laboratory is another STL Laboratory, include the pertinent client information on the COC, i.e. client name, work order number, etc..

8.7 If the sub-contracting laboratory is a private lab, ensure that the client information remains confidential, i.e. the client name will be STL, the work order number will not be noted on the COC, etc.

8.8 One copy of all the paperwork sent with the samples must be made and attached to the original COC.

8.9 The samples with the appropriate paperwork will be packed in a cooler in accordance with proper IATA regulations and delivered to the shipping personnel by 4:00 pm.

9.0 SAMPLE DISPOSAL

If a sample is received broken or is broken in the laboratory, the Environmental Health & Safety Coordinator (EHSC) will be contacted for proper clean-up procedures. If the sample is known to be non-hazardous, personnel may salvage as much sample as possible without contamination and place the original broken container in a plastic bag. The EHSC and the sample custodians will be informed of the incident. The sample custodians will document the breakage on the client paperwork and inform the PM of the incident via an SDR.

The waste sample disposal is handled by waste management department (UWM-001). General requirements for logins sample/digestate/extract disposal procedures are as follows:

- The standard sample disposal time is 30 days after the report is submitted.
- For standard sample disposal, a list is generated from LabNet.
- Samples requiring internal COC are disposed of 60 days after the report has been submitted, unless other arrangements have been specified by the PM.
- The water samples will be taken out of the coolers by the sample custodians. The numbers will be checked on the bottles to make sure they are the correct samples that are ready for disposal.
- The samples will be taken to the disposal room and the bottles emptied.
- Non-water samples are disposed of by the waste disposal group. (SOP UWM-001)
- All hazardous samples will be disposed of commercially or returned to the client.

10.0 SAMPLE BACKLOGS

1. All LabNet backlogs are printed by each department or section manager on a daily basis to review the receipt of additional samples and review holding times.

2. Copies of the COCs and all supporting LabNet paperwork are maintained in the Job's file folder that is maintained in the data management department.

3. Every morning, the Project Manager reviews all of their LabNet jobs to ensure that everything was logged in correctly. If there are corrections to be made, they are changed in the computer, the analyst, section manager, or PM is notified.

11.0 ATTACHMENTS

Attachment 1: Example: Electronic Temperature Monitoring Diagram / Spreadsheet
Attachment 2: Example: STL's Chain-of-Custody
Attachment 3: Example: Sample Handling Guide (i.e., Hold Times; Preservation)
Attachment 4: Example: Shipment Receipt Custody Record
Attachment 5: Example: Internal Sample Transfer Custody Record
Attachment 6: Example: LabNet Computer Login Instructions
Attachment 7: Example: LabNet Sample Receipt Checklist
Attachment 8: Example: Sample Discrepancy Report (SDR)
Attachment 9: Example: Cooler Temperature Control Log
Attachment 10: Example: STL Chicago Sample Acceptance Policy

Historical File:

Revision 05: 04/15/92
Revision 06: 03/11/93
Revision 07: 11/15/93
Revision 08: 07/21/95
Revision 09: 04/08/96
Revision 10: 04/14/97
Revision 11: 11/03/97
Revision 12: 03/16/99
Revision 13: 09/27/00
Revision 14: 05/29/01
Revision 15: 10/22/02
Revision 16: 03/15/04
Revision 17: 04/18/05

Reasons for Change, Revision 17:

- Annual Review – Updated Section 2.2 referencing Attachment 10: STL Chicago's Sample Acceptance Policy
- Updated Section 3.12 with language to clarify/discuss protocol for handling samples with Residual Chlorine.
- Other general review and clarifications on procedures and additional comments on ICOC samples.

U:\QC\SOP\SR\USR-001.doc

Attachment 1.

Example: Electronic Temperature Monitoring Diagram / Spreadsheet

Blue: Below Lower Limit

Red: Above High Limit

Green: Within Normal Limits

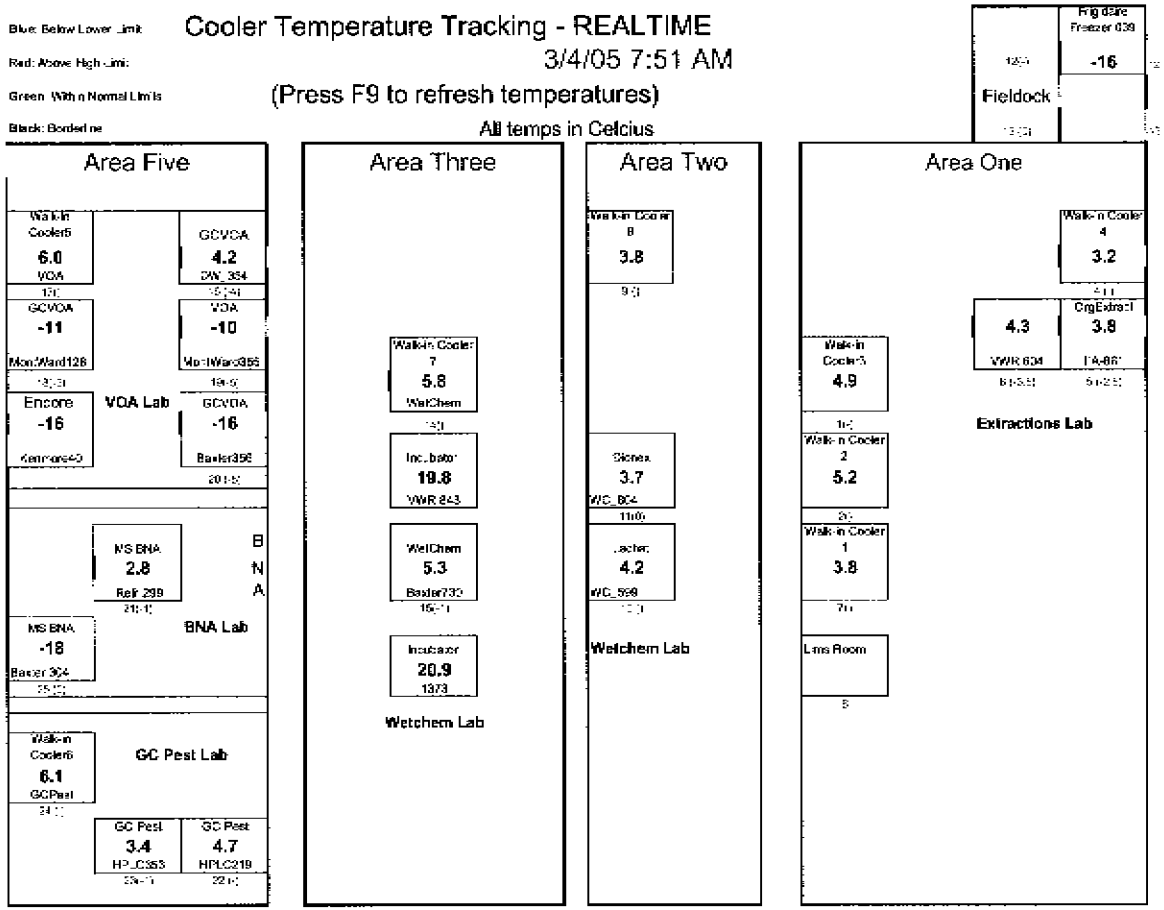
Black: Borderline

Cooler Temperature Tracking - REALTIME

3/4/05 7:51 AM

(Press F9 to refresh temperatures)

All temps in Celcius



Attachment 2.

Example: STL's Chain-of-Custody

SEVERN TRENT SERVICES

STL Chicago
2417 Bond Street
University Park, IL 60466
Phone: 708-534-5200
Fax: 708-534-5211

Report To:

Contact: _____
Company: _____
Address: _____
_____
Phone: _____
Fax: _____
E-Mail: _____

Bill To:

Contact: _____
Company: _____
Address: _____
_____
Phone: _____
Fax: _____
PO#: _____ Quote: _____

Shaded Areas For Internal Use Only ____ of ____

Lab Lot#

Package Sealed	Samples Sealed
Yes No	Yes No
Received on Ice	**Samples Intact**
Yes No	Yes No

Temperature °C of Cooler

Within Hold Time	Preserv. Indicated
Yes No	Yes No NA
pH Check OK	**Res Cl_2 Check OK**
Yes No NA	Yes No NA

Sample Labels and COC Agree
Yes No COC not present

Sampler Name: | **Signature:** | **Project Name:** | **Project Number:** | **Project Location:** | **Lab PM:** | **Date Required** Hard Copy: ____/____/____ Fax: ____/____/____

Laboratory ID	MS-MSD	Client Sample ID	Sampling Date	Sampling Time	Matrix	Comp/Grab	Relng #	# / Cont.	Volume	Preserv	Additional Analyses / Remarks

RELINQUISHED BY	COMPANY	DATE	TIME	RECEIVED BY	COMPANY	DATE	TIME
RELINQUISHED BY	COMPANY	DATE	TIME	RECEIVED BY	COMPANY	DATE	TIME

Matrix Key
WW = Wastewater
W = Water
S = Soil
SL = Sludge
MS = Miscellaneous
O = Oil
A = Air
SE = Sediment
SO = Solid
DS = Drum Solid
DL = Drum Liquid
L = Leachate
WI = Wipe
O = _____

Container Key
1. Plastic
2. VOA Vial
3. Sterile Plastic
4. Amber Glass
5. Widemouth Glass
6. Other _____

Preservative Key
1. HCl, Cool to 4°
2. H_2SO_4, Cool to 4°
3. HNO_3, Cool to 4°
4. NaOH, Cool to 4°
5. NaOH/Zn, Cool to 4°
6. Cool to 4°
7. None

COMMENTS

Date Received ____/____/____
Courier: **Hand Delivered** ☐
Bill of Lading

Attachment 3.

Example: Sample Handling Guide (i.e., Hold Times; Preservation)

SAMPLE HANDLING GUIDE

SEVERN TRENT STL

Inorganic and Conventional Parameters

Parameters	EPA Method*	Container	Recommended Vol. (mL)	Preservative	Holding Time
Acidity	305.1	P	250	4 ± 2°C	14 days
Alkalinity	310.1	P	250	4 ± 2°C	14 days
Ammonia	350.2	P	250	4 ± 2°C, H_2SO_4 to pH <2	28 days
Biochemical Oxygen Demand (BOD)	405.1, SM 5210B	P	1000	4 ± 2°C	48 hours
Bromide	300.0, 9056	P	250	4 ± 2°C	28 days
Chemical Oxygen Demand (COD)	Hach 8000	P	100	4 ± 2°C; H_2SO_4 to pH <2	28 days
Chloride	300.0, 325.2, 9056, 9251	P	200	4 ± 2°C	28 days
Chlorine, Residual	330.4	P	200	4 ± 2°C	Immediately
Chromium VI	7196A; SM 3500CrD	P	250	4 ± 2°C	24 hours
Color	110.2	P	100	4 ± 2°C	48 hours
Cyanide	335.2, 9010B/9014	P	100	4 ± 2°C, ascorbic acid, NaOH to pH > 12	14 days
Fluoride	300.0, 340.2, 9056	P	100	4 ± 2°C	28 days
Hardness	130.2	P	100	4 ± 2°C; HNO_3 to pH < 2	6 months
Metals	6010B, 200 & 7000 series	P	100	4 ± 2°C; HNO_3 to pH < 2	6 months
Mercury	245.1, 7470A, 7471A	P	200	4 ± 2°C; HNO_3 to pH < 2	28 days
Nitrogen, Kjeldahl (TKN)	351.3	P	200	4 ± 2°C, H_2SO_4 to pH < 2	28 days
Nitrate	300.0, 9056	P	200	4 ± 2°C	48 hours
Nitrite	300.0, 354.1, 9056	P	100	4 ± 2°C	48 hours
Nitrate + Nitrite	353.2	P	100	4 ± 2°C, H_2SO_4 to pH < 2	28 days
Oil and Grease	1664RA, 9071B	G	1000	4 ± 2°C, H_2SO_4 to pH < 2	28 days
Phenols	420.2, 9066	G	200	4 ± 2°C, H_2SO_4 to pH < 2	28 days
Phosphorus, Total	365.2, SM 4500 PE	P	100	4 ± 2°C, H_2SO_4 to pH < 2	28 days
Phosphate, Ortho	300.0, 365.2	P	100	4 ± 2°C	48 hours
pH	150.1, 9040B, 9041A, 9045C	P	100	None	Immediately
Solids, Dissolved (TDS)	160.1	P	100	4 ± 2°C	7 days
Solids, Suspended (TSS)	160.2	P	500	4 ± 2°C	7 days
Solids, Volatile (TVS)	160.4	P	100	4 ± 2°C	7 days
Solids, Total (TS)	160.3	P	100	4 ± 2°C	7 days
Specific Conductance	120.1, 9050	P	100	4 ± 2°C	28 days
Sulfate	300.0, 375.4, 9038, 9056	P	200	4 ± 2°C	28 days

SAMPLE HANDLING GUIDE

SEVERN TRENT STL

Inorganic and Conventional Parameters

Parameters	EPA Method*	Container	Recommended Quantity (mL)	Preservative	Holding Time
Sulfide	376.1, 9030B/9034	P	500	4 ± 2°C, Zn acetate, NaOH to pH > 9	7 days
Total Organic Carbon (TOC)	415.1, 9060	P	40	4 ± 2°C, H_2SO_4 to pH < 2	28 days
Total Organic Halides (TOX)	9020B	G-TLC (amber)	200	4 ± 2°C, H_2SO_4 to pH < 2	28 days
Total Petroleum Hydrocarbon (TPH)	1664RA	G-TLC	1000	4 ± 2°C, H_2SO_4 to pH < 2	28 days
Turbidity	180.1	P	100	4 ± 2°C	48 hours

Organic Parameters

Parameters	EPA Method*	Container	Recommended Quantity (mL)	Preservative	Holding Time
Diesel Range Organics (DRO)	8015B; WI DRO; OA-2 (WI DRO req. pre-weighed bottles)	G-TLS	1000	4 ± 2°C	14 days
Gasoline Range Organics (GRO)	8015B; WI GRO; OA-1	G-TLS	2 x 40	4 ± 2°C, HCl to pH < 2	14 days
Volatile Organics	624, 8260B, CLP	G-TLS	2 x 40	4 ± 2°C, HCl to pH < 2	14 days 10 days for CLP
Pesticides (Organochlorine or Organophosphorous) and PCBs	608, 8081A, 8082, 8141A, CLP (where applicable)	G-TLC (amber)	1000	4 ± 2°C, pH 5-8	7 days to extract 40 days to analyze 5/35 days for CLP
Chlorinated Herbicides	8151A	G-TLC (amber)	1000	4 ± 2°C	7 days to extract 40 days to analyze
Semivolatile Organics (BNA), Polynuclear Aromatics	625, 610, 8270C, 8310, CLP (MS Only)	G-TLC (amber)	1000	4 ± 2°C	7 days to extract 40 days to analyze 5/35 days for CLP

TCLP Parameters

Parameters	Holding Time from Collection to TCLP Extraction (days)	Holding Time from TCLP Extraction to Preparative Extraction (days)	Holding Time from TCLP/Preparative Extraction to Analysis (days)	Total Elapsed Time (days)
Volatiles	14	Not Applicable	14	28
Semivolatiles	14	7	40	61
Mercury	28	Not Applicable	28	56
Metals	180	Not Applicable	180	360

References: 40CFR Part 136 Tables IA, IB, IC, ID & IE and Table II, and others.

*The methods listed are for typical EPA references, except for SM, which refers to Standard Methods for the Examination of Water and Wastewater (18th Edition).

For organic parameters, add sodium thiosulfate if residual chlorine is present. Soil samples should be collected in 4-8 oz glass containers with a Teflon®-lined cap and preserved at 4 ± 2°C. No preservative required for waste samples except 4 ± 2°C for volatiles. Teflon® is a registered trademark of E.I. du Pont.

Acronymn Definitions:

P	Polyethylene	G-TLS	Glass with Teflon®-lined septum
G	Glass	PTFE	Fluoropolymer Resin / Teflon®
G-TLC	Glass with Teflon®-lined cap	CLP	EPA Contract Laboratory Program

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Attachment 4.

Example: Shipment Receipt Custody Record

STL Chicago
Shipment Receipt Custody Record

STL Number:_____

Carrier:_____

Shipping Receipt Number:_____

Date Received:_____

Time Received:_____

Project Name:_____

Interim Storage Location[1]:_____

Person Receiving Shipment:_____

COC Tape was present on outer package: Yes No

COC Tape was unbroken on outer package: Yes No

Signature:_____

Date / Time:_____

[1]*Note: The sample cooler(s) (circle one): a) delivered via non-overnight carrier and placed in secured storage prior to processing the next working day.*
b) were placed in secured refrigeration storage with temperatures taken the next working day prior to processing;
c) temperatures were taken upon receipt, placed in secured refrigeration storage with the processing completed on the next working day.

COC Tape was present on outer package: Yes No
COC Tape was unbroken on outer package: Yes No

Relinquished to:
Person Unpacking Shipment:_____ Signature:_____ Date:_____ Time:_____

Comments:_____

_____

_____

_____

Signature:_____ Date:_____

CHI-22-11-010/E-10/02

Attachment 5.

Example: Internal Sample Transfer Custody Record

STL Chicago
Intra-Laboratory Internal Sample Custody Transfer Record

Job No:_____ Client:_____

Sample No.	Analysis	Relinquished by:	Received by:	Date	Time	Comments

CHI-22-11-019/A-12/02

Attachment 6.

Example: LabNet Computer Login Instructions

STL Chicago
Work Instruction for LabNet Computer Log-In

I. Sample Log-In

1. Enter your initials at the "login:" prompt(enter)
2. Enter your password at the "password:" prompt(enter)
3. Enter "LabNet" at the "$" prompt

At the Main Menu:

4. Select 4-LabNet Job Login(enter)
5. At the LabNet Job Login Screen:
6. Select 1-Login Job(enter)

At the Login Job screen:

7. Location code never changes(enter)
8. Enter Job Number-Select "Enter" from your keyboard and LabNet will inquire "Do you want to start a new job?"
9. Select "Y" on your keyboard and LabNet will assign the next available job number.
10. Enter through the Job Status section and the job will be set to "A"(Active).
11. Enter Project Code-Enter the 8 digit code for the particular project you wish to access to log in the job or select F1 to look up from a list of projects.
12. A project description should appear next to the 8 digit number, after entering.
13. Your cursor should now be on the Cust. Project ID. Enter the same information found after the 8 digit project number(as much as possible). It will appear as upper case letters.
14. Enter through the JDE Acct. section
15. Enter through the Login Person section(your initials should appear).
16. Enter through the Report Code section. Note if CLP deliverable,(job should be logged as an "Internal", complete with Internal COC's filled out).
17. Enter through the TAT section(a TAT should be assigned)
18. Enter through the Date Received section(the current date should appear)
19. Enter through the Due section(the due date will be calculated based on the date received and the TAT).
20. At the Login Job Menu:
21. Cursor through the Customer information item
22. Select "Enter" at the Sample Receipt Check List-answer all of the questions with a "Y" or "N", that pertain to this job. If some questions do not pertain, then skip them. Ensure that a temperature is entered on the Check List at the temperature question.
23. Select F2 to save the Check List and return you to the Login Job Menu.
24. Cursor through the Sample Questions item
25. The Jobs' Samples line should be highlighted. Select "Enter" on your keyboard.
26. Your cursor should now be on the blank Sample# column. Select "Enter" and LabNet will inquire "Do you want to Start a New Sample?" Select "Y" on your keyboard and LabNet will bring you to the Sample information screen.

27. At the Customer ID-Enter the customer ID as it appears on the COC(Enter). The Received Date should already appear. Enter the time(Military), of which the samples were received that day(Enter).

28. Enter the Sample Date-Time as they appear on the COC(Enter).
29. Enter through the Sample logger section(your initials should appear).

30. At the Sample Matrix prompt. Select F1 and a menu should appear with a list of Matrices. Select the matrix which applies to that sample(Enter). The menu will go away and the matrix selection you've made should appear.

31. Enter through the Report Sample section.
32. At the QA/QC Required(Y/N) section-if matrix QC is required on this sample, enter "Y". If not, leave as "N".
33. Enter through the Sample Type-one should appear.
34. Enter again and the cursor should be on the first line of the Enter Answers column.
35. At this point, the sample bottles need to be recorded for all samples. While your cursor is on the Enter Answers column, Select SF2 to get to the Job Sample Bottle List.
36. Your cursor should be on the Bottle ID column. Select Enter, and LabNet will ask you "Generate a New Bottle ID?". Select Y(yes) and a new bottle ID Number will appear and the cursor will move to the "Type" column. Select F1 and all the bottle types in the database, will appear. Find the bottle type your looking for by moving your cursor up and down the list. When you find the bottle type you need, select ENTER and that bottle type will appear on the Type line.

37. The cursor will then move to the Preservative column. Select F1 and all of the preservative types in the database will appear. As in the bottle type list, find the correct preservative by moving the cursor, select ENTER and it will appear on the Preserv. line.

38. The cursor will then move to the "F"(filtered), column. Select "Y" or "N" to document that samples' filtration.

39. The cursor will move to the Condition column. This column will accept text only. A brief comment on the condition of the sample(i.e., LTD Volume, To be Filtered, etc..), could be stated here. A blank line will be interpreted as nothing wrong with the sample. If no entry is needed TAB or ENTER through this section.

40. The cursor should now be on the Volume column. This column will also only accept text. A brief description of the volume of the sample is only needed if LTD VOLUME is rec'd. While the client may have sent in a 1-Liter bottle, it may only have 500 mLs of sample in it. A comment here will document and inform the lab.
41. If text is not needed, TAB or ENTER through to the next column.

42. Your cursor should be on the Cont/Bin column. This is where log-in designates which walk-in cooler each bottle will be stored in. Select F1 and cursor up or down to find the correct cooler for where the bottle(s) will be stored. Once the correct cooler is found, Select ENTER and the cooler number will appear and the cursor will move to the Bottle ID column to start the process over for the next bottle.

Note: If you have multiple bottles for one test (i.e., 3 - 40 mL vials for VOA's), enter the information for one of the bottles. The information for that one bottle can be copied. Place your cursor on the Bottle ID column of the first bottle to be copied. Select F1. LabNet will ask: "Would you like to Duplicate bottle ***000000***?"

43. Select "Y" and another screen will ask: "How Many Duplicate Bottles". Enter the number of bottles to be copied and LabNet will assign new numbers to them and copy all of the information from that bottle.

44. Once all of the sample bottles are entered, Select F2(SAVE). LabNet will automatically save the selections and return to the Sample Number Screen.
45. Select F2, Escape, and LabNet will return to the Jobs Samples Screen.
46. Once all sample information is entered for 1 complete sample, it is possible to "copy" that information for other samples. This will ease the sample log-in procedure. To do so, place your cursor on the first sample line. Select F6. LabNet will ask several questions:
47. Would you like to duplicate sample number 1. (select "Y")
48. Do you want to duplicate all of the sample information? Y/N
49. This sample has bottle info-create ID's and Duplicate the bottles?(select "Y")
50. A box should appear asking "how many samples". Enter the number of samples needed to complete the job. Select ENTER. Sample 1 should be duplicated exactly as it was logged. Now, change information per sample (i.e., sample ID, date, time, etc.). If there is one particular sample that has less sample bottles than sample 1, cursor down to that sample, select SF5. This will "zoom" in on that samples bottles. Cursor down to the bottle not needed, and select F3. LabNet will ask "do you want to delete this row?" Select "Y" and the row will be deleted. If you are done, select F2 and the changes will be saved.

51. Selecting Methods
52. From the Job Menu, select "Jobs' Methods and Tests in a Group of Samples"
53. This screen is used to log methods and their tests into a group(range), of samples. The cursor will be on the "Group Number" field. Select ENTER, and LabNet will prompt "Do you want to START a NEW GROUP?". Select "Y" and the system will generate a new group number and move the cursor to the "Description" field.

54. This is a text field. Enter a brief description of the samples about to be logged in (i.e., VOA/Soils, Metals/Waters, EB, TB, etc.). Select ENTER and the system will move the cursor to the "Sample Range" field.

55. Enter the range of the samples to be logged.
56. Pay particular attention to the range selection to be made:
57. Example: If you have soils and waters on the same job, log them into different Groups. If samples 1-5, and 7-10 are soils, the Sample Range should be 1-5,7-10. Use of a comma is needed for LabNet to recognize a separation of sample 5 and sample 7.

CHECK TO ENSURE THE SAMPLE RANGE SELECTED IS CORRECT! ONCE YOU MOVE FROM THE SAMPLE RANGE, IT CAN NEVER BE CHANGED.

58. Select ENTER after the Sample Range has been checked. The cursor should move to the first line under the "Method" column.
59. Select SF1. This will bring up a Menu, "Type of Analysis Zoom", of 3 items:
60. 1)This Jobs' Project Analysis Groups
61. 2)This Jobs Customer Analysis Groups
62. 3)General Systems Analysis Groups

63. ALWAYS SELECT #1!
64. After selecting #1, this will prompt the system to find the analytical methods, selected by the Proj. Mgr., for this Project/Client. There may be several groups of methods to choose from. At this point, cursor through the different groups, and view the methods within the different groups. The methods and their description will appear in the middle of the screen.

65. Once you find the particular group from which to log the methods, select F8 to "Zoom" into that particular group. The cursor should now be on the first method of that group. Cursor down through the group to find the particular method needed to log for the Range you've selected. Select ENTER and that method will be highlighted. If the entire group of methods is to be logged, Select F11. All of the methods within this group will be highlighted.

66. Once the method is highlighted, Select F7 to "Return Selection" to the Range of Samples to be logged for this method. The system will now return to the previous screen(where you selected the Range, its' description and Group#). The method that was highlighted and Returned will appear in the Method column along with its' Description, Method TAT, Destination, etc.
67. To complete the process, Select F2. The system will now SAVE the method to that Range of Samples. Notice at the bottom of the screen that LabNet will tell you it is "Saving Sample 1, 2, 3", etc.

Repeat this process for logging other Groups/Ranges.

II. Jobs' Reviews and Work Processes

68. At the Job Menu, Select Jobs' Reviews and Work Processes.
69. Several Reviews selected by the PM should already appear.
70. There are four (4) Reviews that **EVERY** job will be logged in for:
71. LOGIN
72. PRICING
73. REPORT
74. INVOICE
75. Ensure that these 4 are selected.

76. If there is data to be faxed to the client on a quick TAT:
77. Move cursor to a blank line. Select F1. A list of Reviews will appear. Cursor to the
78. FAXDATA Review and Select ENTER. This will highlight the review chosen. Select F4. This will Return the Selection to the Review list.
79. If you have selected a FAXDATA review, you MUST add a Fax Due Date.
80. Move the cursor to the FAXDATA review. TAB across to the Due Date column.
81. Enter the fax date.
82. Ensure that the DUE DATE DOES NOT fall on a Weekend day or a Holiday.

83. Once a fax date is entered, a Report Due Date *MUST* be entered.
84. Move cursor to the Report review. TAB across to the Due Date column.

85. Enter the Report Due Date. The Report Due Date should match the Hardcopy Date("Due" section at the main job menu).

III. Bottle Labels

86. From the Job Menu, select "Job Login Reports".
87. Cursor to the "Print Folder Labels". Select F4. An "X" will appear.
88. Cursor to the "Print Sample Labels". Select F4. An "X" will appear.
89. Select F7. LabNet will print the Folder label selected. When the print job is finished, another screen will appear with information about the sample labels.
    Total Number of Samples
    Beginning Sample
    Ending Sample
    Labels/Sample
    Description
    Barcode? Y or N

90. Selecting F7 will print all the sample labels for the entire job.

Note: Each label will have a unique number and exact information previously entered for that bottle (type, preservative, filtering information, Bin#, etc..).

91. It is important to ensure that the correct label assigned to a particular bottle, gets placed onto its' exact matching bottle.

    Example:
    A Plastic 1000 mL, Cool 2°C-6°C for Bin-7 label should not go on a VOA, 40 mL preserved w/HCl, for Bin-5.

92. After placing the labels on the proper bottles/jars. Move the samples to their proper walk-in coolers. Put the folder label on a folder. Put the clients' COC and any other documentation in the folder. Place the folder in the bin to be copied and distributed to the laboratory.

This completes the login process for that job. Select F2. This will save the job into the system.

Attachment 7.

Example: LabNet Sample Receipt Checklist

rpjsckl	Job Sample Receipt Checklist Report	V2

Job Number.: 236018 Location.: 57222 Check List Number.: 2 Description.:
Customer Job ID.....: Job Check List Date.: Date of the Report..: 04/26/2005
Project Number.: 20004601 Project Description.: WS PES Study Project Manager.....: tap
Customer.....: STL Chicago Laboratory QA Projects Contact.: Terese Preston

Questions ?	(Y/N)	Comments
Chain-of-Custody Present?.....		
Were samples dropped off at or picked up by STL?..	N	
Custody seal on shipping container?.....	N	
...If "yes", custody seal intact?.....		
Custody seals on sample containers?.....	N	
...If "yes", custody seal intact?.....		
Samples iced?.....	N	
Temperature of cooler acceptable? (4 deg C +/- 2).		
Samples received intact (good condition)?.....		
Volatile samples acceptable? (no headspace).....		
Correct containers used?.....		
Adequate sample volume provided?.....		
Samples preserved correctly?.....		
Samples received within holding-time?.....		
Agreement between COC and sample labels?.....		
Radioactivity at or below background levels?.....		
A Sample Discrepancy Report (SDR) was needed?.....	N	
Residual Chlorine Check Required?		
If samples were shipped was there an air bill #?..		
Sample Custodian Signature/Date.....	Y	tap

Attachment 8.

Example: Sample Discrepancy Report (SDR)

STL CHICAGO

Sample Discrepancy Report (SDR)

Lab Job # _____

Client _____ Analyses _____
Contact _____
Project _____
Phone _____ Matrix ☐ Water ☐ Soil ☐ Other _____
Fax _____

Deliverable **Level** ☐ 1 ☐ 2 ☐ 3 ☐ 4 ☐ MDL'U' ☐ RL'U' ☐ ND ☐ Other

COC Received: ☐ Yes ☐ No Quote from PM: ☐ Yes ☐ No

1A Type of Discrepancy

COC/Sample		LOG-IN	Client	Unit
☐ ID Discrepancy	☐ Rec'd past Hold Time	☐ Log-in past hold time	☐ Changed Analyses	☐ Analyzed past hold time
☐ Incomplete	☐ Improper Preservative	☐ Log-in error	☐ Improper Bottle Type	☐ Missing Sample/Extract
☐ Unreadable	☐ Missing Sample/Extract		☐ Label unreadable	☐ Insufficient Sample
☐ Cooler Temp. _____oC	☐ Container Broken		☐ Insufficient Sample	
☐ Quote Discrepancy	☐-Sample lost			
☐ Bubbles in VOA Vials	☐-Suspect Contamination			
☐ **Other** _____				☐ **EDD**

1B Lab ID COC/Client ID Description of Deficiency or Discrepancy

Initiator: Date:

2A PM Established Action Plan

☐ Cancel	☐ Bottle/jar replaced	☐ Change Test code from: _____	To: _____
☐ Add	☐ Lid replaced	☐ Change due date from: _____	To: _____
☐ Place on Hold	☐ Analyze past hold time	☐ Include in case narrative	
☐ Log-in	☐ Preserve then analyze	☐ Amend EDD	
☐ Subcontract	☐ Analyze	☐ **Other** _____	

2B

Name	Special Actions	Initiator Initial	Initiator Date	Completion Initial	Completion Date
1	1				
2					
3					
4					
5					
6					
7					
8					
9					

3 Distribution

☐ GC	☐ Wet Chem
☐ GC/MS	☐ Digestions
☐ Metals	☐ Extractions
☐ Distribution to QA	☐ **Other** _____

4 Final Approval of All Actions

☐ Send Copy to Client
Notes:

PM Signature:
Date:

Attachment 9.

Example: Cooler Temperature Control Log

STL Chicago
Internal Cooler Temperature Control Log

Job Number: _____ Date Received: _____
Cooler Number: _____
Thermometer ID: _____ Thermometer Correction Factor: _____

Date	Time	Temperature Reading (Not adjusted for correction factor)	Evaulation A – Acceptable; N – Not Acceptable (CA required)	Comment	Initials

Reviewed by: _____ Date: _____

Attachment 10.

Example: STL Chicago Sample Acceptance Policy

SEVERN TRENT STL

STL CHICAGO SAMPLE ACCEPTANCE POLICY

The following describes STL Chicago's Sample Acceptance Policy. Upon receipt of samples at the facility, the laboratory will assess all samples based upon the following criteria. The purpose of such criteria is to maintain the integrity of the samples and ensure that proper sampling and preservation procedures have been followed. Samples found to be in 'non-compliance' will be formally addressed and documented by internal operating procedures. Subsequent analysis of such samples may or may not proceed, and would be determined by discussion with the appropriate parties involved.

Samples are considered 'compromised' if the following conditions are observed upon samples 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.
- Sample ID's on the COC do not match samples received.
- COC is not properly completed or received, as determined by log-in personnel.
- Breakage of any Custody Seal.
- Apparent tampering with cooler and/or samples.
- Headspace in volatile samples.
- Seepage of extraneous water or material into samples.
- Inadequate sample volume.
- Illegible, impermanent or non-unique sample labeling.

This policy will be made available to all STL Chicago clients where applicable.

CHI-22-11-021/A-11/04
Updated: 11/22/04

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Appendix C

STL Labs, Inc.
Laboratory Certifications

STATE OF ILLINOIS

ENVIRONMENTAL PROTECTION AGENCY
NELAP - RECOGNIZED
ENVIRONMENTAL LABORATORY ACCREDITATION

is hereby granted to

STL CHICAGO

2417 BOND STREET

UNIVERSITY PARK, IL 60466-3182

NELAP ACCREDITED
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.

Ron Turpin
Manager
Environmental Laboratory Accreditation Program

Scott D. Siders
Accreditation Officer
Environmental Laboratory Accreditation Program

Certificate No.: 001234
Expiration Date: 04/30/2006
Issued On: 04/12/2005

State of Illinois
Environmental Protection Agency

Certificate No.: 001234

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.

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.

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)

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Drinking Water, Inorganic

USEPA150.1
Hydrogen ion (pH)

USEPA180.1
Turbidity

USEPA200.7R4.4

Aluminum	Arsenic	Barium
Beryllium	Cadmium	Calcium
Chromium	Copper	Hardness (calc.)
Iron	Magnesium	Manganese
Nickel	Silica	Silver
Sodium	Zinc	

USEPA200.9R2.2

Antimony	Arsenic	Cadmium
Chromium	Lead	Selenium
Silver	Thallium	

USEPA245.1R3.0
Mercury

USEPA300.0R2.1

Chloride	Fluoride	Nitrate
Nitrite	Orthophosphate	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	Antimony	Arsenic
Barium	Beryllium	Boron
Cadmium	Calcium	Chromium
Cobalt	Copper	Iron
Lead	Magnesium	Manganese
Molybdenum	Nickel	Potassium
Selenium	Silica	Silver
Sodium	Strontium	Thallium
Tin	Titanium	Vanadium
Zinc		

7041
Antimony

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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

STL Chicago
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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
Malathion	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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Hazardous and Solid Waste, Organic

8260B

1,1,1,2-Tetrachloroethane
1,1,1-Trichloroethane
1,1,2,2-Tetrachloroethane
1,1,2-Trichloroethane
1,1-Dichloroethane
1,1-Dichloroethene
1,1-Dichloropropene
1,2,3-Trichlorobenzene
1,2,3-Trichloropropane
1,2,4-Trichlorobenzene
1,2,4-Trimethylbenzene
1,2-Dibromo-3-chloropropane (DBCP)
1,2-Dibromoethane (EDB)
1,2-Dichlorobenzene
1,2-Dichloroethane
1,2-Dichloropropane
1,3,5-TCB
1,3,5-Trimethylbenzene
1,3-Dichlorobenzene
1,3-Dichloropropane
1,4-Dichlorobenzene
1-Chlorohexane
1-Chlorohexane
2,2-Dichloropropane
2-Butanone (Methyl ethyl ketone, MEK)
2-Chloro-1,3-butadiene (Chloroprene)
2-Chloroethyl vinyl ether
2-Chlorotoluene
2-Hexanone
2-Methyl-1-propanol (Isobutyl alcohol)
2-Methylnaphthalene
2-Nitropropane
4-Chlorotoluene
4-Methyl-2-pentanone (Methyl isobutyl ketone, I
Acetone
Acetonitrile
Acrolein (Propenal)
Acrylonitrile
Allyl chloride
Benzene
Bromobenzene
Bromochloromethane
Bromodichloromethane
Bromoform
Bromomethane
Carbon disulfide
Carbon tetrachloride
Chlorobenzene
Chlorodibromomethane (Dibromochloromethan
Chloroethane
Chloroform
Chloromethane
cis-1,2-Dichloroethene
cis-1,3-Dichloropropene
Dibromomethane
Dichlorodifluoromethane
Dichloromethane (Methylene chloride)
Ethyl acetate
Ethyl ether
Ethyl methacrylate
Ethylbenzene
Hexachlorobutadiene
Isopropyl ether
Isopropylbenzene
Malononitrile
Methacrylonitrile
Methyl ethyl ketone
Methyl iodide (Iodmethane)
Methyl isobutyl ketone
Methyl methacrylate
Methyl-t-butyl ether
m-Xylene
Naphthalene
n-Butylbenzene
n-Propylbenzene
o-Toluidine
o-Xylene
Pentachloroethane
p-Isopropyltoluene
Propionitrile (Ethyl cyanide)
p-Xylene
sec-Butylbenzene
Styrene
tert-Butylbenzene
Tetrachloroethene
Tetrahydrofuran
Toluene
trans-1,2-Dichloroethene
trans-1,3-Dichloropropene
trans-1,4-Dichloro-2-butene
Trichloroethene
Trichlorofluoromethane
Trichlorotrifluoroethane
Vinyl acetate
Vinyl chloride
Vinylidene chloride
Xylenes (Total)

8270C

1,2,4,5-Tetrachlorobenzene
1,2,4-Trichlorobenzene
1,2-Dichlorobenzene
1,2-Diphenylhydrazine
1,3,5-Trinitrobenzene (1,3,5-TNB)
1,3-Dichlorobenzene
1,3-Dinitrobenzene (1,3-DNB)
1,4-Dichlorobenzene
1,4-Dioxane
1,4-Naphthoquinone
1,4-Phenylenediamine
1-Naphthylamine
2,3,4,6-Tetrachlorophenol
2,4,5-Trichlorophenol
2,4,6-Trichlorophenol
2,4-Dichlorophenol
2,4-Dimethylphenol
2,4-Dinitrophenol
2,4-Dinitrotoluene (2,4-DNT)
2,6-Dichlorophenol
2,6-Dinitrotoluene (2,6-DNT)
2-Acetylaminofluorene
2-Chloronaphthalene
2-Chlorophenol
2-Methylnaphthalene
2-Methylpyridine (2-Picoline)
2-Naphthylamine
2-Nitroaniline
2-Nitrophenol
3,3'-Dichlorobenzidine
3,3'-Dimethylbenzidine
3-Methylcholanthrene
3-Nitroaniline
4,6-Dinitro-2-methylphenol
4-Aminobiphenyl
4-Bromophenyl phenyl ether

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Hazardous and Solid Waste, Organic

8270C

4-Chloroaniline
4-Nitrophenol
7,12-Dimethylbenz(a)anthracene
Acetophenone
Anthracene
Benzo(a)anthracene
Benzo(g,h,i)perlyene
Benzyl alcohol
Bis(2-chloroisopropyl) ether
Carbazole
Diallate
Diethyl phthalate
Di-n-octyl phthalate
Ethyl methanesulfonate
Hexachlorobenzene
Hexachloroethane
Indeno(1,2,3-cd) pyrene
m-Cresol (3-Methylphenol)
Methyl methanesulfonate
N-Nitrosodiethylamine
N-Nitrosodi-n-propylamine
N-Nitrosomorpholine
o-Cresol (2-Methylphenol)
p-Cresol (4-Methylphenol)
Pentachloronitrobenzene
Phenanthrene
Pronamide
Pyridine

4-Chlorophenyl phenyl ether
4-Nitroquinoline-1-oxide
Acenaphthene
alpha,alpha-Dimethylphenethylamine
Aramite
Benzo(a)pyrene
Benzo(k)fluoranthene
Bis(2-chloroethoxy) methane
Bis(2-ethylhexyl) phthalate
Chlorobenzilate
Dibenz(a,h)anthracene
Dimethyl phthalate
Dinoseb
Fluoranthene
Hexachlorobutadiene
Hexachlorophene
Isophorone
m-Dinitrobenzene
Naphthalene
N-Nitrosodimethylamine
N-Nitrosodiphenylamine
N-Nitrosopiperidine
o-Toluidine
p-Dimethylaminoazobenzene
Pentachlorophenol
Phenol
Pyrene
Safrole

4-Chloro-3-methylphenol
4-Nitroaniline
5-Nitro-o-toluidine
Acenaphthylene
Aniline
Benzidine
Benzo(b)fluoranthene
Benzoic acid
Bis(2-chloroethyl) ether
Butyl benzyl phthalate
Chrysene
Dibenzofuran
Di-n-butyl phthalate
Diphenylamine
Fluorene
Hexachlorocyclopentadiene
Hexachloropropene
Isosafrole
Methapyrilene
Nitrobenzene
N-Nitrosodi-n-butylamine (N-Nitrosodibutylam
N-Nitrosomethylethylamine
N-Nitrosopyrrolidine
Parathion
Pentachlorobenzene
Phenacetin
p-Phenylenediame
Pyridine

8310

Acenaphthene
Benzo(a)anthracene
Benzo(g,h,i)perylene
Dibenz(a,h)anthracene
Indeno(1,2,3-cd) pyrene
Pyrene

Acenaphthylene
Benzo(a)pyrene
Benzo(k)fluoranthene
Fluoranthene
Naphthalene

Anthracene
Benzo(b)fluoranthene
Chrysene
Fluorene
Phenanthrene

8330

1,3,5-Trinitrobenzene (1,3,5-TBN)
2,4-Dinitrotoluene (2,4-DNT)
4-Amino-2,6-dinitrotoluene (4-Am-DNT)
m-Nitrotoluene (3-Nitrotoluene, 3-NT)
o-Nitrotoluene (2-Nitrotoluene, 2-NT)

1,3-Dinitrobenzene (1,3-DNB)
2,6-Dinitrotoluene (2,6-DNT)
Hexahydro-1,3,5-trinitro-1,3,5-triazine (RDX)
Nitrobenzene
p-Nitrotoluene (4-Nitrotoluene, 4-NT)

2,4,6-Trinitrotoluene (2,4,6-TNT)
2-Amino-4,6-dinitrotoluene (2-Am-DNT)
Methyl-2,4,6-trinitrophenylnitramine (Tetryl)
Octahydro-1,3,5,7-tetranitro-1,3,5,7-tetrazocin

Wastewater, Inorganic

HACH8000

Chemical Oxygen Demand (COD)

SM3500Cr-D,18Ed

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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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Wastewater, Inorganic

USEPA218.2
Chromium

USEPA239.2
Lead

USEPA245.1
Mercury

USEPA270.2
Selenium

USEPA272.2
Silver

USEPA279.2
Thallium

USEPA300.0R2.1
Bromide
Nitrate
Orthophosphate (as P)

USEPA305.1
Acidity

USEPA310.1
Alkalinity

USEPA325.2
Chloride

USEPA330.4
Chlorine

USEPA335.1
Cyanide, Amenable

USEPA335.2
Cyanide

USEPA340.2
Fluoride

USEPA350.2
Ammonia

USEPA351.3
Total Kjeldahl Nitrogen

USEPA353.2
Nitrate (total)

USEPA354.1
Nitrite

USEPA360.1
Oxygen

USEPA365.2

USEPA213.2
Cadmium

Chloride
Nitrate-Nitrite (sum)
Sulfate

Fluoride
Nitrite

Nitrate-Nitrite (sum)

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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

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Wastewater, Organic

USEPA625

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

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

Title: LABORATORY QUALITY MANUAL

STL Chicago
2417 Bond Street
University Park, Illinois 60466-3182
(708) 534-5200

Approved by:	Signature:	Date
Michael J. Healy Laboratory Director	Michael J. Healy	7/6/05
Terese A. Preston Quality Manager	Terese A. Preston	7/08/05

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Full Signature Approvals Are Kept on File
with STL's QA Standard Practice Records

STL Chicago
STANDARD OPERATING PRACTICE (SOP) CHANGE FORM

Original SOP Number/Revision #: UQA-LQM /Rev No. 04 Last Mod ID (circle): NA / _A
SOP Title: STL Chicago Quality Manual
Affected SOP Section Number(s): 1.1
Effective Date: 09/26/05

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Revision Number with Mod ID:

The following SOP change is in effect as of the stated date. This form will remain attached to the referenced SOP until such a time that the SOP is updated, approved, and redistributed, at which time it will become part of the historical SOP record. **Append this form to the front of the SOP copy.**

1. Reason for SOP Change: Self-Declaration for DOD QSM Version 3.0

2. Summary of Procedure Change (circle to indicate if there are attachments to this form: No / Yes: # pages attached = ___)

The following shall be added to Section 1.1 of the LQM: "STL Chicago has reviewed the Department of Defense Quality Systems Manual (DoD QSM) and compared this document to the Laboratory Quality Manual and associated Standard Operating Procedures. STL Chicago has documented its level of compliance with the DoD QSM Standard and identified areas of specific variances between the laboratory's LQM, QSM and associated SOP's. The laboratory will work with all DoD clients to negotiate specific project requirements and data quality objectives, on a project-by-project basis. These requirements will reflect both the laboratory's Quality System and the requirements of the DoD QSM. Project-specific QAPP's will clearly identify and outline all negotiations and ultimate expectations for the specific project. "

Marcella D. Kinney 09/26/05
Initiated/Reviewed By: Name/Date

Initiated/Reviewed By: Name/Date

Jesse A. Preston 9/26/05
Approval Signature/Date: Section Manager

Michael J. Healy 9/26/05
Approval Signature/Date: QA Manager or Designee

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Appendix

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, soil, solid, waste and drinking water.

Associated with this activity are services to ensure client requirements are known, communicated and satisfactorily addressed, and a deliverables package presenting the analytical results meets client needs. 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)
- Department of Defense (DoD)
- Navy Facilities Engineering Service Center (NFESC)
- Clean Water Act (CWA)
- Comprehensive Environmental Response, Compensation, and Liability Act (CERCLA)
- 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 at www.stl-inc.com or available from the laboratory.

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 operations. 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 4.0, February 2005.

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, Final Version 3, March 2005

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 A List of Cited SOPs and Work Instructions Appendix B STL Chicago's Method Capability Listing Appendix C STL Chicago's Description and Floor Space for Analytical Facilities

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. 9601et 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 the method reporting limit or lowest calibration standard. Used in conjunction with the MRL Check standard in LCG 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 ±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 ±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 ½ the spike level used for the MDL Study or ½ the method reporting limit or ½ the lowest calibraton standard.

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 Organizational Chart

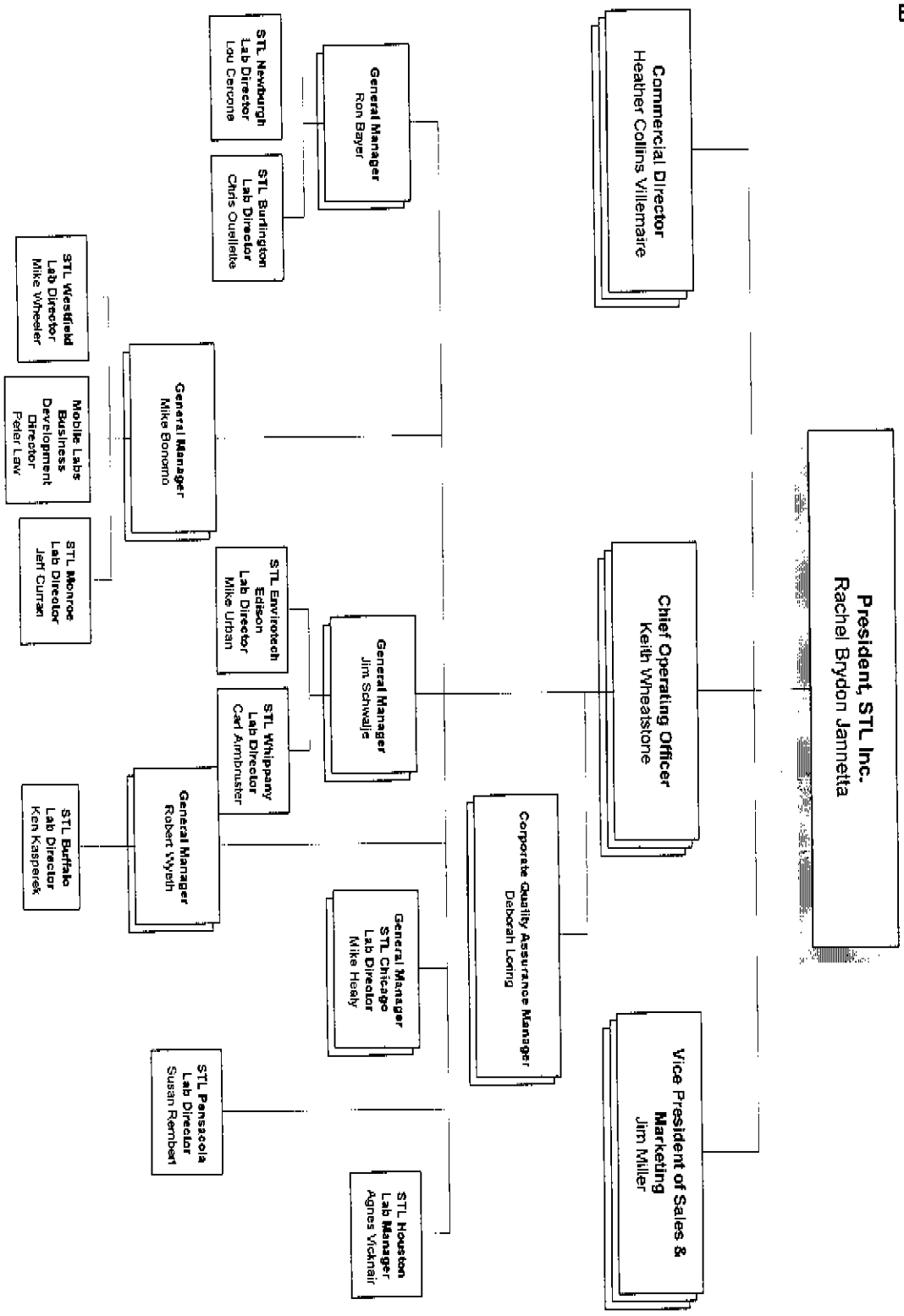


Figure 1. STL Organization Chart

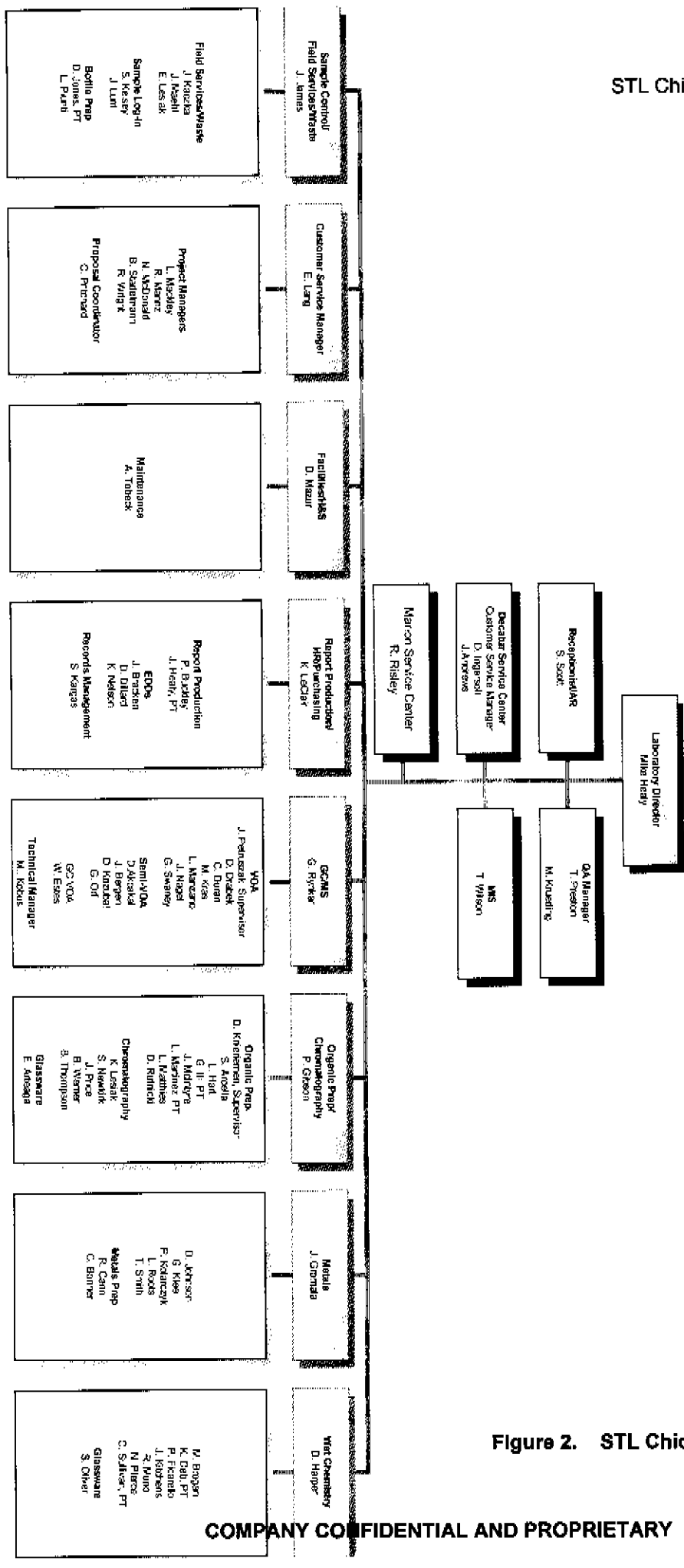


Figure 2. STL Chicago Organizational Chart

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 83 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/MS	GC	HPLC	ICP	ICPMS	AA	CVAA	AutoAnalyzer	IC	TOC	TOX
14	14	6	2	1	3	2	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 ensure 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 ensure 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, verification 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,
- 23 years laboratory experience.
- Terese A. Preston, Quality Assurance Manager, BA Biology,
- 21 years laboratory experience.
- Diane L. Harper, Inorganics Section Manager, MA Biology,
- 25 years laboratory experience.
- Jodi L. Gromala, Metals Section Manager, BS Biology,
- 18 years laboratory experience.
- Patti J. Gibson, Chromatography/Organic Extractions Section Manager, BS Biology,
- 16 years laboratory experience.
- Gary L. Rynkar, GC/MS Section Manager, BS Environmental Biology,
- 17 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 Coordinator

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 ensure 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 contain 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 parenthetic 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
- Ensure 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 yearly basis, which is tracked by an established review schedule (*Approved SOP Listing*; CHI-22-09-SOP). These reviews are conducted by the creator of the SOP and/or Department Manager, QA Specialist and/or QA Manager, Health and Safety Coordinator, and Lab Manager all of whom provide the approval signature for each SOP where appropriate to the subject of the 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 are 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". The laboratory also takes guidance from the STL Corporate MDL SOP (S-Q-003). 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 and/or low level check standard. 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 2-3x the respective MDL to ensure confidence in the value reported. Special project (i.e., ILEPA TACO limits; MIDEQ limits) or program (i.e., AFCEE; LCG; DoD) specific reporting limit requirements are routinely evaluated by the QA and project management staff. Every effort is made to meet project goals or objectives if it is within the laboratory's capability to do so within minimal risk to the quality of the data. Data evaluated below the RL 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. Refer to the laboratories MDL SOP (UQA-017) for additional tools that are used in the MDL evaluation process. If the laboratory continually demonstrates that the method reporting limits are not achieved, equipment, technique, and the method are reviewed to ensure 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, Lab Manager or Project 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 STL's corporate *Testing Solvents and Acids* procedure (S-T-001) for all of the STL laboratories. Notification of approval of specific lot numbers are sent via e-mail to the QA Manager, who subsequently forwards it to the facility manager. A listing of approved lot numbers is also available electronically on the STL Website under Corporate Information / Technology / Approved Solvent Spreadsheet.

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 (for those items indicated on the COC), the LabNet Sample Receipt Checklist and on a Sample Discrepancy Report (SDR) normally as a directed Job Note to the appropriate Project Manager; 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 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), a Customer Complaint Form (CHI-22-09-340) or in a format specifically designed for that purpose (e.g., phone conversation record or e-mail). The Laboratory Director, Project Manager, QA Manager, and Section 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 or electronic 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	- LQM - QMP (Corporate) - QAPPs - SOPs - Work Instructions	- Audits – Internal - Audits - External - Audit Responses - Certifications - PTs - SDR/RDRs - CARs - Review Checklists - Logbooks* - Standard Certificates - Method & Software Validation/Verification - MDL/IDL/IDOC Studies - Statistical Evaluations - Training Records - CDOC Evaluations - QA Reports - Electronic QA Files	- COC - Contracts & Amendments - Correspondence - QAPP - SAP - Telephone Logs - E-mails - Electronic Data - Data Report	- Accounting - Corporate Safety Manual - Permits - Disposal Records - Employee Handbook - Personnel files - Employee Signature & Initials Form -Technical & Administrative Policies

*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		Retention Requirement *
Raw Data	All*	10 Years from completion (Electronic Data Reports - .pdf & EDD) 5 Years from completion for Hardcopy when not available in electronic form 5 Years from archival for electronic raw data
Controlled Documents	All*	5 Years from document retirement date
QC Records	All*	5 Years from archival
Project Records	All*	5 Years from project completion
Administrative Records	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
Specific Client Program / Project	Per contractual requirement

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	QA Department or Designee	**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 approximately the 3rd day of the month, the QA manager prepares a monthly QA report. The report is sent to the Laboratory Director, Project Managers, Section Managers and Department Supervisors. At a minimum, the report content contains the items listed below (Figure 3). A separate report is prepared for and submitted to the Corporate Quality Control Director and the Lab Director. This report contains a narrative summary which includes audit details; revised report details; client complaints; certifications/approvals; preventive actions; QA Highlights and QA Lowlights. Also included are the monthly metrics spreadsheet, PT summary and External Audit Summary. 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. STL Chicago Monthly QA Report Format

1	Audits
	A. External System Audits B. Internal System Audits C. Internal Data Audits
2	Revised Reports / Client Complaints / Client Compliments
	A. Revised Reports (RDR) B. Customer Complaints C. Customer Compliments
3	Certification Changes
	A. Certification Status B. Certified Parameter List
4	Proficiency Testing
	A. PES Results/Scores B. PES Failure Summary C. PES History of Non-Acceptable Scored Analyte/Compound
5	Miscellaneous QA and Operational Issues
	A. Current SOP Status (with 'on-time' percentages calculated for SOPs < 1 year) B. Listing of SOPs > 1 Yr C. Listing of SOPs in Progress
6	QAPP/Project Review Status
7	Holding Time Violations
8	Monthly QA Report Metrics

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, H&S officer, and Section Manager/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. Each administrative/non-technical section also maintains training records for each employee. All Training Records are also kept on file in the QA Department for periodic review with the appropriate Section Manager/Supervisor. 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 Health & Safety officer maintains training documentation related to H&S issues.

The QA Department maintains the following evidence items on file for each employee:

- An Ethics Agreement signed by each staff member (renewed each year). (Figure 6)
- A Confidentiality Agreement signed by each staff member (renewed each year).
- Representative Signature and Initials by each staff member (renewed each year).
- The employee has read and understood the latest, approved version of all test methods and/or SOPs for which the employee is responsible (annual review/sign-off of SOP revisions).
- A training record specific to the job functions performed.
- Copy of external Training seminars or class completion certificates.

The following evidence items are on file (in addition to those listed above) for each technical employee:

- Initial Demonstration of Capability (IDOC) for each method. (CHI-22-09-271) (Figure 4)
- Annual evidence of Continued Demonstration of Capability (CDOC) for each method (CHI-22-09-243) (Figure 5)

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 (Figure 4) is recorded and maintained in the analyst'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 analyst's 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). When an analyst has not yet completed the IDOC requirement, they can perform a task under the supervision of a qualified analyst, 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.

On an annual basis, the analyst's method capabilities must be evaluated, which may include, but is not limited to, successful analysis of a blind sample on the specific test method (PT) or a similar test method; an annual DOC of four successive and acceptable LCSs; Control Chart Evaluations over a given time period. The QA Department in conjunction with the appropriate Section Manager will accumulate specific required information to satisfy the CDOC (Continued Demonstration of Capability) requirement. Documentation will be filed within the analyst training file. *Figure 5* shows an example of a *Continued Demonstration of Method Proficiency* statement (CHI-22-09-243).

Although training is a continual process, initial training is considered complete once the trainee has attended the initial general orientation(includes specific forms to be reviewed and signed, Timesheet Training, Employee Handbook, Drug Policy Form, Ethics/Confidentiality forms, Internet and E-Mail Usage, IT Policy Form, Benefit Info), presentations (ex. Ethics Orientation and Comprehensive Training, QA Orientation-including Manual Integration and Selection of Calibration Points for technical staff, Health & Safety Orientation), and review of those SOP's applicable to the employee's responsibilities. Documentation is appropriate to the training item. Specific training related to the department is assessed and documented within the employee's training record, which is updated over the course of the employee's training progress. This process is applicable to both Technical and Non-Technical employees.

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
Employee Orientation / HR	Week 1	All
Ethics – Corporate Overview	Week 1	All
Environmental Health & Safety	Month 1	All
Ethics Data Integrity	Month 1 Month 1	All Technical and PMs
Ethics Refresher	Annually	All
Quality Assurance	Quarter 1	All
Initial Demonstration of Capability (IDOC)	Prior to unsupervised method Performance	Technical
Continued Demonstration of Capability (CDOC)	Annually	Technical

[1] From the date of initial employment unless otherwise indicated.

The Ethics, Data Integrity and 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.

Further details of the laboratory's training program are described in the Laboratory Training SOP (UQA-014).

Figure 4. Initial Demonstration of Capability Certification Statement

STL Chicago
Initial Demonstration of Method Capability
Certification Statement

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.

_____ _____
Supervisor/Manager Signature Date

_____ _____
QA Signature Date

Figure 5. Continued Demonstration of Method Proficiency

STL Chicago
Continued Demonstration of Method Proficiency

Analyst Name:_____
SOP No.:_____
Analytical Method:_____
Similar Test Methods:_____
Analyte(s):_____

Documentation of Continued Proficiency

Continued Proficiency has been demonstrated by one of the following:

_____ 1. Successful analysis of a blind performance sample (blind to the analyst) on a similar test method using the same technology (Documentation required for one of the test methods).
PT-IS(s): _____
(See attached PT Summary)
_____ 2. Another demonstration of capability.
Description: _____
_____
_____ 3. Successful analysis of a blind performance sample (double-blind to the analyst/QA) on a similar test method using the same technology (Documentation required for one of the test methods).
PT Description: _____ Job#: _____
_____ 4. At least 4 consecutive laboratory control samples with acceptable levels of precision and accuracy.
(See Attached Control Chart)
_____ 5. If Items 1-4 cannot be performed, analysis of authentic samples with results statistically indistinguishable from those obtained by another analyst.

The analyst identified above, using the cited method(s) which is in use at this laboratory and defined within the laboratory's document control system, has read, understood and agrees to perform this most recent version of the test method.

_____ _____
Analyst Signature Date

_____ _____
QA 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 6). 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 6. 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 contact 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 locked at all times with controlled and documented access. Access is controlled by various measures including locked doors, electronic access cards, security codes, and a staffed reception area 8:00 a.m. to 5:00 p.m. Monday through Friday. All visitors sign in and are escorted by STL personnel while at the facility.

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.. Refer to the description of floor space in Appendix C for additional details.

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.

STL Chicago maintains an updated list of all current primary and secondary accreditations. This information is available through the STL web-site (http://stlnet.stl-inc.com). The web-site contains links to all certifications and methods for which the laboratory is currently accredited. In addition, a listing of STL Chicago's Method Capabilities appears in Appendix B *(Methods Capabilities Work Instruction (CHI-22-09-255)*. The table also identifies those methods for which NELAP accreditation is offered and for which the laboratory holds NELAP certification. Certifications are subject to change, and may do so based on laboratory needs and performance. All certifications must be confirmed with appropriate laboratory personnel.

5.3.1 Method Selection

Since numerous methods and analytical techniques are available, continued communication between the client and laboratory is imperative to ensure 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 7).

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
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 7).

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. Where an SOP is based on a published method, the laboratory maintains a copy of the reference method.

Figure 7. Proprietary Information Statement

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 ensure 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).

Validation

Validation is the process of establishing evidence that provides a high degree of assurance that a specific process will consistently produce a product meeting pre-determined specifications and user needs. Software validation involves documentation of original specifications, identity of code, printout of code, software name, software version and any other specific procedures outline in the manufacturers Validation Process. Most often, this documentation exists as a Software Validation Certificate, obtainable from the appropriate manufacturer. Per STL *Policy S-ITQ-007 Software Testing, Verification and Validation*, purchased software that has not been modified at the source code level is not required to be internally validated. As specified in STL Chicago's SOP *UIS-006 Procedures and Processes Related to Entry, Storage, Back-up/Retrieval and Management of Bench Level Electronic Data*, all software related to instrument data gathering was installed in its entirety, with no changes made to base codes or algorithms. Where possible, Software Validation Certificates have been obtained, and are filed within the QA Department.

The Validation of the LabNet system, STL's proprietary LIM's and STL Chicago's end-processing and reporting system, was completed both as a corporate initiative and at STL Chicago during the implementation of the system. The system 'methods' applicable to STL Chicago, containing the algorithms and formulas were tested and documented by the QA Department. Results of this initiative are documented and kept on file in the QA Department.

Verification

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 initial verification of LabNet software programs were conducted by the QA Department with the assistance of the section managers and appropriate personnel. The QA Department also documents the approval of, and verifies, any program modifications. All records of the verification are retained in the QA Department.

Verification of instrumental software was also completed at the time of implementation, either by way of manual comparison to computer generated data or comparison to data generated by the previous system being replaced. Documentation of the most recent systems of all verification procedures is on file in the QA Department. Additionally, an Instrument Validation Checklist (Figure 8, CHI-22-09-286) is provided to each department, which includes a section outlining Software Verification Requirements and both the process and location of such documentation for newly installed systems.

EDD validation and verification is discussed in STL Chicago's SOP *UIS-001 EDD Specifications, Development Generation and Review.*

The above procedures do not apply to general purpose software, except where those applications are used to perform calculations in support of client data. In those cases, verification will be required.

Figure 8: Instrument Validation Checklist

STL-CHICAGO
INSTRUMENT VALIDATON CHECKLIST

Instrument Type: _____
Model#:_____
Serial #:_____
Lab Equip Code: _____ LIMS Equip Code: _____

Installation Date: _____
Installed By: _____

Instrument installed per specifications. Operational and functional per install guidelines.
Signature/Date of installer: _____

Outstanding items yet to be completed (If applicable):_____
_____
_____
_____

Completion Date: _____
Signature/Date Lab Representative: _____

Instrument passes all initial required lab checks and calibrations as appropriate
for method of analysis: _____

Appropriate MDL's as applicable per method complete: _____
*MDL's must be forwarded to the QA Dept. for Validation to be considered complete.

Methods to be analyzed (may change over time): _____
_____

In-Service Date: _____

Signature/Date of Lab Representative: _____
Signature/Date of QA Representative: _____
****

QA Only
Software Verification Required (Y/N)
Verification Completed as Described: _____
_____
_____
_____
Software Verification Documentation Location:_____
Signature/Date of QA Representative: _____

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 (Maintenance Logs may be hard-copy bound or electronic).

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 (hard-copy bound or electronic CHI-22-09-341) 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.

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	Daily
	Replace windows	As required
	Check or change cuvette	Daily
	Check & drain compressor drain	Daily
	Clean atomizer cell/furnace hood	Daily
	Nebulizer cleaned/dried	Weekly or as required
	Check/change marble stones	Weekly
	Clean filters	Weekly
	Change graphite tube/platform	As required
	Empty waste container	Daily
	Remove carbon tube and check wear	Daily
	Check sample introduction probe	Daily
Leeman Mercury Analyzer	Check tubing for wear	Daily
	Fill rinse tank with 10% HCl	Daily
	Insert clean drying tube filled with Magnesium Perchlorate	Daily
	Fill reductant bottle with 10% Stannous Chloride	Daily
ICP	Check pump tubing	Daily
	Check liquid argon supply	Daily
	Check fluid level in waste container	Daily
	Check filters	Weekly
	Clean or replace filters	As required
	Check torch	Daily
	Check sample spray chamber for debris	Monthly
	Clean and align nebulizer	Monthly
	Check entrance slit for debris	Monthly
	Change printer ribbon	As required
	Replace pump tubing	As required
ICP MS	Change pump tubing	Weekly
	Clean torch	Weekly
	Check / clean nebulizer	Weekly
	Clean cones	Daily
	Check air filters	Weekly
	Check multiplier voltages & do cross calibration	Weekly
	Replace sample uptake tubing	Monthly
	Check rotary pump oil	Monthly
	Check oil mist filters	Monthly
	Check chiller water level	Monthly
UV-Vis Spectrophotometer	Clean ambient flow cell	As required
	Precision check/alignment of flow cell	As required
	Wavelength verification check	Semi-annually

Table 9. Major Equipment Maintenance

Instrument	Procedure	Frequency
Auto Analyzers	Clean sampler	Daily
	Check all tubing	Daily
	Clean inside of colorimeter	Daily
	Clean pump well and pump rollers	Quarterly
	Clean wash fluid receptacle	Weekly
	Oil rollers/chains/side rails	Weekly
	Clean optics and cells	Quarterly
Hewlett Packard GC/MS	Ion gauge tube degassing	As required
	Pump oil-level check	Monthly
	Pump oil changing	Annually
	Analyzer bake-out	As required
	Analyzer cleaning	As required
	Resolution adjustment	As required
	COMPUTER SYSTEM AND PRINTER:	
	Air filter cleaning	As required
	Change data system air filter	As required
	Printer head carriage lubrication	As required
	Paper sprocket cleaning	As required
	Drive belt lubrication	As required
Gas Chromatograph	Compare standard response to previous day or since last initial calibration	Daily
	Check carrier gas flow rate in column	Daily via use of known compound retention
	Check temp. of detector, inlet, column oven	Daily
	Septum replacement	As required
	Check system for gas leaks with SNOOP	W/cylinder change as required
		Monthly
	Check for loose/frayed wires and insulation	As Required
	½"Bake injector/column	As Required
	Change/remove sections of guard column	As Required
	Replace connectors/liners	As Required
	Change/replace column(s)	
Electron Capture Detector (ECD)	Detector wipe test (Ni-63)	Semi-annually
	Detector cleaning	As required
Flame Ionization Detector (FID)	Detector cleaning	As required
Flame Photoionization Detector (FPD)	Clean and/or Replace Lamp	As required
Photoionization Detector (PID)	Change O-rings	As required
	Clean lamp window	As required
HPLC	Change guard columns	As required
	Change lamps	As required
	Change pump seals	Semi-annually or as required
	Replace tubing	As required
	Change fuses in power supply	As required
	Filter all samples	Daily
	Change autosampler rotor/stator	As required

Table 9. Major Equipment Maintenance

Instrument	Procedure	Frequency
Balances	Class "S" traceable weight check Clean pan and check if level Field service	Daily, when used Daily At least Annually
Conductivity Meter	0.01M KCl calibration Conductivity cell cleaning	Daily As required
Turbidimeter	Check light bulb	Daily, when used
Deionized/Distilled Water	Conductivity Point Sources Daily conductivity check Check deionizer light Monitor for VOA's System cleaning Replace cartridge & large mixed bed resins	Water Quality SOP UQA-035 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, DoD, 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	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. 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.
	Continuing Calibration	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., ± 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.
Atomic Absorption (GFAA/ CVAA)	Initial Calibration	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 (GFAA) 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. An ICV standard will be analyzed immediately after standardization. The ICV must be within SOP-specified criteria (e.g., ±5% of the true value for drinking water, and ±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. 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.

Table 10. Minimum Instrument Calibration Procedures

Technique	Activity	Minimum Requirements
	Continuing Calibration	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., ±10% recovery of the true value except for mercury within ±20 % of the true value). The CCB must be free of target analytes at or above the concentration reported in samples. If any CCV or CCB exceed their acceptance criteria, corrective action must be taken.
Inorganic Colorimetric Methods	Initial Calibration	An 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 linear range of the method, plus a calibration blank. At least one of the calibration standards will be at a concentration which will enable verification of instrument response at 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. In lieu of an initial curve, a daily calibration verification check may be analyzed. This calibration check 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. 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. 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.
	Continuing Calibration	The initial calibration is verified after every 10 readings and at the end of the analytical shift, with the analysis of a continuing calibration verification standard (CCV) and a blank (CCB). If any CCV or CCB exceed SOP-specified acceptance criteria, appropriate corrective action is taken per SOP. All samples since the last valid calibration verification check are reanalyzed.
Ion Chromato-graphy	Initial Calibration	The ion chromatograph will be calibrated approximately monthly or when any significant change is made to the system. Calibration standards will be prepared from appropriate reference materials and will include a blank and a minimum of three concentrations to cover the linear range of the instrument. At least one of the calibration standards will be at a concentration which will enable verification of instrument response at 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. An ICV will be analyzed on a daily basis, prior to sample analysis and 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.

Table 10. Minimum Instrument Calibration Procedures

Technique	Activity	Minimum Requirements
	Continuing Calibration	The initial calibration is verified after every 10 readings and at the end of the analytical shift, with the analysis of a continuing calibration verification standard (CCV) and a blank (CCB). If any CCV or CCB exceed SOP-specified acceptance criteria, appropriate corrective action is taken per SOP. All samples since the last valid calibration verification check are reanalyzed.
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	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. 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 or BFB. For wastewater programs (600 series methods), the tune expires after 24 hours. Ion abundances will be within the windows dictated by the specific program requirements.
	Initial Calibration	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. 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. 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.
	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.

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	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. Within the analysis sequence, instrument drift will be monitored by analysis of a mid-range calibration standard of varying concentrations 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.

5.5 Measurement Traceability

5.5.1 General

Traceability of measurements is ensured 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/eppendorf 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).

The main DI/RO units are located in a separate area. These include both a DI and RO system. The DI/RO System is connected by modem to the company who maintains the system for STL Chicago (Crossbow Industrial Water). Additionally, there are several Milli-Q Units, described below, which draw directly from this system, in several of the laboratory areas. There are also DI water feeds in each laboratory. The following checks and maintenance are followed.

Daily

Main System

Daily Conductivity Check- check set point
This is recorded in a log kept near the system
Deionizer Light
General Inspection for leaks etc..
Work Instruction for Alarm Re-Setting Posted Near System

Crossbow conducts a thorough monthly check of both systems and provides a full report to STL Chicago. This includes flow and pressure checks, DI Low Alarm Check, RO low pressure switch, pumps and valves, Total Hardness and Cl2 level checks, Control Circuits and finally an entire system check for damage and corrosion. These reports are filed.

Milli-Q Units

Milli-Q Units are located in the GC/MS VOA, Metals, Wet Chem and Extraction Laboratories. These units also contain check point settings. These settings are checked daily, prior to use, by the appropriate laboratory personnel, prior to using the unit to provide water for method blanks or other uses. If the units are not operating at the appropriate set point level, the Facility Manager is called and the appropriate corrective action is taken (change filter on unit, check main DI/RO unit etc...). In this sense, all units are being checked on a daily basis for proper operation and samples of the water being analyzed.

Point Source Checks for Specific Conductivity and pH

The following water point sources are checked on a weekly basis, as they are the first outlet from the water source. This has proven to be an adequate representation of the water used by the lab:

TCLP Laboratory-DI
Metals Digestion- Milli-Q

The following are checked on a quarterly basis:

TCLP Laboratory-DI
Wet Chemistry: General Lab-DI
We Chemistry: Instrumentation Laboratory-DI
Organic Extractions: Dishroom-DI
Organic Extractions-Milli-Q
Metals Digestion-Milli-Q
Wet Chemistry-Milli-Q
GC/MS Volatiles Laboratory-Milli-Q

These procedures and documentation are described in STL Chicago Water Quality SOP UQA-035. The description, as above, will be added to the next revision of the Laboratory Quality Manual LQM.

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.

References 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 $\pm 2^{\circ}C$ (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 ensure 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 or Job Note 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 $4 \pm 2^{\circ}C$. 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. If sub-sampling is required at the login stage it is done with guidance and instruction from the project manager.

General sub-sampling procedure in the laboratory calls for a thorough mixing of the sample within the sample container or to transfer the sample to another suitable container from which a representative sub-sample can be taken to achieve the required sample weight. Any non-homogenous looking material is avoided and noted as such within the sample preparation record. Refer to individual preparation SOPs for additional details.

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 participates in 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	Organics: 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. Inorganics: 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.**
Laboratory Control Sample (LCS)	Use	Measures the accuracy of the method in a blank matrix and assesses method performance independent of potential field sample matrix affects.
	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.

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
		Inorganics
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 ensures that any potential contamination is less than the reporting limit.
ICP Interference Check Samples (ICSA/ICSB)	Sequence Use	Analyzed immediately after the ICV. 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 $\geq$ 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
		Organics
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 ≤ 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), Automated Data Review (ADR), Enviro Data, EQUIS, GISKEY, Excel, Access 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 electronic data 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 initialed and dated by the programmer and kept on file.

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.

6.0 ATTACHMENTS

Appendix A: List of Cited SOPs and Work Instructions
Appendix B: Methods Capabilities Work Instruction
Appendix C: Description and Floor Space for Analytical Facilities

Historical File:		Revision 00: 01/29/99		
		Revision 01: 12/15/00		
		Revision 02: 09/10/02		
		Revision 03: 06/07/04		
		Revision 04: 07/05/05		

Reason for Change: Revision 04:

- Update section 3.0 – Terms & Definitions: Clarification on DLCK and MDLCK and MRL definition
- Update section 4.1 – Organization Chart
- Update section 4.1.1 – Table 3. Major Equipment List
- Update section 4.1.2.4 – Technical Manager's experience level
- Update section 4.4.3.6 – Additonal DQOs: MDL-Reference to Corporate MDL SOP added; RL – Reference to Lab MDL SOP added
- Update section 4.6.1 – Solvent Lot Testing notification / posting location added
- Update section 4.12.2 – Table 4 and 5 – STL Record Types and Record Retention
- Update section 4.15.3 – Monthly QA Report
- Update section 4.7.1 – clarification on documentation practices for compromised samples
- Update section 5.1.2 – Training text updated to include non-technical training, defines what constitutes 'initial training/orientation' and defines who is considered to be a 'qualified analyst'. Basic re-organization of this section and addition of the Continued Demonstration of Method Proficiency form.
- Update Section 5.3 – Test Methods to include discussion and a link to the STL web-site regarding STL Chicago's Methods Capability Listing which was added as Appendix B
- Update/Clarify section 5.3.7 – Verification and Validation discussion; Addition of Instrument Validation Checklist.
- Update section 5.4.2 – Major Equipment maintenance updated to incorporate current electronic maintenance documentation practices also updated to include Conductivity Point Sources and Daily Conductivity Check – referencing the Lab's Water Quality SOP
- Update Table 9 to include ICP MS and DI/RO system text
- Update section 5.5.1 – Measurement Traceability: Discussion added regarding DI/RO systems, Milli-Q Units; Point Source Checks for Specific Conductivity and pH. Added reference to the lab's Water Quality SOP
- Update section 5.9.5 – EDD Discussion
- Addition of Section 6 – Attachments A, B and C
- General Text Clarifications

Appendix A. List of Cited SOPs and Work Instructions

Cited Sec. No(s)	Description	Document No.
1.5	Corporate Quality Management Plan (QMP)	QMP
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
4.4.3.6; 5.3.5	MDL Policy	S-Q-003
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 Report (SDR) / Resubmitted Data Request (RDR) / Corrective Action Report (CAR)	UQA-029
4.8; 4.11	Quality Systems Management Review	UQA-002
4.8	Customer Complaint Form	CHI-22-09-340
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	Initial Demonstration of Capability Certification Statement	CHI-22-09-271
5.1.2	Continued Demonstration of Method Performance	CHI-22-09-243
5.1.2	Training Program: Mechanisms and Documentation Processes Defined by Operational Assessment	UQA-014
5.1.3	Ethics Policy	P-L-006
5.3; 5.3.1	Work Instruction: Methods Capabilities	CHI-22-09-255
5.3.2	SOP Change Protocol	UQA-032
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
5.3.7	Work Instruction: Instrument Validation Checklist	CHI-22-09-286

Appendix A. List of Cited SOPs and Work Instructions

Cited Sec. No(s)	Description	Document No.
5.3.7	Software Testing, Verification & Validation	S-ITQ-007
5.3.7	Procedures & Processes Related to Entry, Storage, Back-up/Retrieval and Management of Bench Level Electronic Data	UIS-006
5.3.7; 5.9.5	EDD Specifications, Development, Generation & Review	UIS-001
5.4.1	Work Instruction: Equipment Tracking Form	CHI-22-09-068
5.4.2	Instrument Tracking Spreadsheet / Maintenance Log	CHI-22-09-341
5.4.2	Instrument and Equipment Out-of-Service Tagging.	UQA-012
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
Table 9; 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; 5.9.6	Data Management: Process Operation	UDM-001

Appendix B. STL Chicago's Methods Capability Listing

STL Chicago
Methods Capability Listing

SEVERN TRENT STL

Unit	Parameter	Method No.	NELAP	Matrix
GCE	Pesticides/PCBs	OLC02.1		W
GCE	Pesticides/PCBs	OLM04.2		W/S
GCE	Pesticides/PCBs	EPA 608	X	W
GCE	Organochlorine Pesticides	SW 8081A	X	W/S
GCE	PCBs	SW 8082	X	W/S
GCE	Organophosphorus Pesticides by GC	SW 8141A	X	W/S
GCE	Chlorinated Herbicides by GC	SW 8151A	X	W/S
GCV	Petroleum Hydrocarbons (DRO)	SW 8015B	X	W/S
GCV	Petroleum Hydrocarbons (GRO)	SW 8015B	X	W/S
HPLC	PAHs by HPLC	E 610	X	W
HPLC	PAHs by HPLC	SW 8310	X	W/S
HPLC	Explosives	SW 8330	X	W/S
M	GFAA Silver	SW 7761	X	W
M	GFAA Arsenic	SW 7060A	X	W/S
M	GFAA Cadmium	SW 7131A	X	W/S
M	GFAA Chromium	SW 7191	X	W/S
M	GFAA Lead	SW 7421	X	W/S
M	GFAA Antimony	SW 7041	X	W/S
M	GFAA Selenium	SW 7740	X	W/S
M	GFAA Thallium	SW 7841	X	W/S
M	GFAA Silver	E 272.2	X	W
M	GFAA Arsenic	E 206.2	X	W
M	GFAA Cadmium	E 213.2	X	W
M	GFAA Chromium	E 218.2	X	W
M	GFAA Lead	E 239.2	X	W
M	GFAA Antimony	E 204.2	X	W
M	GFAA Selenium	E 270.2	X	W
M	GFAA Thallium	E 279.2	X	W
M	GFAA Metals As, Cd, Pb, Sb, Se, Tl, Cr, Ag	E 200.9	X	W
M	Hardness	E 200.7	X	W
M	ICP Metals	E 200.7	X	W

M	CVAA Mercury	SW 7470A	X	W
M	CVAA Mercury	SW 7471A	X	S
M	CVAA Mercury	E 245.1	X	W
M	ICP Metals	SW 6010B	X	W/S
M	Metals-GFAA (As, Pb, Se, Tl)	ILM04.0		W/S
M	Metals-ICP	ILM04.0		W/S
M	Metals-Mercury	ILM04.0		W/S
MSB	GC/MS Semi-Volatiles	E 625	X	W
MSB	GC/MS Semi-Volatiles	SW 8270C	X	W/S
MSB	GC/MS Semi-Volatiles	SW 8270C (SIM)		W
MSB	GC/MS Semi-Volatiles	OLM04.2		W/S
MSB	GC/MS Semi-Volatiles	OLC02.1		W
MSV	VOAs by GC/MS	E 624	X	W
MSV	GC/MS Volatiles	SW 8260B	X	W/S
MSV	GC/MS Volatiles	OLM04.2		W/S
MSV	GC/MS Volatiles	OLC02.1		W
P	GC/MS Soil VOAs in EnCore Samples	SW 5035		S
P	California W.E.T. Test	CA Title 22		S
P	TCLP	SW 1311	X	S
P	SPLP	SW 1312	X	S
P	Extractable Organics; Accel. Liq.-Liq. Waters	SW 3520C		W
P	Extractable Organics; Separatory Funnel	SW 3510C		W
P	Extractable Organics; Accel. Soxhlet	SW 3541A		S
P	Extractable Organics; Sonication	SW 3550B		S
P	Acid Cleanup	SW 3665A		W/S
P	Alumina Cleanup	SW 3610B		W/S
P	Florisil Clean-up	SW 3620B		W/S
P	Gel Permeation Column Clean-up	SW 3640B		S
P	Sulfur Clean-up	SW 3660B		W/S
P	Waste Dilution	SW 3580A		S
P	Metals Digestions; Surface/Ground Water for ICP	SW 3005A		W
P	Metals Digestions; Waters/Extracts for ICP	SW 3010A		W
P	Metals Digestion; Waters/Extracts for GFAA (except As & Se)	SW 3020A		W
P	Metals Digestion; Waters/Extracts for GFAA	SW 3020A (M)		W
P	Metals Digestions; Soils/Wastes for ICP/GFAA	SW 3050B		S

P	Metals Digestions; Waters for As by GFAA	SW 7060		W
P	Metals Digestions; Waters for Se by GFAA	SW 7740		W
W	Alkalinity	EPA 310.1	X	W/S
W	Alkalinity	SM 2320B	X	W/S
W	Ammonia - Nessl.	EPA 350.2	X	W/S
W	Ammonia - Nessl.	SM 4500NH3C		W/S
W	BOD - 5 Day	EPA 405.1	X	W
W	BOD - 5 Day	SM 5210B	X	W
W	Bromide, IC	EPA 300.0	X	W
W	Bromide, IC	SW-846 9056	X	W/S
W	Bromide, IC	SM 4110B		W
W	Carbonaceous BOD	SM 5210B	X	W
W	Chloride, Lachat	EPA 325.2	X	W/S
W	Chloride, Lachat	SM 4500ClE		W/S
W	Chloride, Lachat	SW-846 9251	X	W/S
W	Chloride, IC	EPA 300.0	X	W
W	Chloride, IC	SW-846 9056	X	W/S
W	Chloride, IC	SM 4110B		W
W	Chlorine, Residual	EPA 330.4	X	W
W	Chlorine, Residual	SM 4500 Cl F	X	W
W	COD - High Level	HACH 8000	X	W/S
W	COD - Low Level	HACH 8000	X	W/S
W	Chromium, Hexavalent	SM 3500-CrD	X	W/S
W	Chromium, Hexavalent	SW-846 3060A/7196A	X	W/S
W	Cyanide, Amenable	EPA 335.1	X	W/S
W	Cyanide, Amenable	SM 4500CN G		W/S
W	Cyanide	EPA 335.2	X	W/S
W	Cyanide	SW-846 9010B/9014	X	W/S
W	Cyanide	SM 4500CN C, E	X	W/S
W	Cyanide	ILM04.0		W/S
W	Ferrous Iron	SM 3500 Fe D		W/S
W	Flashpoint	SW-846 1010	X	W/S
W	Fluoride / Fluorine	EPA 340.2	X	W/S
W	Fluoride / Fluorine	SM 4500F C	X	W/S
W	Fluoride, IC	EPA 300.0	X	W
W	Fluoride, IC	SW-846 9056	X	W

W	Langlier Index	SM 2330A+B	X	W/S
W	Nitrate-NO2 (LACHAT)	EPA 353.2	X	W/S
W	Nitrate-NO2 (LACHAT)	SM 4500NO3F	X	W/S
W	Nitrate, IC	EPA 300.0	X	W
W	Nitrate, IC	SW-846 9056	X	W
W	Nitrate, IC	SM 4110B		W
W	Nitrite	EPA 354.1	X	W/S
W	Nitrite	SM 4500NO2B	X	W/S
W	Nitrite, IC	EPA 300.0	X	W
W	Nitrite, IC	SW-846 9056	X	W
W	Nitrite, IC	SM 4110B		W
W	Oil & Grease	E 1664	X	W
W	Oil & Grease (Soil-Soxhlet)	SW-846 9071B	X	W/S
W	Oxygen, Dissolved	EPA 360.1	X	W
W	Oxygen, Dissolved	SM 4500 O C, G		W
W	pH - Low/High	EPA 150.1	X	W
W	pH - Low/High	SM 4500H+B	X	W
W	pH - Low/High	SW-846 9045C / 9040B	X	W/S
W	Paint Filter	SW-846 9095	X	W
W	Phenol (LACHAT)	EPA 420.2	X	W/S
W	Phenol (LACHAT)	SW-846 9066	X	W/S
W	Phosphate, Ortho	EPA 365.2	X	W/S
W	Phosphate, Ortho	SM 4500 PE	X	W/S
W	Phosphate, Ortho , IC	EPA 300.0		W
W	Phosphate, Ortho , IC	SW-846 9056	X	W
W	Phosphate, Ortho , IC	SM 4110B		W
W	Phosphorus	EPA 365.2	X	W/S
W	Phosphorus	SM 4500 PE		W/S
W	Specific Conductance	EPA 120.1	X	W
W	Specific Conductance	SM 2510B	X	W
W	Specific Conductance	SW-846 9050A	X	W/S
W	Specific Gravity	ASTM D2710F		W/S
W	Sulfate / Sulfur - Turbidimetric	EPA 375.4M	X	W/S
W	Sulfate - Turbidimetric	SM 4500SO4E		W/S
W	Sulfate - Turbidimetric	SW-846 9038M	X	W/S
W	Sulfate, IC	EPA 300.0	X	W

W	Sulfate, IC	SW-846 9056	X	W
W	Sulfate, IC	SM 4110B		W
W	Sulfide	EPA 376.1	X	W/S
W	Sulfide	SM 4500SE		W/S
W	Sulfide	SW-846 9030B/9034	X	W/S
W	Sulfide, Reactive	SW 7.3.4.2	X	W/S
W	TDS (Total Dissolved Solids)	EPA 160.1	X	W
W	TDS (Total Dissolved Solids)	SM 2540C	X	W
W	TKN - Nesslerization	EPA 351.3	X	W/S
W	TKN - Nesslerization	SM 4500NorgC		W/S
W	TOC (TIC/DIC) [Organic Carbon; Inorganic & Dissolved]	EPA 415.1	X	W
W	TOC (TIC/DIC) [Organic Carbon; Inorganic & Dissolved]	SM 5310C	X	W
W	TOC (TIC/DIC) [Organic Carbon; Inorganic & Dissolved]	SW-846 9060	X	W
W	TOC (TIC/DIC) [Organic Carbon; Inorganic & Dissolved]	Lloyd Kahn		S
W	TOX (Total Organic Halogens)	SM 5320B		W
W	TOX (Total Organic Halogens)	SW-846 9020B	X	W/S
W	TS - Water (Total Solids)	EPA 160.3	X	W
W	TS - Water (Total Solids)	SM 2540B		W
W	TSS (Total Suspended Solids)	EPA 160.2	X	W
W	TSS (Total Suspended Solids)	SM 2540D		W
W	TDS (Total Dissolved Solids)	EPA 160.1	X	W
W	TDS (Total Dissolved Solids)	SM 2540C	X	W
W	TVS (Total Volatile Solids)	160.4	X	W
W	TVDS (Total Volatile Dissolved Solids)	160.4	X	W
W	TVSS (Total Volatile Suspended Solids)	160.4	X	W

Matrix: W (Water) S (Soil/Solid) O (Other)
Note: NELAP accreditation may be matrix and program specific. Refer to STL Chicago's IL NELAP Certificate No.: 001027
available on the STL web-site.

Appendix C. Severn Trent Laboratories Chicago
Description and Floor Space for Analytical Facilities

Lab Areas	Description	Approx. Feet^2
Organic Extractions	The extraction area has the capacity for performing 36 continuous liquid-liquid extractions, 60 sonification extractions, and 50 separatory funnel extractions each day. The configuration of the extractors and the fume exhausts were designed to facilitate rapid, efficient sample preparation. Separate areas are used for sample cleanups. Contains separate refrigerated sample storage.	2240 (lab) 166 (coolers)
Organic Glassware Cleaning	Dedicated to eliminating cross contamination, this isolated area is equipped with sinks, ample counter space, and pass through shelves for storing clean glassware. Water in this area is supplied by the RO/DI system.	520
GC Extractables and HPLC	The GC Extractables and HPLC area has independent and segregated HVAC systems and a specially designed compressed gas generation and distribution system. One GC is dedicated to Pest/PCB screening. Shares refrigerated storage area with GC/MS BNA.	1080
GC/MS BNA	The GC/MS BNA area is specially designed with independent and segregated HVAC systems to minimize cross contamination. Shares refrigerated storage area with GC Extractables and HPLC.	1050
GC/MS VOA and GC Purge & Trap	The GC/MS VOA and GC Purge & Trap area is specially designed with independent and segregated HVAC systems to minimize cross contamination. One GC and one GC/MS are dedicated to screening. Contains separate refrigerated sample storage area.	GC/MS VOA - 1200 GC P&T - 700
Metals Prep	This isolated room is equipped with sinks, benches and hoods required for performing metals digestion. This area also houses the TCLP extraction apparatus, which can accommodate 52 samples at a time.	590
Inorganic Glassware Cleaning	Dedicated to eliminating cross contamination, this isolated area is equipped with sinks, ample counter space, and pass through shelves for storing clean glassware. Water in this area is supplied by the RO/DI system.	340
Metals - ICP and AA	The Metals Instrumentation area is specially designed with independent and segregated HVAC systems to minimize cross contamination.	2075
Mercury Lab	The Mercury preparation and analysis area has independent and segregated HVAC systems to minimize cross contamination. This area contains a CVAA instrument and a hood for ventilation and sample preparation.	260
Wet Chem Lab	The Wet Chem Lab is specially designed with independent and segregated HVAC systems to minimize cross contamination. Includes a draft free, temperature controlled weigh room. Cyanides, phenols, anions, solids, and other traditional "wet chemistry" analyses are performed here. All distillation procedures are conducted in ventilated hoods. Water in this area is supplied by the RO/DI system.	1500

Appendix D

Field Standard Operating Procedures (SOPs)

Table of Contents

Field SOP 110 - SOIL SAMPLING

SOP PERFORMANCE OBJECTIVES:

- To collect a soil sample that is representative of conditions as they exist at the site:
  - By selecting the appropriate sampling devices(s);
  - By taking measures to avoid introducing contamination as a result of poor sampling and/or handling technique; and
  - By reducing the potential for cross contamination between samples.

1 Introduction

Prior to conducting a soil sampling investigation, a sampling strategy should be developed based on the objectives of the investigation. After developing a soil sampling strategy, the appropriate equipment and techniques must be used to conduct the investigation. This section discusses the various soil sample collection methods, sample handling, and available sampling equipment that has been shown to be technically appropriate.

2 Equipment

Selection of equipment is usually based on the depth of the samples to be collected, but it is also controlled to a certain extent by the characteristics of the soil. Manual techniques and equipment such as hand augers are usually used for collecting surface or shallow, subsurface soil samples. Power operated equipment is usually associated with deep sampling but can also be used for shallow sampling when the bore hole begins to collapse or when the soil is so tight that manual sampling is not practical.

2.1 Precautions for Trace Contaminant Soil Sampling

All soil sampling equipment used for sampling trace contaminants should be constructed of inert materials such as stainless steel where possible. Pans used for mixing should be made of Pyrex® (or equivalent) glass. In no case will chromium, cadmium, galvanized, or plated equipment be used for soil sampling when trace levels of inorganic contaminants are of concern. Similarly, no painted or plastic equipment may be used where trace levels of organic contaminants are of concern. Paint, scaly or heavy rust and grease must be removed before use, most often by sandblasting the equipment. Ancillary equipment such as auger flights may be constructed of other materials since this equipment does not come in direct contact with the samples. The procedures outlined in the Standard Field Cleaning Procedures SOP.

3 Sampling Methodology

This discussion of soil sampling methods reflects both the equipment used to collect the sample as well as how the sample is handled and processed after retrieval. Selection of equipment is usually based on the depth of sampling, but it is also controlled, to a certain extent, by the characteristics of the material. Simple, manual techniques and equipment, such as hand augers, are usually selected for surface or shallow, subsurface soil sampling. As the depth of the sampling interval increases, some type of powered sampling equipment is usually needed to overcome the friction induced by soil resistance and depth. The following is an overview of the various sample collection methods employed over three general depth classifications: surface, shallow

subsurface, and deep subsurface. Any of the deep collection methods described may be used to collect samples from the shallower intervals.

3.1 Manual Collection Techniques and Equipment

These methods are used primarily to collect surface and shallow subsurface soil samples. Surface soils are generally classified as soils between the ground surface and 6 to 12 inches below ground surface. The shallow subsurface interval may be considered to extend from approximately 12 inches below ground surface to a site-specific depth at which sample collection using manual methods becomes impractical.

Surface Soils

Surface soils may be collected with a wide variety of equipment, if constructed of appropriate materials. Spoons or hand-augers are typically used to collect surface soil samples. If a thick, matted root zone is encountered at or near the surface, it should be removed before the sample is collected. The collected soil is placed in a pan, thoroughly mixed, and placed in the appropriate sample container(s). Section 4 contains specific procedures for collecting and handling soil samples for volatile organic compounds analysis.

Shallow Subsurface Soils

Hand augers are the most common equipment used to collect shallow subsurface samples. Typically, 4-inch auger-buckets with cutting heads are pushed and twisted into the ground, then removed as the buckets are filled. The auger holes are advanced one bucket at a time. The practical depth of investigation using a hand-auger depends upon the soil properties. In sand, augering is usually easily performed, but the depth of collection is limited to the depth at which the sand begins to flow. At this depth, the bore hole will usually collapse and cannot be advanced. Deeper sampling must be accomplished using power equipment. Hand augering may also be of limited use in tight clays or cemented sands.

Regardless of the soil type, at depths approaching 20 feet sidewall friction may become so severe that power equipment must be used. Power augers such as the Little Beaver® may be used to advance the borehole where hand augers are impractical. Power augers are a sampling aid, not a sampling device, and can be used to advance a borehole to approximately 20 feet, depending upon soil conditions. If power augers are used to advance the borehole, care must be taken that exhaust fumes, gasoline, and\or oil do not contaminate the borehole. The soil sample may then be collected using a hand auger. After the sample has been collected, the borehole may again be advanced (if necessary), and additional samples collected. The auger bucket must be replaced between samples with a properly decontaminated auger bucket. When a new borehole is advanced, the entire hand auger assembly must be replaced with a properly decontaminated hand auger assembly.

If the borehole is advanced using a hand auger, upon reaching the desired sampling depth replace the bucket with a properly decontaminated bucket. The sample may then be collected. After the sample has been collected, the borehole may be advanced (if necessary) with the bucket that was used to collect the sample. Each sample must be collected using a properly decontaminated bucket. Before the soil is placed in a pan, it is necessary to remove the top several inches of soil to minimize the possibility of cross-contamination of the sample from fall-in of material from the upper portions of the hole. Once the soil is placed in a pan, it is thoroughly mixed, and placed in

the appropriate sample container(s). Section 4 contains specific procedures for collecting and handling soil samples for volatile organic compounds analysis.

3.2 Powered Equipment

Powered equipment may be used to acquire soil samples from any depth (surface, shallow subsurface, and deep subsurface). When power equipment is used to advance the borehole and collect the sample, care must be taken that exhaust fumes, gasoline, and\or oil do not contaminate the borehole and the sample. Among the common types of powered equipment used to collect subsurface soil samples are split-spoon samplers driven with a drill rig drive-weight assembly or pushed using drill rig hydraulics; continuous split-spoon samplers; direct-push rigs; and back-hoes. The use of each of these is described below.

Drill Rigs

Drill rigs offer the capability of collecting soil samples from greater depths. For all practical purposes, the depth of investigation achievable by this method is controlled only by the depth of soil overlying bedrock, which may be in excess of 100 feet. Split-spoon samplers are usually driven either inside a hollow-stem auger or an open borehole after the auger(s) have been temporarily removed. The spoon is driven with a 140-pound hammer through a distance of up to 24 inches and removed. Continuous split-spoon samplers may be used to obtain five-foot long, continuous samples approximately 3 to 5 inches in diameter. These devices are placed inside a five-foot section of hollow-stem auger and advanced with the auger during drilling. As the auger advances, the central core of soil moves into the sampler and is retained. Before the soil is placed in a pan, it is necessary to remove the top several inches of soil to minimize the possibility of cross-contamination of the sample from fall-in of material from the upper portions of the hole. Once the soil is placed in a pan, it is thoroughly mixed, and placed in the appropriate sample container(s). Section 4 contains specific procedures for collecting and handling soil samples for volatile organic compounds analysis.

Direct Push Rigs

This method uses a standard split-spoon modified with a locking tip that keeps the spoon closed during the sampling push. Upon arrival at the desired depth, the tip is remotely released and the push continued. During the push, the released tip moves freely inside of the spoon as the soil core displaces it. This technique is particularly beneficial at highly contaminated sites, because no cuttings are produced. The push rods are generally retrieved with very little residue resulting in minimal exposure to sampling personnel and reduced investigation derived waste (IDW).

Before the soil is placed in a pan, it is necessary to remove the top several inches of soil to minimize the possibility of cross-contamination of the sample from fall-in of material from the upper portions of the hole. Once the soil is placed in a pan, it is thoroughly mixed, and placed in the appropriate sample container(s). Section 4 contains specific procedures for collecting and handling soil samples for volatile organic compounds analysis.

Geoprobe® Large Bore Soil Sampler

Geoprobe® offers several tools for soil sample collection. Among these are the Macro-Core® Soil Sampler and the large bore and dual tube soil sampling systems. The use of the large bore soil sampling system for collection of surface and subsurface soil samples is described herein. The selection of this system does not preclude the use of the other systems at a future time. The

Large Bore sampler is a solid barrel, piston sealed, direct push device for collecting discrete interval samples of unconsolidated materials at depth. The sampler is approximately 30-inches (762 mm) long and has a 1.5-inch (38 mm) outside diameter. The Large Bore sampler is capable of recovering a discrete sample core 22 inches x 1.0 inches (559 mm x 25 mm) contained inside a removable liner. Sample volume measures up to 283 ml. The liner is a 24-inch long by 1.15-inch OD (610 mm x 29 mm) removable/replaceable thin-walled tube that fits inside the Large Bore sample tube. Liners facilitate retrieval of the sample and may be used for storage, when applicable. The Large Bore soil sampler is pushed with 1.25-inch diameter probe rod. The following is a step-by-step description of the components and procedures used to collect a soil sample with the Large Bore sampler.

Assembly of Large Bore Sampler

1. Select a liner tube and push on to cutting shoe (one end of liner should be slightly flared, push this end on to shoe).

2. Insert end of tube opposite cutting shoe into sample tube and screw cutting shoe firmly into sample tube.

3. Thread piston tip onto piston rod then run piston assembly through the end of the sample tube opposite the cutting shoe, seating the piston tip in the cutting shoe. There should be a short section of exposed piston rod sticking out of sample tube.

4. Place drive head over exposed end of piston rod and thread into end of sample tube.

5. Install piston stop-pin in top of drive head. This retains the piston rod assembly during the push.

6. The Large Bore sampler is now fully assembled and ready for sample collection.

Sample Collection Using Large Bore Sampler

1. Attach assembled Large Bore sampler to end of probe rod.

2. Attach drive cap to probe rod and push rod into ground.

3. Add additional rods to push Large Bore sampler to target sampling depth.

4. At the desired target sampling depth, remove the drive cap to access inside of probe rod.

5. Couple extension rods and extension rod handle together and insert into probe rod. Using handle, turn the extension rods inside probe rod. This should engage the piston-stop pin and remove it from the drive head attached to the top of the sample tube. Retrieve extension rods and attached piston-stop pin.

6. Add addition probe rod, if required, reattach drive cap to top of probe rod and push probe rod and Large Bore sampler 24 inches to fill sampler.

7. Attach pull cap and retrieve tool string.

8. When retrieved, remove the piston rod, with piston tip, and the drive head.

9. Using the Large Bore wrench, unscrew the cutting shoe from the down-hole end of the sample tube.

10. Remove the cutting shoe and attached liner and sample from sample tube.

11. Sample has now been collected and is contained in the liner. At this time, it can be sub-sampled, per analytical requirements.

Special Considerations for Large Bore Soil Sampling

Liner Use and Material Selection
Due to the mode of operation, the Large Bore soil sampler must be used with a liner. Liners are available in the following materials: stainless steel, brass, cellulose acetate butyrate (CAB) and Teflon®. For the majority of environmental investigations conducted by EIB, either CAB or Teflon® liners are used. If samples are collected for organic compound analyses, Teflon® liners are required. CAB liners may be used if metals or other inorganic constituents are the object of the investigation.

Sample Orientation

When the liners and associated sample are removed from the sample tubes, it is important to maintain the proper orientation of the sample. This is particularly important when multiple sample depths are collected from the same push. It is also important to maintain proper orientation to define precisely what depth an aliquot was collected from. Maintaining proper orientation is typically accomplished using vinyl end caps. Convention is to place red caps on the top of liner and black caps on the bottom to maintain the proper sample orientation. Orientation can also be indicated by marking on the exterior of the liner with a permanent marker.

Back-Hoes

Back-hoes may be utilized in the collection of shallow subsurface soil samples. Samples may be collected directly from the bucket, or the trench wall (subject to applicable safety procedures). The bucket must be free of rust, grease, and paint. Only soil which has not been in contact with the bucket may be sampled, unless the bucket is cleaned according to the procedures described in Standard Field Cleaning Procedures SOP.

Trenches offer the capability of collecting samples from very specific intervals and allow visual correlation with vertically and horizontally adjacent material. The sample should be collected without entering the trench itself, if possible. To collect the sample without entering the trench, use a stainless steel scoop attached to rigid electrical conduit with a scoop bracket to “dress” (remove surface layer of soil smeared on the trench wall as the bucket passed) the wall of the trench. Replace the scoop with a decontaminated scoop. Collect the soil. The collected soil is placed in a pan, thoroughly mixed, and placed in the appropriate sample container(s). Section 4 contains specific procedures for collecting and handling soil samples for volatile organic compounds analysis

4 Soil/Sediment Sampling (Method 5035)

The following sampling protocol is recommended for site investigators assessing the extent of volatile organic compounds (VOCs) in soils and sediments at a project site. Because of the large number of options available, careful coordination between field and laboratory personnel is

needed. The specific sampling containers and sampling tools required will depend upon the detection levels and intended data use. Once this information has been established, selection of the appropriate sampling procedure and preservation method best applicable to the investigation can be made.

4.1 Equipment

Soil\sediment for VOC analyses may be retrieved using the equipment specified in Section 3 of this SOP. Once the soil\sediment has been obtained, the EnCore™ VOC sampler, Terracore Kits, syringes, stainless steel spatula, standard 2 oz soil VOC container, or pre-prepared 40 mL vials may be used/required for subsampling collection. The specific sample containers and the sampling tools required will depend upon the data quality objectives established for the site or sampling investigation. The various methods are described below.

4.2 Sampling Methodology - Low Concentrations

When total VOC concentrations in the soil/sediment are expected to be less than 200 ug/kg, the samples may be collected directly with the EnCore™ sampler or TerraCore™ syringe. If using the syringes, the sample must be placed in the sample container (40 ml pre-prepared vial) immediately to reduce volatilization losses. The 40 ml vials should contain 10 ml of organic free water for an un-preserved sample or approximately 10 ml of organic free water and a preservative. It is recommended that the 40 ml vials be prepared and weighed by the laboratory (commercial sources are available which supply preserved and tared vials). When sampling directly with the EnCore™ sampler, the vial must be immediately capped. A soil/sediment sample for VOC analysis may also be collected with conventional sampling equipment (as described in Sections 3 of this SOP). A sample collected in this fashion must either be placed in the final sample container (EnCore™ or 40 ml pre-prepared vial) immediately or the sample may be immediately placed into an intermediate sample container with no head space. If an intermediate container (usually 2 oz. soil jar) is used, the sample must be transferred to the final sample container (EnCore™ or 40 ml pre-prepared vial) as soon as possible not to exceed 30 minutes.

NOTE: After collection of the sample into either the EnCore™ Sampler or other container, the sample must immediately be stored in an ice chest and cooled.

Soil\sediment samples may be prepared for shipping and analysis as follows:

EnCore™ Sampler - the sample may simply be capped, locked and secured in a plastic/foil bag.

Syringe (Terra Core Kits) – Terracore Kits are provided by the laboratory and contain a pre-calibrated plastic sampling syringe which only allows 5 grams of soil sample to be obtained. The Terracore Kit contains pre-preserved vials. Add about 3.7 cc (approximately 5 grams) of sample material to 40 ml pre-prepared containers. Secure the containers in a plastic bag. Do not use a custody seals on the container, place the custody seal on the plastic bag. Note: When using the syringes, it is

important that no air is allowed to become trapped behind the sample prior to extrusion, as this will adversely affect the sample. Attachment 1 of this SOP provides additional instructions for collecting soil sample using the Terra Core kit.

4.3 Special Techniques and Considerations

Effervescence

If low concentration samples effervesce from contact with the acid preservative, (see Section 4.2) then either a test for effervescence must be performed prior to sampling, or the investigators must be prepared to collect each sample both preserved or un-preserved as needed, or all samples must be collected un-preserved. To check for effervescence, collect a test sample and add to a pre-preserved vial. If preservation (acidification) of the sample results in effervescence (rapid formation of bubbles) then preservation by acidification is not acceptable, and the sample must be collected un-preserved. If effervescence occurs and only pre-preserved sample vials are available, the preservative solution may placed into an appropriate hazardous waste container and the vials triple rinsed with organic free water. An appropriate amount of organic free water, equal to the amount of preservative solution, should be placed into the vial. The sample may then be collected as an un-preserved sample. Note that the amount of organic free water placed into the vials will have to be accurately measured. The Terra Core Kit eliminates the need for this by collecting an additional 5 gram soil sample placed in a vial preserved in DI water.

Sample Size

While this method is an improvement over earlier ones, field investigators must be aware of an inherent limitation. Because of the extremely small sample size, sample representativeness for VOCs may be reduced compared to samples with larger volumes collected for other constituents. The sampling design and objectives of the investigation should take this into consideration.

Holding Times

Field investigators should note that the holding time for an un-preserved VOC soil/sediment sample is 48 hours. Arrangements should be made to ship the soil/sediment VOC samples to the laboratory by overnight delivery the day they are collected so the laboratory may preserve and\or analyze the sample within 48 hours of collection.

Percent Moisture

Samplers must ensure that the laboratory has sufficient material to determine percent moisture in the VOC soil/sediment sample to correct the analytical results to dry weight. If other analyses requiring percent moisture determination are being performed upon the sample, these results may be used. If not, a separate sample (minimum of 2 oz.) for percent moisture determination will be required. Terra Core Kits contain the separate sample jar for determining % moisture at the lab.

Safety

Methanol is a toxic and flammable liquid. Therefore, methanol must be handled with all required safety precautions related to toxic and flammable liquids. Inhalation of methanol vapors must be avoided. Vials should be opened and closed quickly during the sample preservation procedure. Methanol must be handled in a ventilated area. Use protective gloves when handling the methanol vials. Store methanol away from sources of ignition such as extreme heat or open flames. The

vials of methanol should be stored in a cooler with ice at all times. Terra Core Kits contain specifically labeled vial containing methanol preservative.

Shipping

Methanol and sodium bisulfate are considered dangerous goods, therefore shipment of samples preserved with these materials by common carrier is regulated by the U.S. Department of Transportation and the International Air Transport Association (IATA). The rules of shipment found in Title 49 of the Code of Federal Regulations (49 CFR parts 171 to 179) and the current edition of the IATA Dangerous Goods Regulations must be followed when shipping methanol and sodium bisulfate. Consult the above documents or the carrier for additional information. Shipment of the quantities of methanol and sodium bisulfate used for sample preservation falls under the exemption for small quantities. A summary of the requirements for shipping samples follows. Refer to the code for a complete review of the requirements.

1. The maximum volume of methanol or sodium bisulfate in a sample container is limited to thirty (30) mls.

2. The sample container must not be full of methanol.

3. The sample container must be stored upright and have the lid held securely in place. Note that the mechanism used to hold the cap in place must be able to be completely removed so weight is not added to the sample container, as specified in Method 5035.

4. Sample containers must be packed in a sorbent material capable of absorbing spills from leaks or breakage of the sample containers.

5. The maximum sample shuttle weight must not exceed 64 pounds.

6. The maximum volume of methanol or sodium bisulfate per shipping container is 500 mls.

7. The shipper must mark the sample shuttle in accordance with shipping dangerous goods in acceptable quantities.

8. The package must not be opened or altered until no longer in commerce.

5. Reference

U.S. Environmental Protection Agency, Region 4. 2001. Environmental Investigations Standard Operating Procedures and Quality Assurance Manual. www.epa.gov/region4/sesd/eisopqam/eisopqam.html

FIELD SOP FCP—Standard Field Cleaning Procedures (FCP)

SOP PERFORMANCE OBJECTIVES:

- To remove contaminants of concern from sampling, drilling, and other field equipment using a standard cleaning procedure to concentrations that do not impact study objectives.

1. Introduction

Cleaning procedures are intended for use by field personnel for cleaning sampling and other equipment in the field. Sampling and field equipment cleaned in accordance with these procedures must meet the minimum requirements for Data Quality Objectives (DQO) definitive data collection. Deviations from these procedures should be documented in the approved study plan, field records, and investigative reports. These are the materials, methods, and procedures to be used when cleaning sampling and other equipment in the field.

2. Specifications for Cleaning Materials

Specifications for standard cleaning materials referred to in this SOP are as follows:

- Soap shall be a standard brand of phosphate-free laboratory detergent such as Liquinox®. Use of other detergent must be justified and documented in the field logbooks and inspection or investigative reports.
- Solvent shall be pesticide-grade isopropanol. Use of a solvent other than pesticide-grade isopropanol for equipment cleaning purposes must be justified in the Site Investigation Work Plan (SIWP). Otherwise its use must be documented in field logbooks and inspection or investigation reports.
- Tap water may be used from any municipal water treatment system. Use of an untreated potable water supply is not an acceptable substitute for tap water.
- Analyte free water (deionized water) is tap water that has been treated by passing through a standard deionizing resin column. At a minimum, the finished water should contain no detectable heavy metals or other inorganic compounds (i.e., at or above analytical detection limits) as defined by a standard inductively coupled Argon Plasma Spectrophotometer (ICP) (or equivalent) scan. Analyte free water obtained by other methods is acceptable, as long as it meets the above analytical criteria.
- Organic/analyte free water is defined as tap water that has been treated with activated carbon and deionizing units. At a minimum, the finished water must meet the analytical criteria of analyte free water and should contain no detectable pesticides, herbicides, or extractable organic compounds, and no volatile organic compounds above minimum detectable levels. Organic/analyte free water obtained by other methods is acceptable, as long as it meets the above analytical criteria.
- Other solvents may be substituted for a particular purpose if required. For example, removal of concentrated waste materials may require the use of either pesticide-grade hexane or petroleum ether. After the waste material is removed, the equipment must be subjected to the standard cleaning procedure. Because these solvents are not miscible with water, the equipment must be completely dry prior to use.

Solvents, laboratory detergent, and rinse waters used to clean equipment shall not be reused during field decontamination.

2.1 Handling and Containers for Cleaning Solutions

Improperly handled cleaning solutions may easily become contaminated. Storage and application containers must be constructed of the proper materials to ensure their integrity. Following are acceptable materials used for containing the specified cleaning solutions:

- Soap must be kept in clean plastic, metal, or glass containers until used. It should be poured directly from the container during use.
- Solvent must be stored in the unopened original containers until used. They may be applied using Teflon® squeeze bottles.
- Tap water may be kept in clean tanks, hand pressure sprayers, squeeze bottles, or applied directly from a hose.
- Analyte free water must be stored in clean glass, stainless steel, or plastic containers that can be closed prior to use. It can be applied from plastic squeeze bottles.
- Organic/analyte free water must be stored in clean glass, Teflon®, or stainless steel containers prior to use. It may be applied using Teflon® squeeze bottles, or with the portable system.

Note: Hand pump sprayers generally are not acceptable storage or application containers for the above materials (with the exception of tap water). This also applies to stainless steel sprayers. All hand sprayers have internal oil coated gaskets and black rubber seals that may contaminate the solutions.

2.2 Disposal of Solvent Cleaning Solutions

Procedures for the safe handling and disposition of investigation derived waste (IDW), including used wash water, rinse water, and spent solvents are in the Field SOP for IDW.

2.3 Equipment Contaminated with Concentrated Wastes

Equipment used to collect samples of hazardous materials or toxic wastes or materials from hazardous waste sites, RCRA facilities, or in-process waste streams should be field cleaned before returning from the site. At a minimum, this should consist of washing with soap and rinsing with tap water. More stringent procedures may be required at the discretion of the field investigators.

2.4 Safety Procedures for Field Cleaning Operations

Some of the materials used to implement the cleaning procedures outlined in this SOP can be harmful if used improperly. Caution should be exercised by all field investigators and all applicable safety procedures should be followed. At a minimum, the following precautions should be taken in the field during these cleaning operations:

- Safety glasses with splash shields or goggles, and latex gloves will be worn during all cleaning operations.
- Solvent rinsing operations will be conducted in the open (never in a closed room).
- No eating, smoking, drinking, chewing, or any hand to mouth contact should be permitted during cleaning operations.

2.5 Handling of Cleaned Equipment

After field cleaning, equipment should be handled only by personnel wearing clean gloves to prevent re-contamination. In addition, the equipment should be moved away (preferably upwind) from the cleaning area to prevent recontamination. If the equipment is not to be immediately re-used it should be covered with plastic sheeting or wrapped in aluminum foil to prevent re-contamination. The area where the equipment is kept prior to re-use must be free of contaminants.

3 Field Equipment Cleaning Procedures

Sufficient clean equipment should be transported to the field so that an entire study can be conducted without the need for field cleaning. However, this is not possible for some specialized items such as portable power augers, well drilling rigs, soil coring rigs, and other large pieces of field equipment. In addition, particularly during large scale studies, it is not practical or possible to transport all of the pre-cleaned field equipment required into the field. In these instances, sufficient pre-cleaned equipment should be transported to the field to perform at least one day's work. The following procedures are to be utilized when equipment must be cleaned in the field.

3.1 Specifications for Decontamination Pads

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

- The pad should be constructed in an area known or believed to be free of surface contamination.
- The pad should not leak excessively.
- If possible, the pad should be constructed on a level, paved surface and should facilitate the removal of wastewater. This may be accomplished by either constructing the pad with one corner lower than the rest, or by creating a sump or pit in one corner or along one side. Any sump or pit should also be lined.
- Sawhorses or racks constructed to hold equipment while being cleaned should be high enough above ground to prevent equipment from being splashed.
- Water should be removed from the decontamination pad frequently.
- A temporary pad should be lined with a water impermeable material with no seams within the pad. This material should be either easily replaced (disposable) or repairable.

At the completion of site activities, the decontamination pad should be deactivated. The pit or sump should be backfilled with the appropriate material designated by the field team leader, but only after all waste/rinse water has been pumped into containers for disposal. No solvent rinsates will be placed in the pit. Solvent rinsates should be collected in separate containers for proper disposal. See Field SOP for IDW for proper handling and disposal of these materials. If the decontamination pad has leaked excessively, soil sampling may be required.

3.2 "Classic Parameter" Sampling Equipment

"Classic Parameters" are analyses such as pH meters, oxygen demand, nutrients, certain inorganics, sulfide, flow measurements, etc. For routine operations involving classic parameter analyses, water quality sampling equipment such as Kemmerers, buckets, dissolved oxygen dunkers, dredges, etc., may be cleaned with the sample or analyte-free water between sampling locations. A brush may be used to remove deposits of material or sediment, if necessary. If

analyte-free water is unavailable the samplers should be flushed at the next sampling location with the substance (water) to be sampled, before the sample is collected. Flow measuring equipment such as weirs, staff gages, velocity meters, and other stream gauging equipment may be cleaned with tap water between measuring locations, if necessary. **The previously described procedures are not to be used for cleaning field equipment to be used for the collection of samples undergoing trace organic or inorganic constituent analyses.**

3.3 Sampling Equipment used for the Collection of Trace Organic and Inorganic Compounds

The following procedures are to be used for all sampling equipment used to collect routine samples undergoing trace organic or inorganic constituent analyses:

1. Clean with tap water and soap using a brush if necessary to remove particulate matter and surface films. Equipment may be steam cleaned (soap and high pressure hot water) as an alternative to brushing. Sampling equipment that is steam cleaned should be placed on racks or saw horses at least two feet above the floor of the decontamination pad. PVC or plastic items should not be steam cleaned.
2. Rinse thoroughly with tap water.
3. Rinse thoroughly with analyte free water.
4. Rinse thoroughly with solvent. Do not solvent rinse PVC or plastic items.
5. Rinse thoroughly with organic/analyte free water. If organic/analyte free water is not available, equipment should be allowed to completely dry. Do not apply a final rinse with analyte water.
6. Remove the equipment from the decontamination area and cover with plastic. Equipment stored overnight should be wrapped in aluminum foil and covered with clean, unused plastic.

3.4 Well Sounders or Tapes

1. Wash with soap and tap water.
2. Rinse with tap water.
3. Rinse with analyte free water.

3.5 Automatic Sampling Pump Cleaning Procedure

CAUTION - During cleaning always disconnect the pump from the generator.
The automatic sampling pump should be cleaned prior to use and between each monitoring well. The following procedure is required:

1. Using a brush, scrub the exterior of the contaminated hose and pump with soap and tap water.
2. Rinse the soap from the outside of the pump and hose with tap water.
3. Rinse the tap water residue from the outside of pump and hose with analyte-free water.
4. Place the pump and hose in a clean plastic bag.

3.6 Automatic Sampler Tubing

The tubing previously used in the automatic samplers may be field cleaned as follows:

1. Flush tubing with tap water and soap.
2. Rinse tubing thoroughly with tap water.

3. Rinse tubing with analyte free water.

4. Downhole Drilling Equipment

These procedures are to be used for drilling activities involving the collection of soil samples for trace organic and inorganic constituent analyses, and for the construction of monitoring wells to be used for the collection of groundwater samples for trace organic and inorganic constituent analyses.

4.1 Introduction

Cleaning and decontamination of all equipment should occur at a designated area (decontamination pad) on the site. The decontamination pad should meet the specifications of Section 3.1. Tap water (potable) brought on the site for drilling and cleaning purposes should be contained in a pre-cleaned tank. A steam cleaner and/or high pressure hot water washer capable of generating a pressure of at least 2500 PSI and producing hot water and/or steam (200_F plus), with a soap compartment, should be obtained.

4.2 Preliminary Cleaning and Inspection

The drill rig should be clean of any contaminants that may have been transported from another hazardous waste site, to minimize the potential for cross-contamination. Further, the drill rig itself should not serve as a source of contaminants. In addition, associated drilling and decontamination equipment, well construction materials, and equipment handling procedures should meet these minimum specified criteria:

- All downhole augering, drilling, and sampling equipment should be sandblasted before use if painted, and/or there is a buildup of rust, hard or caked matter, etc., that cannot be removed by steam cleaning (soap and high pressure hot water), or wire brushing. Sandblasting should be performed prior to arrival on site, or well away from the decontamination pad and areas to be sampled.
- Any portion of the drill rig, backhoe, etc., that is over the borehole (kelly bar or mast, backhoe buckets, drilling platform, hoist or chain pulldowns, spindles, cathead, etc.) should be steam cleaned (soap and high pressure hot water) and wire brushed (as needed) to remove all rust, soil, and other material which may have come from other hazardous waste sites before being brought on site.
- Printing and/or writing on well casing, tremie tubing, etc., should be removed before use. Emery cloth or sand paper can be used to remove the printing and/or writing. Most well material suppliers can supply materials without the printing and/or writing if specified when ordered.
- The drill rig and other equipment associated with the drilling and sampling activities should be inspected to insure that all oils, greases, hydraulic fluids, etc., have been removed, and all seals and gaskets are intact with no fluid leaks.
- PVC or plastic materials such as tremie tubes should be inspected. Items that cannot be cleaned are not acceptable and should be discarded.

4.2.1 Drill Rig Field Cleaning Procedure

Any portion of the drill rig, backhoe, etc., that is over the borehole (kelly bar or mast, backhoe buckets, drilling platform, hoist or chain pulldowns, spindles, cathead, etc.) should be steam cleaned (soap and high pressure hot water) between boreholes.

4.2.2 Field Cleaning Procedure for Drilling Equipment

The following is the standard procedure for field cleaning augers, drill stems, rods, tools, and associated equipment. This procedure does not apply to well casings, well screens, or split-spoon samplers used to obtain samples for chemical analyses, which should be cleaned as outlined in Section 3.3.

1. Clean with tap water and soap, using a brush if necessary, to remove particulate matter and surface films. Steam cleaning (high pressure hot water with soap) may be necessary to remove matter that is difficult to remove with the brush. Drilling equipment that is steam cleaned should be placed on racks or saw horses at least two feet above the floor of the decontamination pad.
Hollow-stem augers, drill rods, etc., that are hollow or have holes that transmit water or drilling fluids, should be cleaned on the inside with vigorous brushing.
2. Rinse thoroughly with tap water.
3. Remove from the decontamination pad and cover with clean, unused plastic. If stored overnight, the plastic should be secured to ensure that it stays in place.

When there is concern for low level contaminants it may be necessary to clean this equipment between borehole drilling and/or monitoring well installation using the procedure outlined in Section 3.3.

5. Reference

U.S. Environmental Protection Agency, Region 4. 2001. Environmental Investigations Standard Operating Procedures and Quality Assurance Manual. www.epa.gov/region4/sesd/eisopqam/eisopqam.html

FIELD SOP COC—Field Chain of Custody (COC)

SOP PERFORMANCE OBJECTIVES:

- To maintain and document the possession of samples from the time of collection until they or the received by the laboratory.

1 Introduction

Chain-of-custody procedures are comprised of the following elements; 1) maintaining sample custody and 2) documentation of samples for evidence. To document chain-of-custody, an accurate record must be maintained to trace the possession of each sample from the moment of collection to its introduction into evidence.

2 Sample Custody

A sample or other physical evidence is in custody if:

- It is in the actual possession of an investigator;
- It is in the view of an investigator, after being in their physical possession;
- It was in the physical possession of an investigator and then it was secured to prevent loss or tampering; and/or
- It is placed in a designated secure area.

3 Documentation of Chain-of-Custody

Sample Tag/Label

A sample tag/label should be completed for each sample using waterproof, nonerasable ink.

Chain-of-Custody Record

The field Chain-Of-Custody Record is used to record the custody of all samples or other physical evidence collected and maintained by investigators. All physical evidence or sample sets shall be accompanied by a Chain-Of-Custody Record. This Chain-Of-Custody Record documents transfer of custody of samples from the sample custodian to another person, to the laboratory, or other organizational elements. To simplify the Chain-of-Custody Record, as few people as possible should have custody of the samples during the investigation.

The Chain-Of-Custody Record also serves as a sample logging mechanism for the laboratory sample custodian. A separate Chain-of-Custody Record should be used for each final destination or laboratory utilized during the investigation.

The following information must be supplied in the indicated spaces to complete the field Chain-Of-Custody Record.

1. The project number.
2. The project name.
3. The project manager.

4. If the individual serving as the field sample custodian is different from the individual serving as the project leader, the field sample custodian's name and the title of the sample custodian (e.g., Jane Doe, Sample Custodian) should be recorded in the Remarks/AirBill" section of the Chain-of-Custody Record. This section may also be used to record airbill numbers, registered or certified mail serial numbers, or other pertinent information.

5. All samplers or sampling team leaders (if applicable) must sign in the designated signature block.

6. The sampling station ID (if positional data is recorded for the sample), Station ID, Media Code, date, and time of sample collection, grab or composite sample designation, and a brief description of the type of sample and/or the sampling location must be included on each line. One sample should be entered on each line and a sample should not be split among multiple lines.

7. If multiple sampling teams are collecting samples, the sampling team leader's name should be indicated in the "Remarks" column.

8. The total number of sample containers must be listed in the "Total Containers" column for each sample. The number of individual containers for each analysis must also be listed. There should not be more than one sample type per sample. Required analyses should be circled or entered in the appropriate location as indicated on the Chain-of- Custody Record.

9. The tag/label numbers for each sample and any needed remarks are to be supplied in the "Tag/Label Number" column.

10. The sample custodian and subsequent transferee(s) should document the transfer of the samples listed on the Chain-of-Custody Record. The person who originally relinquishes custody should be the sample custodian. Both the person relinquishing the samples and the person receiving them must sign the form. The date and time that this occurred should be documented in the proper space on the Chain-of-Custody Record.

11. Usually, the last person receiving the samples or evidence should be the laboratory sample custodian or their designee(s).

The Chain-of-Custody Record is a serialized document. Once the Record is completed, it becomes an accountable document and must be maintained in the project file. The suitability of any other form for chain-of-custody should be evaluated based upon its inclusion of all of the above information in a legible format.

4 Transfer of Custody with Shipment

- Samples shall be properly packaged for shipment in accordance with the procedures outlined in the Shipment SOP.

- All samples shall be accompanied by the Chain-Of-Custody Record. The original and one copy of the Record will be placed in a plastic bag inside the secured shipping container if samples are shipped. When shipping samples via common carrier, the "Relinquished By" box should be filled in; however, the "Received By" box should be left blank. The laboratory sample custodian is responsible for receiving custody of the samples and will fill in the "Received By" section of the Chain-of-Custody Record. One copy of the Record will be retained by the project manager. The original Chain-of-Custody Record

will be transmitted to the project manager after the samples are accepted by the laboratory. This copy will become a part of the project file.

5 Reference

U.S. Environmental Protection Agency, Region 4. 2001. Environmental Investigations Standard Operating Procedures and Quality Assurance Manual.
www.epa.gov/region4/sesd/eisopqam/eisopqam.html

FIELD SOP IDW—Investigation Derived Waste

SOP PERFORMANCE OBJECTIVES:

- Ensure proper management and disposal of Investigation Derived Waste.

1. Introduction

Investigation-derived wastes (IDW) are defined as any by-product to field activities that is suspected or known to be contaminated with any hazardous substance. The performance of field activities will produce waste products that may be non-hazardous or hazardous IDW.

2. Types of IDW

Materials which may become IDW are:

- Personnel protective equipment (PPE) -- This includes disposable coveralls, gloves, booties, respirator canisters, splash suits, etc.
- Disposable equipment -- This includes plastic ground and equipment covers, aluminum foil, conduit pipe, composite liquid waste samplers (COLIWASAs), Teflon® tubing, broken or unused sample containers, sample container boxes, tape, etc.
- Soil cuttings from drilling or hand augering.
- Drilling mud or water used for water rotary drilling.
- Ground water obtained through well development or well purging.
- Cleaning fluids such as spent solvents and washwater.
- Packing and shipping materials.

3. Management of Non-Hazardous IDW

Disposal of non-hazardous IDW from hazardous waste sites should be addressed in the study plan. If the waste is from an active facility, permission should be sought from the operator of the facility to place the non-hazardous PPE, disposable equipment, and/or paper/cardboard wastes into the facilities' dumpsters. If necessary, these materials may be placed into municipal dumpsters, with the permission of the owner. These materials may also be taken to a nearby permitted landfill. On larger studies, waste hauling services may be obtained and a dumpster located at the study site. Non-hazardous IDW may also be buried on site near the contamination source, with the burial location noted in the field logbook. Disposal of non-hazardous IDW such as drill cuttings, purge or development water, decontamination washwater, drilling muds, etc., should be specified in the approved study plan. It is recommended that these materials be placed into a unit with an environmental permit such as a landfill or sanitary sewer. These materials must not be placed into dumpsters. If the facility at which the study is being conducted is active, permission should be sought to place these types of IDW into the facilities treatment system. It may be feasible to spread drill cuttings around the borehole, or if the well is temporary, to place the cuttings back into the borehole. Cuttings, purge water, or development water may also be placed in a pit in or near the source area. Monitoring well purge or development water may also be poured onto the ground downgradient of the monitoring well. Purge water from private potable wells which are in service may be discharged directly onto the ground surface.

At a minimum the requirements of the management of non-hazardous IDW are as follows:

- Liquid and soil/sediment IDW must be containerized and analyzed before disposal.

- The collection, handling, and proposed disposal method must be specified in Site Investigation Work Plan (SIWP).

4. Management of Hazardous IDW

Disposal of hazardous or suspected hazardous IDW must be specified in the SIWP. Hazardous IDW must be disposed as specified in US-EPA regulations. If appropriate, these wastes may be placed back in an active facility waste treatment system. These wastes may also be disposed of in the source area from which they originated, if doing so does not endanger human health and the environment. If on-site disposal is not feasible, and if the wastes are suspected to be hazardous, appropriate tests must be conducted to make that determination. If they are determined to be hazardous wastes, they must be properly contained and labeled. They may be stored on the site for a maximum of 90 days before they must be manifested and shipped to a permitted treatment or disposal facility. Generation of hazardous IDW must be anticipated, if possible, to permit arrangements for proper containerization, labeling, transportation, and disposal/treatment in accordance with US-EPA regulations. The generation of hazardous IDW should be minimized.. Most routine studies should not produce any hazardous IDW, with the exception of spent solvents and possibly purged ground water. Care should be taken to keep non-hazardous materials segregated from hazardous waste contaminated materials. The volume of spent solvents produced during equipment decontamination should be controlled by applying only the minimum amount of solvent necessary, and capturing it separately from the washwater.

At a minimum the requirements of the management of hazardous IDW are as follows:

- Spent solvents must be properly disposed or recycled.
- All hazardous IDW must be containerized. Proper handling and disposal should be arranged prior to commencement of field activities.

5. Reference

U.S. Environmental Protection Agency, Region 4. 2001. Environmental Investigations Standard Operating Procedures and Quality Assurance Manual. www.epa.gov/region4/sesd/eisopqam/eisopqam.html

FIELD SOP ___100—MINNIERAE 2000 PORTABLE VOC MONITOR (PGM-7600) OPERATION & MAINTENANCE

SOP PERFORMANCE OBJECTIVES:

- Ensure that the instrument will provide the highest available accuracy and results of VOC monitoring during the project.
- Ensure that the instrument will operate as it is designed, given the field conditions that it is subjected during the project.
- Ensure that the instrument will be maintained according to its required specifications, thereby providing dependability and accuracy for the duration of the project.

1 Introduction

The basis methods and procedures for the operation and maintenance of the MinnieRae 2000 VOC monitor are contained in SOP ___100. All of specific procedures and methods discussed in this SOP should first be reviewed in the MinnieRae 2000 VOC Monitor Operation & Maintenance Manual. The SOPs to be used for this instrument should be applied for the duration of the project, which will ensure dependability and accuracy of the field sampling media evaluations, as well as personal health & safety requirements.

2 Calibration Methods

Prior to the field operation of the VOC monitor, proper calibration of the instrument is recommended. The calibration is done by exposing the sensor(s) to a known concentration of calibration gas. Typically, this is done at the beginning of each day. The calibration can be performed on a two-point process; (1) fresh air and (2) standard reference gas. The fresh air calibration should read no detectable VOCs (0.0 ppm), and is used to set the zero point for the sensor. The standard reference point of a known concentration and gas will be used for the second point of reference. Up to eight measurement gases can be stored into the instrument memory.

3 Operation & Maintenance Methods

To turn on the instrument, the “Mode” key needs to be depressed for a few seconds. A single beep will sound, and a series of readings will cycle on the instrument display screen. The “Mode” key is also used in turning off the instrument, and needs to be depressed during a 5-second countdown. The final reading shall read “Off” after the countdown is completed. During the operation of the instrument, special care should be taken to ensure that the sensor probe is not restricted. Any restriction of air supply to the sampling pump shall cause a repeating beeping alarm. The instrument should be turned off, and then turned back on to resume operation.

The VOC monitor instrument is very sensitive to high humidity and moisture conditions. Also, soil particles can easily block the sensor tube and possibly get sucked on the lamp and sensor itself. In the event that these conditions occur, the instrument will need to be disassembled and cleaned. GC grade methanol may be used for cleaning the instrument. After reassembly, the instrument may need to be re-calibrated prior to normal operation.

FIELD SOP 601 ___ MINI-RAM AERESOL DUST MONITOR
OPERATION & MAINTENANCE

SOP PERFORMANCE OBJECTIVES:

- Ensure that the instrument will provide the highest available accuracy and results of aerosol dust monitoring during the project.
- Ensure that the instrument will operate as it is designed, given the field conditions that it is subjected during the project.
- Ensure that the instrument will be maintained according to its required specifications, thereby providing dependability and accuracy for the duration of the project.

1 Introduction

Field measurements may be collected using portable real time aerosol meter (Mini-RAM) that measures aerosols. The Mini-RAM is used to measure dust particulates in ambient air. All of specific procedures and methods discussed in this SOP should first be reviewed in the Mini-Ram Aerosol Monitor Operation & Maintenance Manual. The SOPs to be used for this instrument should be applied for the duration of the project, which will ensure dependability and accuracy of the field sampling media evaluations, as well as personal health & safety requirements.

2 Calibration Methods

The Mini-RAM will be calibrated to manufacturer's specification including daily zero in zero bag before each day's sampling activities begin and as needed throughout the day if irregularities in the readings become apparent.

3 Operation & Maintenance Methods

- Press MEAS key (always starting from OFF on the display).
- Wait about 36 seconds after which MINIRAM will indicate concentration in mg/ m3, update every 10 seconds.
- At anytime during a run, pressing the TWA key, the MINIRAM will indicate the timeweighted average concentration for the run up to present time. Pressing the TWA does not influence the run operation of the MINIRAM. As soon as the TWA key is released the display returns to the 10-second concentration indications.
- To terminate a run, press OFF key. MINIRAM will retain in memory the TWA value up to the time the OFF key was pressed. The last 7 such TWA values are always retained in memory and can be played back by pressing PBK key for more than two seconds (always starting from OFF display).

If at any time MINIRAM display shows a small bar to the left of the BAT, the battery should be recharged for at least 12 hours. Only use charger supplied with MINIRAM. Allow MINIRAM battery to discharge completely before recharging: just leave it in MEAS until it shuts off automatically. Do not "top off" battery charge as that will reduce its capacity.

Appendix E

Field Forms

Table of Contents

Company Name		20004329
Deisan & Associates		
Sample ID		
Project	Sample Date	Sample Time
Bottle Desc. Plastic 1000mL Oblong, HDPE		
Preservatives: COOL 2C-6C		Filtered ____ Unfiltered X
Analysis Requested Comments: TDS/PH		

Custody Seal

DATE

SIGNATURE

SEVERN TRENT STL 704755

SEVERN TRENT STL 704755

SEVERN TRENT **STL**

STL Chicago
2417 Bond Street
University Park, IL 60466
Phone: 708-534-5200
Fax: 708-534-5211

Report To:

Contact: _____
Company: _____
Address: _____
_____
Phone: _____
Fax: _____
E-Mail: _____

Bill To:

Contact: _____
Company: _____
Address: _____
_____
Phone: _____
Fax: _____
PO#: _____ Quote: _____

Lab Lot#

Package Sealed Yes No	**Samples Sealed** Yes No
Received on Ice Yes No	**Samples Intact** Yes No
Temperature °C of Cooler	

Sampler Name: | **Signature:**

Project Name: | **Project Number:**

Project Location:

Lab PM:

Date Required
Hard Copy: ____/____/____
Fax: ____/____/____

Refrg #															
# / Cont.															
Volume															
Preserv															
Laboratory ID	**MS-MSD**	**Client Sample ID**	**Sampling Date**	**Sampling Time**	**Matrix**	**Comp/Grab**									**Additional Analyses / Remarks**

Within Hold Time Yes No | **Preserv. Indicated** Yes No NA

pH Check OK Yes No NA | **Res Cl_2 Check OK** Yes No NA

Sample Labels and COC Agree Yes No | **COC not present**

RELINQUISHED BY	COMPANY	DATE	TIME	RECEIVED BY	COMPANY	DATE	TIME
RELINQUISHED BY	COMPANY	DATE	TIME	RECEIVED BY	COMPANY	DATE	TIME

Matrix Key

WW	= Wastewater	SE	= Sediment
W	= Water	SO	= Solid
S	= Soil	DS	= Drum Solid
SL	= Sludge	DL	= Drum Liquid
MS	= Miscellaneous	L	= Leachate
OL	= Oil	WI	= Wipe
A	= Air	O	= _____

Container Key

1. Plastic
2. VOA Vial
3. Sterile Plastic
4. Amber Glass
5. Widemouth Glass
6. Other _____

Preservative Key

1. HCl, Cool to 4°
2. H2SO4, Cool to 4°
3. HNO3, Cool to 4°
4. NaOH, Cool to 4°
5. NaOH/Zn, Cool to 4°
6. Cool to 4°
7. None

COMMENTS

Date Received ____/____/____

Courier: **Hand Delivered** ☐

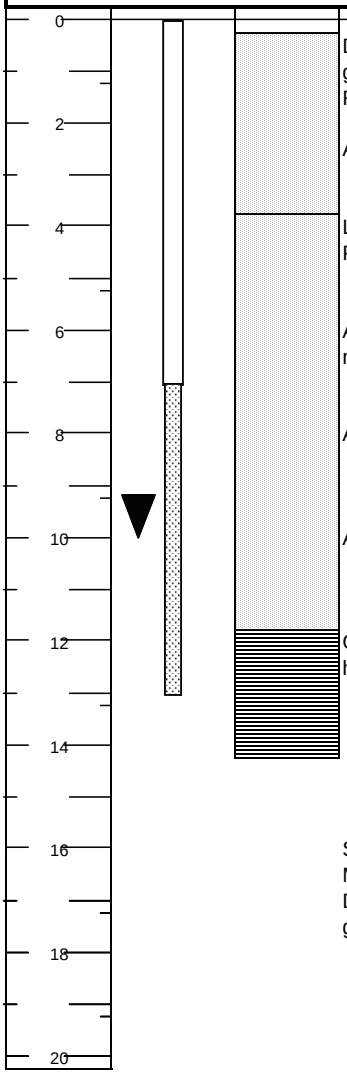
Bill of Lading

EXAMPLE BORING/WELL LOG--Note this information may also be recorded in the field log book--then prepared in the office.

Deigan & Associates

BORING NUMBER MW-2

PROJECT Former Fansteel/VR Wesson Property	PROJECT NO.
LOCATION Waukegan, Illinois	BOREHOLE DIA. 8 inches
TOTAL DEPTH 14 ft.	DEPTH TO WATER 10.04' TOC (583.58' MSL)
TOC ELEV. 593.62	DRILLING METHOD Truck Mounted Rig - HSAs
COMPANY	DATE DRILLED November 17, 2004
DRILLER	GEOLOGIST Kerry Van Allen
LOCATION North-Central Portion of Parcel 6; 11283.20/5368.03	
COMMENTS	



Depth (ft)	Well Record	Graphic Log	Description Soil Classification	Sample Int.	Sample Type
0–2			Dark brown to black silty coarse to fine sand, some coarse to fine gravel, cinder, coal, slag, fill, medium dense, moist. PID = 0.1 ppm Recovery = 23"		SM
2–4			As above, fill, moist. PID = NA Recovery = 0		SM
4–6			Light brown medium to fine sand, poorly graded, loose, moist. PID = 2 to 3 ppm Recovery = 14"		SP
6–8			As above. Below 7.2', brown coarse to fine sand, well graded, loose, moist. PID = 0.5 ppm Recovery = 17"		SW
8–10			As above, medium dense, moist. PID = ND Recovery = 15"		SW
10–12			As above, dense, saturated. PID = 0.5 ppm Recovery = 15"		SW
12–14			Gray silty clay, some coarse to fine sand, occasional fine gravel, very hard, moist. PID = ND Recovery = 22"		CL

Set well screen from 8 to 13' bgs, with sand pack to 7' bgs.
Materials used include Schedule 40 PVC, 2" diameter w/ flush mount
Developed well on November 19, 2004 using peristaltic pump. Removed 2.5 gallons before emptying well.

Legend

SILTY CLAY
SANDY CLAY
Organic topsoil
SAND
SILT

CC = Continuous Core
SS = Split Spoon
ST = Shelby Tube
AS = AugerSample
GP = Geo-Probe
HSA = Hollow-Stem Auger

Appendix B
Health and Safety Plan

Site Health & Safety Plan

Lake Shore Foundry
653 Market Street
Waukegan, Illinois 60085

April 27, 2007

Prepared by:

Deigan & Associates, LLC
Environmental Consultants
100 S. Genesee St.
Waukegan, IL. 60085

www.deiganassociates.com

SITE-SPECIFIC HEALTH AND SAFETY PLAN

Environmental Soil Sampling Work
Lake Shore Foundry
653 Market Street
Waukegan, Illinois

1.0 PURPOSE

In accordance with OSHA 29 CFR 1910.120 and Deigan & Associates, LLC (D&A) policy, a Site-Specific Safety and Health Plan (HASP) has been completed prior to engaging in sampling activities of sites where hazardous constituents or conditions have been confirmed or may potentially be present. This HASP has been designed to help identify, evaluate, and control safety and health hazards and provide for emergency response. To ensure that proper health and safety measures are implemented during sampling activities at the above referenced site, all on-site D&A personnel are required to adhere to the contents of this HASP. This HASP will also be provided to all other parties involved with the subject project, including but not limited to D&A's subcontractors, the site owner, and the owner's authorized representatives. All parties other should have and implement their own HASP.

2.0 APPLICABILITY

This Plan has been developed in compliance with all applicable regulations, including Occupational Safety and Health Administration (OSHA) standards (29 Code of Federal Regulations (CFR) 1910 and 1926).

D&A will require that all sub-contractors and sub-consultants follow applicable health and safety requirements promulgated by OSHA, this Plan, and those listed below:

- Employees must have the appropriate training (i.e., 40-hour OSHA 29 CFR 1910.120) health and safety course for hazardous waste workers/operators.
- Personnel working at this site must have had an annual physical (or physician's waiver for biennial physical), be certified by a qualified physician "fit for duty" and "fit for respirator use", if necessary, and be in a medical monitoring program, when applicable to their duties at the site.
- Proof of training and physical must be provided upon request.

- Personnel must have appropriate personal protective equipment (PPE) for the specific job (e.g., hard hat, safety shoes, protective eye and/or face protection, respirator, hearing protection, gloves, etc.).

- All equipment and field operations must meet applicable safety standards and satisfy an inspection by D&A's Site Safety Officer and/or Project Manager. Unsafe equipment or operations will not be tolerated and repeated safety and health violations will necessitate shut-down of the job/work.

  Subcontractors may operate under their own Site- Specific Health and Safety Plan, provided that such Plan is at least as stringent as the provisions contained within this HASP. Such Plans must be submitted to D&A and approved in advance by the D&A's Site Safety Officer and/or Project Manager.

3.0 SITE CHARACTERIZATION AND ANALYSIS

Soils at the site have been evaluated in preliminary site investigations in an effort to identify areas potentially impacted by contaminants due to historical operations. Potential environmental concerns have been identified within the project boundaries and at varying degrees of impact. Based on information available from 2004 sampling activities conducted by the USEPA, the primary contaminant on the site is lead.

The principal health hazards associated with lead are presented in Attachment A.

3.1 Site Location

The site is located at 653 Market Street, Waukegan, Lake County, Illinois. The dimensions of the property are approximately 270 feet north-south and 135 feet east-west. The 0.77 acre LSF property contains a single corrugated metal building. The Facility is located on the western shoreline of Lake Michigan. The Elgin, Joliet, and Eastern railroad borders the facility on the west and north sides. Lake Michigan borders the facility on the east side. A City ROW is south of the facility. The ground surface is relatively flat with fill soil covering much of the ground throughout the facility property. The LSF property and adjoining properties have a 100+ year history of heavy industrial uses, including Moen, US Steel, Fansteel/VR Wesson, Waukegan Paint & Lacquer, Diamond Scrap Yard and numerous other factories and warehouses.

3.2 Objective

The objective of the project is the completion of a soil investigation at various locations throughout the site. The site-work portion of this project will involve the preparation and collection of soil samples, utilizing a coordinate grid system. Soil borings will be collected using a Geoprobe soil-boring machine or a truck-mounted auger. Soil borings will be advance to pre-determined depths ranging between the ground surface and 11 to 14 feet. Upon completion of the soil borings, soil samples will secured for laboratory analysis.

3.3 Material Types

The materials to be handled during this project are concrete, asphalt, and soil materials from the soil boring locations. It is also believed that fill debris will be encountered. It is believed that the soil materials and groundwater encountered will contain low level metal contaminants or non-detectable contaminants.

3.4 Characteristics

Some of borings will penetrate through several inches of concrete and/or asphalt, followed by soil borings below grade. The remaining boring locations will be advanced at locations where concrete and/or asphalt surface materials do not exist.

Soil characteristics and historic contaminant findings are summarized in the Interim Measures Work Plan.

3.5 Unusual Site Features

The site consists of approximately 0.77 acres. Most of the property is covered by a building. The rest of the site is used for parking or is undeveloped land that is covered with low lying vegetation.

Overhead power lines are present at various locations bordering the site. Extreme care must be taken to prevent contact of personnel or drilling equipment with overhead lines or any other energized conductors or equipment. Accordingly, all utilities will be clearly marked prior to drilling activities.

No soil borings or sampling will be conducted in confined or enclosed spaces during the course of this project.

3.6 Brief Summary of Hazard Evaluation

The surficial and subsurface soil materials to be sampled are expected to contain lead. Samples are expected to be moist, and inhalation exposure to contaminated soil dust is unlikely. However due to the potential for lead entrainment in ambient air, concentrations in the employee breathing zone will be closely monitored; however, significant exposures are not expected. Encountering "hot spots" or additional contaminants not previously identified may increase exposure potential.

Respiratory protection will not be required when lead concentrations measured with a micro-R meter are at or below "background" levels. Work practices, PPE, and other necessary protective clothing are required to prevent excessive skin contact with contaminated soils.

4.0 SITE DESCRIPTION

The subject site and project is referred to as the Lake Shore Foundry, which is an operating

facility. The site is located at 653 Market Street, Waukegan, Lake County, Illinois. The dimensions of the property are approximately 270 feet north-south and 135 feet east-west. The 0.77 acre LSF property contains a single corrugated metal building. The Facility is located on the western shoreline of Lake Michigan. The Elgin, Joliet, and Eastern railroad borders the facility on the west and north sides. Lake Michigan borders the facility on the east side. A City ROW is south of the facility. The ground surface is relatively flat with fill soil covering much of the ground throughout the facility property. The LSF property and adjoining properties have a 100+ year history of heavy industrial uses, including Moen, US Steel, Fansteel/VR Wesson, Waukegan Paint & Lacquer, Diamond Scrap Yard and numerous other factories and warehouses.

Products presently produced by Lake Shore Foundry include brass, bronze & aluminum sand & permanent mold castings. The facility previously manufactured red brass and tin bronze, products which contained lead. Previous investigations by EPA in September 2004 measured lead in soil at levels exceeding regulatory limits.

5.0 COMPREHENSIVE WORK PLAN

The project objective is to conduct soil borings and collect soil samples.

Soil borings are scheduled to be advanced to further characterize surface and subsurface soil impact at this site. Borings will be collected using a Geoprobe or truck-mounted auger. All samples collected will be screened and secured on-site for subsequent delivery to, or pick-up by the laboratory for analysis.

Under normal circumstances, the following tasks are to be performed during drilling operations at the site:

- Mobilization
- Geoprobe/Drilling Equipment Positioning
- Drilling Operations Using the Geoprobe or Auger Equipment
- Soil Sample Extraction
- Soil Screening
- Collection, Securing, and Packing of Soil Samples
- Cleaning and Decontamination of Drill Stocks
- Placement of Soil in Disposal Drums for Removal and Disposal (as Required)
- Disposal of Cleaning/Decontamination Solutions and other Wastes Arising from Decontamination of Equipment and PPE

5.1 Schedule of Work

Surface and sub-surface soil boring installation work on this site is scheduled to be conducted in Spring/Summer 2007.

5.2 Location of Sampling Areas

The locations of sampling areas are identified in the "Interim Measure Work Plan".

6.0 ON-SITE ORGANIZATION AND COORDINATION

D&A will assign a Project Manager and a Site Safety Officer for the site work. The Project Manager will have overall responsibility for the work, including the coordination of sub-contractor activities. The Site Safety Officer will have responsibility for overall site safety, including making recommendations to sub-contractors regarding all issues related to safety and health. These assigned individuals will be identified prior to site mobilization on the "Responsible Persons" forms included as Attachment B.

Sub-contractors shall identify their own "Responsible Persons" or supervisors. These "Responsible Persons" will be knowledgeable of their work and associated hazards. Sub-contractors must have the authority to stop work whenever unsafe conditions exist or when ordered to stop work by the D&A Project Manager or Site Safety Officer.

All persons working on-site (including listing their job responsibilities) must be identified on the forms presented in Attachment B/C prior to the start of work. Attachment B/C will be updated by the D&A Project Manager or Site Safety Officer as on-site personnel change.

7.0 SITE CONTROL

The D&A Project Manager has been designated to coordinate access to the site, including security, as reasonably should be expected. An "Exclusion Zone" (EZ) of approximately 25-foot radius will be established around each boring location. Only authorized persons, wearing designated PPE, will be permitted within this zone. Smoking, eating, drinking, or application of cosmetics is prohibited in this zone.

A "Contamination Reduction Zone" (CRZ) will be established outside of the EZ. The purpose of the CRZ is to provide an area around the EZ to decontaminate tools and equipment, and also to offer protection of the workers within the work area. A CRZ will be established by the D&A Site Safety Officer or Project Manager around each boring location using paint, traffic cones, barrier tape and/or other site features, as appropriate.

There will not be an office trailer or other temporary structure on-site during this project. The D&A Project Manager and/or Site Safety Officer will keep a cellular phone activated at all times in case of emergencies. The drilling sub-contractor will be equipped with a first aid kit and fire extinguisher.

Due to the site's proximity to Lake Michigan, prevailing winds change frequently and can be

from any direction. Prevailing winds in the summer months in the Midwest are typically from the southwest and may be variable. The perimeters of the EZ and CRZ may require adjustment to account for windy conditions.

8.0 HAZARD EVALUATION

Surface and sub-surface soils at the site have been evaluated in preliminary site investigations in an effort to identify areas potentially impacted by contaminants due to historical operations. Potential environmental concerns have been identified within the project boundaries and at varying degrees of impact. Based on information available from 2004 sampling activities conducted by the USEPA, the primary contaminant on the site is lead.

The principal health hazards associated with lead are presented in Attachment A.

9.0 PERSONAL PROTECTIVE EQUIPMENT

Based on the evaluation of potential hazards, the following levels of personal protection have been designated for use for the following work tasks:

Location	Job Function	Level of Protection
Exclusion Zone	Drilling	Modified Level D*
Exclusion Zone	Sample Preparation	Modified Level D*
Contamination Reduction Zone	Decontamination of tools and equipment	Modified Level D*
Safe Zone	Various	Hard hat, safety shoes and eye protection are required throughout the site

*Note: Under normal operations, the minimum PPE required by personnel performing soil boring and monitoring well installation work is "Modified Level D". Modified Level D protection includes a hard hat, protective eyewear (safety glasses with side shields), steel-toed footwear, hearing protection (muffs or plugs), and disposable latex gloves (soil handling) and household-type work gloves (equipment handling). Respiratory protection is not required unless warranted by air monitoring.

PPE suitable for normal operations will be worn as described above. PPE will be upgraded to Level C (addition of appropriate air-purifying respirator) if air monitoring indicates sustained exposures in the personal breathing zone of 25 ug/m^3 lead. At any exposure above

50% of lead's OSHA Permissible Exposure Limit (PEL, which is 50 ug/m^3 for lead), work activities will be stopped to review safety procedures to make any appropriate changes.

10.0 COMMUNICATION PROCEDURES

Communication procedures will include face-to-face verbal communication and the use of cellular phones. In the event of an emergency, the emergency procedures presented in Section 12.3 will be followed.

11.0 DECONTAMINATION PROCEDURES

11.1 Equipment Decontamination

In order to prevent cross-contamination of samples and to minimize exposure to personnel, drill rigs, stock, and sampling equipment shall be thoroughly decontaminated before being removed from the EZ of each boring location.

Procedures for equipment decontamination are specified in the Section 4.4 of the Quality Assurance Project Plan (QAPP).

Decontamination of equipment shall be performed in Modified Level D PPE.

11.2 Personnel Decontamination

Prior to relocating the drill rig and supporting equipment, personnel shall perform decontamination as follows:

- Complete equipment decontamination prior to personal decontamination
- Rinse gross contamination from outer gloves into waste drum
- Decontaminate other PPE as needed
- Remove barriers and proceed with relocation

Prior to leaving the EZ for breaks, lunch, or at end of work shift, personnel shall perform decontamination as follows:

- Rinse gross contamination from outer gloves into waste drum
- Decontaminate other PPE as needed
- Remove gloves and dispose in waste drum

A personal wash station shall be established outside the CRZ. The wash station will be equipped with potable water and soap. All employees who have entered the EZ or CRZ must, upon leaving the EZ or CRZ, use the personal wash station to wash hands

and face prior to eating, drinking or smoking.

Persons will avoid direct contact with and will take steps to avoid inhalation of decontamination chemicals, including sprayed water, when performing decontamination.

12.0 SITE SAFETY AND HEALTH PLAN

The Site Safety Officer is directly responsible for implementation of all site safety recommendations.

12.1 Emergency Medical Location/Procedure

Local Waukegan ambulance/fire service is available by calling 911. Their response time is approximately 2-5 minutes.

The following phone list represents the emergency phone numbers for this project:

- Ambulance: 911
- Waukegan Fire Department: 911
- Waukegan Police Department: 911
- Vista Medical Center, East
  1324 N. Sheridan Road, Waukegan, Illinois: 1-847-360-3000
  1-847-360-4181
  (Emergency Dept.)

  The Vista Medical Center, East is approximately 2.5 miles from the center of the Project site. See Figure 1.

  <u>Directions:</u> WEST on Belvidere Road. Turn RIGHT onto N SHERIDAN RD. End at 1324 N. Sheridan Road.

- Poison Control Center: 1-800-382-9097
- Illinois Emergency Management Agency: 1-800-782-7860
- IEPA-Emergency Response Unit: 1-800-424-9300
- Center for Disease Control: 1-404-633-5313
- National Response Center: 1-800-424-8802

First aid/safety equipment will be available on-site as follows:

- First aid kit will be available on drilling equipment.
- Emergency eyewash station will be available on drilling equipment.
- Fire extinguishers will be available on drilling equipment.

Material Safety Data Sheets (MSDSs) will be available that provide information on chemicals likely to be stored or used on-site. The MSDSs provide emergency medical information for exposure.

12.2 Environmental Monitoring

The following environmental monitoring equipment will be used on-site at specified intervals:

Photoionization Detector (PID), with 10.6eV or 11.7eV lamp-

- Performs periodic measurements in worker breathing zone
- Perform "fresh air zero" and calibrate daily using 100 ppm isobutylene calibration gas
- Mini-RAM aerosol monitor performs periodic measurements for particulates in worker breathing zone.

12.3 Emergency Procedures

The Site Safety Officer shall be notified of any on-site emergencies and shall be responsible for ensuring that appropriate procedures are followed.

Any person who becomes ill or injured inside the EZ or the CRZ shall be decontaminated to the maximum extent possible before being transported from the site. If the injury or illness is minor, full decontamination should be completed and first aid treatment rendered to the affected individual. If the condition is serious, partial decontamination should still be possible prior to the removal of the worker from the EZ or the CRZ.

In the event of a major fire or explosion all persons should be removed from the area immediately. The Waukegan Fire Department shall be alerted by calling 911. All persons must stay clear of the work area until the Waukegan Fire Department and the Project Manager have cleared the area and deemed it safe to resume work.

12.4 PPE Failure

If a site worker experiences personal equipment failure, or if they determine that the

PPE is not adequate for the task, they shall immediately decontaminate and leave the area. The worker shall not re-enter the area until the Project Manager and/or Site Safety Officer is notified and the problem is corrected.

12.5 Other Equipment Failure

If equipment on-site fails to operate properly or is determined to be inadequate, the Project Manager shall be notified immediately. The Project Manager will then determine the effect the failure will have on continuing operations.

12.6 Personal Monitoring

Personal exposure sampling will be conducted using a PID in the approximate breathing zone of a potentially affected worker to make an initial exposure determination. Personal monitoring should be periodically repeated during activities involving exposure to contaminated soil. A sustained PID reading of 5 ppm or more above background levels in the worker breathing zone will require upgrading to Level C PPE.

If further monitoring is indicated, personal sampling with organic vapor monitors or other suitable means will be conducted on affected workers.

Monitoring for particulates will be performed with a Mini-RAM aerosol monitor.

12.7 Medical Monitoring

Applicable employees working at the site who are engaged in work operations where potential exposure to hazardous substances or other health hazards exist, must be in compliance with a medical surveillance program in accordance with 29 CFR 1910.120 (f).

Employees working at the site who wear, or may be required to wear, respiratory protection must have received a medical evaluation to determine their ability to wear a respirator. The medical evaluation must meet the requirements of 29 CFR 1910.134 (e).

If it is determined that heat stress is an issue, a work/rest regimen will be implemented in accordance with the requirements of the National Institute of Occupational Safety and Health (NIOSH). Procedures to monitor worker body temperature, pulse, and fluid intake may be implemented to determine the potential for heat stress.

12.8 Hazard Communication

A list of anticipated contaminants and an overview of their respective health effects is included in Attachment A, Principal Health Hazards (Preliminary Hazard Evaluation). Contractors bringing hazardous materials onto the work site must provide the Project Manager or Site Safety Officer with MSDSs for each hazardous chemical brought on-

site.

12.9 Respiratory Protection

It is not anticipated that respiratory protection will be required during the work. However, respiratory protection will be required if lead levels exceed sustained levels of 25 ug/m^3 within the worker's breathing zone and/or if organic contaminant levels exceed sustained levels of 5 ppm within the worker's breathing zone.

12.10 Lockout/Tagout

A Lockout/Tagout Program for the isolation of energy sources and energized/powered equipment does not apply to this project or the work of this project.

12.11 Confined Space Entry

OSHA defines a confined space as having the following characteristics:

- They are large enough to enter
- They have limited means of entrance and egress
- They are not designed for continuous human occupancy

Confined spaces do not exist at the subject site nor are applicable to the work scheduled to be performed.

13.0 EMPLOYEE TRAINING

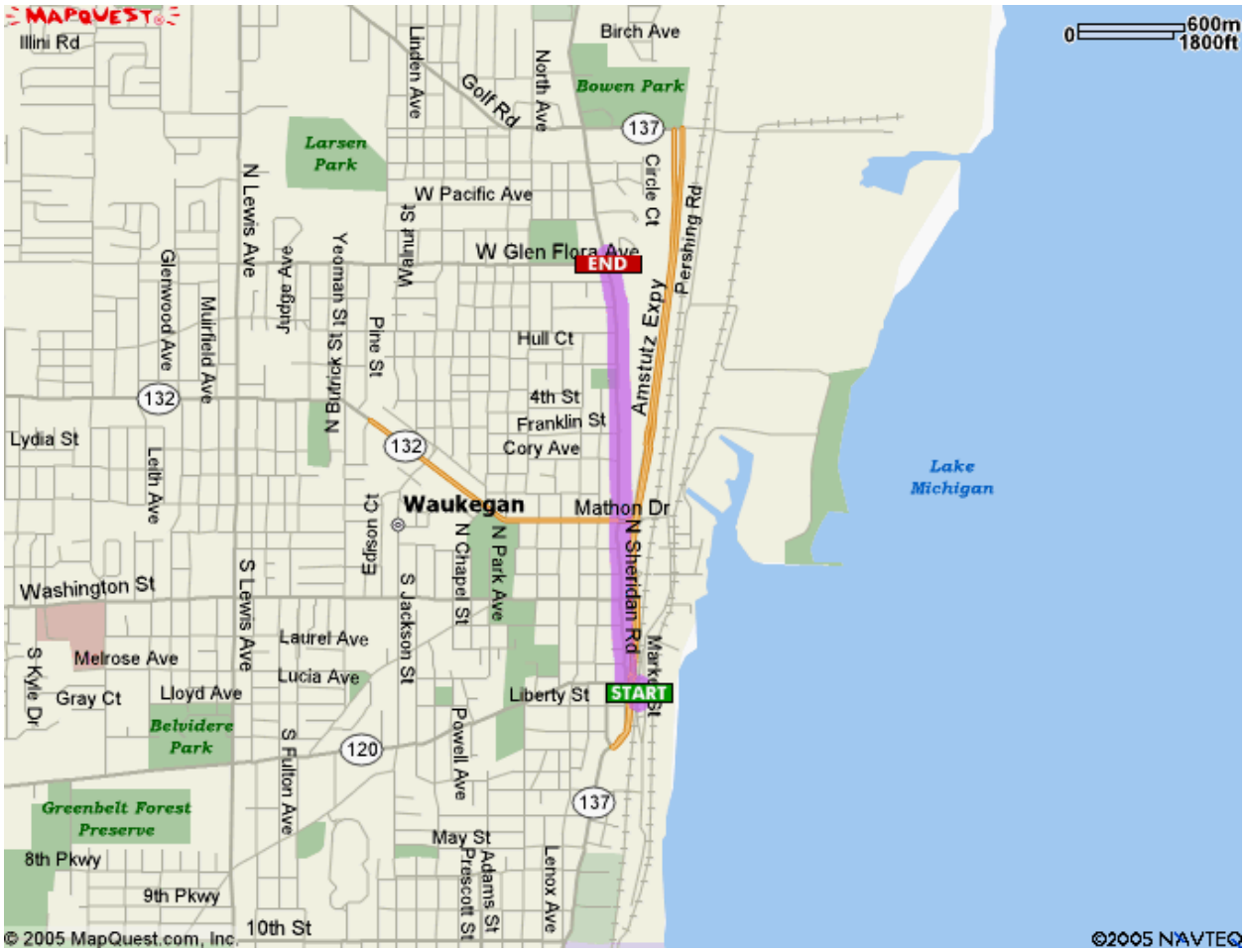
Applicable employees working on the site shall have received 40-hour hazardous waste operations and emergency response (HAZWOPER) training in accordance with 29 CFR 1910.120 (e)(3)(i).

Workers on the site must also complete site-specific safety orientation training where they will be instructed on the provisions of this HASP, their employer's HASP (as applicable) and their individual responsibilities as related to the project. Records of this training will be documented to ensure all persons on the site are aware of the specific hazards present on the site and the provisions of this HASP.

Daily safety meetings (Toolbox Meetings) will also be conducted to address certain specific safety issues relevant to the upcoming work of the day and any specific hazards that may be encountered.

All training records, including daily meetings minutes, should be retained on-site for the duration of the project.

Figure 1—Map from Site to Vista Medical Center



ATTACHMENT A
PRINCIPAL HEALTH HAZARDS
(PRELIMINARY HAZARD EVALUATION)

Lead is known o be present on-site. The primary hazards associated with this substance are inhalation, contact and ingestion hazards. Effects include weakness, insomnia, abdominal pain, anemia, tumors, hypotension, etc. Target organs include the GI tract, central nervous system, kidneys and tissue.

The effects of lead are the same whether it enters the body through breathing or swallowing. The main target for lead toxicity is the nervous system, both in adults and children. Long-term exposure of adults to lead at work has resulted in decreased performance in some tests that measure functions of the nervous system. Lead exposure may also cause weakness in fingers, wrists, or ankles. Lead exposure also causes small increases in blood pressure, particularly in middle-aged and older people. Lead exposure may also cause anemia. At high levels of exposure, lead can severely damage the brain and kidneys in adults or children and ultimately cause death. In pregnant women, high levels of exposure to lead may cause miscarriage. High-level exposure in men can damage the organs responsible for sperm production. We have no conclusive proof that lead causes cancer (is carcinogenic) in humans. Kidney tumors have developed in rats and mice that had been given large doses of some kind of lead compounds. The Department of Health and Human Services (DHHS) has determined that lead and lead compounds are reasonably anticipated to be human carcinogens based on limited evidence from studies in humans and sufficient evidence from animal studies, and the EPA has determined that lead is a probable human carcinogen (ATSDR Public Health Statement for Lead Draft, September 2005, http://www.atsdr.cdc.gov/toxprofiles/phs13.html)

The OSHA PEL/TWA is 50 ug/m^3. The employer shall assure that no employee is exposed to lead at concentrations greater than fifty micrograms per cubic meter of air (50 ug/m^3) averaged over an 8-hour period.

ATTACHMENT B
RESPONSIBLE PERSONS

NAME	POSITION/FUNCTION
Gary Deigan	Project Manager
Kerry Van Allen	Project Geologist/Site Safety Officer

FOR ENVIRONMENTAL DRILLER (CS DRILLING):

NAME	POSITION/FUNCTION
Gerry Butkus	Responsible Person
	Driller
	Driller/Tech

ATTACHMENT C
SITE-SPECIFIC ORIENTATION CERTIFICATION

The undersigned individuals hereby certify that:

1. I have read the Site-Specific Health and Safety Plan for the Environmental Sampling Work, Lakeshore Foundry Site, Waukegan, Illinois, and am familiar with its provisions.

2. I have been provided with site-specific orientation training.

3. I agree to comply with all provisions of the Plan, applicable government regulations, and recommendations of the Project Manager and Site Safety Officer.

SIGNATURE	PRINTED NAME	ORGANIZATION