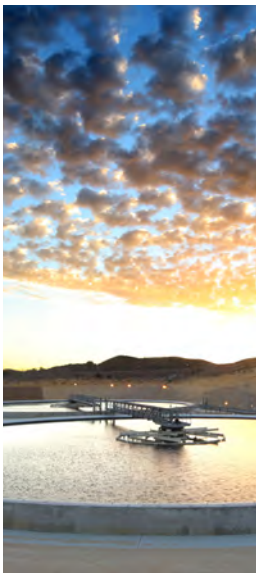
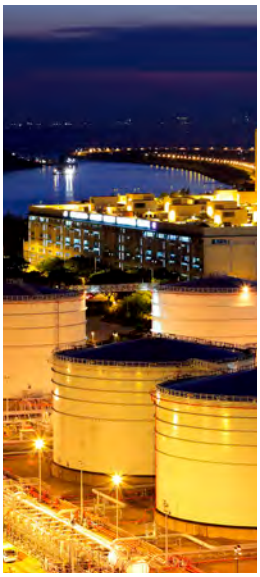


Quality Assurance Project Plan (QAPP)

OU1 Removal Activities

Pilsen Area of Chicago, Illinois

1801 Old Highway 8 Northwest, Suite 114 St. Paul Minnesota 55112

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Quality Assurance Project Plan (QAPP)

Project Title: QAPP
OU1 Removal Activities
Pilsen Area of Chicago, Illinois

Revision Number: 0

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Prepared For: USEPA

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Ramon Medoza

Approved By: _____ Date: _____
USEPA QA/QC Coordinator
TBD

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Ron Frehner – GHD	1
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Grant Anderson – GHD	1
Richard Wright – TestAmerica - Chicago	1
Shawn Hayes - TestAmerica - Cedar Falls	1
Terese Preston – TestAmerica - Chicago	1
Ramon Mendoza – USEPA	1
QA/QC Coordinator – USEPA	1

List of Acronyms and Short Forms

bgs	Below Ground Surface
CAA	Correction Action Agreement
CFR	Code of Federal Regulations
DQOs	Data Quality Objectives
EDD	Electronic Data Deliverable
EPA	Environmental Protection Agency
GHD	GHD Services, Inc.
GIS	Geographic Information System
ICP	Inductively Coupled Plasma
IRM	Interim Response Measure
LCS	Laboratory Control Sample
MS/Dup	Matrix Spike/Laboratory Duplicate
MS/MSD	Matrix Spike/Matrix Spike Duplicate
NELAP	National Environmental Laboratory Accreditation Program
NIST	National Institute of Standards and Technology
PARCCS	Precision, Accuracy, Representativeness, Comparability, Completeness, Sensitivity
PPE	Personnel Protective Equipment
%R	Percent Recovery
QA	Quality Assurance
QA/QC	Quality Assurance/Quality Control
QAPP	Quality Assurance Project Plan
QC	Quality Control
RPD	Relative Percent Difference
Site	OU1 Removal Activities - Pilsen Area of Chicago, Illinois
SOPs	Standard Operating Procedures
SRM	Standard Reference Material
USEPA	United States Environmental Protection Agency

1. Introduction

The Quality Assurance Project Plan (QAPP) is a planning document that provides a "blueprint" for obtaining the type and quantity of data needed to support environmental decision making. The QAPP integrates all technical and quality aspects of a project and documents all quality assurance (QA), quality control (QC), and technical activities and procedures associated with planning, implementing, and assessing environmental data collection operations.

This QAPP has been prepared by GHD Services, Inc. (GHD) in accordance with the QAPP guidance documents "EPA Requirements for Quality Assurance Project Plans", EPA QA/R-5, March 2001 and "EPA Guidance for Quality Assurance Project Plans", EPA QA/G-5, December 2002. In accordance with these documents, there are four basic groups of elements that must be included in a QAPP. These four groups and associated elements are:

- Group A - Project Management. The elements in this group include all aspects of project management, project objectives, and project history.
- Group B - Data Generation and Acquisition. The elements in this group include descriptions of the design and implementation of all measurement systems that will be used during the project.
- Group C - Assessment/Oversight. The elements in this group encompass the procedures used to ensure proper implementation of the QAPP.
- Group D - Data Validation and Usability. The elements in this group cover the QA activities that occur after the data collection phase of the project is completed.

The elements that comprise project management, data generation and acquisition, assessment/oversight, and data validation and usability for the sampling activities being conducted at the OU1 Removal Activities - Pilsen Area of Chicago, Illinois (Site) are documented in this QAPP.

1.1 Project Organization

The responsibilities of management, QA personnel, field personnel, and laboratory personnel are provided in the following subsections. Additionally, any special training/certification requirements for the project are identified, and an organization chart that identifies the lines of communication among the participants in the OU1 removal activities is presented herein.

1.2 Management responsibilities

GHD is the technical consultant for the OU1 removal activities at the Site. GHD has technical responsibility for the data collection activities at the Site. GHD's Project Manager is ultimately responsible for ensuring that the project objectives are achieved. Specific management responsibilities follow:

Ron Frehner - Project Manager – GHD

- Overview of field activities
- Overview of laboratory activities
- Advise on corrective actions
- Preparation and review of reports

- Coordinate GHD's technical staff
- Final evidence file custodian
- Approval of the QAPP

The analytical laboratory's Project Manager is responsible for ensuring that the project objectives are achieved by the laboratory. The laboratories selected for this project are:

TestAmerica Laboratories, Inc. (TestAmerica - Chicago)
2417 Bond Street
University Park, Illinois 60484
Phone: 708-534-5200

And

TestAmerica Laboratories, Inc. (TestAmerica - Cedar Falls)
704 Enterprise Drive
Cedar Falls, Iowa 50613
Phone: 319-277-2401

TestAmerica - Chicago will analyze investigative soil samples. TestAmerica - Cedar Falls will analyze air filter samples. TestAmerica's Project Managers and his/her specific responsibilities follow:

Richard Wright - Project Manager - TestAmerica - Chicago/
Shawn Hayes - Project Manager - TestAmerica - Cedar Falls

- Ensure all resources of the laboratory are available on an as-required basis
- Review of final analytical reports
- Approve final reports prior to submission to GHD
- Approval of the QAPP

The United States Environmental protection Agency (USEPA) Federal On-Scene Coordinator is responsible for overview of this project. This person also is responsible for submitting this QAPP and any subsequent revisions or amendments to the appropriate USEPA personnel for review and approval and for providing approval of the QAPP. Ramon Mendoza is the USEPA Federal On-Scene Coordinator for the OU1 removal activities at the Site.

1.3 Quality Assurance Responsibilities

Project team members with QA responsibilities include GHD's QA officer, GHD's Field QA officer, and TestAmerica's QA officer. These individuals and their specific responsibilities are as follows:

Grant Anderson - Quality Assurance officer - GHD

- Overview and review field QA/QC
- Review laboratory QA/QC
- Coordinate and review data validation and assessment
- Advise on laboratory corrective action procedures
- Preparation and review of QA reports

- QA/QC representation of project activities
- Approval of QAPP

Walt Pochron - Field Quality Assurance officer - GHD

- Management of field activities and field QA/QC
- Field data assessment
- Internal field technical system audits
- Technical representation of field activities
- Implement and document field corrective actions, if necessary
- Approval of QAPP

Terese Preston – QA officer - TestAmerica - Chicago/
Tom Tjaden - QA officer - TestAmerica - Cedar Falls

- Coordinate and overview of laboratory systems audits
- Overview of QA/QC documentation
- Conduct detailed data review
- Implement and document laboratory corrective actions, if required
- Technical representation of laboratory QA procedures
- Oversee preparation of laboratory SOPs
- Approval of QAPP

The USEPA Quality Assurance Reviewer is responsible for reviewing and providing final approval of the QAPP.

1.4 Field Responsibilities

GHD will conduct all field sampling during the OU1 activities. The specific procedures for field sample collection and field measurements are presented in Sections 2.2 through 2.5 of this QAPP.

GHD's field sampling team will consist of technical staff from GHD's Chicago, Illinois office. GHD's Field QA officer (Project Coordinator) will be responsible for documenting any non-conformances and subsequent corrective actions. The Field QA officer or any field team member can identify and report non-conformances.

1.5 Laboratory Responsibilities

The laboratories selected for this project are TestAmerica - Chicago in University Park, Illinois and TestAmerica - Cedar Falls in Cedar Falls, Iowa. TestAmerica - Chicago will analyze soil samples and TestAmerica - Cedar Falls will analyze filter air samples.

The specific responsibilities of additional laboratory personnel involved in the project follow:

Jodi Gromala - Operations Manager - TestAmerica - Chicago/
Lorna Bormann - Operations Manager - TestAmerica - Cedar Falls

- Coordinate laboratory analyses
- Supervise in-house chain-of-custody (COC)
- Schedule sample analyses
- Oversee data review
- Oversee preparation of analytical reports

Sherri Scott - Sample Custodian - TestAmerica - Chicago/
Chad Timmins - Sample Custodian - TestAmerica - Cedar Falls

- Receive and inspect the incoming sample containers
- Record the condition of the incoming sample containers
- Sign appropriate documents
- Verify correctness of chain-of-custody documentation
- Notify project manager of any non-conformances identified during sample receipt and inspection
- Assign a unique identification number to each sample, and enter the client identification number and sample identification numbers into the sample receiving log
- Initiate transfer of the samples to appropriate laboratory sections
- Control and monitor access/storage of samples and extracts

The project organizational chart for the OU1 removal activities is presented on Figure 1.1.

1.6 Problem Definition

This QAPP has been developed to outline the procedures and methodologies for

- Collecting soil samples from treated soils to ensure the treated soils are below the objective of 5 milligrams/per liter (mg/L) for Toxicity Characteristic Leaching Procedure (TCLP) lead
- Conduct downwind particulate monitoring during earth moving activities associated with the removal action
- Conduct construction personnel air filter sampling for lead

The specific area associated with the removal action are outlined in the Removal Plan for Alley and Railroad and this QAPP has been defined by the USEPA as Operational Unit 1 (OU1) (Site). The Site (OU1) location is shown on Figure 1.2 and the limits of OU1 are shown on Figure 1.3.

1.7 Project/Task Description

The specific tasks to be completed during the removal action are outlined in the Removal Plan for Alley and Railroad.The following sampling and monitoring activities will be performed for the removal action activities.

- Collecting soil samples from treated soils to ensure the treated soils are below to objective of 5 milligrams/per liter (mg/L) for TCPL lead
- Conduct downwind particulate monitoring during earth moving activities associated with the removal action
- Conduct construction personnel air filter sampling for lead

Table 1.1 provides a summary of the sampling and analysis program.

1.8 Quality Objectives and Criteria

Data quality objectives (DQOs) are qualitative and quantitative statements derived from the outputs of each step of the DQO process. The DQO process is a series of planning steps based on the scientific method that is designed to ensure that the type, quantity, and quality of environmental data used in decision making are appropriate for the intended application.

There are seven steps in the DQO process which include:

1 State the problem

2 Identify the decision

3 Identify inputs to the decision

4 Define the study boundaries

5 Develop a decision rule

6 Specify limits on decision errors

7 Optimize the design for obtaining data

The DQOs derived from this process are used to develop a scientific and resource-effective sampling design. The DQOs were developed in part using the USEPA guidance document EPA QA/G-4 (September 1994).

1.8.1 Statement of the Problem

The results of sampling completed in the alley and railroad have identified lead at concentrations above the industrial and commercial property objective of 800 milligrams per kilogram (mg/kg) and exceeded the TCLP lead regulatory limit of 5.0 mg/L within a limited portion of the Site. The results of sampling completed in the alley and railroad are presented in the following documents:

- Site Assessment Report for Pilsen Soil Assessment Area: Rail Road/Alley Chicago, Cook County, Illinois Addendum 1; dated November 3, 2014
- Pilsen Soils OU1 Railroad Spur and Alley Site: Western Areas, Rail Road Spur Soil Sample Results: USEPA Memorandum dated May 22, 2015
- Pilsen Soils OU1 Railroad Spur and Alley Site: Western Areas, Rail Road Spur Reanalysis of Soil Sample ID PA-RR26—0624 for TCLP Lead: USEPA Memorandum dated August 21, 2015

1.8.2 Identify the Decision

Environmental data is collected to:

- Confirm that treated soil meets the objective of 5.0 mg/L for TCLP lead.
- Confirm that that removal earth working activities do not generate unacceptable levels of particulate matter.
- Confirm construction workers are not exposed to unacceptable levels of lead in dust. The current compound of concern is lead in soil.

1.8.3 Identify Inputs to the Decision

This section describes how to identify information necessary to make the decisions listed above.

- Collecting soil samples for laboratory analysis from treated soils to ensure the treated soils are below to objective of 5 milligrams/per liter (mg/L) for TCLP lead
- Conduct daily downwind particulate monitoring during earth moving activities associated with the removal action
- Conduct personnel air filter sampling for lead and submit the sample for lead analyses

The analytical methods for the analyses can be found in Section 2.11 of this QAPP.

1.8.4 Define the Study Boundaries

This area is divided into the following ten parts based generally on land ownership and use as shown of Figure 1.3 and listed as follows:

1. **Area 1 Revised - Railroad West of Loomis (West Part)**: This part is approximately 18 feet in width (defined as 9 feet on each side of the centerline of the rail road tracks) 490 feet long between Laflin and Loomis and is owned by the City of Chicago. This area has lead levels above 800 mg/kg but EPA samples collected in this area were below the TCLP lead criteria within the area. The rails and ties are in place and the spur is inactive.
2. **Area 2 Revised - Railroad West of Loomis (East Part)**: This part is triangular in shape and approximately 120 feet long and between 18 and 45 feet wide at its widest point (defined as 9 feet on each side of the centerline of the rail road tracks with the area between the two sets of tracks at the east end included). This area is directly adjacent to Loomis and is owned by the City of Chicago. This area has lead levels above 800 mg/kg. The rails and ties are in place and the spur is inactive.
3. **Area 3 -Loomis Crossing**: This is the paved street section of Loomis where the railroad tracks formerly crossed the road. The rails and ties have been removed and there is street pavement or concrete sidewalks covering this area.
4. **Area 4 - Railroad East of Loomis (North)**: This part is approximately 95 feet long and owned by H. Kramer and was used by BNSF. This part lies between Loomis and 21st Place (entrance to H Kramer). The rails and ties are still present. This area exceeds 800 mg/kg lead and has TCLP[1] lead within the area. The rail spur is inactive.
5. **Area 5- 21st Place**: - This part represents an approximate 135 foot by 75 foot area east of Loomis which is the entrance to H Kramer and is currently owned by the City. This area exceeds 800 mg/kg lead and also has TCLP lead within the area. The rail spur is inactive.

[1] Toxicity Characteristic Leaching Procedure (TCLP) lead concentrations above 5.0 milligrams per liter (mg/L)

6. **Area 6 - Railroad East of Loomis (South)**: This part represents an approximate 110 foot long section of railroad tracks used by BNSF and owned by H. Kramer. This part lies between the east-west alley and 21st Place (entrance to H Kramer). The rails and ties are still present. This area exceeds 800 mg/kg lead but EPA samples collected in this area were below the TCLP lead criteria within the area. The rail spur is inactive.

7. **Area 7- North South Alley**: This part is approximately 110 feet by 25 feet in area and is owned by the City. It has a gravel/fill surface and has lead above 800 mg/kg but EPA samples collected in this area were below the TCLP lead criteria within the area.

8. **Area 8 - Unpaved East- West Alley**: This part represents an approximate 325 feet of unpaved alley along the western part and is owned by the City. This area has lead levels above 800 mg/kg and has TCLP lead within the area.

9. **Area 9 - Paved East West Alley**: This part represents an approximate 175 feet of paved alley along the eastern part and is owned by the City. This area has lead levels above 800 mg/kg. Recent inspection of this area indicates that the pavement in this area is in poor shape.

10. **Area 10 - Railroad South of Alley**: This approximately 120 feet long railroad segment is owned by DeTrinh and 1358 Cermak LLC and was used by BNSF. This part lies between the east-west alley to the north and Cermak Road to the south. The rails and ties are still present. This area exceeds 800 mg/kg lead but EPA samples collected in this area were below the TCLP lead criteria within the area. The rail spur is inactive.

Figure 1.3 shows the remediation area.

1.8.5 Develop a Decision Rule

The decision criteria for the sampling and monitoring are as follows:

- The treated soil criterial is 5.0 mg/L for TCLP lead
- Downwind dust/particulate action level is 3,000 micrograms per cubic meter (ug/m^3) and the OSHA's PEL is 15,000 µg/m^3
- Filter standards OSHA's action level is 30 µg/m^3 and the PEL is 50 µg/m^3 per 8-hour day

1.8.6 Specify Limits on Decision Errors

QA/QC samples will be collected during the investigation. Particulate monitoring will be conducted on a daily basis. Monitoring equipment and air sampling pumps will be tested and calibrated in accordance with manufactures recommended procedures.

Optimize the Design for Obtaining Data

This QAPP, the Removal Plan and the HASP outline the protocols that are implemented to perform all field activities associated with the OU1 Removal Plan.

1.9 Special Training/Certification Requirements

GHD's field sampling team members are required to have received the 40-hour Hazardous Waste Operations and Emergency Response (HAZWOPER) safety training and annual 8-hour refresher courses required by 29 CFR Parts 1910 and 1926. On-Site subcontractor personnel involved in

invasive activities (e.g., excavation) are required to have received the same training. The subcontractor is responsible for compliance of their personnel with the applicable regulations.

The laboratory performing sample analysis for the OU1 removal activities is required to be accredited by the National Environmental Laboratory Accreditation Program (NELAP) to demonstrate compliance with USEPA's requirement that the laboratory have a documented quality system that complies with ANSI/ASQC E4-94 ("Specifications and Guidelines for Quality System for Environmental Data Collection and Environmental Technology Programs", January 1995) and EPA QA/R-2 ("EPA Requirements for Quality Management Plans", March 2001). The laboratory will also be certified in the State of Illinois. TestAmerica is accredited by NELAP and certified in the State of Illinois for the analyses identified in this QAPP.

1.10 Documentation and Records

The documents, records, and reports generated during the OU1 removal activities are identified in the following subsections.

1.10.1 Field and Laboratory Records

Documents and records generated during the project include sample collection records, QC sample records, field measurement records, laboratory records, and data handling records. A brief description of these documents and records is provided below. Detailed information on these records is provided in subsequent sections of this QAPP.

Sample collection records that will be used during the sampling activities include computer tablets, field logbooks and forms, chain-of-custody records, and shipping papers.

QC sample records that will be used during the project to document the generation of QC samples include computer tablets; field logbooks; and forms for recording equipment blank samples, field duplicate samples, matrix spike/matrix spike duplicates (MS/MSD), and/or MS/Dup samples. TestAmerica will maintain appropriate documentation of sample integrity information. Records of sample preservation will be maintained in field logbooks and by TestAmerica.

Field measurements will be recorded in computer tablets, bound logbooks, or on standard field forms. Calibration data, where applicable, will also be recorded in these logbooks or forms.

Laboratory records that will be maintained for the project include sample receipt documentation, field and laboratory chain-of-custody documentation, sample container cleanliness certifications, reagent and standard reference material certifications, sample preparation records, sample analysis records (e.g., run logs), instrument/raw data, QC data, calibration data, corrective action reports, and final reports.

Data handling records that will be maintained include verification of computer programs used to manipulate or reduce raw data into final results and data validation reports. TestAmerica will maintain documentation of data verification and reduction procedures as necessary for the analyses used during the OU1 removal activities. GHD will maintain checklists, notes, and reports generated during the external data validation process.

1.10.2 Data Reporting Format

Field data will be recorded in computer tablets, bound logbooks, or on standard forms. The details for recording field data are provided in Section 2.5 of this QAPP. Field data primarily will be from field observations.

Laboratory reports for samples collected during OU1 removal activities to determine the nature and extent of contamination or for risk assessment purposes will consist of the following data deliverables:

1. Case Narrative
    a. Date of issuance
    b. Any deviations from intended analytical strategy
    c. Laboratory batch number
    d. Number of samples and respective matrices
    e. Project name and number
    f. Condition of samples "as received"
    g. Discussion of whether or not sample holding times were met
    h. Discussion of technical problems or other observations that may have created analytical difficulties
    i. Discussion of any laboratory quality control checks that failed to meet project criteria
2. Chemistry Data Package
    a. Dates of sample collection, receipt, preparation, and analysis
    b. Cross-reference of laboratory to project sample identification numbers
    c. Description of data qualifiers used
    d. Methods of sample preparation and analysis
    e. Sample results in tabular format
    f. MS/MSD and MS/Dup data, laboratory control sample (LCS) data, method blank data
    g. Fully executed chain-of-custody document

Method detection limit studies, instrument detection limit studies, and method performance and validation studies will be maintained by the laboratory.

1.10.3 Data Archiving and Retrieval

Evidential files for the entire project will be maintained by GHD and will consist of the following:

1. Project plan
2. Project logbooks
3. Field data records
4. Sample identification documents
5. Chain-of-custody records
6. Correspondence
7. References, literature
8. Final data packages
9. Miscellaneous - photos, maps, drawings, etc.
10. Final report

The evidentiary file materials will be the responsibility of the evidentiary file custodian with respect to maintenance and document removal.

The laboratory will be responsible for maintaining analytical logbooks and laboratory data. Raw laboratory data files will be inventoried and maintained by the laboratory for a period of five years, at which time GHD will advise the laboratory regarding the need for additional storage.

2. Data Generation and Acquisition

Details of the sample collection and monitoring procedures are presented in the following sections.

2.1 Sampling Process Design

The sampling process design is discussed in Section 1.8.

2.2 Sampling Methods

The following protocols will be employed during treated soil and filter sampling throughout this program:

- Sampling activities will be conducted in accordance with the Health and Safety Plan (HASP).
- Sampling equipment will be cleaned in accordance with the protocols presented in Section 2.3 prior to sampling at each location.
- A new pair of disposable nitrile gloves will be used at each location to be sampled for chemical analyses. Additional glove changes will be undertaken as conditions warrant.
- Sampling-generated wastes such as gloves will be collected and containerized for subsequent disposal as non-hazardous waste.
- Samples collected for off-Site chemical analyses will be placed in laboratory-supplied coolers after collection and labeling. Any remaining space will be filled with packing material to cushion

the containers within the shipment coolers. Each cooler will be sealed with a custody seal. The cooler will then be sealed with packing tape.

- Samples will be shipped to the off-Site laboratory by commercial courier or sampling personnel within 2 business days from the time of collection or as necessary to meet sample hold time requirements. In the event that samples are collected on a Saturday or during or prior to a holiday, the samples will remain in the custody of the sample team until shipped.
- Samples will remain under the control of the sampler until relinquished to the laboratory or commercial courier under chain-of-custody documentation. An example of a typical chain-of-custody record is provided as Appendix A.
- Samples not shipped will be placed in secured storage at the end of each day.
- Samples will not be stored overnight in areas other than a secured location.

Additional protocols specific to each sampling method are presented in the following sections.

Monitoring equipment and air sampling pumps will be tested and calibrated in accordance with manufactures recommended procedures.

2.3 Equipment Cleaning Protocols

All equipment used for the collection of soil samples for chemical analysis will be cleaned according to the following protocol:

- Wash thoroughly with Alconox or equivalent
- Potable water rinse
- Air dry

Rinse water will be discharged to the ground surface.

2.4 Sample Handling and Custody

Custody is one of several factors which are necessary for the admissibility of environmental data as evidence in a court of law. Custody procedures help to satisfy the two major requirements for admissibility: relevance and authenticity. Sample custody is addressed in three parts: field sample collection, laboratory analysis, and final evidence files. Final evidence files, including all original laboratory reports, are maintained under document control in a secure area.

A sample or evidence file is in a person's custody if:

- The item is in actual possession of a person
- The item is in the view of the person after being in actual possession of the person
- The item was in actual physical possession but is locked up to prevent tampering
- The item is in a designated and identified secure area

2.5 Field Custody Procedures

Logbooks or field sheets will be used to record field data collection activities. Entries into field logbooks or field sheets will be described in as much detail as possible to ensure that a particular

situation could be reconstructed solely from the entries. Field logbooks or field sheets will be stored at GHD's office when not in use. Each logbook or sheet will be identified by the project-specific document number.

The logbook entries or field sheets will contain the following information:

- Name(s) of personnel performing the work
- Project number
- Project name
- Date

Entries into the computer tablet, logbook, or field sheets will contain a variety of information. At the beginning of each day's entry, the date, start time, weather, and names of all sampling team members present will be entered. The names of individuals visiting the site or field sampling team and the purpose of their visit will also be recorded in the logbook or field sheets.

All field measurements obtained and samples collected will be recorded. All entries will be made in ink, initialed, and dated with no erasures or into a computer tablet. If an incorrect entry is made, the incorrect information will be crossed out with a single strike mark which is initialed and dated by the person making the erroneous entry. The correct information will be entered into the computer tablet, logbook, or field sheet adjacent to the original entry.

Whenever a sample is collected or a measurement is made, a detailed description of the location will be recorded in the computer tablets, logbook, or sheets.

The equipment used to collect samples, time of sample collection, sample description, and parameters to be analyzed will be recorded in the field logbook or sheets.

The sample packaging and shipping procedures summarized below will ensure that the samples arrive at the laboratory with the chain-of-custody intact:

1. The field sampler is personally responsible for the care and custody of the samples until they are transferred to another person or the laboratory. As few people as possible will handle the samples.
2. All sample containers will be identified by using sample labels which include the date of collection, unique sample number, and analyses to be performed.
3. Sample labels will be completed for each sample using waterproof ink.
4. Samples will be placed in coolers immediately after collection (if required).
5. Samples will be accompanied by a properly completed chain-of-custody form (see Appendix A). The sample identification numbers will be listed on the chain-of-custody form. When transferring the possession of samples, the individuals relinquishing and receiving the samples will sign and record the date and time on the form. The chain-of-custody form documents sample custody transfers from the sampler to another person, to the laboratory, or to/from a secure storage area.
6. Sample shipments will be accompanied by the chain-of-custody form identifying its contents. The form is completed by the sampling team, which retains a copy after signing and

relinquishing custody to the laboratory. A copy is retained by the laboratory and the fully executed copy is returned as part of the data deliverables package.

7. Samples will be properly packaged for shipment and dispatched to the appropriate laboratory for analysis, with a signed chain-of-custody form enclosed in one of the sample coolers. Shipping coolers will be secured with custody seals for shipment to the laboratory. The custody seals are then covered with clear plastic tape to prevent accidental damage to the custody seals and to keep coolers sealed during shipping.
8. If the samples are sent by common carrier, an electronic record will be retained as permanent documentation. Commercial carriers are not required to sign the chain-of-custody form as long as the form is sealed inside the sample cooler and the custody seals remain intact.
9. If samples are not shipped to the laboratory the same day the samples are collected in the field, the coolers will be sealed and kept in a designated secure area until they are shipped to the laboratory as described above.

2.6 Sample Labeling and Control

Sample labeling and control will be consistent with USEPA requirements and GHD procedures. These procedures are discussed below.

2.6.1 Labeling of Samples

A unique numbering system will be used to identify each collected sample. This system will provide a tracking number to allow retrieval and cross-referencing of sample information. A listing of the sample identification numbers with written descriptions of sample location, type, time, and date will be maintained by the on-Site sampling personnel. The sample number system to be used is described as follows:

Example: S-YYMMDD-AA-XXX

Where:

S designates sample type (WG - groundwater, W – water, A - air, S - soil)

YYMMDD date of collection (year, month, day)

AA sampler initials

XXX sequential number starting with 001 for each event

QC samples will also be numbered with a unique location number using this numbering system (except MS/MSD samples).

The on-Site sampling personnel will be responsible for recording the sampling activities for each day and will record in the computer tablet, logbook, or field sheets the following with respect to each sample:

- Unique sample identification number
- Sampling location identification
- Date/time of sample collection

- Sampling data/remarks

2.6.2 Sample Shipment

Samples will be placed in laboratory-supplied coolers after collection and labeling. Cushioning material, such as bubble wrap, will be placed around the sample containers in the cooler to protect them from breakage. All samples will be shipped within two business days and delivered overnight to the laboratory by commercial courier.

Each cooler will be sealed with a custody seal containing the sampler's initials. The cooler will then be sealed with packing tape.

Table 2.1 presents a summary of sample collection, preservation, and shipping requirements.

2.6.3 Chain-of-Custody Records

Chain-of-custody records will be used to track all samples from the time of sampling to the arrival of samples at the laboratory. Two original copies of the chain-of-custody record will accompany the sample shipment to the laboratory and will be signed and retained by the receiving laboratory's sample custodian. The chain-of-custody record will be retained by the shipper (GHD). One completed copy will be returned to the sampling contractor by the laboratory. A typical chain-of-custody form is presented on Appendix A.

2.7 Handling of Materials Generated During OU1 Removal Activities

Personal protective equipment (PPE) and other sampling generated wastes such as gloves, used filters, etc. will be collected and containerized for subsequent disposal as non-hazardous waste.

2.8 Laboratory Custody Procedures

Laboratory sample custody begins when the samples are received at the laboratory. The sample custodian will assign a unique laboratory sample identification number to each incoming sample. The field sample identification numbers, laboratory sample identification numbers, date and time of sample collection, date and time of sample receipt, and requested analyses will be entered into the sample receiving log. The laboratory sample log-in, custody, storage, and document control procedures are detailed in the appropriate SOPs in Appendix B.

2.9 Storage of Samples

Following log-in, all samples will be stored within an access-controlled location and will be maintained properly preserved until completion of all laboratory analyses. Unused sample aliquots and sample extracts/digestates/distillates will be maintained properly preserved for a minimum of 30 days following receipt of the final report by GHD. The laboratory will be responsible for the disposal of unused sample aliquots, sample containers, and sample extracts/digestates/distillates in accordance with all applicable local, state, and federal regulations.

2.10 Final Evidence Files Custody Procedures

The final evidence file for the project will be maintained by GHD and will consist of the following:

1. Project plan

2. Project logbooks/computer tablet files
3. Field data records
4. Sample identification documents
5. Chain-of-custody records
6. Correspondence
7. References, literature
8. Final laboratory reports
9. Miscellaneous - photos, maps, drawings, etc.
10. Final report

The final evidence file materials will be the responsibility of the evidentiary file custodian with respect to maintenance and document removal.

The laboratory will be responsible for maintaining analytical logbooks and laboratory data. Raw laboratory data files will be inventoried and maintained by the laboratory for a minimum period of 5 years, after which time GHD will advise the laboratory regarding the need for additional storage.

2.11 Analytical Methods

Laboratory analysis of soil samples will be performed by TestAmerica Laboratories, Inc. in University Park, Illinois. The street address and phone number are as follows:

TestAmerica Laboratories, Inc.
2417 Bond Street
University Park, Illinois 60484
Phone: 708-534-5200

The samples collected for TCLP lead analyses will be analyzed using method SW-846 1311/6010C. SOPs for laboratory methods are provided in Appendix B.

Laboratory analysis of filter air samples will be performed by TestAmerica Laboratories, Inc. in Cedar Falls, Iowa. The street address and phone number are as follows:

TestAmerica Laboratories, Inc.
704 Enterprise Drive
Cedar Falls, Iowa 50613
Phone: 319-277-2401

The samples collected for lead in air analyses will be analyzed using NIOSH method 7300. Samples will be analyzed off-site by TestAmerica. The following presents a brief discussion of each analytical technique that will be used for chemical analysis of samples collected during the OU1 removal activities.

Samples analyzed for metals will be acid digested and the digestates will be analyzed using inductively coupled plasma (ICP) emission spectrometry.

TestAmerica's SOPs for the analytical methods are presented in Appendix B. Method validation and detection limit study information for the analyses is included in TestAmerica's SOPs.

2.11.1 Level of QC Effort

To assess the quality of data resulting from the field sampling program a field duplicate sample will be collected and submitted to the laboratory.

Field blank samples for air sampling will be collected at a frequency of one per ten samples collected or one per day of sampling, whichever is greater.

Field duplicate samples for soil sampling will be collected at a frequency of one per 20 or fewer investigative soil samples. Field duplicate samples will be analyzed to check for sampling and analytical reproducibility. Field duplicate samples will be used to measure precision throughout the sampling event.

The level of QC effort provided by the laboratories for analysis of the samples will be equivalent to the level of QC effort specified in the analytical methods specified in Table 1.1.

2.12 Field Quality Control

Monitoring equipment and air sampling pumps will be tested and calibrated in accordance with manufactures recommended procedures.

2.13 Laboratory Quality Control

Internal laboratory QC procedures for the sample analyses are specified in TestAmerica's laboratory SOPs located in Appendix B. These specifications include the types and frequencies of QC checks required (e.g., method blanks, reagent/preparation blanks, matrix spike and matrix spike duplicates, calibration standards, specific calibration check standards, laboratory duplicate/replicate analysis), compounds and concentrations to be used for each QC check, and the QC acceptance criteria.

2.14 Instrument/Equipment Testing, Inspection, and Maintenance

2.14.1 Field Instruments

Monitoring equipment and air sampling pumps will be tested and calibrated in accordance with manufactures recommended procedures.

2.14.2 Laboratory Instruments

As part of their QA/QC program, the laboratory conducts a routine preventive maintenance program to minimize the occurrence of instrument failure and other system malfunctions. Designated laboratory employees will regularly perform routine scheduled maintenance and repair of (or coordinate with the instrument manufacturer for the repair of) all instruments. All maintenance that is performed will be documented in the laboratory's maintenance logbooks. All laboratory instruments are maintained in accordance with manufacturer's specifications.

Table 2.2 provides examples of routine preventive maintenance procedures and frequency at which components of key analytical instruments or equipment will be serviced. The laboratory SOPs provide complete details for instrument preventive maintenance.

Calibration of laboratory equipment will be based on approved written procedures. Records of calibration, repairs, or replacement will be filed and maintained by the designated laboratory

personnel performing quality control activities. These records will be filed at the location where the work is performed and will be subject to QA audit. For all instruments, the laboratory will maintain a properly trained repair staff with in-house spare parts or will maintain service contracts with vendors.

The records of calibration will be kept as follows:

1. Instrument calibration is tracked in the laboratory information management system (LIMS).
2. A log assigned to each instrument showing description, manufacturer, model numbers, date of last calibration, and the signature of the person who calibrated the instrument, due date of next calibration, and compensation or correction figures, as appropriate.
3. A stepwise calibration procedure will be available for each piece of test and measurement equipment.
4. Any instrument that is not calibrated to the manufacturer's original specification will display a warning tag or will otherwise be removed from service, as appropriate.

Specific calibration procedures and frequencies are detailed in the laboratory SOPs in Appendix B.

2.14.3 Inspection and Acceptance Requirements for Supplies and Sample Containers

The field supplies for the sampling activities consist of detergent (Alconox) for equipment cleaning, deionized water for sample collection equipment rinsing.

2.15 Data Acquisition Requirements (Non-Direct Measurements)

The results of sampling completed in the alley and railroad have identified lead at concentrations above the industrial and commercial property objective of 800 milligrams per kilogram (mg/kg) and exceeded the TCLP lead regulatory limit of 5.0 mg/L within a limited portion of the Site. The results of sampling completed in the alley and railroad are presented in the following documents:

- Site Assessment Report for Pilsen Soil Assessment Area: Rail Road/Alley Chicago, Cook County, Illinois Addendum 1; dated November 3, 2014
- Pilsen Soils OU1 Railroad Spur and Alley Site: Western Areas, Rail Road Spur Soil Sample Results: USEPA Memorandum dated May 22, 2015
- Pilsen Soils OU1 Railroad Spur and Alley Site: Western Areas, Rail Road Spur Reanalysis of Soil Sample ID PA-RR26—0624 for TCLP Lead: USEPA Memorandum dated August 21, 2015

2.16 Project Criteria

Project-specific criteria are presented in Table 2.3.

2.17 Measurement Performance Criteria

The measurement performance criteria for precision, accuracy, representativeness, completeness, and comparability are provided in the following subsections.

2.17.1 Precision

Precision is a measure of the degree to which two or more measurements of the same characteristic (i.e., analyte, parameter) under the same or similar conditions are in agreement.

Precision of measurements will be assessed by determining the Relative Percent Difference (RPD) between the data from either duplicate spiked sample analyses or duplicate sample analyses. RPD is calculated by dividing the absolute value of the difference between two numbers by their arithmetic mean and multiplying by 100. RPD data is used to evaluate the analytical precision of two replicate measurements (e.g., matrix spike/matrix spike duplicate). RPD data control charts are developed and maintained on a matrix and analyte-specific basis. RPD is calculated using the following simplified formula:

$$RPD = \frac{|R_1 - R_2|}{R_1 + R_2} \text{ X } 200$$

where: R_1 = value of first result
R_2 = value of second result

2.17.1.1 Laboratory Precision Criteria

Laboratory precision will be assessed through the calculation of RPDs for replicate/duplicate sample analyses. In general, these will be MS/MSD samples or lab duplicates. Precision control limits for the analyses are presented in Table 2.4.

2.17.2 Accuracy

Accuracy is the extent of agreement between an observed value (i.e., sample result) and the accepted or true value for the parameter being measured.

Accuracy of measurements will be assessed by determining the percent recovery (%R) of analytes spiked into samples or blank matrix materials. The %R of a parameter is calculated by dividing the amount recovered by the known amount added and multiplying by 100. The %R data are evaluated to establish the analytical accuracy of a measurement. Percent recovery control charts are developed and maintained on a matrix and analyte-specific basis. Percent recovery is calculated using the following formula:

$$\%R = \frac{SSR - SR}{SA} \text{ X } 100$$

where: SSR = Spiked Sample Result
SR = Sample Result or Background
SA = Spike Added

Precision and accuracy limits for the project are listed in Table 2.4.

2.17.2.1 Field Accuracy Criteria

Accuracy also will be ensured by adhering to all sample handling procedures, sample preservation requirements, and holding time periods.

2.17.2.2 Laboratory Accuracy Criteria

Laboratory accuracy will be assessed by determining percent recoveries from the analysis of LCSs or standard reference materials (SRMs). Accuracy relative to the sample matrix will be assessed by determining percent recoveries from the analysis of MS/MSD or MS/Dup samples. Accuracy control limits are presented in Table 2.4.

2.17.3 Representativeness

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

2.17.3.1 Field Representativeness Criteria

Representativeness is dependent upon the proper design of the sampling program. The representativeness criteria for field sampling will be to ensure that the sample locations are properly established on-site and off-site (as applicable) and that the sampling procedures are followed. The sampling programs were designed to provide data representative of Site conditions. During development of these programs, consideration was given to past practices, existing analytical data, physical setting and processes.

2.17.3.2 Laboratory Representativeness Criteria

The representativeness criteria for laboratory data will be to ensure that the proper analytical procedures are used for sample preparation (e.g., homogenizing the sample prior to subsampling) and sample analysis, and that sample holding times are met. Additionally, the accuracy and precision of the laboratory data affect representativeness. The laboratory representativeness criteria will include achieving the accuracy and precision criteria for the sample analyses.

2.17.4 Comparability

Comparability is an expression of the confidence with which one data set can be compared with another.

2.17.4.1 Field Comparability Criteria

The criteria for field comparability will be to ensure and document that the sampling networks designed for the CA activities are properly implemented and the sampling procedures in this QAPP are followed for the duration of the sampling program and any amendments to this QAPP.

2.17.4.2 Laboratory Comparability Criteria

The criteria for laboratory data comparability will be to ensure that the analytical methods used for the sampling and analysis events that are comparable to the methods used for previous sampling events. The analytical methods identified in Section 2.11 of this QAPP are comparable to the methods used to generate data for previous investigations.

2.17.5 Completeness

Completeness is a measure of the amount of valid data obtained from a measurement system compared to the amount that was expected to be obtained under normal conditions. Percent completeness is calculated using the following formula:

$$\% \text{ Completeness} = \frac{\text{Number of Valid (usable) measurements}}{\text{Number of Measurements Planned}} \text{ X } 100$$

The criteria for laboratory completeness will be that 85 percent or greater of the laboratory data are determined to be valid (usable) for the intended purpose. The procedure for determining laboratory data validity is provided in Section 4 of this QAPP.

2.17.6 Sensitivity

Sensitivity is the ability of a method or instrument to detect a parameter to be measured at a level of interest.

The sensitivity requirements for the laboratory analyses are provided in Table 2.3. The analytical methods are sufficiently sensitive for the project.

2.18 Data Management

The procedures for managing data from generation to final use and storage are detailed in subsections that follow.

2.18.1 Data Recording

Laboratory data are recorded in a variety of formats. Data from instruments are recorded on magnetic media, strip charts, or bench sheets. The laboratory SOPs in Appendix B provide the data recording requirement for each preparation and analysis method.

2.18.2 Data Validation

Validation of the analytical data will be performed by GHD's QA officer or their designee based on applicable guidance in "USEPA Contract Laboratory Program National Functional Guidelines for Inorganic Superfund Data Review", USEPA 540-10-011, January 2010. The evaluation and action criteria specified in this document (referred to hereafter as the National Functional Guidelines) will be used for validating the data. However, the acceptance limits for QC data will be the control limits determined statistically by the laboratory, not the control limits specified in the National Functional Guidelines. Qualifiers assigned to the data will be consistent with the data qualifiers specified in the National Functional Guidelines.

The following QC data deliverables will be evaluated on 100 percent of the data.

Inorganic Analyses

- Technical Holding Times
- Blanks
- MS/MSD or MS/Dup results
- LCS Results

- Field Duplicates
- Field Blanks

The results of the data validation process will be documented in a memorandum that specifies all limitations on the usability of the analytical data.

2.18.3 Data Transformation/Data Reduction

Field data reduction procedures will be minimal in scope compared to those implemented for laboratory data.

Laboratory data reduction procedures will be followed according to the following protocol:

1. Raw data produced and checked by the responsible analyst is turned over for independent review by another analyst.
2. The area supervisor or senior chemist reviews the data for attainment of quality control criteria established by the QAPP.
3. The area supervisor will decide whether any sample re-analysis is required.
4. Upon completion of all reviews and acceptance of the raw data by the area supervisor, a report will be generated and sent to the laboratory Project Manager.
5. The laboratory Project Manager will complete a thorough inspection of all reports.
6. Following review and approval of the preliminary report by the laboratory Project Manager, final reports will be generated and signed by the laboratory Project Manager.

Specific equations and procedures used for data reduction are contained in the SOPs in Appendix B.

2.18.4 Data Transmittal/Transfer

TestAmerica (at GHD's request) will provide electronic data deliverables (EDDs) in the EQuIS™ 4-file format. EQuIS™ is a database product from EarthSoft that uses Microsoft Access as the database engine. The laboratory data are downloaded into the EDDs directly from the LIMS, thus eliminating the possibility of manual transcription errors. The EDDs are imported into EQuIS™ and the data are maintained in the database for manipulation and presentation.

GHD's QA officer is responsible for verifying the correctness of the analytical database after the laboratory data for each event have been imported. If discrepancies between the database and hardcopy analytical reports are detected, a complete verification of the database will be performed or a new EDD will be submitted, imported, and verified as described previously.

2.18.5 Data Analysis

Soil chemical data will be tabulated and evaluated for possible soil cleanup criteria exceedences.

2.18.6 Data Assessment

Assessment of laboratory data by TestAmerica will be performed using the procedures detailed in the SOPs in Appendix B. These assessments included determining the mean, standard deviation,

relative standard deviation (RSD), percent difference, RPD, and percent recovery for certain QC elements.

Assessment of QC data for data validation purposes will include determining the mean, standard deviation, RSD, percent difference, percent recovery, RPD, and percent completeness. The statistical equations to determine percent recovery, RPD, and percent completeness are provided in Section 2.17 of this QAPP.

2.18.7 Data Tracking

Laboratory data tracking procedures are provided in the SOPs in Appendix B. These SOPs provide the procedures for tracking data from generation to reporting. TestAmerica's LIMS also provides a means for tracking data in the laboratory. The laboratory Operations Manager is ultimately responsible for data tracking in the laboratory.

Tracking of analytical data in the EQuIS™ database includes recording the laboratory generating the data, the date when the EDD was received and imported, the date when qualifiers were applied to the results, and the level of data validation performed. GHD's Project Manager is ultimately responsible for tracking data from entry into the database to reporting.

2.18.8 Data Storage and Retrieval

Electronic laboratory data are archived for a period of 5 years. Electronic instrument data are maintained on magnetic media (i.e., magnetic tape). TestAmerica's records manager is responsible for data archiving and retrieval.

GHD's Project Manager is responsible for project data storage and retrieval. Field logbooks and field sheets will be maintained at GHD's office between sampling events. Upon completion of the OU1 removal activities, the final evidence file will be archived in GHD's electronic archiving system.

2.18.9 Data Security

Laboratory data security is the responsibility of TestAmerica's records manager. Archived data cannot be accessed without authorization and the name and purpose of personnel accessing archived data are recorded. TestAmerica's LIMS is password protected and access rights are restricted by job function.

GHD's data security procedures include limiting project database access to database analysts and general building security procedures.

3. Assessment/Oversight

The following subsections describe the procedures used to ensure proper implementation of this QAPP and the activities for assessing the effectiveness of the implementation of the project and associated QA/QC activities.

3.1 Assessments and Response Actions

Assessments consisting of internal and external audits may be performed during the project. Internal technical system audits of both field and laboratory procedures will be conducted to verify

that sampling and analysis are being performed in accordance with the procedures established in the QAPP. External field and laboratory audits may be conducted by the USEPA.

An internal field technical system audit of field activities, including sampling and field measurements, may be conducted by the Field QA officer or his designee at the beginning of the field sampling activities to identify deficiencies in the field sampling and documentation procedures. The field technical system audit may include examining field sampling records and chain-of-custody documentation. In addition, sample collection, handling, and packaging in compliance with the established procedures will be reviewed during a field audit. Any deficiencies identified will be documented and corrective actions will be taken to rectify the deficiencies.

Corrective action resulting from internal field technical system audits will be implemented immediately if data may be adversely affected due to unapproved or improper use of approved methods. The Field QA officer will identify deficiencies and recommended corrective action to the Project Manager. Implementation of corrective actions will be performed by the Field QA officer and field team. Corrective action will be documented in the field logbook and/or the project file. Follow-up audits will be performed as necessary to verify that deficiencies have been corrected and that the QA/QC procedures described in this QAPP are maintained throughout the project.

An external field technical system audit may be conducted by the USEPA any time during the field operations. These audits may or may not be announced and are conducted at the discretion of the USEPA.

An internal laboratory technical system audit will be conducted by the TestAmerica QA officer or their designee. The laboratory technical system audit is conducted on an annual basis and includes examining laboratory documentation regarding sample receiving, sample log-in, storage and tracking, chain-of-custody procedures, sample preparation and analysis, instrument operating records, data handling and management, data tracking and control, and data reduction and verification. The laboratory QA officer will evaluate the results of the audit and provide a final report to section managers and the Operations Manager that includes any deficiencies and/or noteworthy observations.

Corrective action resulting from deficiencies identified during the internal laboratory technical system audit will be implemented immediately. The Operations Manager or section leaders, in consultation with the laboratory supervisor and staff, will approve the required corrective action to be implemented by the laboratory staff. The laboratory QA officer will ensure implementation and documentation of the corrective action. All problems requiring corrective action and the corrective action taken will be reported to the laboratory Project Manager. Follow-up audits will be performed as necessary to verify that deficiencies have been corrected and that the QA/QC procedures described in the QAPP are maintained throughout the project.

An external laboratory audit may be conducted by the USEPA. These audits may or may not be announced and are at the discretion of the USEPA. The external laboratory audits will include, but not be limited to, reviewing laboratory analytical procedures, laboratory on-site audits, and/or submitting performance evaluation samples to the laboratory for analysis.

An external laboratory audit may be conducted prior to the initiation of the sampling and analysis activities.

3.2 Reports to Management

Management will receive reports on the performance of the measurement system and data quality following each sampling round and at the conclusion of the project.

Minimally, these reports will include:

- Assessment of measurement quality indicators (i.e., data accuracy, precision, and completeness)
- Results of system audits
- QA problems and recommended solutions
- Amendments to QAPP

The GHD QA officer will be responsible within the organizational structure for preparing these reports. The final report for the project will include a separate QA section which will summarize data quality information contained in the periodic QA/QC reports to management and details an overall data assessment and validation in accordance with the data quality objectives outlined in this QAPP.

4. Data Verification/Validation and Usability

The QA activities that will be performed to ensure that the CA data are scientifically defensible, properly documented, of known quality, and meet the project objectives are described in the following sections.

4.1 Data Review, Verification, and Validation Requirements

All laboratory data will be reviewed and verified/validated. The procedures and criteria used to verify and validate laboratory data will consist of evaluating the data to the measurement performance criteria in Section 2.17 of this QAPP. Field data and logbooks will be reviewed to ensure that the requirements of the sampling program, including the number of samples and locations, sampling procedures, and sample handling, were fulfilled. Acceptable departures from the planned sampling program, such as collecting a sample from an adjacent location because of a subsurface obstruction, will not impact the data usability.

Sample collection procedures will be reviewed for compliance with the requirements of the QAPP. If alternate sampling procedures are used, the acceptability of the procedures will be evaluated to determine the effect on the usability of the data. Data usability will not be affected if the procedure used is determined to be an acceptable alternative that fulfills the measurement performance criteria in Section 2.17 of this QAPP. Data generated from sampling procedures that do not provide representative samples will be qualified or rejected.

Sample handling records will be reviewed to ensure that sample integrity remained intact from collection to laboratory receipt and that samples were properly preserved. Chain-of-custody documentation and sample condition upon laboratory receipt will be reviewed. The data from samples for which the chain-of-custody or sample identification cannot be verified will be rejected. The data for samples that were not properly preserved will be qualified or rejected depending on the severity of the deviation from the requirements of the QAPP. The criteria for rejecting improperly

preserved samples will be that the sample has been rendered unsuitable for analysis. An example of this situation is preserving a water sample designated for metals analysis with base. If minor pH adjustments are required at the laboratory to account for sample buffering affects, data qualification may be required. The criteria for qualifying or rejecting data for samples that are received at the laboratory without being properly preserved, but not rendered unsuitable for analysis, will be based on the sample holding time period evaluation criteria for unpreserved samples specified in the National Functional Guidelines. Data qualification will be consistent with the action specified in the National Functional Guidelines.

Laboratory data will be verified to ensure that the methods used to analyze the samples were consistent with the requirements of this QAPP. Data generated from the use of unapproved methods will be rejected.

QC data will be reviewed to determine compliance with the acceptance criteria in Sections 2.16 and 2.17 of this QAPP as well as Tables 2.3 and 2.4. QC data that do not meet the acceptance criteria will result in sample data qualification. Significant departures from the QC acceptance criteria may result in rejected data. Situations that result in data rejection include samples analyzed beyond twice the technical holding time period, inorganic LCS analyte recoveries less than 40 percent if the analyte is not detected in the associated samples, and inorganic matrix spike analyte recoveries less than 30 percent if the analyte is not detected in the associated samples.

4.2 Verification and Validation Methods

TestAmerica will internally verify the laboratory data by reviewing and documenting sample receipt, sample preparation, sample analysis (including internal QC checks), data reduction, and reporting. Any deviations from the acceptance criteria, corrective actions taken, and data determined to be of limited usability (i.e., laboratory-qualified data) will be noted in the case narrative of the laboratory report.

Data validation will be conducted by GHD consistent with the procedure identified in Section 2.18.2 of this QAPP. The data validation procedure will identify data as being acceptable, of limited usability (qualified as estimated), or rejected. The conditions that result in data being qualified as estimated or rejected are identified in Section 4.1 of this QAPP. The results of the data validation will be provided in data validation memoranda that are provided to GHD's Project Manager and are included in Quality Assurance Management Reports.

Data determined to be unusable may require that corrective action to be taken. Potential types of corrective action may include resampling by the field team or reanalysis of samples by the laboratory. The corrective actions taken are dependent upon the ability to mobilize the field team and whether the data are critical for project DQOs to be achieved. Should GHD's QA officer identify a situation requiring corrective action during data verification/validation, GHD's Project Manager will be responsible for approving the implementation of the corrective action.

4.3 Usability/Reconciliation with Data Quality Objectives

The overall usability of the data will be assessed by evaluating the PARCCS of the data set to the measurement performance criteria in Section 2.17 of this QAPP using basic statistical quantities as applicable. The procedures and statistical formulas to be used for these evaluations are presented in the following subsections.

4.3.1 Sensitivity and Quantitation Limits

The quantitation limits for the sample data will be reviewed to ensure that the sensitivity of the analyses was sufficient to achieve Target Cleanup Criteria. The method/preparation blank sample data and LCSs percent recovery data will be reviewed to assess compliance with the measurement performance criteria specified in Section 2.17 and Tables 2.3 and 2.4 of this QAPP.

Overall sensitivity will be assessed by comparing the sensitivity for each monitoring program to the detectability requirements for the analyses. Overall sensitivity will be considered acceptable if quantitation limits for the samples are less than the applicable evaluation criteria in Table 2.3.

It should be noted that quantitation limits may be elevated as a result of high concentrations of target compounds, non-target compounds, and matrix interferences (collectively known as sample matrix effects). In these cases, the sensitivity of the analyses will be evaluated on an individual sample basis relative to the applicable evaluation criteria. The need to investigate the use of alternate analytical methods may be required if the sensitivity of the analytical methods identified in this QAPP cannot achieve the evaluation criteria because of sample matrix effects.

4.3.2 Data Limitations and Actions

Data use limitations will be identified in data quality assessment reports. Data that do not meet the measurement performance criteria specified in this QAPP will be identified and the impact on the project quality objectives will be assessed and discussed in these reports. Specific actions for data that do not meet the measurement performance criteria depend on the use of the data and may require that additional samples are collected or the use of the data be restricted.

Data quality assessment reports will be prepared at the conclusion of each sampling event. Determination of the overall data quality for a specific sampling program will be conducted at the completion of the program. Data quality assessment reports will be included with the project reports.

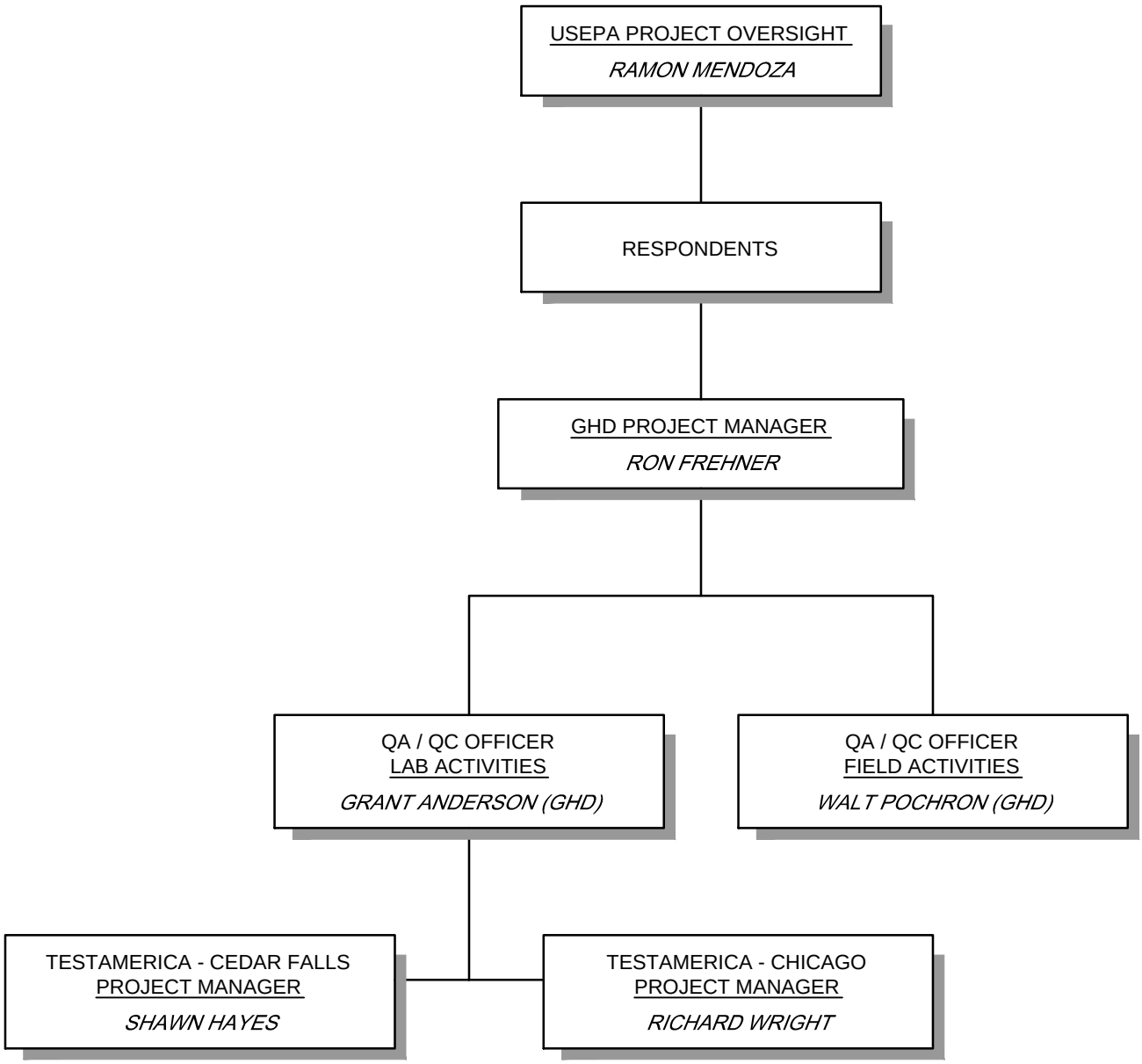
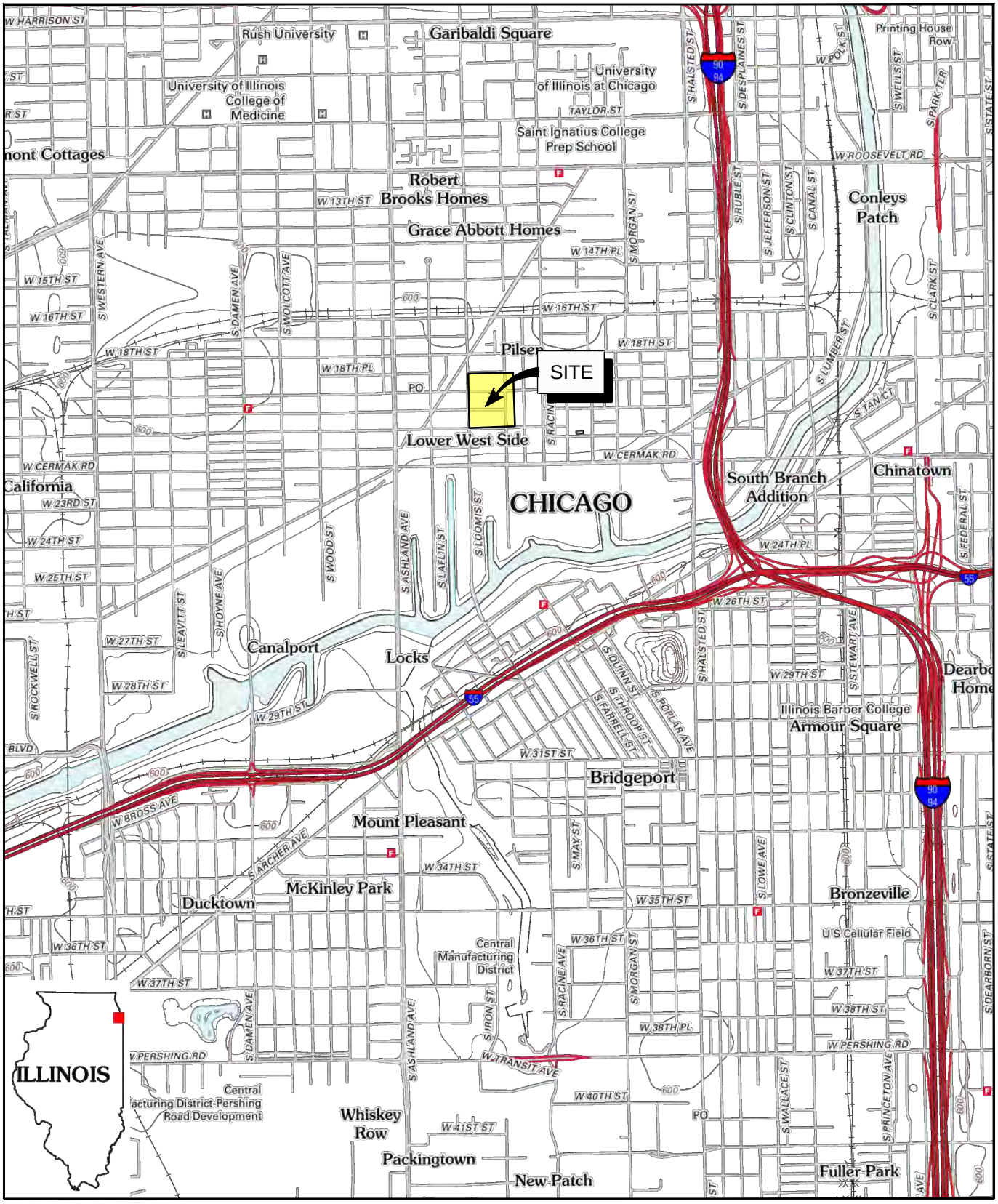


figure 1.1

PROJECT ORGANIZATION
OU1 PILSEN AREA
Chicago, Illinois



BASE SOURCE: USGS 7.5 MINUTE TOPOGRAPHIC QUADRANGLE; ENGLEWOOD, ILLINOIS 2012

0 1200 2400ft

OU1 PILSEN AREA
CHICAGO, ILLINOIS

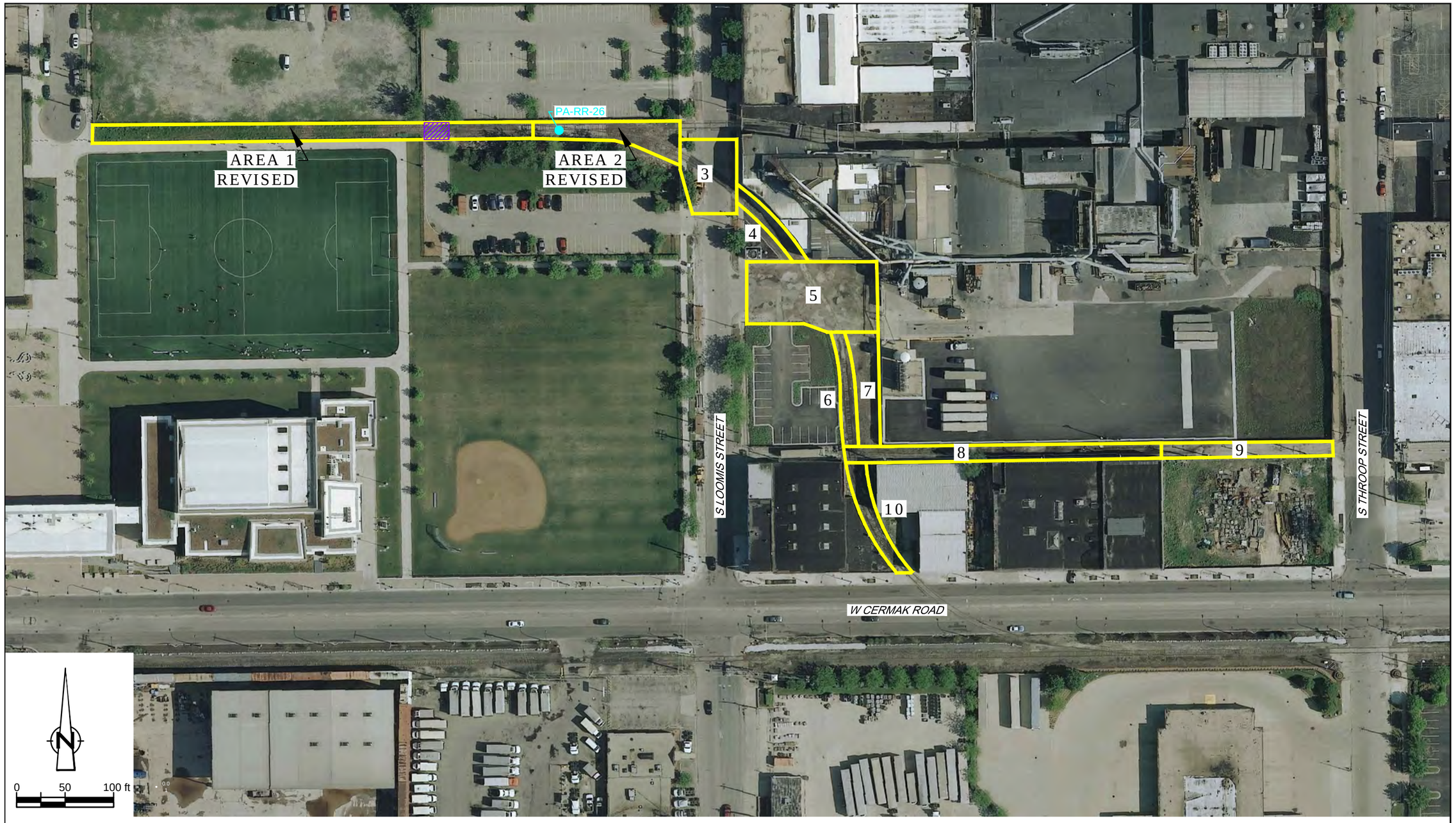
SITE LOCATION

39826-01

Sep 16, 2015

FIGURE 1.2

CAD File: I:\DRAWINGS\30000s\39826\39826-REPORT\39826-01(018)\39826-01(018)GN\39826-01(018)GN-CO001.dwg



LEGEND:

1 REMEDIATION AREA

PA-RR-26 EPA SAMPLE LOCATION AND IDENTIFIER

PAVED DRIVEWAY IS NOT INCLUDED IN THE OUI REMEDIATION AREA

GHD

figure 1.3

OUI REMEDIATION AREAS
PILSEN SITE
Chicago, Illinois

39826-01(018)GN-CO002 SEP 17/2015

Table 1.1

Summary of Sampling and Analysis Program
OU1 Pilsen Area
Chicago, Illinois

Sample Matrix	Field Parameters	Laboratory Parameters	Investigative Samples	Field Duplicates	Field Blanks	Total Number of Samples
Soil	None	TCLP Lead (SW 1311/6010C)	8	1	0	9
Air	None	Lead in air (NIOSH 7300)	30	0	1 in 10[(1)]	33

Note:

[(1)] - Field blanks for lead in air will be collected 1 in 10 samples collected or minimum 1 per day of sampling

Table 2.1

Container, Preservation, Shipping and Packaging Requirements
OU1 Pilsen Area
Chicago, Illinois

Analyses	Sample Containers	Preservation	Maximum Holding Time From Sample Collection[(1)]	Volume of Sample	Shipping	Normal Packaging
Soil						
TCLP Lead	One 8-oz jar	None	6 months to TCLP extraction/ 6 months from extraction to analysis	Fill to neck of jar	Overnight	Foam Liner or Bubble-wrap
Air						
Lead in air	Filter	--	6 months	varies	Overnight	Foam Liner or Bubble-wrap

Notes:

[(1)] - Maximum holding time periods were consistent with methods referenced in SW-846, "Test Methods for Evaluating Solid Waste", Third Edition, 1986.

Table 2.2

Routine Preventive Maintenance Procedures and Schedules
OU1 Pilsen Area
Chicago, Illinois

Instrument	Maintenance Procedures/Schedule	Spare Parts in Stock
Inductively Coupled Plasma Spectrometer	1. Clean torch assembly and mixing chamber when discolored or after 8 hours of running high dissolved solid samples 2. Clean nebulizer as needed 3. Check to ensure the gas supply is sufficient for the day's activity and the delivery pressures are set as described in the SOP	1. Torch and mixing chamber 2. Nebulizer
Autoanalyzer	1. Inspect pump tubes after each 8-hour run; replace if discolored or distorted 2. Inspect colorimeter daily; replace lamp as necessary	1. Pump tubes 2. Colorimeter lamp
TCLP Extractor	1. Keep area and equipment clean and free of contaminants 2. Check pH probe periodically for bubbles. Replace as needed.	1. Spare pH probe

Table 2.3

Targeted Project Criteria
OU1 Pilsen Area
Chicago, Illinois

Method	Parameter	Targeted Quantitation Limit[(1)] (mg/L)	Method Detection Limit[(2)] (mg/L)	Soil Cleanup Criteria (mg/L)
SW 1311/6010C	TCLP Lead	0.0500	0.00750	5
NIOSH 7900	Lead in air	2.5 µg/filter	0.14 µg/filter	--

Notes:

(1) - Targeted quantitation limits are presented for guidance and may not be achievable for all samples as a result of matrix interferences or high concentrations of target and non-target compounds.

(2) - Method detection limits are presented for guidance; these limits are updated periodically by the lab.

Table 2.4

Percent Recovery and Relative Percent Difference Control Limits
OU1 Pilsen Area
Chicago, Illinois

	% Recovery Control Limits[(1)]	
Soil Analyses	**LCS**	**MS/MSD or MS/DUP**
TCLP Lead	80-120 (20)	50-150 (20)
Lead in air	85-110 (10)	NA

Note:

(1) - Values in parentheses are the maximum RPD values allowed for LCS/LCSD and MS/MSD analyses. Laboratory control limits are updated on a periodic basis and the control limits in effect when the samples are analyzed will be used for data validation purposes.

Appendix A
Example Chain-of-Custody

CHAIN OF CUSTODY RECORD

COC NO.: PL-24567

PAGE ___ OF ___

(See Reverse Side for Instructions)

Project No/ Phase/Task Code:	Laboratory Name:	Lab Location:	SSOW ID:
Project Name:	Lab Contact:	Lab Quote No:	Cooler No:
Project Location:			Carrier:
Chemistry Contact:			Airbill No:
Sampler(s):			Date Shipped:

Item	Sample Identification (Containers for each sample may be combined on one line)	Date (mm/dd//yy)	Time (hh:mm)	Sample Type: Matrix Code (see back of COC)	Sample Type: Grab (G) or Comp (C)	Container Quantity & Preservation: Unpreserved	Hydrochloric Acid (HCl)	Nitric Acid (HNO_3)	Sulfuric Acid (H_2SO_4)	Sodium Hydroxide (NaOH)	Methanol/Water (Soil VOC)	EnCores 3x5-g, 1x25-g	Other:	Total Containers/Sample	Analysis Requested (See Back of COC for Definitions)											MS/MSD Request	Comments/ Special Instructions:
1																										
2																										
3																										
4																										
5																										
6																										
7																										
8																										
9																										
10																										
11																										
12																										
13																										
14																										
15																										

TAT Required in business days (use separate COCs for different TATs):

☐ 1 Day ☐ 2 Days ☐ 3 Days ☐ 1 Week ☐ 2 Week ☐ Other:

Total Number of Containers:

All Samples in Cooler must be on COC

Notes/ Special Requirements:

Relinquished By	Company	Date	Time	Received By	Company	Date	Time
1.				1.			
2.				2.			
3.				3.			

THE CHAIN OF CUSTODY IS A LEGAL DOCUMENT – ALL FIELDS MUST BE COMPLETED ACCURATELY

Distribution: WHITE – Fully Executed Copy YELLOW – Receiving Laboratory Copy PINK – Shipper GOLDENROD – Sampling Crew

Appendix B
Laboratory Standard Operating Procedures

August 29, 2014

Laboratory ID: 101044

Michael McGee
TestAmerica Laboratories, Inc.
Cedar Falls Division
704 Enterprise Drive
Cedar Falls, IA 50613

Dear Mr. McGee:

Congratulations! The AIHA Laboratory Accreditation Programs (AIHA-LAP), LLC's Analytical Accreditation Board (AAB) has approved TestAmerica Laboratories, Inc. as an accredited Industrial Hygiene laboratory.

Accreditation documentation includes the IHLAP accreditation certificate, scope of accreditation document and a copy of the current AIHA-LAP, LLC license agreement (if your completed agreement is not on file at AIHA-LAP, LLC). The accreditation logo has been designed for use by all AIHA-LAP, LLC accredited laboratories. If your laboratory chooses to use the logo in its advertising the laboratory's accreditation, you must complete and return the AIHA-LAP, LLC license agreement to a Laboratory Accreditation Specialist. Once submitted, an electronic copy of the accreditation logo will be sent to you. Please inform us if your laboratory does not wish to use the logo in advertising.

Laboratory accreditation shall be maintained by continued compliance with IHLAP requirements *(see Policy Modules 2B and 6)*, which includes proficient participation in AIHA-LAP, LLC approved proficiency testing, demonstration of competency, or round robin program as indicated on the AIHA-LAP "Approved PT and Round Robin" webpage, its associated PT-Scope table, and as required in Policy Module 6, for all Fields of Testing (FoTs) for which the laboratory is accredited. An accredited laboratory that wishes to expand into a new FoT must submit an updated accreditation application to AIHA-LAP, LLC for review by the AAB.

Any changes in ownership, laboratory location, personnel, FoTs/Methods, or significant procedural changes shall be reported to AIHA-LAP, LLC in writing within twenty (20) business days of the change.

The accreditation certificate is the property of AIHA-LAP, LLC and must be returned to us should your laboratory withdraw or be removed from the Industrial Hygiene.

Again, congratulations. If you have any questions, please contact Lauren Maher, Laboratory Accreditation Specialist, at (703) 846-0716.

Sincerely,

Cheryl O. Morton

Cheryl O. Morton
Managing Director
AIHA Laboratory Accreditation Programs, LLC

AIHA Laboratory Accreditation Programs, LLC

acknowledges that

TestAmerica Laboratories, Inc.

Cedar Falls Division, 704 Enterprise Drive, Cedar Falls, IA 50613

Laboratory ID: 101044

along with all premises from which key activities are performed, as listed above, has fulfilled the requirements of the AIHA Laboratory Accreditation Programs (AIHA-LAP), LLC accreditation to the ISO/IEC 17025:2005 international standard, *General Requirements for the Competence of Testing and Calibration Laboratories* in the following:

LABORATORY ACCREDITATION PROGRAMS

✓	**INDUSTRIAL HYGIENE**	Accreditation Expires: 11/01/2016
☐	**ENVIRONMENTAL LEAD**	Accreditation Expires:
☐	**ENVIRONMENTAL MICROBIOLOGY**	Accreditation Expires:
☐	**FOOD**	Accreditation Expires:
☐	**UNIQUE SCOPES**	Accreditation Expires:

Specific Field(s) of Testing (FoT)/Method(s) within each Accreditation Program for which the above named laboratory maintains accreditation is outlined on the attached **Scope of Accreditation**. Continued accreditation is contingent upon successful on-going compliance with ISO/IEC 17025:2005 and AIHA-LAP, LLC requirements. This certificate is not valid without the attached **Scope of Accreditation**. Please review the AIHA-LAP, LLC website (www.aihaaccreditedlabs.org) for the most current Scope.

Gerald R Schultz

Gerald Schultz, CIH
Chairperson, Analytical Accreditation Board

Cheryl O. Morton

Cheryl O. Morton
Managing Director, AIHA Laboratory Accreditation Programs, LLC

Revision 14: 03/26/2014

Date Issued: 08/29/2014

AIHA Laboratory Accreditation Programs, LLC
SCOPE OF ACCREDITATION

TestAmerica Laboratories, Inc.
Cedar Falls Division, 704 Enterprise Drive, Cedar Falls, IA 50613

Laboratory ID: **101044**
Issue Date: 08/29/2014

The laboratory is approved for those specific field(s) of testing/methods listed in the table below. Clients are urged to verify the laboratory's current accreditation status for the particular field(s) of testing/Methods, since these can change due to proficiency status, suspension and/or withdrawal of accreditation.

Industrial Hygiene Laboratory Accreditation Program (IHLAP)

Initial Accreditation Date: 12/01/1985

IHLAP Scope Category	Field of Testing (FoT)	Technology sub-type/ Detector	Published Reference Method/Title of In-house Method	Method Description or Analyte (*for internal methods only*)
Chromatography Core	Gas Chromatography	GC/FID	NIOSH 1003	
			NIOSH 1005	
			NIOSH 1022	
			NIOSH 1300	
			NIOSH 1500	
			NIOSH 1501	
			NIOSH 1550	
			NIOSH 1615	
			OSHA 07	
	Gas Chromatography (Diffusive Samplers)		NIOSH 1003	
			NIOSH 1005	
			NIOSH 1022	
			NIOSH 1300	
			NIOSH 1500	
			NIOSH 1501	
			NIOSH 1550	
			NIOSH 1615	
			OSHA 07	
Spectrometry Core	Inductively-Coupled Plasma	ICP/AES	NIOSH 7303	
			NIOSH 9102	
Miscellaneous Core	Gravimetric		NIOSH 0500	
			NIOSH 0600	

A complete listing of currently accredited Industrial Hygiene laboratories is available on the AIHA-LAP, LLC website at: http://www.aihaaccreditedlabs.org

Title: Determination of Metals and Trace Elements in Water, Wastes and IH Media by Inductively Couples Plasma-Atomic Emission Spectrometry (ICP-AES)

Computer File Name: F:\TjadenT\QA Folder\SOP\Metals\CF-EMA-01_3Mod1.doc

Approvals (Signature/Date):

Lorna Bormann Inorganics Technical Manager	8/27/12 Date
Chad Timmins Environmental Health & Safety Coordinator	8-27-12 Date
Michael McGee Laboratory Director	08/27/12 Date
Thomas Tjaden Quality Assurance Manager	8-27-12 Date

Modification:

Section 9.2.6 is modified as follows and section 9.2.6.1 has been added:

9.2.6 Linear Dynamic Range (LDR): The upper limit of the LDR must be established for each wavelength utilized. It must be determined from a linear calibration prepared in the normal manner using the established analytical operating procedure for the instrument. The LDR should be determined by analyzing succeedingly higher standard concentrations of the analyte until the observed analyte concentration is no more than 10% below the stated concentration of the standard or if there is a significant reduction from the typical recovery. Determined LDRs must be documented and kept on file. The LDR which may be used for the analysis of samples should be judged by the analyst from the resulting data. Determined sample analyte concentrations that are greater than 90% of the determined upper LDR limit must be diluted and reanalyzed. The LDRs should be verified ~~annually or~~ whenever in the judgment of the analyst, a change in analytical performance caused by either a change in instrument hardware or operating conditions would dictate that they be redetermined.

9.2.6.1. At a minimum, the range should be checked every six months using a single point standard prepared at the upper limit, analyzed and quantitated against the normal calibration curve. The calculated value should be within 10% (±10%) of the true value.

Facility Distribution No.: CF **Distributed To:** _____

TestAmerica
THE LEADER IN ENVIRONMENTAL TESTING

Title: Determination Of Metals And Trace Elements In Water, Wastes, and IH Media by Inductively Coupled Plasma-Atomic Emission Spectrometry (ICP-AES)

Computer File Name: F:\DeimerlyC\QA Folder\SOP\Metals\CF-EMA-01_3.doc

Operations Manager Approval — Date: 2/7/12	Environmental Health and Safety Approval — Date: 2-7-12
	Laboratory Director Approval — Date: 02/07/12
	Quality Assurance Manager Approval — Date: 2-7-12

Facility Distribution No.: CF **Distributed To:** _____

This method may involve hazardous materials, operations and equipment. This method does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of the method to follow appropriate safety, waste disposal and health practices under the assumption that all samples and reagents are potentially hazardous. Safety glasses, gloves, lab coats and closed toe shoes are a minimum. For specific hazard(s) see reagents, materials and procedure sections of this SOP.

Method Reference: "Methods for the Determination of Metals in Environmental Samples – Supplement I," EPA Doc. 600/R-94/111, May 1994
METHOD #: EPA 200.7 Revision 4.4 and EPA 200.2 Revision 2.8

Method Reference: *Test Methods for Evaluating Solid Waste Physical Chemical Methods* (SW-846) Third Edition, as amended by Update III, December 1996, EPA Office of Solid Waste and Emergency Response
METHOD #: EPA 6010B

Method Reference: *Test Methods for Evaluating Solid Waste Physical Chemical Methods* (SW-846) Third Edition, as amended by Update IV, February 2007, EPA Office of Solid Waste and Emergency Response
METHOD #: EPA 6010C

Modifications:

Item	Method	Modification

1. Scope and Application

1.1 Analytes: This method is applicable to the following analytes:

Analyte	Symbol	CASRN	Reporting Limit Aqueous (mg/L)	Reporting Limit Solid (mg/Kg)	Reporting Limit Cassette (µg)	Reporting Limit Wipe (µg)
Aluminum	Al	7429-90-5	0.10	5.0	5.0	5.0
Antimony	Sb	7440-36-0	0.10	5.0	2.5	5.0
Arsenic	As	7440-38-2	0.080	4.0	5.0	20.0
Barium	Ba	7440-39-3	0.010	0.50	0.25	0.50
Beryllium	Be	7440-41-7	0.010	0.50	0.25	0.50
Boron	B	7440-42-8	0.10	5.0	NA	NA
Cadmium	Cd	7440-43-9	0.020	1.0	0.50	1.0
Calcium	Ca	7440-70-2	1.0	50	25	5.0
Chromium	Cr	7440-47-3	0.020	1.0	2.5	1.0
Cobalt	Co	7440-48-4	0.020	1.0	0.50	1.0
Copper	Cu	7440-50-8	0.020	1.0	0.50	1.0
Indium	In	7440-74-6	0.50	25	NA	NA
Iron	Fe	7439-89-6	0.10	5.0	10	20
Lead	Pb	7439-92-1	0.10	5.0	2.5	5.0
Lithium	Li	7439-93-2	0.050	2.5	2.5	5.0
Magnesium	Mg	7439-95-4	1.0	50	25	50
Manganese	Mn	7439-96-5	0.010	0.50	0.25	0.50
Molybdenum	Mo	7439-98-7	0.050	2.5	2.5	2.5
Nickel	Ni	7440-02-0	0.050	2.5	1.2	5.0
Potassium	K	7440-02-0	1.0	50	25	125
Selenium	Se	7782-49-2	0.15	7.5	5.0	7.5
Silver	Ag	7440-22-4	0.020	1.0	0.50	1.0
Sodium	Na	7440-23-5	1.0	50	25	195
Strontium	Sr	7440-24-6	0.10	5.0	2.5	5.0
Thallium	Tl	7440-28-0	0.1	10	25	50
Tin	Sn	7440-31-5	0.10	5.0	2.5	10
Titanium	Ti	7440-32-6	0.050	2.5	1.2	2.5
Vanadium	V	7440-62-2	0.050	2.5	1.2	2.5
Zinc	Zn	7440-66-6	0.020	1.0	2.5	10

1.2 Matrices: Ground and surface water, wastewater, soil, sludge, TCLP leachates, and drinking waters. Digestates prepared from Industrial Hygiene matrices are also analyzed according to procedures in this SOP.

1.3 Reporting Limits and Units: See the table in Section 1.1.

1.4 Method Detection Limit: The most recent MDL study is located near the analyzing instrument. The Quality Assurance Manual (CF-QA-01) describes the procedure for determining a MDL

2. Summary of Method

2.1 A sample aliquot is accurately measured for sample processing. For total metal analysis refluxing with a varying combination of nitric acid, hydrochloric acid, and hydrogen peroxide first solubilizes analytes. After cooling, the sample is made up to volume for analysis. For the determination of dissolved analytes in a filtered aqueous sample aliquot, or for the "direct analysis" determination of total metal analytes in drinking water where sample turbidity is < 1 NTU, no sample preparation steps are required. Drinking water with a turbidity >1 will require digestion by method 200.2

2.2 The analysis described in this method involves multi-elemental determinations by ICP-AES using sequential or simultaneous instruments. The instruments measure characteristic atomic-line emission spectra by optical spectrometry. Samples are nebulized and the resulting aerosol is transported to the plasma torch. Element specific emission spectra are produced by a radio-frequency inductively coupled plasma. The spectra are dispersed by a grating spectrometer, and the intensities of the line spectra are monitored at specific wavelengths by a photosensitive device. Photocurrents from the photosensitive device are processed and controlled by a computer system. A background correction technique is required to compensate for variable background contribution to the determination of the analytes. Background must be measured adjacent to the analyte wavelength during analysis. Various interferences must be considered and addressed appropriately as discussed in Sections 5, 7, 9, 10, and 11.

3. 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

3.1.1 The ICP plasma emits strong UV light and is harmful to vision. All analysts must avoid looking directly at the plasma.

3.1.2 When working with concentrated acids, employees must wear safety glasses with permanently affixed side shields, a labcoat, nitrile gloves, and a face shield.

3.1.3 Concentrated acids must be used in a hood.

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.
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. 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." Waste disposal procedures are incorporated by reference to the laboratory's Waste Disposal SOP (CF-WD-01).

4.1 Waste Streams Produced by the Method

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

Acid waste consisting of sample and rinse solution: Employees are to transfer acid waste to SAA bottles in the waste room to use for neutralization of other lab samples. If the SAA is full, neutralize and dispose through the sanitary sewer system.

5. Interferences/Comments/Definitions

5.1 Spectral interferences are caused by background emission from continuous or recombination phenomena, stray light from the line emission of high concentration elements, overlap of a spectral line from another element, or unresolved overlap of molecular band spectra.

5.2 Subtracting the background emission determined by measurement(s) adjacent to the analyte wavelength peak can usually compensate for background emission and stray light. Spectral scans of samples or single element solutions in the analyte regions may indicate not only when alternate wavelengths are desirable because of severe spectral interference, but also will show whether the most appropriate estimate of the background emission is provided by an interpolation from measurements on both sides of the wavelength peak or by the measured emission on one side or the other. The location(s) selected for the measurement of background intensity will be determined by the complexity of the spectrum adjacent to the wavelength peak. The location(s) used for routine measurement must be free of off-line spectral interference (interelement or molecular) or adequately corrected to reflect the same change in background intensity as occurs at the wavelength peak.

5.3 Spectral overlaps may be avoided by using an alternate wavelength or can be compensated for by equations that correct for interelement contributions, which involves measuring the interfering elements. Some potential on-line spectral interference observed for the recommended wavelengths are given in Attachment 3. When operative and uncorrected, these interferences will produce false-positive determinations and be reported as analyte concentrations. The interferences listed are only those that occur between method analytes. Only interferences of a direct overlap nature that were observed with a single instrument having a working resolution of 0.035 nm are listed. More extensive information on interferant effects at various wavelengths and resolutions is available in Boumans' Tables in Method 200.7. Users may apply interelement correction factors determined on their instruments within tested concentration ranges to compensate (off-line or on-line) for the effects of interfering elements.

5.4 When interelement corrections are applied, there is a need to verify their accuracy by analyzing spectral interference check solutions as described in Section 7.6. Interelement corrections will vary for the same emission line among instruments because of differences in resolution, as determined by the grating plus the entrance and exit slit widths, and by the order of dispersion. Interelement corrections will also vary depending upon the choice of background correction points. Selecting a background correction point where an interfering emission line may appear should be avoided when practical. Interelement corrections that constitute a major portion of an emission signal may not yield accurate data. Users should not forget that some samples might contain uncommon elements that could contribute spectral interferences.

5.5 The interference effects must be evaluated for each individual instrument whether configured as a sequential or simultaneous instrument. For each instrument, intensities will vary not only with optical resolution but also with operating conditions (such as power, viewing height and argon flow rate). When using the recommended wavelengths given in Table 1, the analyst is required to determine and document for each wavelength the effect from the known interferences given in

Table 2, and to utilize a computer routine for their automatic correction on all analyses. To determine the appropriate location for off-line background correction, the user must scan the area on either side adjacent to the wavelength and record the apparent emission intensity from all other method analytes. This spectral information must be documented and kept on file. The location selected for background correction must be either free of off-line interelement spectral interference or a computer routine must be used for their automatic correction on all determinations. If a wavelength other than the recommended wavelength is used, the user must determine and document both the on-line and off-line spectral interference effect from all method analytes and provide for their automatic correction on all analyses. Tests to determine the spectral interference must be done using analyte concentrations that will adequately describe the interference. Normally, 100 mg/L single element solutions are sufficient, however, for analytes such as iron that may be found at high concentration a more appropriate test would be to use a concentration near the upper LDR limit. See Section 10.3.3.4.2 for required spectral interference test criteria.

5.6 Physical interferences are effects associated with the sample nebulization and transport processes. Changes in viscosity and surface tension can cause significant inaccuracies, especially in samples containing high dissolved solids or high acid concentrations. If physical interferences are present, they must be reduced by such means as a high-solids nebulizer, diluting the sample, using a peristaltic pump, or using an appropriate internal standard element. Another problem that can occur with high dissolved solids is salt buildup at the tip of the nebulizer, which affects aerosol flow rate and causes instrumental drift. A high-solids nebulizer, wetting the argon prior to nebulization, using a tip washer, or diluting the sample can control this problem. Also, it has been reported that better control of the argon flow rates, especially for the nebulizer, improves instrument stability and precision; this is accomplished with the use of mass flow controllers.

5.7 Chemical interferences include molecular-compound formation, ionization effects, and solute-vaporization effects. Normally, these effects are not significant with the ICP-AES technique. If observed, they can be minimized by careful selection of operating conditions (such as incident power and observation height), by buffering of the sample, by matrix matching, and by standard-addition procedures. Chemical interferences are highly dependent on matrix type and the specific analyte element.

5.8 Memory interferences result when analytes in a previous sample contribute to the signals measured in a new sample. Memory effects can result from sample deposition on the uptake tubing to the nebulizer, and from the buildup of sample material in the plasma torch and spray chamber. The site where these effects occur is dependent on the element and can be minimized by flushing the system with a rinse blank between samples. The possibility of memory interferences should be recognized within an analytical run and suitable rinse times should be used to reduce them. The rinse times necessary for a particular element must be estimated prior to analysis. This may be achieved by aspirating a standard containing elements corresponding to either their LDR or a concentration ten times those usually encountered. The aspiration time should be the same as a normal sample analysis period, followed by analysis of the rinse blank at designated intervals. The length of time required to reduce analyte signals to within a factor of two of the method detection limit should be noted. Until the required rinse time is established, this method requires a rinse period of at least 60 sec between samples and standards. If memory interference is suspected, the sample must be re-analyzed after a long rinse period. Memory effects are suspected if the %RSD of 3 consecutive analyses for an element is >10%.

5.9 The total metal sample digestion procedure given in this method will solubilize and hold in solution only minimal concentrations of barium in the presence of free sulfate. For the analysis of barium in samples having varying and unknown concentrations of sulfate, analysis should be completed as soon as possible after sample preparation.

5.10 Definitions

5.10.1 Calibration Blank - A volume of reagent water acidified with the same acid matrix as in the calibration standards. The calibration blank is a zero standard and is used to calibrate the ICP instrument.

5.10.2 Calibration Standard (CAL) - A solution prepared from the dilution of stock standard solutions. The CAL solutions are used to calibrate the instrument response with respect to analyte concentration (Sect. 7.8).

5.10.3 Dissolved Metal Analyte - The concentration of analyte in an aqueous sample that will pass through a 0.45-um membrane filter assembly prior to sample acidification. The concentration of analyte determined either by "direct analysis" of filtered acid preserved water sample (w/out color or odor), or by analysis of the solution extract of a solid sample or an unfiltered aqueous sample following digestion by refluxing with hot dilute mineral acid(s).

5.10.4 Field Reagent Blank (FRB) - An aliquot of reagent water or other blank matrix that is placed in a sample container in the laboratory and treated as a sample in all respects, including shipment to the sampling site, exposure to the sampling site conditions, storage, preservation, and all analytical procedures. The purpose of the FRB is to determine if method analytes or other interferences are present in the field environment.

5.10.5 Instrument Detection Limit (IDL) - The concentration equivalent to the analyte signal which is equal to three times the standard deviation of a series of ten replicate measurements of the calibration blank signal at the same wavelength.

5.10.6 Instrument Performance Check (IPC) Solution - A solution of method analytes, used to evaluate the performance of the instrument system with respect to a defined set of method criteria (Sects. 7.6 & 9.2.7). This is also known as the continuing calibration verification (CCV) and will be referred to as such throughout the remainder of this SOP.

5.10.7 Internal Standard - Pure analyte(s) added to a sample, extract, or standard solution in known amount(s) and used to measure the relative responses of other method analytes that are components of the same sample or solution. The internal standard must be an analyte that is not a sample component.

5.10.8 Laboratory Duplicates (LD1 and LD2) - Two aliquots of the same sample taken in the laboratory and analyzed separately with identical procedures. Analyses of LD1 and LD2 indicate precision associated with laboratory procedures, but not with sample collection, preservation, or storage procedures.

5.10.9 Laboratory Fortified Blank (LFB) - An aliquot of LRB to which known quantities of the method analytes are added in the laboratory. The LFB is analyzed exactly like a sample, and its purpose is to determine whether the methodology is in control and whether the laboratory is capable of making accurate and precise measurements (Sect. 9.1.2). This is also known as the Laboratory Check Sample (LCS) and will be referred to as such throughout the remainder of this SOP.

5.10.10 Laboratory Fortified Sample Matrix (LFM) - An aliquot of an environmental sample to which known quantities of the method analytes are added in the laboratory. The LFM is analyzed exactly like a sample, and its purpose is to determine whether the sample matrix contributes bias to the analytical results. The background concentrations of the analytes in the sample matrix must be determined in a separate aliquot and the measured values in the LFM corrected for background concentrations (Sect. 9.1.3). This is also known as the Matrix Spike (MS). If a duplicate of the MS is prepared and analyzed, the duplicate is the Matrix Spike Duplicate (MSD) and will be referred to as such throughout the remainder of this SOP.

5.10.11 Laboratory Reagent Blank (LRB) - An aliquot of reagent water or other blank matrices that are treated exactly as a sample including exposure to all glassware, equipment, solvents, reagents, and internal standards that are used with other samples. The LRB is used to determine if method analytes or other interferences are present in the laboratory environment, reagents, or apparatus (Sect. 9.1.1). This is also known as the Procedure Blank and will be referred to as such throughout the remainder of this SOP.

5.10.12 Linear Dynamic Range (LDR) - The concentration range over which the instrument response to an analyte is linear (Sect. 9.2.6).

5.10.13 Method Detection Limit (MDL) - The minimum concentration of an analyte that can be identified, measured, and reported with 99% confidence that the analyte concentration is greater than zero.

5.10.14 Plasma Solution - A solution that is used to determine the optimum height above the work coil for viewing the plasma.

5.10.15 Quality Control Sample (QCS) - A solution of method analytes of known concentrations. The QCS is obtained from a source external to the laboratory and different from the source of calibration standards. It is used to check either laboratory or instrument performance. This is also known as the Initial Calibration Verification (ICV) solution and will be referred to as such throughout the remainder of this SOP.

5.10.16 Reporting Limit Verification Standard – A solution of method analytes of known concentrations which is used to validate instrument response at the reporting limit. The RLVS is prepared from a dilution of the stock standard.

5.10.17 Spectral Interference Check (SIC) Solution - A solution of selected method analytes of higher concentrations which is used to evaluate the procedural routine for correcting known interelement spectral interferences with respect to a defined set of method criteria.

5.10.18 Standard Addition - The addition of a known amount of analyte to the sample in order to determine the relative response of the detector to an analyte within the sample matrix. The relative response is then used to assess either an operative matrix effect or the sample analyte concentration.

5.10.19 Stock Standard Solution - A concentrated solution containing one or more method analytes prepared in the laboratory using assayed reference materials or purchased from a reputable commercial supplier. See Attachment 1 for a list of typical stock solutions used at the laboratory.

5.10.20 Total Metal Analyte - The concentration of analyte determined either by "direct analysis" of an unfiltered acid preserved drinking water sample with turbidity of < 1 NTU, or by analysis of the solution extract of a solid sample or an unfiltered aqueous sample following digestion by refluxing with hot dilute mineral acid(s).

5.10.21 Water Sample - For the purpose of this method, a sample taken from one of the following sources: drinking, surface, ground, storm runoff, industrial or domestic wastewater.

6. Equipment and Supplies

6.1 Inductively coupled plasma emission spectrometer

6.2 Computer-controlled emission spectrometer with background-correction capability. The spectrometer must be capable of meeting and complying with the requirements described and referenced in Section 5.2.

6.3 Radio-frequency generator compliant with FCC regulations.

6.4 Argon gas supply - High purity grade (99.99%). When analyses are conducted frequently, liquid argon is more economical and requires less frequent replacement of tanks than compressed argon in conventional cylinders.

6.5 A variable speed peristaltic pump is required to deliver both standard and sample solutions to the nebulizer.

6.6 Mass flow controllers to regulate the argon flow rates, especially the aerosol transport gas, are highly recommended. Their use will provide more exacting control of reproducible plasma conditions.

7. Reagents and Standards

7.1 Hydrochloric Acid, concentrated

7.1.1 Hydrochloric Acid (1:1): 100 mL of concentrated Hydrochloric Acid added slowly to 100 mL of deionized water.

7.2 Nitric Acid, concentrated

7.2.1 Nitric Acid (1:1): 100 mL of concentrated Nitric Acid added slowly to 100 mL of deionized water.

7.3 Standard Stock Solutions - Stock standards may be purchased or prepared from ultra-high purity (UHP) grade chemicals (99.99 to 99.999% pure). The laboratory purchases stock standard solution from a commercial vendor. For preparation instructions utilizing the UHP grade chemicals, refer to Method 200.7, Revision 4.4 in EPA Document 600/R-94/111. See Attachment 1 for a list of common stock solutions typically used at the laboratory. See Attachment 2 for typical working standard preparation for CCV, ICV, SIC and calibration solutions found in the following sections.

7.4 Continuing Calibration Verification (CCV) Solution - The CCV solution is used to periodically verify instrument performance during analysis. It should be prepared in the same acid mixture as the calibration standards by combining method analytes at appropriate concentrations. The CCV solution should be prepared from the same standard stock solutions used to prepare the calibration standards and stored in an FEP bottle. Agency programs may specify or request that additional instrument performance check solutions be prepared at specified concentrations in order to meet particular program needs.

7.5 Initial Calibration Verification (ICV) - Analysis of an ICV is required for initial and periodic verification of calibration standards or stock standard solutions in order to verify instrument performance. The ICV must be obtained from an outside source different from the standard stock solutions and prepared in the same acid mixture as the calibration standards. The concentration of the analytes in the ICV solution should be >1 mg/L. The ICV solution should be stored in a FEP bottle and analyzed as needed to meet data quality needs.

7.6 Spectral Interference Check (SIC) Solutions - When interelement corrections are applied, SIC solutions are needed containing concentrations of the interfering elements at levels that will provide an adequate test of the correction factors. SIC solutions containing (a) 300 mg/L Fe; (b) 200 mg/L Al; (c) 50 mg/L Ba; (d) 50 mg/L Be; (e) 50 mg/L Cd; (f) 50 mg/L Co; (g) 50 mg/L Cr; (h) 50 mg/L Cu; (i) 50 mg/L Mn; (j) 50 mg/L Mo; (k) 50 mg/L Ni; (l) 50 mg/L Sn; (l) 50 mg/L Ti; (m) 50 mg/L Tl and (n) 50 mg/L V should be prepared in the same acid mixture as the calibration standards and stored in FEP bottles. The concentrations of the elements in the SIC solutions may or may not match what is listed above. However, the concentrations of elements in these solutions must be less than the LDR. These solutions can be used to periodically verify a partial list of the on-line (and possible off-line) interelement spectral correction factors for the recommended wavelengths given in Table 1. Other solutions could achieve the same objective as well.

7.7 Internal Standard Solutions: Internal standards are pure analyte(s) added to a sample, extract, or standard solution in known amount(s) and used to measure the relative responses of other method analytes that are components of the same sample or solution. The internal standard must be an analyte that is not a sample component. Internal standard solution preparation instructions can be found in Attachment 2.

7.8 Mixed Calibration Standard Solutions - For the analysis of total metal digested samples prepare mixed calibration standard solutions (see Attachment 2) by combining appropriate volumes of the stock solutions in 100mL volumetric flasks containing 8mL conc. HNO3 and 4mL conc. HCl and dilute to volume with reagent water. Care should be taken when preparing the mixed standards to ensure that the elements are compatible and stable together. To minimize the opportunity for contamination by the containers, it is recommended to transfer the mixed-standard solutions to acid-cleaned, never-used FEP fluorocarbon (FEP) bottles for storage. Fresh mixed standards should be prepared, as needed, with the realization that concentrations can change on aging. Calibration standards not prepared from primary standards must be initially verified using a certified reference solution.

7.9 Reporting Limit Verification Standard Solutions – For the analysis of metal samples prepare RLVS (see Attachment 2) by combining the appropriate volumes of stock and calibration solutions in 100 mL volumetric flasks containing appropriate concentrations of acid. Care should be taken when preparing the mixed standards to ensure that the elements are compatible and stable together. To minimize the opportunity for contamination by the containers, it is recommended to transfer the mixed-standard solutions to acid-cleaned, never-used FEP fluorocarbon (FEP) bottles for storage. Fresh mixed standards should be prepared, as needed, with the realization that concentrations can change on aging.

8. Sample Collection, Preservation, Shipment and Storage

Parameter	Container	Preservative	Minimum Volume	Maximum Holding Time
Metals (Aqueous)	1L high density polyethylene bottle	HNO_3 pH<2	200 mL	180 days
Metals (Solid)	Solids – 4 oz widemouth glass jar with telfon closure.	NA	100 g	180 days

9. Quality Control

9.1 Sample QC

9.1.1 Procedure Blank: The procedure blank is an aliquot of reagent or a combination of reagents that are prepared in the same manner as the preparation procedures for the field samples. One procedure blank is prepared with each batch of field samples. See the laboratory's SOP *Sample Preparation for Metals Analysis by ICP* (CF-EMA-04) for the preparation procedure for aqueous and solid samples. Analyze the sample as specified in Section 10.3. If the blank contains a result greater than the reporting limit for that metal then the entire preparation batch should be re-prepared and re-analyzed for that metal. Alternatively, the sample results for that metal must be flagged appropriately.

9.1.2 Laboratory Control Sample: The LCS consists of an aliquot of reagent water. The LCS is spiked with the same analytes at the same concentrations as the matrix spike and carried through the same entire preparation scheme as the samples, including sample digestion. See the laboratory's SOP CF-EMA-04 for preparation procedures for solid or liquid samples. See the laboratory's SOP CF-IH-03 for preparation procedures for wipe and cassette samples. When the results of the matrix spike analysis indicates a potential problem due to the sample matrix itself, the LCS results are used to verify that the laboratory can perform the analysis in a clean matrix.

9.1.2.1 Calculate the percent recovery of the spiked analytes. See the laboratory's Quality Assurance Manual (CF-QA-01) for determining the percent recovery. The percent recoveries of the spiked analytes should be within laboratory established control limits. See the laboratory's Quality Assurance Manual for specifics on generating control limits.

9.1.2.2 If the percent recovery of a target analyte falls outside the control limit, the LCS should be re-analyzed once. If the percent recovery of the same target is out of control the sample batch must be re-prepared and re-analyzed. Alternatively all samples in the batch can be appropriately flagged.

9.1.3 Matrix Spike: Sample homogeneity and the chemical nature of the sample matrix can affect analyte recovery and the quality of the data. Taking separate aliquots from the sample for replicate and fortified analyses can in some cases assess the effect. See the laboratory's SOP *Sample Preparation for Metals Analysis by FLAA/ICP* (CF-EMA-04) for the preparation procedure.

9.1.3.1 The analyst will choose a sample from the preparation or run batch that has the lowest numerical sample ID, and has sufficient volume/mass for one initial (unspiked) analysis and a MS/MSD analysis. This sample matrix will be representative of the preparation or run batch.

9.1.3.2 The laboratory must add a known amount of each analyte to a MS sample and a MSD sample. A minimum of 1 MS/MSD pair is prepared per batch. In each case the MS or MSD aliquot must be a duplicate of the aliquot used for sample analysis and for total metal analyte determinations added prior to sample preparation. For water samples, the added analyte concentration must be the same as that used in the LCS (Sect. 9.1.2). For solid samples, however, the concentration added should be expressed as mg/kg. Over time, samples from all routine sample sources should be fortified.

9.1.3.3 Calculate the percent recovery of the spiked analytes. See the laboratory's Quality Assurance Manual for determining the percent recovery. Recovery calculations are not required if the concentration added is less than 30% of the sample background concentration. The percent recoveries of the spiked analytes should be within laboratory established control limits. See the laboratory's Quality Assurance Manual for specifics on generating control limits and SOP CF-QA-04 Quality Control Limits Summary for these limits.

9.1.3.4 Calculate the relative percent difference (RPD) between the MS and the MSD result for each target analyte. See the laboratory's Quality Assurance Manual for instruction on determining the RPD.

9.1.3.5 If the recovery or RPD of any analyte falls outside the acceptance recovery range, the recovery problem encountered with the fortified sample is judged to be matrix related, not system related. The sample results for that analyte should then be flagged appropriately.

9.1.4 Duplicates: To help determine the precision of the instrument, two separate aliquots of the same prepared sample must be analyzed. The relative percent difference (RPD) between the two measurements for each target metal must be ≤20% (or calculated ranges). See the laboratory's QA Manual for a description of the determination of RPD. If the RPD exceeds 20%, the duplicate samples are re-analyzed. If the RPD exceeds 20% a second time, the data for the affected analyte is flagged appropriately. The duplicate is performed on batches of samples that are prepared for dissolved metals or if the aqueous sample turbidity is <1. One duplicate is performed for every ten samples.

9.1.4.1 The analyst will choose a sample from the preparation or run batch that has the lowest numerical sample ID, and has sufficient volume/mass for one initial (unspiked) analysis and a DUP analysis. This sample matrix will be representative of the preparation or run batch.

9.1.5 Analytical Spike: To determine the accuracy of the analyses, at least one sample in each analytical batch is spiked with the target analytes after the preparation procedure. This analysis is referred to as post digestion spike or the analytical spike. The analytical spike is performed only on batches of samples that are prepared for dissolved metals or if the sample turbidity is <1. Calculate the percent recovery for each spiked target analyte (see the laboratory's Quality Assurance Manual for determining the percent recovery). The recovery must be 85-115% for each target analyte or within calculated ranges. If the recovery for any target falls out of the range, the sample should be re-analyzed. If the recovery still fails the acceptance criteria, the sample results should be flagged to indicate matrix interference. One analytical spike is performed for every ten samples.

9.1.6 Internal Standards: The internal standard solution (see Attachment 2 for internal standard solution preparation information) is continuously mixed with all samples analyzed on the instrument. A percent recovery is calculated by the instrument software and is used to determine whether or not there is potential matrix interference

9.1.6.1 Currently there is no method listed acceptance criteria for the percent recovery of the internal standards. The laboratory has developed criteria for the ICP analysts to follow. The internal standard percent recovery is monitored throughout the instrument run. The laboratory monitors the percent recovery of both Yttrium and Gallium, the % RSD for both Yttrium and Gallium and the

difference in the Yttrium and Gallium % RSD. Analyst knowledge and run history is very important in interpreting the Yttrium and Gallium results.

9.1.6.2 The optimal percent recovery for both Yttrium and Gallium should be 90-110%. If it is outside this range it does not mean there is a problem but the analyst should monitor the recovery closely. If the recoveries stay consistent and the other QC indicators are all within protocol it is OK to continue with the run. If the percent recovery for either Yttrium or Gallium drops from one sample to the next by more than 20% there may be an interference or a problem with the sample delivery system. At this point the analyst will investigate. If it is determined to be due to the sample matrix the analyst will rerun the sample with a dilution. If the problem is from the sample delivery system, the problem will be fixed and the affected samples reanalyzed.

9.1.6.3 The % RSD of the internal standards is also a useful tool in determining possible interferences. If the % RSDs are greater than 5% or if the % RSD is significantly different between Yttrium and Gallium there may be possible interferences. Once again the analyst will investigate. Problems with the sample delivery system will be corrected and affected samples re-analyzed. If it is determined to be due to the sample matrix the analyst will rerun the sample with a dilution.

9.1.7 Summary of QC Frequency

	Procedure Blank	Matrix Spike/ Matrix Spike Duplicate	Lab Duplicate	(LCS)[1]	Analytical Spike	Internal Standards
Frequency	5 %/EAB[2]	5%/EAB	As needed	5%/EAB; IH: 20%/EAB	As needed	Each analysis
Acceptance Limits	<RL	Refer to the LIMS	RPD ≤ 20%	Refer to the LIMS	85-115% Recovery	Analyst dependant (not established by this SOP at this time)
Corrective Action	Re-prepare the sample batch or flag	If the LCS is acceptable, flag the data to indicate matrix interference; if LCS not acceptable, re-prepare the sample batch.	Re-analyze the duplicate samples; if the criteria fails a second time, flag the affected analytes appropriately	Re-prepare the sample batch or flag data accordingly	Flag the data to indicate matrix interference	Dilute sample and re-analyze; if still low, flag data for matrix interference.

1. Laboratory Control Sample
2. Each Analytical Batch

9.2 Instrument QC

9.2.1 Initial Calibration Verification (ICV): When beginning the use of this method, on a quarterly basis, after the preparation of stock or calibration standard solutions or as required to meet data quality needs, verify the calibration standards and acceptable instrument performance with the preparation and analyses of an ICV (see Attachment 2 for ICV preparation information). To verify the calibration standards the determined mean concentrations from 3 repetitions of the ICV must be within ±5% (±10% for 6010 B and C) of the stated values. If the calibration standard cannot be verified, performance of the determinative step of the method is unacceptable. The source of the problem must be identified and corrected before either proceeding on with analyses.

9.2.2 Low Level Initial Calibration Verification Standard (LLICV): An LLICV must be analyzed when running samples per SW-846 6010C. For purposes of verifying the initial calibration, only the mid-level ICV needs to be prepared from a source other than the calibration standards. To make the LLICV, prepare a standard at a concentration

expected to be the lower limit of quantitation by diluting the calibration standards. The suggested acceptance criteria for the LLICV is ±30% of its true value. Quantitative sample analyses should not proceed for those analytes that fail the second source standard initial calibration verification. However, analyses may continue for those analytes that fail the criteria with an understanding these results should be qualified and would be considered estimated values. Once the calibration acceptance criteria is met, either the lowest calibration standard or the LLICV concentration can be used to demonstrate the lower limit of quantitation and sample results should not be quantitated below this lowest standard. In some cases depending on the stated project data quality objectives, it may be appropriate to report these results as estimated; however, they should be qualified by noting the results are below the lower limit of quantitation. There, the quantitation limit cannot be reported lower than either the LLICV standard used for the single point calibration option or the low calibration and/or verification standard used during initial multi-point calibration. If the calibration curve cannot be verified within these specified limits for the mid-range ICV and LLICV analyses, the cause needs to be determined and the instrument recalibrated before samples are analyzed. The analysis data for the initial calibration verification analyses should be kept on file with the sample analysis data. The LLICV will also serve as the Reporting Limit Verification Standard (RLVS).

9.2.3 Continuing Calibration Verification (CCV): solution - For all determinations the laboratory must analyze the CCV solution (see Attachment 2 for CCV solution preparation information) immediately following daily calibration, after every tenth sample and at the end of the sample run. Analysis of the CCV solution immediately following calibration must verify that the instrument is within ±5% (±10% for SW-6010 B and C) of calibration with a relative standard deviation <3% from replicate integrations. Subsequent analyses of the CCV solution must be within ±10% of calibration.

9.2.3.1 If the calibration cannot be verified within the specified limits, reanalyze the CCV solution.

9.2.3.2 If the second analysis of the CCV solution confirms calibration to be outside the limits, sample analysis must be discontinued, the cause determined, corrected and/or the instrument recalibrated.

9.2.3.3 All samples following the last acceptable CCV solution must be reanalyzed. The analysis data of the CCV solution must be kept on file with the sample analyses data.

9.2.4 Low Level Continuing Calibration Verification Standard (LLCCV): An LLCCV must be analyzed when running samples per SW-846 6010C. It should be analyzed at the end of each analysis batch. A more frequent LLCCV analysis (i.e., every 10 samples) may be necessary if low-level sample concentrations are anticipated and the system stability at the low end of the calibration is questionable. In addition, the analysis of an LLCCV on a more frequent basis will minimize the number of samples for re-analysis should the LLCCV fail if only run at the end of the analysis batch. The LLCCV standard should be made from the same source as the initial calibration standard at the established lower limit of quantitation as reported by the laboratory. The acceptance criteria for the LLCCV standard should be ±30% of its true value. If the calibration cannot be verified with these specified limits, the analysis of samples containing the affected analytes at similar concentration cannot continue until the cause is determined and the LLCCV standard successfully analyzed. The instrument may need to be recalibrated or the lower limit of quantitation adjusted to a concentration that will ensure a compliant LLCCV analysis. The analysis data for the LLCCV standard should be kept on file with the sample analysis data.

9.2.5 Reagent Blank: A reagent blank is analyzed immediately following daily calibration, after every tenth sample (or more frequently, if required) and at the end of the sample run. Analysis of the reagent blank should always be less than the analyte Reporting Limit (RL).

9.2.5.1 If a target analyte result is greater than the RL, reanalyze the reagent blank.

9.2.5.2 If the same target analyte result is greater than the RL in the second analysis of the reagent blank, sample analysis must be discontinued, the cause determined, corrected and/or the instrument recalibrated. If not related to the instrument the batch must be re-digested. Samples with results less than the RL may be reported without re-digestion.

9.2.5.3 All samples following the last acceptable CCV solution must be reanalyzed. The analysis data of the reagent blanks must be kept on file with the sample analyses data.

9.2.6 Linear Dynamic Range (LDR): The upper limit of the LDR must be established for each wavelength utilized. It must be determined from a linear calibration prepared in the normal manner using the established analytical operating procedure for the instrument. The LDR should be determined by analyzing succeedingly higher standard concentrations of the analyte until the observed analyte concentration is no more than 10% below the stated concentration of the standard or if there is a significant reduction from the typical recovery. Determined LDRs must be documented and kept on file. The LDR which may be used for the analysis of samples should be judged by the analyst from the resulting data. Determined sample analyte concentrations that are greater than 90% of the determined upper LDR limit must be diluted and reanalyzed. The LDRs should be verified annually or whenever, in the judgment of the analyst, a change in analytical performance caused by either a change in instrument hardware or operating conditions would dictate they be re-determined.

9.2.7 Spectral Interference Check (SIC) Solutions - When interelement corrections are applied, SIC solutions are needed containing concentrations of the interfering elements at levels that will provide an adequate test of the correction factors. See Attachment 2 for the SIC solution preparation information.

9.2.7.1 Interelement Correction Verification: On a daily basis, the interelement corrections are verified by analyzing spectral interference check (SIC) solutions. If the apparent concentration of a line being checked is ± ½ the reporting limit, the IEC factor is changed as followed:

$$\frac{\text{Apparent Concentration}*1000}{\text{Interferant Concentration}}$$ (this will put the factor in ppb/ppm of interferant)

9.2.7.2 If the apparent concentration was false positive, the new factor is added to the IEC factor in the computer method. If the apparent concentration was a negative result, the new factor is subtracted from the IEC factor in the computer method. The new correction factors are then entered into and saved with the computer's analytical method. The raw data of the entire analytical sequence following the IEC factor update is processed using the new IEC factors.

9.2.8 Calibration Acceptance Summary

Step	Standards	Type	Control Limit		Frequency
Method # 200.7, 6010 B, C IH Methods: NIOSH 7300 & NIOSH 9102			*EPA 200.7*	*SW 6010 B, C* *IH Methods*	
Initial Cal	Calibration blank and 1 standard	Linear			Daily
ICV	1 Standard	Single Point	95-105%	90-110%	Once, Immediately following curve
LLICV / RLVS	1 Standard at RL	Single Point	70-130%	70-130%	Once, Immediately following curve
CCV	See Attachment 2 for CCV standard conc.'s	Single Point	Initial CCV: 95-105%; Subsequent CCV: 90-110%	90-110%	Immediately following curve and once every 10 samples
LLCCV	Standard at RL	Single Point	70-130%	70-130%	At end of run or more frequently to minimize number of re-analyses
IEC & SIC	See Attachment 2	Single Point	80-120%	80-120%	Twice, Immediately following curve and at end of run

9.3 Method Detection Limit Study (MDLs) See the laboratory's Quality Assurance Manual (CF-QA-01) for MDL determination and calculation.

9.4 Training Requirements: See the laboratory's Quality Assurance Manual for experience requirements for analysts performing this test and for training requirements

10. Procedure

10.1 Sample Preparation – Refer to SOP #'s CF-EMA-04 *Sample Preparation for Metals Analysis by ICP-AES* and CF-IH-03 *Elements by ICP-AES for Industrial Hygiene*.

10.2 Calibration – Profile and calibrate the instrument according to the instrument manufacturer's recommended procedures, using the typical mixed calibration standard solutions described in Attachment 2. Typically, the concentration of the analytes in the standard is 10 ppm. Flush the system with the calibration blank between each standard or as the manufacturer recommends. (Use the average intensity of multiple exposures for both standardization and sample analysis to reduce random error.) The calibration curve must consist of a minimum of a blank and a standard.

10.3 Sample Analysis

10.3.1 Set up the instrument with proper operating parameters established as detailed below. The instrument must be allowed to become thermally stable before beginning (usually requiring at least 30 minutes of operation prior to calibration). See Attachment 4 for a list of typical plasma operating conditions.

10.3.2 Specific wavelengths are listed in Attachment 3. Other wavelengths may be substituted if they can provide the needed sensitivity and are corrected for spectral interference. Because of differences among various makes and models of spectrometers, specific instrument operating conditions cannot be provided. The instrument and operating conditions utilized for determination must be capable of providing data of acceptable quality to the program and data user. The analyst should follow the instructions provided by the instrument manufacturer unless other conditions provide similar or better performance for a task. Operating conditions for aqueous solutions usually vary from 1100 to 1200 watts forward power, 14 to 18 mm viewing height, 15 to 19 liters/mm argon coolant flow, 0.6 to 1.5 L/min argon nebulizer flow, 1 to 1.8 mL/min sample pumping rate with a 1 minute pre-flush time and measurement time near 10 seconds per sample for simultaneous instruments.

10.3.3 The plasma operating conditions need to be optimized prior to use of the instrument. This routine is not required on a daily basis, but only when first setting up a new instrument or following a change in operating conditions. The following procedure is recommended or

follows manufacturer's recommendations. The purpose of plasma optimization is to provide a maximum signal to background ratio for some of the least sensitive elements in the analytical array. The use of a mass flow controller to regulate the nebulizer gas flow or source optimization software greatly facilitates the procedure.

10.3.3.1 Ignite the radial plasma and select an appropriate incident RF power. Allow the instrument to become thermally stable before beginning, about 30 to 60 minutes of operation. While aspirating a 1000 µg/L solution of yttrium, follow the instrument manufacturer's instructions and adjust the aerosol carrier gas flow rate through the nebulizer so a definitive blue emission region of the plasma extends approximately from 5 to 20 mm above the top of the load coil Record the nebulizer gas flow rate or pressure setting for future reference. The yttrium solution can also be used for coarse optical alignment of the torch by observing the overlay of the blue light over the entrance slit to the optical system.

10.3.3.2 After establishing the nebulizer gas flow rate, determine the solution uptake rate of the nebulizer in mL/min by aspirating a known volume of calibration blank for a period of at least three minutes. Divide the volume aspirated by the time in minutes and record the uptake rate; set the peristaltic pump to deliver the rate in a steady even flow.

10.3.3.3 Profile the instrument to align it optically as it will be used during analysis. The following procedure can be used for both horizontal and vertical optimization in the radial mode, but is written for vertical. Aspirate a solution containing 10 µg/L of several selected elements. These elements can be As, Se, TI or Pb as the least sensitive of the elements and most needing to be optimize or others representing analytical judgment (V, Cr, Cu, Li and Mn are also used with success). Collect intensity data at the wavelength peak for each analyte at 1 mm intervals from 14 to 18 mm above the load coil. (This region of the plasma is referred to as the analytical zone.) Repeat the process using the calibration blank. Determine the net signal to blank intensity ratio for each analyte for each viewing height setting. Choose the height for viewing the plasma that provides the best net intensity ratios for the elements analyzed or the highest intensity ratio for the least sensitive element. For optimization in the axial mode, follow the instrument manufacturer's instructions.

10.3.3.4 The instrument operating condition finally selected as being optimum should provide the lowest reliable instrument detection limits and method detection limits.

10.3.3.4.1 If either the instrument operating conditions such as incident power or nebulizer gas flow rate are changed, or a new torch injector tube with a different orifice internal diameter is installed, the plasma and viewing height should be re-optimized.

10.3.3.4.2 After completing the initial optimization of operating conditions, but before analyzing samples, the laboratory must establish and initially verify an interelement spectral interference correction routine to be used during sample analysis. A general description concerning spectral interference and the analytical requirements for background correction in particular is discussed in the section on interferences (Section 5.2). Criteria for determining an interelement spectral interference is an apparent positive or negative concentration for the analyte that falls within $\pm$ one reporting limit from zero. Once established the entire routine must be verified annually. Only a portion of the correction routine must be verified more frequently or on a daily basis. Initial and periodic verification

of the routine should be kept on file. Special cases where continual verification is required are described elsewhere.

10.3.3.4.3 Before daily calibration and after the instrument warm-up period, the nebulizer gas flow rate must be reset to the determined optimized flow. If a mass flow controller is being used, it should be set to the recorded optimized flow rate, In order to maintain valid spectral interelement correction routines the nebulizer gas flow rate should be the same (<2% change) from day to day.

10.3.3.5 Sensitivity, instrumental detection limit, precision, linear dynamic range, and interference effects must be established for each individual analyte line on each particular instrument. All measurements must be within the instrument linear range where the correction equations are valid.

10.3.4 For each sample analyzed, aspirate 3 replicates. The %RSD must be <10% for samples which the analyst has determined that a 'memory effect' is a concern (e.g. an unusually high concentration of a target analyte was detected in the sample).

11. Calculations/Data Reduction and Interpretation

The ICP data systems calculate the final concentration of each sample automatically. If dilutions were performed, the appropriate factors must be applied to sample values. All results should be reported with up to three significant figures.

12. Method Performance

See the laboratory's Quality Assurance Manual for required precision and accuracy data for this method.

12.1 The Quality Assurance Manual (CF-QA-01) describes the process to be used to generate control limits

12.2 The most recent control limits for quality control indications for this method are also posted in LIMS (Element). As the control limits are update, the limits in Element will also be updated.

12.3 The Quality Assurance Manual also includes the control limits for this method. The control limits listed are updated when the Quality Assurance Manual is updated, or if the newly calculated limits are wider than the limits currently posted in the applicable table.

13. References/Cross-References

13.1 Laboratory's QA Manual (CF-QA-01)

13.2 Laboratory's SOP *Sample Preparation for Metals Analysis by ICP-AES* (CF-EMA-04)

13.3 Laboratory's SOP *Elements by ICP-AES for Industrial Hygiene* (CF-IH-03)

13.4 Laboratory's Waste Disposal SOP (CF-WD-01)

14. Contingencies

If for any reason a part of this SOP can not be followed, seek the guidance of the Operations Managers, Laboratory Manager, or Quality Assurance Manager. Document all deviations on a Corrective Action Report and submit it to the QA Manager.

15. Attachments

15.1 Attachment 1: Typical Stock solutions Received from CPI, Spex, and Ultra Scientific

15.2 Attachment 2: Typical Preparations for Working Standard Solutions

15.3 Attachment 3: Wavelengths and Potential Interferants

15.4 Attachment 4: ICP Plasma Operating Parameters

16. Revision History

16.1 Revision 0, dated 01/17/2008

- Initial release

16.2 Revision 1, dated 11/17/2008

- Revised Safety and Waste Management sections

16.3 Revision 1, Modification 1, dated 12/31/2008

- Section 9.1.6 - Added control criteria for internal standards

16.4 Revision 2, dated 11/12/2010

- Added Modification 1.1 to current revision
- Added Updated IV references

16.5 Revision 3, dated 02/08/2012

- Corrected several Update IV requirements for LLICV throughout SOP
- Updated Attachments 1 & 2 to include customs RLVS standards

Attachment 1 – Stock Solutions

Stock target analyte standards are typically received as certified solutions from a manufacturer or supplier. Typical standards received from typical suppliers are:

Stock Solution	Supplier	Name / Concentration	Catalog Number	Preservative
Internal Standard	Ultra	1,000 µg/mL Y	ICP-039	1%HNO_3
		10,000 µg/mL Ga	ICP-131	5% HNO_3
Stock Calibration I	Spex	Custom Multi-element Standard	XNETIA-1	5% HNO_3
	Ultra	1,000 µg/mL In Standard	ICP-049	1% HNO_3
Stock Calibration II	Spex	Quality Control Standard 21	QC-21-500	5% HNO_3
	Ultra	1,000 µg/mL Sn	ICP-050	2% HNO_3
Calibration III		1,000 µg/mL Ag	ICP-047	2% HNO_3
ICV I	CPI	Various	P/NS4400-0005	5% HNO_3
		Various	P/N4400-132515	15% HCl
ICV II		Various	P/N4400-132715	5% HNO_3
ICV III		1,000 µg/mL Ag	P/N4400-1000511	2% HNO_3
IEC A	Spex	Interferants A	INT-A1	5% HNO_3
IEC AB		Interferants A	INT-A1	5% HNO_3
		Analytes B	INT-B1	5% HNO_3
SIC I	Ultra	1,000µg/mL Mo	ICP-042	2%NH_40H
SIC II		1,000 µg/mL Co	ICP-027	2% HNO_3
		1,000 µg/mL Cr	ICP-024	2% HNO_3
		1,000 µg/mL Cu	ICP-029	2% HNO_3
		1,000 µg/mL Mn	ICP-025	2% HNO_3
		1,000 µg/mL V	ICP-023	2% HNO_3
SIC III		1,000 µg/mL Al	ICP-013	2% HNO_3
		1,000 µg/mL Fe	ICP-026	2% HNO_3
		1,000 µg/mL Ni	ICP-028	2% HNO_3
SIC IV		1,000 µg/mL Ba	ICP-056	2% HNO_3
		1,000 µg/mL Be	ICP-004	2% HNO_3
		1,000 µg/mL Cd	ICP-048	2% HNO_3
		1,000 µg/mL Sn	ICP-050	2% HNO_3
		1,000 µg/mL Ti	ICP-022	2% HNO_3
		1,000 µg/mL Tl	ICP-081	2% HNO_3
CCV I	Spex	Custom Multi-element Standard	XNETIA-1	5% HNO_3
	Ultra	1000 µg/mL In	ICP-049	1% HNO_3
CCV II	Spex	Quality Control Standard 21	QC-21-500	5% HNO_3
	Ultra	1,000 µg/mL Sn	ICP-050	2% HNO_3
	Spex	1,000 µg/mL Ag	ICP-047	2% HNO_3
Custom RLVS A	CPI	Various	4400-091216RH02	2% HNO_3
Custom RLVS B	CPI	Various	4400-091216RH02	5% HNO_3 + 0.1% HF

Attachment 2 – Typical Preparations for Working Standard Solutions

Typical preparation, final volume and concentration of solutions for calibration, quality control/assurance made from stock reagents and standards in Attachment 1 are:

Standard	Preparation Procedure	Final Volume	Matrix
Internal standard	20mL Y + 4mL Ga	**200mL**	8% HNO_3 / 4% HCl
Calibration Standard 1	10mL Custom Multi-element + 1mL In	100mL	
Calibration Standard 2	10mL Quality Control Standard 21 + 1mL Sn		
Calibration Standard 3	0.2mL Ag Standard		
ICV 1	5mL stock standard P/NS4400-0005 + 5mL stock standard P/N4400-132515		
ICV II	10mL stock standard P/N4400-132715		
ICV III	0.1mL stock standard P/N4400-1000511		
IEC A	6mL Interferants A		
IEC AB	6mL Interferants A + 1mL Analytes B		
SIC I	5mL Mo		
SIC II	1mL Co + 2mL Cr + 2mL Mn +2mL V + 4mL Cu		
SIC III	2mL Ni + 3mL Al + 15mL Fe		
SIC IV	2mL Ba + 2mL Be + 2mL Cd + 2mL Sn + 5mL Ti + 5mL Tl		
CCV I	5mL Custom Multi-element Standard + 0.5mL In		
CCV II	5mL Quality Control Standard 21 + 0.5mL Sn + 0.1mL Ag Standard		
CCV III	2mL Custom Multi-element STD + 2mL Quality Control STD 21 + 0.25mL In + 0.2mL Sn		
RLVS	0.1mL of Custom RLVS A + 0.1mL Custom RLVS B		

Attachment 3 – Wavelengths and Potential Interferants

Analyte	λ (nm)	Interferant
Ag	328.068	Ce,Ti,Mn
Al	308.213	V,Mo,Ce,Mn
As	193.695	V,Al,Co,Fe,Ni
Ba	233.528	None
Be	234.861	V,Ce
B	249.772	Al, Ca, Co, Fe, Na, V
Ca	315.885	Co,Mo,Ce
Cd	214.438	Ni,Ti,Fe,Ce
Co	228.614	Ti,Ba,Cd,Ni,Cr,Mo,Ce
Cr	267.706	Be,Mo,Ni
Cu	324.753	Mo,Ti
Fe	238.864	None
Ga	294.363	None
In	230.606	Ba,Co,Fe,Mo,Ni,Ti
K	766.491	None
Li	670.790	None
Mg	279.079	Ce
Mn	257.608	Ce
Mo	202.030	Ce
Na	589.602	None
Ni	231.604	Co,Tl
Pb	220.351	Co,Al,Ce,Cu,Ni,Ti,Fe
Sb	206.836	Cr,Mo,Sn,Ti,Ce,Fe
Se	196.025	Fe
Sn	189.927	Mo,Ti,Fe,Mn,Si
Sr	460.734	None
Tl	190.794	Ti,Mo,Co,Ce,Al,V,Mn
Ti	368.519	None
V	292.396	Mo,Ti,Cr,Fe,Ce
Y	371.032	None
Zn	213.856	Ni,Cu,Fe

Attachment 4 – ICP Plasma Operating Parameters

Source Equilibration Delay: 15 seconds
Elements: ALL
Plasma (L/min): 15
Auxilary (L/min): 0.3
Nebulizer (L/min): 0.95
Power (Watts): 1425
View Height (mm): 15
Plasma View: Radial

Title: Elements by ICP/AES for Industrial Hygiene

Computer File Name: G:\QA\QA Official Docs\SOP\IH\CF-IH-03_2.doc

Approvals (Signature/Date):

12/30/13	12-30-13
Lorna Bormann Inorganics Technical Manager — Date	Chad Timmins Environmental Health & Safety Coordinator — Date
12/30/13	12/30/13
Brian Graettinger IH Technical Manager — Date	Michael McGee Laboratory Director Approval — Date
	12-30-13
	Thomas Tjaden Quality Assurance Manager — Date

Facility Distribution No.: CF **Distributed To:**

This method may involve hazardous materials, operations and equipment. This method does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of the method to follow appropriate safety, waste disposal and health practices under the assumption that all samples and reagents are potentially hazardous. Safety glasses, gloves, lab coats and closed toe shoes are a minimum. For specific hazard(s) see reagents, materials and procedure sections of this SOP.

Method Reference: NIOSH Manual of Analytical Methods (NMAM), 4th Edition

METHOD #: 7300, Issue 3 (2003): ELEMENTS by ICP (Nitric/Perchloric Acid Ashing)

Method Reference: NIOSH Manual of Analytical Methods (NMAM), 4th Edition

METHOD #: 9102, Issue 1 (2003): ELEMENTS ON WIPES

Modifications:

Item	Method	Modification
1	NIOSH 7300	The digestion procedure in the method specifies “ashing acid”, a combination of conc. nitric acid and conc. perchloric acid, as the acid for digesting the membrane filters. The laboratory uses only conc. nitric acid to digest the samples.
2	NIOSH 7300	The method specifies ICP calibration using elements in 4% HNO_3, 1% $HClO_4$. The laboratory calibrates using elements in 8% HNO_3, 4%HCl.
3	NIOSH 9102	The digestion procedure in the method specifies “ashing acid”, a combination of conc. nitric acid and conc. perchloric acid, as the acid for digesting the membrane filters. The laboratory uses conc. nitric acid and 30% H_20_2 to digest the samples.
4	NIOSH 9102	The method specifies ICP calibration using elements in 4% HNO_3, 1% $HClO_4$. The laboratory calibrates using elements in 8% HNO_3, 4%HCl.

1. Scope and Application

1.1 Analytes: This method is applicable to the analytes listed in Table 1 in Attachment 1

1.2 Matrices: Filters, 0.8µm, mixed cellulose ester membrane; Ghost Wipes

1.3 Reporting Limits and Units: See Table 1, Section 1.1

1.4 Method Detection Limit: The most recent MDL study is on file in the QA department and tracked in the LIMS. The laboratory’s Detection Limit SOP (CF-QA-05) describes the procedure for determining an MDL.

2. Summary of Method

Cassettes: Metals are collected from air on a membrane filter that is positioned in a cassette filter holder.
Wipes: Metals are collected by wiping an area using a Ghost Wipe.

The membrane filter or wipe is digested with conc. HNO_3, diluted with water and analyzed by ICP. The sample preparation procedures are outlined below. However, refer to analytical SOP CF-EMA-01 for the ICP analytical method. The procedures specified in CF-EMA-01 for method SW-846 6010B are also applicable to the analysis of the digestates.

3. Safety

Employees must abide by the policies and procedures in the Corporate Safety Manual, the facility-specific addendum to the CSM, and this document.

3.1 Specific Safety Concerns or Requirements

All concentrated acids must be used in a fume hood.

Samples that contain high concentrations of carbonates or organic material or samples that are at elevated pH can react violently when acids are added.

3.2 Primary Materials Used

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

Material (1)	Hazards	Exposure Limit (2)	Signs and symptoms of exposure
Hydrochloric Acid	Corrosive Poison	5 ppm-Ceiling	Inhalation of vapors can cause coughing, choking, inflammation of the nose, throat, and upper respiratory tract, and in severe cases, pulmonary edema, circulatory failure, and death. Can cause redness, pain, and severe skin burns. Vapors are irritating and may cause damage to the eyes. Contact may cause severe burns and permanent eye damage.
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. 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." Refer also to the laboratory's Waste Disposal SOP (CF-WD-01).

4.1 Waste Streams Produced by the Method

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

4.1.1 Acidic waste containing nitric acid generated by the extraction. Employees are to transfer waste to SAA bottles in waste room for neutralization of other lab samples. If SAA is full, neutralize and dispose through the sanitary sewer system.

4.1.2 Contaminated disposable materials utilized for the analysis.

- Samples high in mercury are given to the waste coordinator.
- Neutralize and dump aqueous samples.
- Soils and solids are placed in appropriate SAA of waste room.

5. Interferences/Comments/Definitions

Spectral interferences are the primary interferences encountered in ICP-AES analysis. Judicious wavelength selection, interelement correction factors and background correction minimize these. High levels of iron may interfere with some elements. See SOP CF-EMA-01 (Section 13.4) for more information on ICP-AES interferences.

6. Equipment and Supplies

6.1 Blank mixed cellulose ester membrane filter, 0.8-mm pore size, 37-mm diameter; for QC

6.2 Blank Ghost Wipe, Environmental Express Cat# 4210 or Cat# 4250; for QC

6.3 Smear Tabs, low-ash and acid hardened, Grade 50, Whatman Cat# 1450-993 or equivalent

6.4 ICP-AES, equipped as specified by the manufacturer for analysis of elements of interest (see SOP CF-EMA-01)

6.5 Regulator, two-stage, for argon.

6.6 Beakers, 100mL, 150mL with watchglass covers.

6.7 Volumetric flasks, various volumes.

6.8 Assorted volumetric pipettes as needed.

6.9 Hotplate

6.10 Disposable Plastic Funnels

6.11 Glass wool

7. Reagents and Standards

7.1 Conc. nitric acid; acid should be certified to have impurities <MDL for the target analytes

7.2 Hydrogen Peroxide, 30%

7.3 Reagent Water, High Quality (Standard Methods 1080 C-2011, Table 1080:II)

7.4 Argon

7.5 Spiking Solutions (see SOP CF-SS-02, Standards/Reagents Documentation and Tracking, for expiration times for the stock and prepared standards).

7.5.1 LCS1 Stock Solution (All analytes in 5% HNO_3 + tr. HF)

Analytes	Conc. (mg/L)
Al, As, Ca, Cu, Fe, Mg, Mo, Ni, Pb, Sr, Tl	100
Ba, Be, Cd, Co, Cr, Mn, V, Zn	50
Na	150
Se, K	200

7.5.2 LCS 5 Stock Solution (all analytes in 20% HCl)

Analytes	Conc. (mg/L)
Sb, Sn, Li, In, B, Ti	100

7.5.3 LCS3 Stock Solution: Ag (silver) at 50mg/L in 4% HNO_3

8. Sample Collection, Preservation, Shipment and Storage

Media	Container	Preservative	Minimum Quantity	Maximum Holding Time
Cassette	Filter Holder	NONE	1 Filter	NONE
Wipe	Hard Walled Plastic	NONE	1 Wipe	NONE

9. Quality Control

9.1 Sample QC

9.1.1 Method (or Preparation) Blank: The method or preparation blank is a membrane filter and smear tab (cassette analysis) or Ghost Wipe (wipe analysis) digested with the same reagents at the same concentration as a sample. The blank is prepared along with a batch of 20 samples using the procedure specified in Section 10. Each batch of samples has a blank. See SOP CF-EMA-01 for acceptance criteria and corrective action to be taken if the Preparation Blank does not meet the acceptance criteria.

9.1.2 Laboratory Control Sample: A Laboratory Control Standard (LCS) is a new membrane filter and smear tab or a new Ghost Wipe to which a known concentration of analyte is added. The LCS is taken through all of the preparation steps and is analyzed just like a sample. A LCS/LCSD pair must be prepared for every 10 samples. The LCS is prepared by spiking a membrane filter or Ghost Wipe with 1mL each the LCS stock solutions listed in Section 7.5. If silver is not a target analyte then the sample is not spiked with LCS3 (Section 7.5.3). If Antimony, Tin and Titanium are not target analytes then the sample is not spiked with LCS5 (Section 7.5.2). See SOP CF-EMA-01 for acceptance criteria and corrective action to be taken if the LCS does not meet the acceptance criteria.

9.2 Instrument QC: See SOP CF-EMA-01 for required instrument QC, the acceptance criteria, and the corrective action to be taken if a QC parameter fails the acceptance criteria. The criteria specified for SW-846 Method 6010B is applicable to this method.

9.3 Method Detection Limit (MDL) Study (MDL) See the laboratory's Quality Manual for MDL determination and calculation.

9.4 Training Requirements: See the laboratory's Quality Assurance Manual for experience requirements for analysts performing this test and for training requirements.

10. Procedure

10.1 Sample Preparation for Cassette Samples

10.1.1 Cut the band surrounding the cassette with a razor, gently pry the halves of the cassette apart with an opening tool, and transfer the filter to a clean, acid washed 100mL glass beaker using a non-metal tweezers. Cassette filters are NEVER to be touched with fingers.

10.1.1.1 Some cassettes are labeled MW for matched weight. These will contain 2 filters. Remove the top filter, transfer it to the beaker and check the bottom filter. If the top filter has split or "broken through", transfer the bottom filter to the beaker also.

10.1.1.2 If both filters are used for digestion of a field sample, then both filters from the client-supplied field blank must be used also.

10.1.2 Rinse the inside of the top half of the cassette body with a small stream of reagent water from a narrow-tip bottle. Dump the rinse water into the appropriate beaker. Making sure to wear clean nitrile gloves, use a smear tab (Section 6.3) and wipe the inside of the top half of the cassette body. Place the smear tab in the same beaker as the filter and rinse water.

10.1.3 To the beaker containing the sample filters, add 3mL of reagent water and 2mL of concentrated nitric acid (Section 7.1). Cover the beaker with a watchglass. Heat on the hotplate until the sample filter dissolves. Some orange colored fuming may occur. The digestion process should take approximately 1 hour. Check the sample digestion process approximately every 10 minutes to ensure the watchglass has not become displaced. If the watchglass has become displaced, add another aliquot of reagent water, re-cover the beaker with the watch glass and continue the digestion. **If the contents of a sample beaker evaporate to dryness, the sample digestion is invalid.**

10.1.4 When the digestion is completed, remove the beaker from the hotplate and allow cooling. Remove the watchglass and use reagent water to rinse the watchglass into the digestate. Dilute the digestate to 25mL with reagent water. If the sample digestate is cloudy or contains particulate matter, filter the digestate using a disposable funnel (Section 6.10) and glass wool (Section 6.11).

10.2 Sample Preparation for Wipe Samples

10.2.1 Transfer the wipe into a 150mL glass beaker. Wipes are NEVER to be touched with fingers – wear a glove if handling is required. Rinse the sample container with 25mL Millipore water and transfer to the beaker. Add 2.5mL of concentrated HNO_3 and cover with a watch glass.

10.2.2 Put the beakers on the hotplate and stir samples occasionally. After 30 minutes remove samples from hotplate and allow cooling.

10.2.3 After the samples have cooled, add 2.5mL of conc. HNO_3 (Section 7.1) and put back on hotplate. Care must be taken to avoid dryness. Continue heating until the wipe is fully dissolved. This may take up to an hour.

10.2.4 Remove samples from hotplate and cool. Rinse the watch glass into the beaker and place back on hotplate.

10.2.5 Add 5mL of H_20_2 (Section 7.2) and allow samples to continue heating on hotplate for an additional 15 minutes. If a sample has a large amount of particulate matter, add the H_20_2 slowly.

10.2.6 Dilute samples to 50mL and filter (Section 6.1) through glass wool (Section 6.11) as necessary.

10.3 Sample Analysis: Refer to SOP CF-EMA-01 for calibration and analytical procedures for this method.

11. Calculations/Data Reduction and Interpretation

11.1 Obtain the solution concentrations for the sample(s) and the preparation blank from the instrument.

11.2 Cassettes: Calculate the amount of metal per filter and/or per volume of air using the following equations:

$$[C]\ (mg/filter) = \frac{[(C_sV_S)\times DF] - C_BV_B)}{1{,}000}$$

$$[C]\ (mg/m^3) = \frac{[(C_sV_S)\times DF] - C_BV_B)}{V_A}$$

where:

C_s = Concentration of the target analyte in the sample digestate solution (taken from the instrument, in µg/mL);

V_S = Solution final volume of the sample (in mL);

C_B = Concentration of the target analyte found in the method blank, if applicable (taken from the instrument, in µg/mL);

V_B = Solution final volume of the method blank (in mL);

V_A = Volume of air sampled (in L); and

DF= Dilution factor of sample, if necessary.

11.3 Wipes: Calculate the amount of metal per wipe using the following equation:

$$[C]\ (\mu g/wipe) = [(C_sV_S)\times DF] - C_BV_B$$

where all symbols are defined above in Section 11.2.

12. Method Performance

See the laboratory's Industrial Hygiene Quality Assurance Manual (CF-QA-02) for required precision and accuracy data for this method.

12.1 The laboratory's Control Limit SOP (CF-QA-04) describes the process to be used to generate quality control limits.

12.2 The laboratory's Measurement Uncertainty SOP (CF-IH-13) describes the process to be used to estimate method uncertainty.

12.3 The most recent control limits for quality control indications for this method are posted in LIMS (TALS). As the control limits are updated, the limits on LIMS will also be updated.

13. References/Cross-References

13.1 Laboratory's Detection Limit SOP (CF-QA-05)

13.2 Laboratory's IH QA Manual (CF-QA-02)

13.3 Laboratory's Waste Disposal SOP (CF-WD-01)

13.4 Laboratory's Determination of Metals and Trace Elements by ICP-AES SOP (CF-EMA-01)

13.5 Laboratory's Standard Tracking SOP (CF-SS-02)

14. Contingencies

If for any reason a part of this SOP can not be followed, seek the guidance of the Technical Managers, Laboratory Director, or Quality Assurance Manager. Document all deviations in a Non-Conformance Memo (NCM) in TALS and submit for review.

15. Attachments

15.1 Attachment 1: Table of Target Analytes

15.2 Attachment 2: Data Review Sheet for ICP Metals for Industrial Hygiene Analyses

16. Revision History

16.1 Revision 0, dated 6/26/2009

16.1.1 Initial release

16.2 Revision 1, dated 9/9/2011

16.2.1 Section 11 – updated equations used to calculate final concentrations of metals in cassette and wipe samples.

16.2.2 Attachment 2 – Updated Secondary Data Review Checklist

16.3 Revision 2, dated 12/31/2013

16.3.1 Section 7.3 – updated specification of reagent water from Standard Methods 18th Edition to Standard Methods Online.

16.3.2 Section 10.1 – added instructions for wiping inside of cassette body

16.3.3 Section 11 – modified equations to account for detections in method blank

Attachment 1: Table of Target Analytes

Element	CAS #	Molecular Weight	Cassette Reporting Limit (µg/sample)	Wipe Reporting Limit (µg/sample)
Aluminum (Al)	7429-90-5	26.98	5.0	5.0
Antimony (Sb)	7440-36-0	121.75	2.5	5.0
Arsenic (As)	7440-38-2	74.92	5.0	20.0
Barium(Ba)	7440-39-3	137.34	0.25	0.50
Beryllium (Be)	7440-41-7	9.01	0.25	0.50
Boron (B)	7440-42-8	10.81	10.0	20.0
Cadmium (Cd)	7440-43-9	112.41	0.50	1.0
Calcium (Ca)	7440-70-2	40.08	25.0	100
Chromium (Cr)	7440-47-3	52.00	2.5	1.0
Cobalt (Co)	7440-48-4	58.93	0.50	1.0
Copper (Cu)	7440-50-8	63.55	0.50	1.0
Indium (In)	7440-74-6	114.82	12.0	25.0
Iron (Fe)	7439-89-6	55.85	5.0	20.0
Lead (Pb)	7439-92-1	207.19	2.5	5.0
Lithium (Li)	7439-93-2	6.94	2.5	5.0
Magnesium (Mg)	7439-95-4	24.31	25.0	200
Manganese (Mn)	7439-96-5	54.94	0.25	0.50
Molybdenum (Mo)	7439-98-7	95.94	2.5	2.5
Nickel (Ni)	7440-02-0	58.69	1.2	5.0
Potassium (K)	7440-09-7	39.10	25.0	125
Selenium (Se)	7782-49-2	78.96	5.0	7.5
Silver (Ag)	7440-22-4	107.87	0.50	1.0
Sodium (Na)	7440-23-5	22.99	25.0	300
Strontium (Sr)	7440-24-6	87.62	2.5	5.0
Thallium (Tl)	7440-28-0	204.37	25.0	50.0
Tin (Sn)	7440-31-5	118.69	2.5	10.0
Titanium (Ti)	7440-32-6	47.90	1.2	2.5
Vanadium (V)	7440-62-2	50.94	1.2	2.5
Zinc (Zn)	7440-66-6	65.38	2.5	100

Attachment 2 – Data Review Sheet for ICP metals for Industrial Hygiene Analyses (Front)

Document: CF-IH-RF-003
Revision: 2
Revision Date: 12/27/2013

NIOSH 7300
NIOSH 9102

TestAmerica Secondary Data Review Checklist

ICP Metals for IH Data Review

Date Analyzed:	**REF: CF-IH-03 and CF-EMA-01**
Batch Number(s) Cassettes: **Wipes:**	**Instrument:** ☐ **4300 DV** ☐ **7300 DV**

Review Item	Yes	No	N/A
A. Prep Log			
1. Is the prep batch filled out correctly and completely?			
2. All standard identification numbers and reagent numbers identified?			
3. Are the MB and LCS/LCSD performed at the proper frequency?			
4. Are the analyst(s) performing the preparation and analysis clearly identified?			
B. Initial Calibration			
1. Does the curve consist of a calibration blank and one calibration standard?			
2. Is the ICV within control limits? (90-110%)			
3. Is the ICV prepared from a second source?			
4. Is the RLVS/CRI within control limits? (60-140%)			
5. ICSA/ICSAB control			
6. ICSA/ICSB frequency			
C. Continuing Calibration			
1. Are the CCVs ran at the correct frequency? (every 10 samples and at end of analytical batch)			
2. Are the CCVs within control limits? (90-110%)			
3. Is the CCB concentration less than the RL?			
4. ICSA/ICSAB control			
5. ICSA/ICSB frequency			
D. Sample Analysis			
1. Are the sample IDs clearly identified?			
2. Is the sample duplicate run at the desired frequency and is the RPD within QC limits?			
E. QC Samples			
1. Is the Method Blank run at the desired frequency and is its concentration for target analytes less than the RL?			
2. Is the Laboratory Control Sample within percent recovery QC limits?			

Attachment 2 – Data Review Sheet for ICP metals for Industrial Hygiene Analyses (Back)

Document: CF-IH-RF-003
Revision: 2
Revision Date: 12/27/2013

Review Item	Yes	No	N/A
F. Others			
1. Are all nonconformances included and noted?			
2. Did analyst sign/date the appropriate printouts and report sheets?			
3. Were 10% of samples and all QC checked for calculation and data entry into TALS?			
4. If a result was calculated or entered incorrectly the entire batch must be checked. If applicable was that completed?			

Comments on any "No" response:

Second-Level Reviewer:_____ **Date:**_____

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

Approvals (Signature/Date):

Debbie Johnson 11/3/14
Debbie Johnson Date
Supervisor, Metals Supervisor

Terese A. Preston 10/30/14
Terese A. Preston Date
Quality Assurance Manager

Michael J. Healy 10-30-14
Michael J. Healy Date
Laboratory Director

Chris Hoham 10/30/14
Chris Hoham Date
Env. Health & Safety Coor.

Facility Distribution No: _____ **Distributed To:** Uncontrolled

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", Update IV, Method 6010C as a reference. The hardness calculation found in section 9.4 is from Standard Methods 2340 B-97.

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), referred to as the detection limit (DL) in NELAC, 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" with additional details are provided in the TestAmerica Corporate SOP, *CA-QS-006, Detection Limits* and the TestAmerica Chicago SOP, *UP-QA-017, Method Detection Limit Studies.* 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; the MDL will be verified on an annual basis.

1.1.2 Demonstration of Capability

Before the laboratory may institute a new method and begin reporting results, the laboratory shall confirm that it can properly operate the method. A demonstration of capability is performed whenever there is a change in instrument type, method or personnel. An Initial Demonstration of Capability (IDOC) must be thoroughly documented and approved by the Department Manager/Supervisor and QA Manager prior to independently analyzing client samples. All associated documentation must be retained in the QA Department and in the Analyst Training files. For additional details on the demonstration of capability procedures followed, refer to the laboratory SOP, *UP-QA-QAM, Quality Assurance Manual, Sections 20.4.2 and 20.4.3.*

1.1.3 Instrument Detection Limits

Instrument Detection Limits (IDLs) are performed on a quarterly basis for each element and for each instrument. These limits are used to gauge instrument sensitivity and when routinely evaluated, instrument performance without the introduction of method variance can be determined. Refer to the IDL SOP, *UP-QA-010* for additional details.

1.1.4 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.

1.1.5 Definitions

Refer to Section 3.0 of the Laboratory's Quality Assurance Manual (UP-QA-QAM).

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 iCAP 6500 is capable of analyzing simultaneously 29 different elements on a sample, and the Trace 61E analyzes 28 elements simultaneously.

2.0 INTERFERENCES

Spectral, Physical and Chemical Interferences are the three main interferences that are commonly present on the ICAP.

2.1 Spectral Interferences

Spectral interferences are 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 IECs. See Section 6.2. Interferent Check Standard A (ICSA) is monitored to detect instrument drift requiring the IEC equations to be corrected.

2.2 Physical Interferences

Physical interferences are 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. A high solids nebulizer, a mass flow controller and argon humidifier is used prior to nebulization of the sample to minimize physical interferences. If internal standard counts indicate matrix interference a dilution on the sample will be performed.

2.3 Chemical Interferences

Normally there are not significant chemical interferences 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.

The ICP can have some ionization effects caused by torch positioning. To eliminate these effects, Cesium may be added to the internal standard solution (100 mLs / 1-Liter).

Most interference 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 ICP/MS 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, Lab Specific Addendum to the CSM, and this document. This procedure may involve hazardous material, operations and equipment. This SOP does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of the method to follow appropriate safety, waste disposal and health practices under the assumption that all samples and reagents are potentially hazardous. Safety glasses, gloves, lab coat and closed-toe, nonabsorbent shoes are a minimum.

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.
- Individuals with pacemakers must be aware that the instrument radio frequency generator may interfere with pacemaker operation.

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

1) Thermo Fisher ICAP 61E Trace Analyzer. This simultaneous ICAP currently has 31 analytical wavelengths, with additional wavelengths available. It is equipped with a peristaltic pump for sample uptake and an autosampler, and uses TJA software version 6.2. TestAmerica Chicago Lab ID: ICP5

2) Thermo Fisher iCAP 6500. Duo, with Cetac ASX-520 autosampler, and Neslab Thermo Flex 900 water chiller. Currently has 32 analytical wavelengths with additional wavelengths available. The instrument employs iTEVA Analyst – TA 2009B (Revision 47) software. TestAmerica Chicago Lab ID: ICP6

All ICP instruments use the following high solids nebulizer: Thermo Scientific C2 Aerosalt 35 PSI Concentric Nebulizer Argon and Sample Disonnect.

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
- Internal Standards: Ytrium (Y); Indium (In); Profile Standard Arsenic(As)– Inorganic Ventures

*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. The suppliers that are currently used are Inorganic Ventures, Hi Purity, CPI and Ultra. 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 TALS.

5.2.1 Calibration Standards

Prepared with Milli-Q water that has been acidified with 1% HNO_3 and 5% HCl. Internal standard is automatically mixed into all standards, QC solutions and samples at an approximate concentration of 1 ppm. 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)

Note: A blank and a 3-standard curve is done for each element on the Trace 61E, but a blank and single standard are used to calibrate the iCAP 6500.

The trace 61E calibration curve must have a correlation coefficient of ≥ 0.998.

Trace 61E Calibration

Standard	Preparation
S1	• Add ~50 mLs of Milli-Q water to a 200 mL Class A volumetric flask. • Re-pipette 2 mLs of concentrated HNO_3 into the flask. • Re-pipette 10 mLs of concentrated 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 of concentrated HNO_3 into the flask. • Re-pipette 10 mLs of concentrated 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 of concentrated HNO_3 into the flask. • Re-pipette 10 mLs of concentrated HCl into the flask. • Using Eppendorf pipettes, add 1.0 mL 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 of concentrated HNO_3 into the flask. • Re-pipette 10 mLs of concentrated 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 of concentrated HNO_3 into the flask. • Re-pipette 10 mLs of concentrated 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 of concentrated HNO_3 into the flask. • Re-pipette 10 mLs of concentrated 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.

ICAP 6500 Calibration

Standard	Preparation
S1	• Add ~50 mLs of Milli-Q water to a 100ml Class A volumetric flask • Re-pipette 1 mL of conc. HNO_3 into the flask. • Re-pipette 5 mLs of conc. HCl into the flask. • Using Eppendorf pipettes, add 1.0 mL each of: STDIL-STD-1 STDIL-STD-2 • Using an Eppendorf pipette, add 0.50 mL of RFW-ICPT-STD-2B • Using an Eppendorf pipette, add 0.250 mL of RFW-ICPT-STD-2A • Using Eppendorf pipettes, add 0.10 mL each of: Bi, Si and Ag from a 1000 ug/mL stock solution • Using Eppendorf pipette, add 1.0 mL from Li 100 ug/mL stock solution • Dilute to volume with Milli-Q water and mix thoroughly.
S2	• Add ~50 mLs of Milli-Q water to a 100ml Class A volumetric flask • Re-pipette 1 mL of conc. HNO_3 into the flask. • Re-pipette 5 mLs of conc. HCl into the flask. • Using Eppendorf pipettes, add 2.00 mL of RFW-ICPT-STD-2B • Using Eppendorf pipettes, add 1.00 mL of RFW-ICPT-STD-2A • Dilute to volume with Milli-Q water and mix thoroughly.

5.2.2 Instrument QC Solutions (Refer to Attachment 2 for element concentrations.)

All QC solutions are recorded in the TALS reagent module and are prepared as follows:

QC Solution	Preparation To a 1-L Class A volumetric flask filled w/ ~500 mLs of Milli-Q water, add the following for each QC Solution:
Reporting Limit Check Std. (ICVL/CCVL)	• 10 mLs of concentrated HNO_3. • 50 mLs of concentrated HCl. • 1.0 mL of TACHI-1 • 1.0 mL of TACHI-2 • 50 uLs Bi from a 1000 µg/mL stock • 100 uLs Li from 100 ug/mL stock solution • Dilute to volume with Milli-Q water and mix thoroughly.
Initial Calibration Verification (ICV)	• 10 mLs of concentrated HNO_3. • 50 mLs of concentrated HCl. • 8 mLs each of: CCV Solution A CCV Solution A1 CCV Solution B • 1.6 mLs of 10,000 µg/mL Fe • 1.6 mLs of 10,000 µg/mL Na • 1.68 mLs of 10,000 µg/mL Mg • 3.6 mLs of 10,000 µg/mL Al • 1.84 mLs of 10,000 µg/mL Ca • 3.6 mLs of 10,000 ug/mL K • Dilute to volume with Milli-Q water and mix thoroughly.
Continuing Calibration Verification (CCV)	• 10 mLs of concentrated HNO_3. • 50 mLs of concentrated HCl. • 10 mLs each of: CCV Solution A CCV Solution A1 CCV Solution B • 4.5 mLs of 10,000 µg/mL Al • 2.3 mLs of 10,000 µg/mL Ca • 2.0 mLs of 10,000 µg/mL Fe • 2.0 mLs of 10,000 µg/mL Na • 4.5 mLs of 10,000 µg/mL K • 2.1 mLs of 10,000 µg/mL Mg • Dilute to volume with Milli-Q water and mix thoroughly.
CRI [Contract Required Detection Limit (CRDL) Standard for ICAP]	• 10 mLs of concentrated HNO_3 . • 50 mLs of concentrated HCl. • 2.0 mL of TACHI-1 • 2.0 mL of TACHI-2 • 100 uLs of 1,000 µg/mL Bi • 200 uLs of 100 ug/mL Li • Dilute to volume with Mill-Q water and mix thoroughly.
ICSA (Interferent Check Standard)	• 10 mLs of concentrated HNO_3. • 50 mLs of concentrated HCl. • 100 mLs of CLPP-ICS-A. • Dilute to volume with Milli-Q water and mix thoroughly.

QC Solution	Preparation To a 1-L Class A volumetric flask filled w/ ~500 mLs of Milli-Q water, add the following for each QC Solution:
ICSAB (Interferent Check Standard)	• 10 mLs of concentrated HNO_3. • 50 mLs of concentrated HCl. • 100 mLs of CLPP-ICS-A • 10 mLs of CLPP-ICS-B4 • Dilute to volume with Milli-Q water and mix thoroughly.

Note: Some of the volume amounts of the single-element spikes in the above table appear to be reduced from the obvious volume necessary to achieve the final desired concentration. This is to compensate for the presence of those metals in the mixed standards also added to the working standard.

6.0 CALIBRATION (NON-DAILY)

6.1 Linear Range Standard (LRS)

ICAP analysis involves a Linear Dynamic Range (LDR) and a Linear Calibration Range (LCR). At TestAmerica Chicago, the high standard of the LCR is set below the established upper limit of the LDR for all elements. In order to establish, verify, and document linearity at or near that upper limit, a Linear Range Study (LRS) is done every six months on each instrument. This is done by running a single standard at the upper limit of the anticipated linear range of measurement after the instrument has been calibrated in the usual daily manner. The acceptance limits for these single-element verification standards is 95-105%. During routine analysis, all results, target or non-target, that are above the LDR are flagged by the instrument software, aiding the analyst in making appropriate dilution decisions. All samples for which a target analyte or an interfering element result is found to be above the LDR are diluted and re-analyzed until the concentration falls within the instrument's linear range.

6.2 Inter-Element Correction (IEC)

Correction factors for spectral interference will be determined at least annually for all wavelengths used for each analyte reported, any time the ICAP is adjusted in any way that may affect the IECs, or as needed based on continuing observation of Interferent Check Standard A (ICSA) and Interferent Check Standard B (ICSAB). The correction factor is manually calculated from the result ratios between the affected analyte and a known interferent analyzed simultaneously at an appropriately high concentration. The factor, documented with the instrument records, is entered into the method at the instrument PC and becomes part of the software algorithm. See Section 9.5 for calculation details.

Correction factors for spectral interferences other than Al, Ca, Fe, and Mg are recommended and are performed as needed.

7.0 PROCEDURE

7.1 Batch Quality Control Checks

The following section summarizes the quality control (QC) samples associated with routine ICAP analysis.

QC Sample	Frequency[6]	Control Limit [1]
Method Blank (MB)	1 per 20 samples	≤ Reporting Limit (<1/2 RL per QAPP)
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) [3,5]	1 per 20 samples	75 – 125%; 20 RPD
Duplicates (DU) [4,6]	1 per 20 samples	20 RPD
Serial Dilution (SD) (5x dilution)	1 per 20 samples – required if MS or MSD fails	± 10% of the original result if analyte conc. >10X MDL
Post Digestion Spike (PDS)	1 per 20 samples - recommended if SD or MS or MSD fails	80-120%

[1] Refer to Section 8 for additional details.
[2] LCS Duplicate (LCSD) is run 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] 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.
[6] Some programs, including IDEM, require 1/10 frequency.

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 UP-SP-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

Trace 61E (ICP5):

Set up the instrument with the proper operating conditions as defined in the 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 by aspiration and selecting the automatic profile feature from the Thermo Fisher software. The peak position reading should be within +/- 0.1. If the reading is acceptable, record the peak area in the maintenance 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 maintenance logbook and rinse. The instrument is now ready to calibrate.

ICAP 6500 (ICP6):

Set up the instrument with the proper operating conditions as defined in the instrument manual. The instrument must be allowed to become thermally stable (~1/2-hour) prior to calibration. Alignment of the torch or sensor chip is done as needed. A 2ppm Zinc solution is aspirated to align the torch as needed. An autopeak adjust is run only if some part of the induction system is changed such as pump speed, nebulizer pressure, etc.

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 Trace 61E instrument is standardized using a calibration blank and 3 calibration standards, the 6500 uses a calibration blank and one standard. The results are given in intensities.

Standard	Frequency	Control Limit
Calibration Curve	Initially	Corr. Coeff. ≥ 0.998 (Trace only)
High Standards (S1, S2)	After the Calibration Curve	± 5% of the Known Conc.
Initial Cal. Verif. (ICV)	After the Calibration Curve – mid-range	± 10% of the Known Conc. <3% RSD
Low-level ICV (ICVL)	After mid-range ICV - RL level	± 30% of Known Conc.
Initial. Cal. Blank (ICB)	After the ICV	≤ Reporting Limit (≤ ½ RL per QAPP)
ICSA / ICSAB	Daily	*± 20% of the Known Conc. or absolute value <RL for all non-spiked elements. (< ½ RL per QAPP)
Cont. Cal. Verif. (CCV)	Every 10 reading; End of each run – mid-range	± 10% of the Known Conc.
Low-level CCV (CCVL)	After mid-range CCV – at RL	± 30% of Known Conc.
Cont. Cal. Blank (CCB)	Every 10 readings; End of each run	≤ Reporting Limit (≤1/2 RL per QAPP)

* Note: Samples with extremely high "A" concentrations and low "B" concentrations may warrant further evaluation.
The Low-level ICV and Low-level CCV are only required at the beginning and the end of a run, but may be run with every CCV since reported data must be successfully bracketed. Any exceedance of the ICVL / CCVL method limits of 70-130% recovery requires and NCM and approval of the project manager prior to acceptance and the reporting of the data to the client. The 60-140% alternate limit will be used only if approval from the project manger is obtained otherwise re-analysis of the samples associated with a failed ICVL/CCVL is mandatory.

7.5 Preventive Maintenance

The required preventive maintenance is listed in the preventive maintenance logbooks which are kept at the instrument. All maintenance is recorded along with the date and the signature of the analyst performing the maintenance. Documentation of "Return to Control" must be entered into the maintenance log book. The instrument is under a full service contract with the manufacturer for all major repairs and an annual PM services. A copy of the service maintenance record must be attached to the maintenance logbook.

7.5.1 Daily Maintenance

Minimally 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

Minimally 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. Check the air filters to be cleaned or replaced.

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
- ICVL Low-level ICV
- ICB Initial Calibration Blank
- ICSA Interferent Check Standard A
- ICSAB 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 (SD)
- Sample (5) Matrix Duplicate (DU)
- Sample (6) Matrix Spike (MS)
- Sample (7) Matrix Spike Duplicate (MSD)
- Sample (8)
- Sample (9)
- Sample (10)
- CCV Continuing Calibration Verification
- CCVL Low-level CCV
- CCB Continuing Calibration Blank

If the CCV, CCVL 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. Any exceedance of the ICVL / CCVL method limits of 70-130% recovery requires and NCM and approval of the project manager prior to acceptance and the reporting of the data to the client. The 60-140% alternate limit will be used only if approval from the project manger is obtained otherwise re-analysis of the samples associated with a failed ICVL/CCVL is mandatory.

7.7 Documentation

7.7.1 Instrument Sequence-Log

The analysis of each day's samples and standards is documented within the instrument sequence log (Attachment 3) in the TALs LIMs system and is supported by the instrument print-out.

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 LIMS and is issued a unique source ID#. The manufacturer, lot #, date received, expiration date, date of verification and the initials of the recording analyst are also entered. The preparation data for all intermediate standards prepared in house are also entered into TALS. Unique bar-coded labels are generated that are affixed to the stock or standard containers. The unique standard IDs are scanned or manually entered into the TALS preparation and analytical batches along with the volumes used for spiking the various QC standards. The standard IDs appear in the TALS-generated raw data.

7.7.3 Data Review

Analytical data goes through a 200% review cycle. As results are generated, analysts review their work to ensure that the results generated meet QC requirements and relevant EPA methodologies. The analyst transfers the data into TALs in the Analyst Desktop module. Where non-compliance is observed, the analyst creates Non-Conformance Memos (NCMs) in TALs. Flags and data qualifiers can be method, project, program or QAPP specific. The analyst documents the initial review on a data review checklist (Attachment 4) and sets the batch status in LIMs to 1st level review. The second level or peer review of the data is conducted by another individual who has been trained on the review process. This secondary review is documented on the same checklist, making any necessary corrections to the data or additions to the NCMs as necessary. The batch is then set to 2nd level review. The raw data, including the checklist, instrument print-outs, and manual entries, and electronic files are retained for easy retrieval in accordance with the laboratory's record and retention policy outlined in the SOP, *UP-QA-QAM, Section 15.*

Examples of items included in the above reviews are as follows:

- QC data are outside the specified control limits for accuracy and precision
- Unusual detection limit changes are observed
- Samples having unusually high results
- Samples exceeding a known regulatory limit
- Raw data indicating some type of contamination or poor technique
- Transcription errors
- Results outside of calibration range

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. 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 ±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 (DU)

A duplicate sample will be prepared at a frequency of 5% (1 in 20 samples). A 20 RPD is set as the acceptance limits.

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

The MS / MSD will be prepared at a frequency of 5% (1 in 20 samples). The recovery must be within 75–125%. (Exception allowed if the sample concentration exceeds 4 times the spike added concentration.)

TCLP - If the MS recovery is <50% and the concentration does not exceed the regulatory limit or the sample concentration is within 20% of the regulation level, the Method of Standard Addition (MSA) is required. Three aliquots of the sample are spiked at 50%, 100% and 150% of the sample concentration or, if the sample concentration is < RL, the MSA is at 50%, 100% and 150% of the MS level. The data is subjected to linear regression whereas the concentration of the unknown is the x-intercept and the correlation coefficient value must be ≥ 0.995.

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 >10 times the MDL, results should agree within +/- 10% of the original results.

8.1.6 Post Digestion Spikes

If the MS/MSD is not within limits, a Post Digestion Spike must be analyzed to confirm the matrix effect. Recoveries must fall between 80-120%.

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 is 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 NCM;
- document on the NCM 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/ICVL	• 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. • any exceedance of the ICVL method limits of 70-130% recovery requires and NCM and approval of the project manager prior to acceptance and the reporting of the data to the client. The 60-140% alternate limit will be used only if approval from the project manger is obtained otherwise re-analysis of the samples associated with a failed ICVL is mandatory.
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.
LCS	If the LCS is low: • 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 an NCM. If the LCS is high: • 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 an NCM.

QC Indicator	Suggested Corrective Actions
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; • report results at least 10x > the MB or < RL where applicable; • in the extreme case where all samples in the set are at least 10x > the MB or < RL for all metals, reanalysis will not be required; however, an NCM will be written and approved by the supervisor or section manager.
DU	• an NCM will be written and approved by the supervisor or section manager.
MS / MSD	• an NCM will be written and approved by the supervisor or section manager.
Serial Dilution (SD)	• prepare a new serial dilution to verify proper preparation; • an NCM will be written and approved by the supervisor or section manager.
CCV/CCVL	• 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. • any exceedance of the CCVL method limits of 70-130% recovery requires and NCM and approval of the project manager prior to acceptance and the reporting of the data to the client. The 60-140% alternate limit will be used only if approval from the project manger is obtained otherwise re-analysis of the samples associated with a failed CCVL is mandatory.
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.
Additional CAs	• If any of the ICV, ICB, ICSA, ICSAB, 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 is 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 DU 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. • NCMs can be created at any time in LIMS, most frequently as the data are initially reviewed in AD. They are automatically forwarded to the appropriate managers as emails and the text appears in the final report to the client.

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 the TALs LIMs analytical batch at the time the data is processed.

9.4 Calculations for SiO2 and Hardness

Calculation of SiO2

(Si) Silicon Raw (mg/L) * 2.1392 * PF

PF=Prep Factor

Calculation of Magnesium Hardness

(Mg) Magnesium Raw (mg/L) * 4.118 * PF

Calculation of Calcium Hardness

(Ca) Calcium Raw (mg/L) * 2.497 * PF

Calculation of Total Hardness

(((Ca) Calcium Raw (mg/L) * 2.497) + ((Mg) Magnesium Raw (mg/L) * 4.118)) * PF

9.5 Calculation of Inter-element Correction Factors

Spike a blank with the known interfering element at an appropriate concentration, typically at the upper level of the linear range. For ICP5, divide the observed result of the affected element by the observed result of the interfering element. Since the IEC study was done with the previous IEC factor applied, add (or subtract if negative) this result to the current correction factor value in the method. The result is the new IEC for the affected element, equal to the adjustment made on 1 ppm of the affected element. ICP6 has an optional auto-IEC calculation feature in the software.

10.0 POLLUTION CONTROL

It is TestAmerica's policy to evaluate each method and look for opportunities to minimize waste generated (i.e., examine recycling options, ordering chemicals based on quantity needed, preparation of reagents based on anticipated usage and reagent stability). Employees must abide by the policies in section 13 of the Corporate Safety Manual for "Waste Management and Pollution Prevention."

10.1 Waste Management

Waste management practices are conducted consistent with all applicable rules and regulations. Excess reagents, samples and method process wastes are disposed of in an accepted manner. Waste description rules and land disposal restrictions are followed. Waste disposal procedures are incorporated by reference to UP-WM-001. The following waste streams are produced when this method is carried out.

- Waste from this procedure will enter the "Corrosive Wastewater" waste stream.

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(A&B) Calibration Stock Solutions
Attachment 2. Stock QC Solutions
Attachment 3. Example: Analysis Sequence Log / Maintenance Log
Attachment 4. Example: Data Review Checklist
Attachment 5. Known Digested Quality Control

14.0 REVISION HISTORY

- Revision 05 updated on 10/30/14
- Annual Review
- No Changes

Table 1
Element and Reporting Limits

Element	ICAP 61E Wavelength (nm)	ICAP 6500 Wavelength (nm)	Reporting Limit (RL)[1] (ug/L)	Soils (mg/kg)
Al	308.2	308.2	200	20.0
Sb	206.8	206.8	20	2.0
As	189.0	189.0	10	1.0
Ba	493.4	455.4	10	1.0
Be	313.0	234.8	4	0.40
Bi	N/A	223.0	20	2.0
B	249.6	208.9	50	5.0
Ca	317.9	317.9	200	20.0
Cd	226.5	228.8	2	0.20
Cr	267.7	267.7	10	1.0
Co	228.6	228.6	5	0.50
Cu	324.7	324.7	10	1.0
Fe	271.4	271.4	200	20.0
Pb	220.3	220.3	5	0.5
Li	N/A	670.7	10	1.0
Mg	279.0	279.0	100	10.0
Mn	257.6	257.6	10	1.0
Mo	202.0	202.0	10	1.0
Ni	231.6	231.6	10	1.0
K	766.4	766.4	500	50.0
Se	196.0	196.0	10	1.0
Si	288.1	212.4	200	20.0
Ag	328.0	328.0	5	0.50
Na	588.9	589.5	1,000	100
Sr	421.5	421.5	5	0.50
Tl	190.8	190.8	10	1.0
Sn	189.9	189.9	40	4.0
Ti	334.9	334.9	5	0.50
V	292.4	292.4	5	0.50
Zn	206.2	206.2	20	2.0
Y[3]	371.0	224.3/360.0/371.0	N/A	N/A
In[3]	N/A	230.6	N/A	N/A

[1] These are routine ICAP reporting limits (RL). Lower RLs may be available to use per client request. Sample RLs will vary depending on sample volume, dilution factors, and changes in MDLs. Contact the laboratory for the most current RLs based on annual MDL determinations.
[3] Y on the ICAP 61E and Y and In on the ICAP 6500, are used as internal standards and are introduced continuously to all samples (including standards and QC samples) via the peristaltic pump at an approximate concentration of 5 ppm. The ICP can have some ionization effects caused by torch positioning. To eliminate these effects, Cesium may be added to the internal standard solution (100 mLs / 1-Liter).

Attachment 1A.

Trace 61E Calibration Stock Standard 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	1.0			
		Mo	100	0.4	0.5	1.0			
		Si	100	0.4	0.5	1.0			
		Sn	100	0.4	0.5	1.0			
		Ti	100	0.4	0.5	1.0			
Inorganic Ventures	RFW-ICPT-STD-1C	Al	1,000	---	5	10			
		Fe	1,000	---	5	10			
		K	1,000	---	---	---			
		Na	1,000	---	---	---			
		Li	800	3.2	4	8			
		Mg	800	---	4	8			
		Ca	400	---	---	---			
Inorganic Ventures	RFW-ICPT-STD-1D	As	100	0.4	0.5	1.0			
		Ba	100	0.4	0.5	1.0			
		Be	100	0.4	0.5	1.0			
		Bi	100	0.4	0.5	1.0			
		B	100	0.4	0.5	1.0			
		Cd	100	0.4	0.5	1.0			
		Cr	100	0.4	0.5	1.0			
		Co	100	0.4	0.5	1.0			
		Cu	100	0.4	0.5	1.0			
		Pb	100	0.4	0.5	1.0			
		Ni	100	0.4	0.5	1.0			
		Se	100	0.4	0.5	1.0			
		Ag	100	0.4	0.5	1.0			
		Sr	100	0.4	0.5	1.0			
		Tl	100	0.4	0.5	1.0			
		Zn	100	0.4	0.5	1.0			
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 1B.

iCAP 6500 Calibration Stock Standard Solutions

Vendor	Stock Name	Element	Conc. (mg/L)	S1 mg/L	S2 mg/L
Inorganic Ventures	STDIL-STD-2	Sb	100	1	
		Mo	100	1	
		Sn	100	1	
		Ti	100	1	
Inorganic Ventures	STDIL-STD-1	As	100	1	
		Ba	100	1	
		Be	100	1	
		B-	100	1	
		Cd	100	1	
		Co	100	1	
		Cr	100	1	
		Cu	100	1	
		Mn	100	1	
		Ni	100	1	
		Pb	100	1	
		Se	100	1	
		Sr	100	1	
		Tl	100	1	
		V-	100	1	
		Zn	100	1	
Inorganic Ventures	CGBI1-1	Bi	1000	1	
	CGSI1-1	Si	1000	1	
	CGAG1-1	Ag	1000	1	
Inorganic Ventures	RFW-ICPT-STD-2A	Al	10,000		100
		K	10,000		100
Inorganic Ventures	RFW-ICPT-STD-2B	Ca	5,000		100
		Fe	5,000		100
		Mg	5,000		100
		Na	5,000		100
Inorganic Ventures	MSLI-100ppm	Li	100	1	

Note: Vendors and stock names may vary.

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 A1	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)	Daily LR DoD (mg/L)
Inorganic	Aluminum	Al	*See ICSA*[1]	500 (ICSA)[1]
Ventures	Antimony	Sb	1,000	10
	Arsenic	As	1,000	5
	Barium	Ba	1,000	10
	Beryllium	Be	1,000	5
	Bismuth[1]	Bi	1,000	5
	Boron	B	1,000	10
	Cadmium	Cd	1,000	5
	Calcium	Ca	*See ICSA*[1]	500 (ICSA)[1]
	Chromium	Cr	1,000	10
	Cobalt	Co	1,000	10
	Copper	Cu	1,000	10
	Iron	Fe	*See ICSA*[1]	200 (ICSA)[1]
	Lead	Pb	1,000	10
	Lithium[2]	Li	100	
	Magnesium	Mg	*See ICSA*[1]	500 (ICSA)[1]
	Manganese	Mn	1,000	10
	Molybdenum	Mo	1,000	10
	Nickel	Ni	1,000	10
	Potassium	K	*See S2*[1]*/10,000*	100 (S2)[1]/500
	Selenium	Se	1,000	5
	Silicon	Si	1,000	10
	Silver	Ag	*See S1*[1]	1 (S1)[1]
	Sodium	Na	*See S2*/10,000[3]	50 (S2)[1]/400[3]
	Strontium	Sr	*See S1*[1]	1 (S1)[1]
	Thallium	Tl	1,000	5
	Tin	Sn	1,000	5
	Titanium	Ti	1,000	5
	Vanadium	V	1,000	10
	Zinc	Zn	1,000	10

[1] Bismuth is run on ICP6 only.
[2] Lithium is run on ICP6 only.

Attachment 2.
(continued)
Examples of Stock QC Solutions

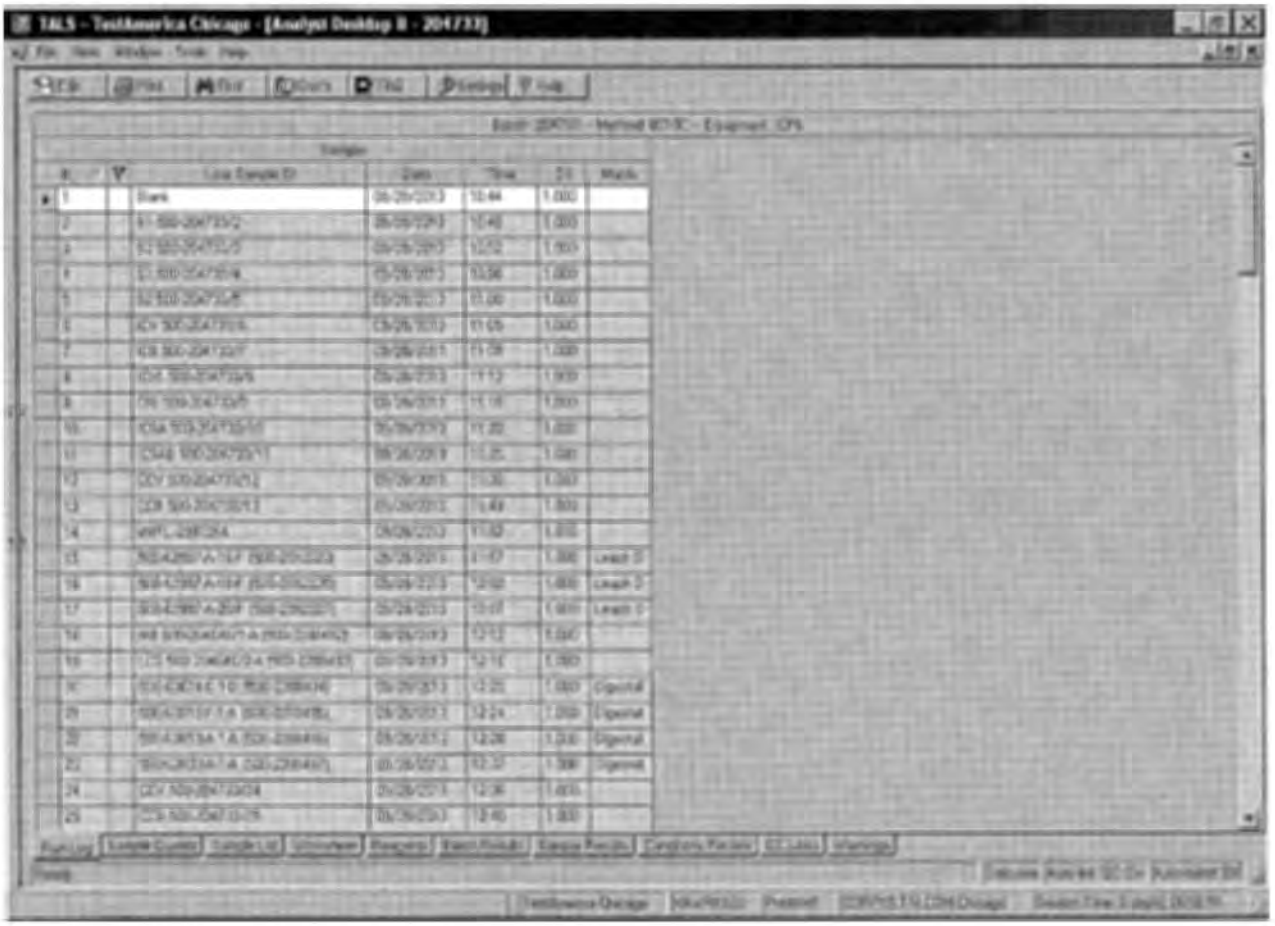
Vendor	Stock Name	Element	Conc. (mg/L)	ICVL/CCVLConc. (mg/L)	CRI (mg/L)
Inorganic	TACHI-2	Al	200	0.200	0.400
Ventures	TACHI-1	Sb	20	0.020	0.040
	TACHI-2	As	10	0.010	0.020
	TACHI-2	Ba	10	0.010	0.020
	TACHI-2	Be	4	0.0040	0.008
	Bi Stock	Bi	1,000	0.020	0.040
	TACHI-2	B	50	0.050	0.100
	TACHI-2	Cd	2	0.0020	0.0040
	TACHI-2	Ca	200	0.200	0.400
	TACHI-2	Cr	10	0.010	0.020
	TACHI-2	Co	5	0.0050	0.010
	TACHI-2	Cu	10	0.010	0.020
	TACHI-2	Fe	200	0.20	0.40
	TACHI-2	Pb	5	0.0050	0.010
	TACHI-2	Mg	100	0.10	0.20
	TACHI-2	Mn	10	0.010	0.020
	TACHI-1	Mo	10	0.010	0.020
	TACHI-2	Ni	10	0.010	0.020
	TACHI-2	K	500	0.500	1.00
	TACHI-2	Se	10	0.010	0.020
	TACHI-1	Si	200	0.20	0.40
	TACHI-2	Ag	5	0.0050	0.010
	TACHI-2	Na	1000	1.00	2.00
	TACHI-2	Sr	5	0.0050	0.010
	TACHI-2	Tl	10	0.010	0.020
	TACHI-1	Sn	40	0.040	0.080
	TACHI-1	Ti	5	0.0050	0.010
	TACHI-2	V	5	0.0050	0.010
	TACHI-2	Zn	20	0.020	0.040

Attachment 2.
(continued)
Stock QC Solutions

Vendor	Stock Name	Element	Conc. (mg/L)	ICSA 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
				ICSAB 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 Sequence Log / Maintenance Log (026-001)



TestAmerica Chicago
TJA Trace ICAP (61E) – ICP5
Instrument Maintenance Log

Page No._____

	Date/Initials	Date/Initials	Date/Initials	Date/Initials	Date/Initials	Date/Initials	Date/Initials
Daily Maintenance:							
Check/Change Pump Tubing							
Check Waste Container							
Check Torch for buildup (Note Cleaning)							
Check Nebulizer / Spray Chamber							
Internal Standard Filename / As =							
Internal Standard Filename / Y =							
Internal Standard Filename / As =							
Internal Standard Filename / Y =							
Internal Standard Filename / As =							
Internal Standard Filename / Y =							

Non-Routine Maintenance:*	Date	Initials	Maintenance Details	Return to Control

Comments:_____

_____

Any Maintenance/Repair/Part Replacement performed that is not listed above must be documented in the Non-Routine Maintenance sections

Reviewer Signature:_____ Date:_____ **CHI-22-14-063/C-09/12**

(100420)

Attachment 4.

Example: Data Review Checklist
(027-001 to 027-002)

TestAmerica Chicago

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ICP Data Review Checklist

LIMS Batch Number:	**Filename:**	**Instrument ID (circle one):** ICP5 ICP6 ICP8
Analyst/1st Reviewer:	**Method (circle):** 200.7 6010B 6010C	**QC Type (circle):** Standard QAPP Other
Matrix (circle): Drinking Water Non-potable Water Solid Leachate		**Circle:** Total Solubles

Review Items	NA	Yes	No	2nd Rev	If No, why is data reportable?
A. Calibration/Instrument Run QC					
1. Instrument calibrated per lab SOP?					
2. For multi-point calibrations: $r \geq 0.995$ (6010B); $r \geq 0.998$ (6010C); $\lvert$x-intercept$\rvert \leq$ RL					
3. Reanalysis of High Standard before samples: 95-105% recovery					
4. ICB/CCB: run before samples, 10% frequency, & closing Result < RL (routine) Result < ½ RL (special project)					If no, list details:
5. ICV/CCV: frequency initial, 10%, & closing 90-110% recovery, 5% RSD (6010) 95-105% recovery, 3% RSD (200.7 SDWA)					If no, list details:
6. ICVL/CCVL: 70-130% recovery (6010C) (MRL Check Std) 80-120% recovery (special project) CRI run, 50-150% (other)					If no, list details:
7. ICSA/ICSAB: run before samples ICSA detections for non-spiked <RL (<LOD for special project) ICSA/ICSAB for spiked elements 80-120% *If no, list analytes:*					□ Results outside limits due to contamination. □ Interfering elements not present in sample at level which would result in false result >±1x RL. □ Concentration of affected analyte in sample is more than 10x analyte signal in ICSA.
8. RL-level check standard: 70-130% recovery (6010C) (MRL Check Std) 80-120% recovery (special projects) CRI run at 2x RL, 50-150% (other)					If no, list details:
B. Client Sample and QC Sample Results					
1. Samples with target element concentrations > linear range diluted and reanalyzed?					Comments:
2. Were all hits reported from a run with interfering elements < linear range?					Comments:
3. Internal standard (IS) response ± 50% of ICB IS? *If no, list details*					□ High IS response. Sample(s) rerun at dilution. □ Low IS response. Sample(s) reanalyzed.
C. Preparation/Matrix QC					
1. Method Blank: one per preparation batch Result < RL (routine); Result < ½ RL (special project) *If no, list blank ID & explain:*					□ No analyte > RL in associated samples □ Sample results >10x blank □ Insufficient sample for reanalysis
2. LCS one per preparation batch 80-120% recovery (routine); 85-115% recovery (200.7) special project limits (other) _____ *If no, list LCS ID & explain:*					□ Insufficient sample for reanalysis □ LCS %R > QC limits & samples < RL
3. MS/MSD or MS/Dup frequency: a pair per batch (routine) a pair per 10 samples (200.7) *If no, list QC ID & explain:*					□ Insufficient sample – LCS/LCSD analyzed
4. MS/MSD recovery & RPD: 75-125% recovery (routine) 70-130% recovery (200.7) 20% RPD project limits (other) *If no, list MS or MSD ID & explain:*					□ LCS acceptable – matrix effects □ Native analyte > 4x spike level □ Matrix effect and native analyte > 4x spike

(027-001)

Review Items	NA	Yes	No	2nd Rev	If No, why is data reportable?
5. If TCLP MS <50% and sample result 80-100% of toxicity characteristic limit, was MSA run?					Comments:
6. Serial dilution: present for each prep batch (routine) Required if MS/MSD fail (special project) run at same dilution as parent sample ≤ 10% difference *If no, list details:*					□ Sample result < 50 x MDL
7. Post digestion spike: Required for 200.7-85-115% recovery; if IS not used Required if MS/MSD fail (6010C); Optional for earlier versions of 6010; *If no, list details:*					
D. Raw Data & TALS Data Entry					
1. Raw Data					
a. Unused data is clearly identified					
b. All crossed out data is initialed and dated (manual entry data only) Note: When the instrument raw data is electronically uploaded into the TALs batch, analyst identification is in raw data file.)					
c. Out of control QC is clearly identified					
d. Any data that has a qualifier tick is commented on with appropriate action taken					
e. The first page of the run includes the filename, instrument, and analyst.					
2. Run Log					
a. Unused data is clearly identified (manual entry data only)					
b. All crossed out data is initialed and dated (manual entry data only)					
c. Analyst initials/signature provided (manual entry data only)					
3. TALS Samples Tab					
a. LIMS Sample IDs / Containers are correct					
b. Method and matrix are correct					
c. Date and time match raw data					
d. Dilutions are correct					
e. Correct suffix designated (where applicable)					
4. TALS Worksheet Tab is complete and correct					
5. TALS Reagent Tab is complete and correct					
6. TALS QC Links Tab is correct					
7. TALS Sample Results Tab					
a. All unused data are marked Rejected or Accepted					
b. All reported analytes are marked Primary or Secondary					
8. TALS Batch Information Screen documentation is complete					
9. TALS Status set to appropriate review level					
E. Final Report and NCMs (2nd level review only)					
1. Were all job/project requirements met?					
2. Results for samples and QC correct on final report?					
3. Are all necessary scanned documents in TALS?					
4. NCMs reviewed for applicability, correct references to batches, grammar/typographical errors?					

2nd Reviewer: _____ **Review Date:** _____

Comments: _____

_____

_____

(027-002)

Attachment 5.

Known Digested QC Values (mg/L)

Element	LCS/Matrix 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
Li	0.5	---
Mg	10	---
Mn	0.5	---
Mo	1	---
Ni	0.5	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%
Matrix Spike: 75 - 125%
TCLP Spike: >50%

TITLE: SAMPLE PREPARATION
Toxicity Characteristic Leaching Procedure (TCLP)

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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.

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

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 Assurance Manual (UP-QA-QAM).

1.2 Summary of Method

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). Note: If elements not contained in the TCLP spiking solutions are requested, the sample will be spiked (with the appropriate solution) by the metals prep analyst.

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 must abide by the policies and procedures in the Corporate Safety Manual, Radiation Safety Manual, Lab Specific Addendum to the CSM, and this document. This procedure may involve hazardous material, operations and equipment. This SOP does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of the method to follow appropriate safety, waste disposal and health practices under the assumption that all samples and reagents are potentially hazardous. Safety glasses, gloves, lab coat and closed-toe, nonabsorbent shoes are a minimum.

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.

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 SDS for each of the materials listed in the table. A complete list of materials used in the method can be found in the reagents and materials section. Employees must review the information in the SDS for each material before using it for the first time or when there are major changes to the SDS.

Material (1)	Hazards	Exposure Limit (2)	Signs and symptoms of exposure
Hydrochloric Acid	Corrosive Poison	5 ppm-Ceiling	Inhalation of vapors can cause coughing, choking, inflammation of the nose, throat, and upper respiratory tract, and in severe cases, pulmonary edema, circulatory failure, and death. Can cause redness, pain, and severe skin burns. Vapors are irritating and may cause damage to the eyes. Contact may cause severe burns and permanent eye damage.
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.
Sodium Hydroxide	Corrosive Poison	2 ppm, 5 mg/m^3	This material will cause burns if comes into contact with the skin or eyes. Inhalation of Sodium Hydroxide dust will cause irritation of the nasal and respiratory system.

Material (1)	Hazards	Exposure Limit (2)	Signs and symptoms of exposure
Acetic Acid	Corrosive Poison Flammable	10 ppm-TWA	Contact with concentrated solution may cause serious damage to the skin and eyes. Inhalation of concentrated vapors may cause serious damage to the lining of the nose, throat, and lungs. Breathing difficulties may occur.
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

- The extractor is a custom made rotary-type design that meets the specifications of tumbling the samples at a rate of 30±2 RPMs. The tumbler rate is checked weekly with the tumbler full of samples and documented on the TCLP Rotation Check Sheet (CHI-22-09-357).
- 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 Appendix 3 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, the filters shall be acid-washed prior to use by placing them in a container of 1 N nitric acid, soaking them for approximately 10 minutes and then performing 3 consecutive rinses with deionized (DI) water (a minimum of 1 L per rinse is recommended).

NOTE: After a single use, Teflon tumbler jars and ZHEs shall be completely disassembled and all the parts washed in hot soapy water, followed by 3 rinses in tap water and three rinses in DI water, prior to re-assembly for the next sample. If there has been contamination with an oily substance, a methanol rinse may be necessary, followed by oven drying (ZHEs only) to evaporate the solvent.

- Lab balance capable of reading ± 0.01 g
- Tedlar Bags® / 40 mL VOA Vials
- 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).
- 40 mL VOA Vials
- Non-Coring stainless steel needle w/female luer lock. Male stainless steel luer lock adapter, non-coring stainless steel vent tube.

5.0 REAGENTS AND STANDARDS

All purchased acids, reagents, and dry chemicals used in this protocol must be ACS Reagent grade or better.

5.1 Hydrochloric Acid (HCl), 1.0 N

To a 1-L Class A volumetric flask containing ~500 mLs of DI water, carefully add 83 mLs of concentrated hydrochloric acid. Swirl the flask to mix. Dilute to volume with DI 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 DI water, carefully add 64 mLs of concentrated nitric acid. Swirl the flask to mix. Dilute to volume with DI 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 DI 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 DI 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 DI 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 DI water. The pH of this extraction fluid should be 4.93 $\pm$ 0.05.

Note: For convenience, TestAmerica Chicago typically prepares this fluid in 20 liter batches, using a 20 liter carboy, a class A graduated cylinder for adding the NaOH, and a re-pipettor to dispense the proportional amount of acetic acid.

- 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 DI water, carefully add 5.7 mL of glacial acetic acid. Swirl the flask to mix. Dilute to volume with DI water. The pH of this Extraction Fluid should be 2.88 $\pm$ 0.05.

Note: For convenience, TestAmerica Chicago typically prepares this fluid in 20 liter batches, using a 20 liter carboy and a re-pipettor to dispense the proportional amount of acetic acid. Any volume can be prepared as long as the components are added proportionally and the pH is acceptable.

- 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 Attachment 3 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. When a malfunctioning ZHE is discovered, that particular ZHE or part will be identified with an 'Out-of-Service' tag or equivalent and removed from service.

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 that contains any visible sediment, even if logged in as a water sample, or if it is multi-phasic, liquid/solid separation is required. The only exception will be when a client has specifically agreed that the sample is to be analyzed as a water sample after a documented discussion with the laboratory.

This involves the filtration device outlined in Sections 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 ± 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 the sample is a liquid and contains any visible sediment, even if it is logged in as a water sample, the percent solids will be determined as follows. The only exception will be when a client has specifically agreed that the sample is to be analyzed as a water sample after a documented discussion with the laboratory.

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

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

- If the dry 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 dry solid phase is ≥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, cutting, or grinding until it is < 9.5 mm. Note: It is not necessary to cut up filamentous material such as gloves or waste filters for all the pieces to fit through the sieve if the surface area per gram is ≥3.1 cm^2. It is not necessary or recommended to attempt to quantify this, but the analyst should use discretion.

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, using the example 1 mm grid as a visual reference.
- Transfer a 5.0 g portion to a 250 mL beaker recording the sample weight on the sample container and on the TCLP Backlog. The pre-test sample weight will be transferred to the TCLP pH Measurement Logbook for samples that need to follow the BP LaMP protocol and upon client request.
- 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 ≤ 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+/-5°C. Continue heating at 50+/-5°C for ten minutes.
- Let the solution cool to room temperature and record the pH. If pH is ≤ 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 ± 2 hours. Note: If pressure builds up in the extractor bottle, especially initially, it is permissible to vent the bottle.

The ambient room temperature shall be maintained at 23 ± 2°C during the extraction period. A Min/Max Thermometer is used to establish that the TCLP extraction room temperature requirement has been met during the TCLP sample extraction process. If temperature exceeds the 23 ± 2 °C, an NCM will be written to document the issue and the PM will be notified via an NCM in TALs of the exceedence. The PM will determine if additional corrective action is necessary.

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. The volume of the extraction fluid filtered (recovered) is recorded in the TCLP Extraction Logbook (CHI-22-15-003).

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. The results may be combined mathematically.

7.6.1.16 Measure and record the pH of the extracts and initiate an Internal Chain of Custody (ICOC) for those extracts which require it. TCLP extracts, accompanied by the extracted blank, are ready for transfer to metals digestion and/or organic extractions for further preparation according to the procedures for the particular analysis (organics or metals). *Note: For BP samples that require LaMP protocols, each analytical or prep group must also be supplied with a sufficient volume of extraction fluid to prepare an LCS.*

If metals analysis is required, immediately remove and acidify (nitric acid to pH <2) an appropriate portion and reserve for analysis. If precipitation is observed upon the addition of nitric acid to a small aliquot of the extract, then the remaining portion of the extract for metals analyses shall not be acidified and the extract shall be analyzed as soon as possible. The analyst will initiate an NCM to document this event. Refrigerate the remaining 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, cutting, or grinding if possible until the particle size is < 9.5 mm. Note: It is not necessary to cut up filamentous material such as gloves or waste filters for all the pieces to fit through the sieve if the surface area per gram is $\geq$3.1 cm^2. It is not necessary or recommended to attempt to quantify this, but the analyst should use discretion.

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 ± 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 10 ± 5 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 ± 2 hours. The temperature of the room shall be maintained at 23 ± 2°C during agitation. A Min/Max Thermometer is used to establish that the TCLP extraction room temperature requirement has been met during the TCLP sample extraction process. If temperature exceeds the 23 ± 2 °C, an NCM will be written to document the issue and the PM will be notified via an NCM in TALs of the exceedence. The PM will determine if additional corrective action is necessary.

7.6.2.12 Following the 18 hour extraction of the sample, 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 or the pressure is not within 5 psi of the initial pressure) 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 and the final pressure is within 5 psi of the initial pressure, 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

Important extraction information including dates, temperatures, filter lot numbers, container identification numbers, etc., are recorded in the TCLP extraction logbook and completed for each day's analysis (see 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 POLLUTION CONTROL

It is TestAmerica's policy to evaluate each method and look for opportunities to minimize waste generated (i.e., examine recycling options, ordering chemicals based on quantity needed, preparation of reagents based on anticipated usage and reagent stability). Employees must abide by the policies in section 13 of the Corporate Safety Manual for "Waste Management and Pollution Prevention."

10.1 Waste Streams Produced by the Method

Waste management practices are conducted consistent with all applicable rules and regulations. Excess reagents, samples and method process wastes are disposed of in an accepted manner. Waste description rules and land disposal restrictions are followed. Waste disposal procedures are incorporated by reference to UP-WM-001.

Waste from this procedure will enter the "Waste Water" waste stream.

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" waste stream.
- 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" waste stream.

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
Attachment 3: pH Calibration Work Instruction

14.0 REVISION HISTORY

- Revision 21, was updated on 08/31/15
- Audit Response
- Section 4.0 was updated to reflect the weekly tumbler rate check conducted while the tumbler is full of samples.
- Section 4.0 was updated to reference the use of a methanol rinse for ZHE's
- Section 7.6.1.14 was updated to instruct analyst to record the amount of fluid recovered in the TCLP Logbook
- Section 7.6.1.16 was updated to provide instructions on what to do when a precipitate is observed when nitric acid is added for metals analysis.
- Sections 7.6.1.13 and 7.6.2.11 were updated with the SOP change form Min/Max Thermometer text.

Figure 1.

TCLP Flowchart

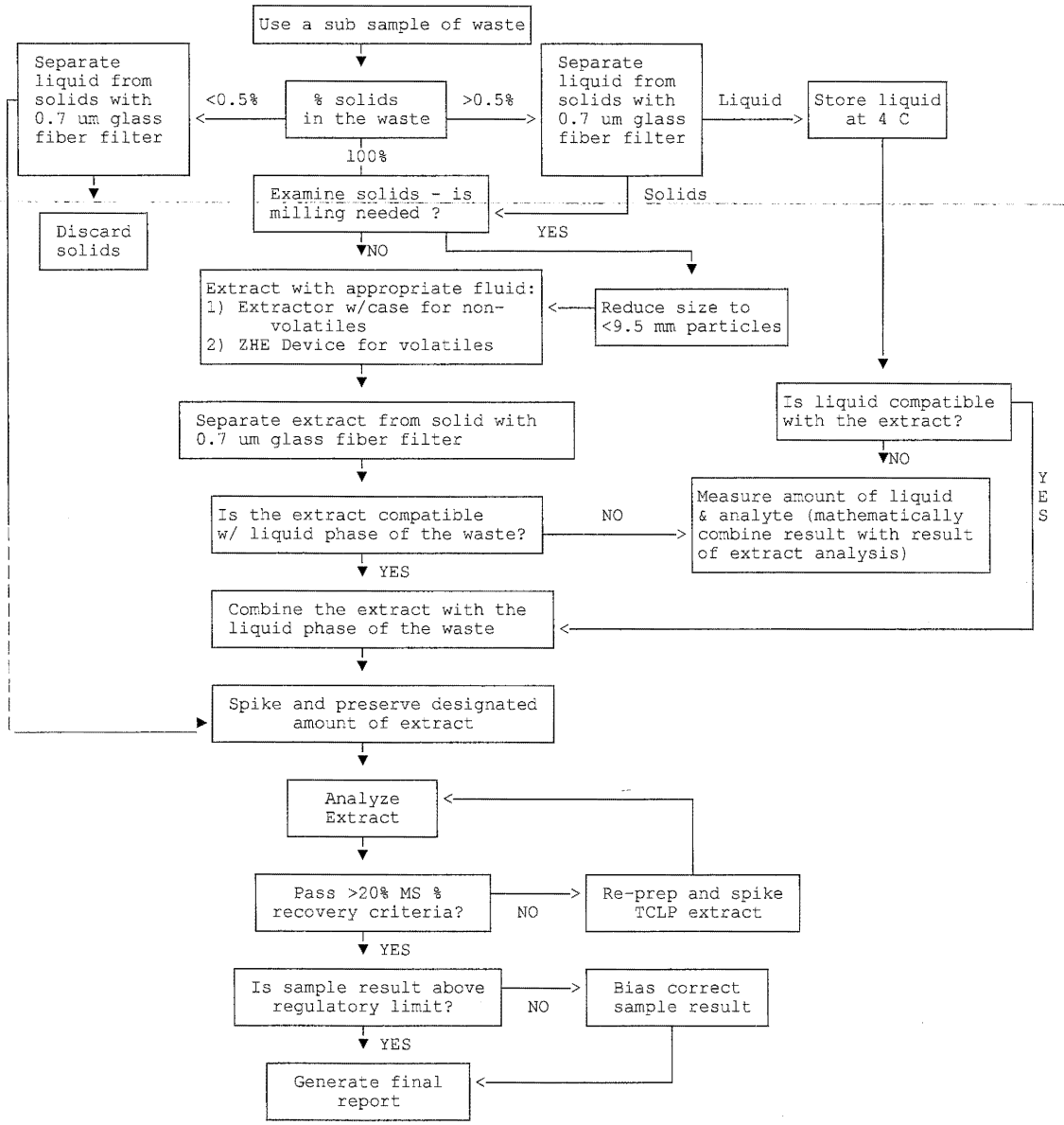

Table 1.

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 the Metals Digestion Standard Spiking solution as referenced in SOP UP-SP-3000, Appendix A, into the TCLP extract.
- Preserve the TCLP spiked extract with 2 mLs of concentrated nitric acid.
- Store at 4 $\pm$ 2°C.

Attachment 2.

Example: TCLP Extraction Logbook

TestAmerica Chicago
TCLP Extraction Logbook

Page Number:_____

Rotator RPM : 30 ± 2 RPMs ID#: _____ Extraction Start Date / Time / Temp: _____/_____/_____ °C Filtration Start Time: _____

Group Number:_____ Extraction End Date / Time / Temp: _____/_____/_____ °C Filtration End Time: _____

LIMS Batch No.:_____ Min.Temp: Uncorrected/Corrected____/____ °C Max.Temp: Uncorrected/Corrected____/____ °C Filter Paper Lot #: _____

Sample Size Specifications:____<9.5 mm__ Control Limits: 10 ± 5 PSI; 23 ± 2 °C Thermometer ID: _____

Sample Type (Circle): TCLV SPLP TCLP ASTM

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 (Lot # _____)

Analyst: _____ Date:_____

Reviewer:_____ Date:_____

Attachment 3.

Example: pH Calibration Work Instruction

Calibration Instructions

Thermo Scientific: Orion Star A211 Benchtop pH Meter

C-2621

The meter **must be re-calibrated daily prior to use,** documenting the calibration in the TCLP pH logbook.

1. Bring buffers pH 2.0. 4.0, 7.0 and 10.0 to room temperature and pour fresh daily.
2. In measurement mode, press **f1 (call)**
3. Rinse the pH electrode and any other electrodes in use with distilled water, blot dry with a lint-free tissue and immerse in the pH buffer to a depth of ~1.5" in the pH 4.0 buffer.
4. When the electrode and buffer are ready, press **f3 (start)**.
5. Wait for the pH value on the meter to stabilize and stop flashing and perform one of the following actions:

a. Press **f2 (accept)** to accept the displayed pH value.

b. Press **f3 (edit)** to access the numeric entry screen and edit the pH buffer value.

i. Press **setup ^** **mode >** **log/print >** **or** **hold <**

To highlight a number, decimal point or negative sign; **press f3 (enter)** to select the highlighted item and repeat until the buffer value at the measured temperature is shown above the numeric entry screen.

ii. Press **f2 (done)** to exit the numeric entry screen

iii. Press **f3 (accept)** to accept the entered pH value.

6. Press **f2 (next)** to proceed to the next buffer 7.0 and then 10.0 and repeat steps 3 through 5 for each then press **f3 (cal done)** to save and end calibration
7. The meter will display the calibration summary including the average slope. Press **f1 (meas)** to export the data to the calibration log. The meter will automatically proceed to the measurement mode.
8. Verify calibration by reading back the 2.0, 4.0, 7.0 and 10.0 buffers, and bracket every 10 sample readings with an alternate-source pH 7.0 QC buffer. The calibration verification is documented in the TCLP pH logbook.

For additional information, more detailed instructions, and a trouble-shooting guide, see the Instruction Manual C-2621 stored near the pH meter.

TestAmerica Chicago
STANDARD OPERATING PRACTICE (SOP) CHANGE FORM

Original SOP Number/Revision #: UP-SP-3000, Rev. ~~24~~ 25 ng 7/2/15 Last Mod ID (circle): NA/ _____

SOP Title: **SAMPLE PREPARATION: Metals Digestion by SW-846 3000 Series**

Affected SOP Section Number(s): 7.3.1
Effective Date of Change: 06/30/15

CONTROLLED DISTRIBUTION

COPY # :
ISSUED TO : **Uncontrolled**

Full Signature Approvals Are Kept on File with TestAmerica Chicago Standard Practice Records

Revision Number with Mod ID: UP-SP-3000, Rev. ~~24A~~ 25A ng 7-2-15

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: Audit Response_BP LaMP PT

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

7.3.1 Sample Preparation (Dissolved)

Samples that are to be reported as "Dissolved" which have **NOT** been filtered in the field, are logged with a "Filtration" pre-Prep in TALs and will be filtered in the laboratory.

The filtration procedure is as follows:

1. An unpreserved sample aliquot of approximately 250 mL is pressure filtered through a 0.45 um filter into a 250 ml plastic sample bottle. (Document Filter Lot # in the filtration batch in TALs)
2. For metals analysis the filtrate is preserved with HNO3 to pH < 2. (Document Acid Lot # in filtration batch in TALs)
3. For metals analysis a sticker is placed on the filtrate stating "Do not analyze for 24 hours".
4. Each sample filtrate is issued a unique sample ID TALs label ensuring that the sample is traceable to the filtration batch.
5. The filtration apparatus is cleaned after each use.

NOTE: The LCS and MB must be filtered through the 0.45 um filter prior to being analyzed with samples logged for dissolved metals.

Paul Kalm 6/29/15
Initiated/Reviewed By: Name/Date

Initiated/Reviewed By: Name/Date

Approval Signature/Date: Section Manager

Teresa A. Preston 6/29/15
Approval Signature/Date: QA Manager or Designee

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

Approvals (Signature/Date):

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Michael J. Healy 1/21/15
Michael J. Healy Date
Laboratory Director

Facility Distribution No: _____ **Distributed To:** Uncontrolled

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 Inductively Coupled Argon Plasma / Mass Spectrometry (ICP-MS). 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, ICP-MS
3010A	Waters and extracts for analysis by Trace ICP
3030C	See note below.
3050B	Soil and waste samples for analysis by Trace ICP

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.

Note: The state of North Carolina requires the use of Standard Methods 3030C for the preparation of wastewater samples.

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 Assurance Manual (UP-QA_QAM).

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 ICP-MS.

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 types 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, Lab Specific Addendum to the CSM, and this document. This procedure may involve hazardous material, operations and equipment. This SOP does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of the method to follow appropriate safety, waste disposal and health practices under the assumption that all samples and reagents are potentially hazardous. Safety glasses, gloves, lab coat and closed-toe, nonabsorbent shoes are a minimum.

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 SDS for each of the materials listed in the table. A complete list of materials used in the method can be found in the reagents and materials section. Employees must review the information in the SDS for each material before using it for the first time or when there are major changes to the SDS.

Material (1)	Hazards	Exposure Limit (2)	Signs and symptoms of exposure
Hydrochloric Acid	Corrosive Poison	5 ppm-Ceiling	Inhalation of vapors can cause coughing, choking, inflammation of the nose, throat, and upper respiratory tract, and in severe cases, pulmonary edema, circulatory failure, and death. Can cause redness, pain, and severe skin burns. Vapors are irritating and may cause damage to the eyes. Contact may cause severe burns and permanent eye damage.
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.
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
- Fume hood(s)
- Eppendorf Pipettes
- Watch glasses (ribbed & non-ribbed)
- Filters and plunger apparatus[1]
- 100 & 50 mL digestate vessels[1] (which are checked to ensure volume markings at 25 mL, 50 mL, and 100 mL are within 2.5% Tolerance).
- 100 mL Snap-Cap containers for digestates (which are checked to ensure volume markings are within 2.5% Tolerance).
- Teflon strips (soils)

[1]Each time the metals digestions staff opens a new case/box of filters or digestion tubes, the certificate must be removed and the lot number compared with previous lot numbers. If the case/box has a new lot number, the filters or digestion tubes must be verified as free of metals contamination to the lowest reporting level by in-house testing. This will be accomplished as follows:

1. Prep analyst identifies new lot and documents the prep batch number for the first ICP and ICPMS MB created on the vendor supplied quality certificate.
2. The lot number will be documented in the comments section of the LIMs prep batch.
3. The certificate will be forwarded to the instrument analyst attached to the metals digestion sheet.
4. After the analysis of the MB, the raw data will be reviewed to determine if the acceptance criteria of results falling below ½ the current RL are met.
5. If the acceptance criteria are met, the analyst or supervisor will initial and date the certificate to indicate approval and document the analysis run and batch where the results may be found.
6. If the criteria are not met, the analyst must notify their supervisor immediately who will then initiate corrective action measures.
7. The certificate will be maintained on file in the Metals Department.

Note: Any consumable items for which a lot number is required to be tracked, must be stored with the lot number present. **Do Not** Separate the containers (boxes/bags) with the lot number identified from the consumable item until that consumable item is all used up.

5.0 REAGENTS AND STANDARDS

5.1 Reagents

- Concentrated Nitric Acid (Instra Pure)
- Concentrated Hydrochloric Acid (Instra Pure)
- 30% Hydrogen Peroxide Solution
- 30% Ultrex II Ultra Pure 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 ICP and ICP-MS Intermediate Standards

These standards are prepared from multi-element solutions purchased from vendors. Single element stocks are also required. These purchased solutions expire 1-year from the date of receipt.

Standard	Preparation
ICP Spike Solution And ICP-MS Spike Solution	**For both ICP and ICPMS**, add ~400 mLs of Milli-Q water to a 1-L Class A volumetric flask. **For ICP**, add 100 mLs each of HP1381-A-500, HP1381-B-500 and HP1381-C-500 **For ICPMS**, add 100 mLs each of HP2930-A-500, HP2930-B-500, HP2930-C-500 **For both ICP and ICPMS**, add the following: • 9 mLs of 1,000 ppm Se; • 8 mLs of 1,000 ppm Pb; • 6 mLs of 1,000 ppm As; • 5 mLs of 1,000 ppm Tl; and • 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.

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 5 - 10 mLs of Milli-Q water. [1]	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 (DU) [2]	Aliquot of the same field sample that is digested independently.	1 per 20 or fewer samples.
Laboratory Control Sample (LCS) [3]	For soil sample batches, use a Teflon strip, 5 - 10 mLs of Milli-Q water and spike as listed below. [1, 4]	1 per 20 or fewer samples.
	For water sample batches, use 50 mLs of Milli-Q water and spike as listed below. [4]	1 per 20 or fewer samples.
Matrix Spike (MS); MS Duplicate (MSD) [2]	Aliquot of the same field sample that is spiked as listed below [4] and digested independently.	1 per 20 or fewer samples.

[1] For those programs, clients, and projects mandating the use of a 'soil' matrix for the preparation of Soil MB's and LCS's, Teflon Strands will be incorporated. The lot number of the Teflon strips will be documented in the preparation batch notes.
[2] The sample selection for MS/MSD/DU is rotated among client samples so that various matrix problems may be noted and/or addressed.
[3] LCS Duplicate (LCSD) is performed when requested by the client, contract or QAP.
[4] 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
ICP or ICP-MS	0.5 mL of ICP or ICP-MS Intermediate Spiking Solution.	1 mL of ICP or ICP-MS 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 SOP No. UP-SP-1311.

- **Matrix spikes for solid matrices are to be spiked prior to adding the water.**
- **Matrix spikes for TCLP extracts are added after filtration of the TCLP extract and before preservation. Refer to UP-SP-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

Note: 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 24 hours".

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 to provide a uniform matrix from which to take a sample aliquot. Sample size is approximately 1.0 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 analyst 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 in TALS.

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 mL 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. Do not allow the samples to boil.
- 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. Transfer the digestate to a digestion vessel, washing down the sides of the beaker with Milli-Q water as needed. Dilute the sample to a 50 mL final volume using Milli-Q water and filter using the plunger apparatus.

7.6.1.1 Method 3005A-Modified for ICP-MS

- Transfer 50 mLs of the well-mixed (homogenized) sample into a 50 mL digestion vessel.
- Add 1 mL of InstraPure nitric acid and ***0.5 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:** Method 3005A was modified for ICP-MS to matrix match the preparation with the analytical standards. In this regard, a volume of 0.5 mL of InstraPure hydrochloric acid is added to the digestion vessel in place of 2.5 mLs. Hydrochloric acid is a known interferent on the ICP-MS.

7.6.2 Method 3010A

Used for aqueous samples and extracts for ICP analysis.

- 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 (approximately 20 mLs) – just enough to cover the bottom of the vessel. **The sample should not boil or evaporate completely on any portion of the bottom of the vessel. If this should happen, a re-digestion must be done.**
- Remove the vessel from the hot block and allow to cool.
- Add another 1.5 mLs 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.
- 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. Transfer the digestate to a digestion vessel, washing down the sides of the beaker with Milli-Q water as needed. Dilute the sample to a 50 mL final volume using Milli-Q water and filter using the plunger apparatus.

7.6.3 Method 3030C

- Transfer 50 mLs of well mixed (homogenized) sample into a digestion vessel.
- Add 1.25 mLs of InstraPure hydrochloric acid.
- Heat 15 minutes on hot block, at 90-95 degrees C.
- Cool sample
- Filter through a pre-rinsed (with DI water) 0.45 um membrane filter.
- Bring sample up to volume of 50 mL and mix.
- Sample is now ready for analysis.

7.6.4 Method 3050B

Used for soils, sludge, sediments, wipes, paint chips, and other miscellaneous solid samples, such as crushed wood or bricks.

- Weigh out a portion of homogenized sample, generally 1.0 – 1.2 grams, into a 100 mL vial. The exact weight is recorded in the TALS digestion spreadsheet. More sample weight may be used if the liquid content is high, as long as the digestion is complete.

NOTE: When using the hot plate/beaker combination, soils are generally weighed to 1.00-2.00 grams. All other volumes are the same as for hot block digestions.

- Add 5 mLs of InstraPure nitric acid and 5 mLs of Milli-Q water.
- Cover the vial with a non-ribbed watch glass and place on a preheated hot block set at 95° ± 5° C for 15 minutes without boiling.
- Remove the vial from the hot block and allow to cool.
- Add 5 mLs of InstraPure nitric acid and reflux for 30 minutes with a non-ribbed watch glass.

- 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 vial. **Do not allow the sample to boil.**
- Remove the vial from the hot block and allow to cool.
- Add 2 mLs of Milli-Q water and 3 mLs of 30% hydrogen peroxide. If for soil samples, the element Tin is included in the analytes to be reported, the 30% Ultrex II Ultra pure hydrogen peroxide must be used.
- Cover the vial with a non-ribbed watch glass and heat until the reaction is complete.
- Remove the vial from the hot block 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 95° ± 5° C for 2-hours without boiling.
- Maintain a covering of solution on the bottom of the vial (or beaker) at all times.

If the sample is being analyzed by ICP:

- Allow the sample to cool.
- Add 10 mLs of InstraPure hydrochloric acid.
- Place the vial on the hot block and heat for 15 minutes without boiling.
- Remove the vial from the hot block and allow to cool.
- Wash down the sides of the vial 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.

7.7 Documentation

7.7.1 TALS Digestion Spreadsheets

Sample digestion and standard traceability are documented within the TALS ADII spreadsheets. The spreadsheets must be completed for each day's work in real time as the samples are measured and spiked prior to putting them on the hot block. Sample IDs must be scanned into the batches to reduce transcription errors. Any notes relevant to the digestion batch must be made directly in ADII.

The following information must be recorded in the Batch Notes when applicable: Filter Lot #, HCl Lot #, HNO_3 Lot #, H_2O_2 Lot #, Hot Block ID # and Temperature, Thermometer ID #, Start Time, End time, Digestion Tube Lot # and Teflon strips Lot #. Refer to Appendix B for examples of 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 TALs LIMS database and is issued 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 (DU)	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

A Non-Conformance Memo (NCM) will be initiated by the analyst in TALS anytime there is a deviation from the routine preparation procedures, as outlined within this SOP. This includes, but is not limited to: limited sample volume, sample loss due to spills, extremely vigorous reactions, spiking issues or any other issues that may occur during the preparatory procedure. The Section Manager will review the NCM and document any corrective action needed at that time. All analytical 'out-of-control' situations are identified as indicated in the supporting analytical SOP's proper.

9.0 DATA ANALYSIS AND CALCULATIONS

Not Applicable.

10.0 POLLUTION CONTROL

It is TestAmerica's policy to evaluate each method and look for opportunities to minimize waste generated (i.e., examine recycling options, ordering chemicals based on quantity needed, preparation of reagents based on anticipated usage and reagent stability). Employees must abide by the policies in section 13 of the Corporate Safety Manual for "Waste Management and Pollution Prevention."

10.1 Waste Management

Waste management practices are conducted consistent with all applicable rules and regulations. Excess reagents, samples and method process wastes are disposed of in an accepted manner. Waste description rules and land disposal restrictions are followed. Waste disposal procedures are incorporated by reference to UP-WM-001.

- 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: ICP; ICP-MS TALs LIMS Digestion Spreadsheets

14.0 REVISION HISTORY

- Revision 25 was updated on 01/19/15
- Annual Review
- Sections 1.0 and 7.6.5 were updated to remove reference to the AVS-SEM method.
- Appendix A was updated to reflect current spiking solutions.

Appendix A.

Metals Digestion Standard Spike Concentration

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

ICP-MS

Vendor	Stock Name	Elements	Conc. (mg/L)
Environmental Express	HP2930-A-500	Al, Ba	2,000
		Ca, Mg, K, Na	10,000
	HP2930-B-500	Se	10
		Pb	20
		As	40
		Tl, Be, Cd	50
		Cr	200
		Cu	250
		Co, Ni, V, Mn, Zn	500
		B, Fe, Sr	1,000
	HP2930-C-500	Ag	50
		Sb	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

Appendix B.

Example: TALs LIMS Digestion Spreadsheets (014-001 to 014-007)

3005A Prep Worksheet

(Used for Collecting Prep Info)

Batch Number: 500-273161 Analyst: Cain_Vocu, Jennifer L Batch Open: 1/22/2015 7:50:00AM

Batch End: 1/22/2015 8:41:08AM

Preparation, Total Recoverable or Dissolved Metals

	Input Sample Lab ID	Input Sample Lab ID (Analytical Method)	SDG	Matrix	Initial Amount	Final Amount	Due Date	Analytical TAT	Dlv Rank	Comments
1		MB~500-273161/1 N/A	N/A		50 mL	50 mL	N/A	N/A	N/A	
2		LCS~500-273161/2 N/A	N/A		50 mL	50 mL	N/A	N/A	N/A	
3		500-91080-A-3 (6020)	N/A	Water	50 mL	50 mL	1/23/15	2_Days	N/A	
4		500-91127-E-1 (6020A)	N/A	Water	50 mL	50 mL	1/30/15	6_Days	N/A	
5		500-91127-E-1~DU (6020A)	N/A	Water	50 mL	50 mL	1/30/15	6_Days	N/A	
6		500-91127-E-1~MS (6020A)	N/A	Water	50 mL	50 mL	1/30/15	6_Days	N/A	
7		500-91127-E-1~MSD (6020A)	N/A	Water	50 mL	50 mL	1/30/15	6_Days	N/A	
8		500-91127-E-2 (6020A)	N/A	Water	50 mL	50 mL	1/30/15	6_Days	N/A	
9		500-91127-E-3 (6020A)	N/A	Water	50 mL	50 mL	1/30/15	6_Days	N/A	
10		500-91127-E-4 (6020A)	N/A	Water	50 mL	50 mL	1/30/15	6_Days	N/A	
11		500-91127-E-5 (6020A)	N/A	Water	50 mL	50 mL	1/30/15	6_Days	N/A	

(100-h10)

3005A Prep Worksheet

(Used for Collecting Prep Info)

Batch Number: 500-273161 Analyst: Cain_Vocu, Jennifer L Batch Open: 1/22/2015 7:50:00AM

Batch End: 1/22/2015 8:41:08AM

Batch Notes

Digestion Tube/Cup Lot #	1408268
Hot Block ID number	2602
Hood ID or number	3
Lot # of Nitric Acid	93717
Lot # of hydrochloric acid	86340
Uncorrected Temperature	94
Oven, Bath or Block Temperature 1	94
ID number of the thermometer	a1103x
Uncorrected Temperature 2	n/a
Oven, Bath or Block Temperature 2	n/a
Filter Paper Lot Number	filter/plunger 40723760
Pipette ID	2850
Person who witnessed spiking	fg
First Start time	750
First End time	820
pH Paper Lot Number	n/a
Batch Comment	n/a

(200-4010)

3010A Prep Worksheet

(Used for Collecting Prep Info)

Batch Number: 500-273086 Analyst: Hacek, Phillip J Batch Open: 1/21/2015 4:45:00PM

Batch End: 1/21/2015 5:15:00PM

Preparation, Total Metals

	Input Sample Lab ID	Input Sample Lab ID (Analytical Method)	SDG	Matrix	Initial Amount	Final Amount	Due Date	Analytical TAT	Dlv Rank	Comments
1	MB 500-273086/1	MB~500-273086/1 N/A	N/A		50 mL	50 mL	N/A	N/A	N/A	
2	LCS 500-273086/2	LCS~500-273086/2 N/A	N/A		50 mL	50 mL	N/A	N/A	N/A	
3	500-91053-A-1	500-91053-A-1 (6010B)	N/A	Water	5 mL	50 mL	1/30/15	8_Days	N/A	
4	500-90735-C-12	500-90735-C-12 (6010B)	N/A	Water	50 mL	50 mL	1/30/15	12_Days	N/A	
5	500-90735-D-12	500-90735-D-12 (6010B)	N/A	Filtrate	50 mL	50 mL	1/30/15	12_Days	N/A	
6	500-90735-D-12 DU	500-90735-D-12~DU (6010B)	N/A	Filtrate	50 mL	50 mL	1/30/15	12_Days	N/A	
7	500-90735-D-12 MS	500-90735-D-12~MS (6010B)	N/A	Filtrate	50 mL	50 mL	1/30/15	12_Days	N/A	
8	500-90735-D-12 MSD	500-90735-D-12~MSD (6010B)	N/A	Filtrate	50 mL	50 mL	1/30/15	12_Days	N/A	
9	500-90735-C-13	500-90735-C-13 (6010B)	N/A	Water	50 mL	50 mL	1/30/15	12_Days	N/A	
10	500-90735-D-13	500-90735-D-13 (6010B)	N/A	Filtrate	50 mL	50 mL	1/30/15	12_Days	N/A	

(04-003)

3010A Prep Worksheet

(Used for Collecting Prep Info)

Batch Number: 500-273086 Analyst: Hacek, Phillip J Batch Open: 1/21/2015 4:45:00PM

Batch End: 1/21/2015 5:15:00PM

Batch Notes

Lot # of hydrochloric acid	86340
Lot # of Nitric Acid	93717
Vendor of Reagent used	n/a
Digestion Tube/Cup Lot #	1408268
Hot Block ID number	2602
ID number of the thermometer	a1103x
Uncorrected Temperature	99
Oven, Bath or Block Temperature 1	99
Uncorrected Temperature 2	n/a
Oven, Bath or Block Temperature 2	n/a
Filter Paper Lot Number	40723760
Pipette ID	2850
Person who witnessed spiking	MD
First Start time	1645
First End time	1715
pH Paper Lot Number	n/a
Batch Comment	n/a

(500-410)

3050B Prep Worksheet

(Used for Collecting Prep Info)

Batch Number: 500-273174 Analyst: Cain_Vocu, Jennifer L Batch Open: 1/22/2015 9:15:00AM

Batch End: 1/22/2015 9:45:00AM

Preparation, Metals

	Input Sample Lab ID	Input Sample Lab ID (Analytical Method)	SDG	Matrix	Initial Amount	Final Amount	Due Date	Analytical TAT	Dlv Rank	Comments
1	MB 500-273174/1	MB~500-273174/1 N/A	N/A		1.0 g	100 mL	N/A	N/A	N/A	
2	LCS 500-273174/2	LCS~500-273174/2 N/A	N/A		1.0 g	100 mL	N/A	N/A	N/A	
3	500-91112-F-1	500-91112-F-1 (6010B)	N/A	Solid	1.1593 g	100 mL	1/27/15	4_Days	N/A	
4	500-91112-F-2	500-91112-F-2 (6010B)	N/A	Solid	1.1061 g	100 mL	1/27/15	4_Days	N/A	
5	500-91112-F-2 DU	500-91112-F-2~DU (6010B)	N/A	Solid	1.0221 g	100 mL	1/27/15	4_Days	N/A	
6	500-91112-F-2 MS	500-91112-F-2~MS (6010B)	N/A	Solid	1.0729 g	100 mL	1/27/15	4_Days	N/A	
7	500-91112-F-2 MSD	500-91112-F-2~MSD (6010B)	N/A	Solid	1.0065 g	100 mL	1/27/15	4_Days	N/A	
8	500-91115-F-1	500-91115-F-1 (6010B)	N/A	Solid	1.1478 g	100 mL	1/27/15	4_Days	N/A	
9	500-91115-F-2	500-91115-F-2 (6010B)	N/A	Solid	1.0302 g	100 mL	1/27/15	4_Days	N/A	
10	500-91115-F-3	500-91115-F-3 (6010B)	N/A	Solid	1.0101 g	100 mL	1/27/15	4_Days	N/A	
11	500-91105-A-1	500-91105-A-1 (6010C)	N/A	Solid	1.0172 g	100 mL	2/2/15	8_Days	N/A	
11	500-91105-A-1	500-91105-A-1 (6010C)	N/A	Solid	1.0172 g	100 mL	2/2/15	8_Days	N/A	
12	500-91106-A-1	500-91106-A-1 (6010B)	N/A	Solid	1.0342 g	100 mL	2/2/15	8_Days	N/A	

(500-91104)

3050B Prep Worksheet

(Used for Collecting Prep Info)

Batch Number: 500-273174 Analyst: Cain_Vocu, Jennifer L Batch Open: 1/22/2015 9:15:00AM

Batch End: 1/22/2015 9:45:00AM

Batch Notes

Digestion Tube/Cup Lot #	1408268
Blank Soil Lot Number	n/a
Balance ID	1966
Hood ID or number	2
Perform Calculation (0=No, 1=Yes)	n/a
Nominal Amount Used	n/a
Hot Block ID number	2403
Lot # of hydrochloric acid	86340
Lot # of Nitric Acid	93717
Logbook ID for diluted Nitric	n/a
Hydrogen peroxide lot number	4406382
ID number of the thermometer	2012558
Temperature	96/95
Filter Paper Lot Number	9581416
Acid used for pH adjustment	n/a
Pipette ID	1752
Analyst	jcv
First Start time	915
First End time	945
Person's name who witnessed reagent drop	fg

(900-410)

3050B Prep Worksheet

(Used for Collecting Prep Info)

Batch Number: 500-273174 Analyst: Cain_Vocu, Jennifer L Batch Open: 1/22/2015 9:15:00AM

Batch End: 1/22/2015 9:45:00AM

Uncorrected Temperature	96
Oven, Bath or Block Temperature 1	95
Uncorrected Temperature 2	n/a
Oven, Bath or Block Temperature 2	n/a
Snap Cap Lot #	27814010
Batch Comment	n/a

Comments

(400-410)

TITLE: SAMPLE RECEIPT Handling and Processing Procedures

Approvals (Signature/Date):

Jeffrey A. James 3-26-15
Date
Supervisor, Sample Receipt & Container Mgmt

Chris Hoham 3/30/15
Date
Env. Health & Safety Coordinator

Eric Lang 3/30/15
Date
Manager of Project Management

Terese A. Preston 3/30/15
Date
Quality Assurance Manager

Michael J. Healy 3/26/15
Date
Laboratory Director

Facility Distribution No: _____ **Distributed To:** Uncontrolled

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 TestAmerica Chicago.

2.0 TEMPERATURE MONITORING

2.1 Sample Storage

The temperatures of the refrigerated areas are maintained at <6°C but not frozen; 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 readings).

Any out-of-control refrigerator storage temperatures are recorded within a Corrective Action Log (electronic) within the QA Department. Primary Corrective Action may involve thermostat adjustment or defrosting. The temperatures are rechecked for the out-of-control units later the same day or in the case of defrosting, the following morning. 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 recorded in the electronic CAR Log in the QA Department. Further Secondary Corrective Action (required maintenance by outside contractor) is pursued at that time if required and recorded in the CAR Log. 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. Resolution and return to control are recorded in the same log once the issue has been resolved.

2.2 Sample Receipt

All samples that are not hand-delivered directly from the sample site will have temperatures taken in accordance with the Sample Login: Cooler Receipt Temperature Recording Procedure (Attachment 11). Using the cooler temperature blank bottle or if a temperature blank bottle is not present in the cooler, a random sample bottle, the Sample custodians will document the sample receipt temperature readings on the designated client chain-of-custody (COC-Attachment 2) and within LIMS Sample Receipt Checklist (Attachment 6). These readings will be reported to the client in the final data report. Please note that it is a requirement to record both the "uncorrected" and "corrected" temperature readings, the container type (P/G) on the Sample Receipt Cooler Temperature Tracking Log (Attachment 12). Note that before recording the temperature of the sample allow the IR Gun temperature to reach equilibrium for several seconds. If the sample temperature observed is outside the acceptance limits, a second or third temperature reading from the cooler may be necessary.

"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 criterion is required by a QAPP, contract, etc.., the client will be notified and a Non-Conformance Memo (NCM) in TALs LIMs will be written. The documentation will also be recorded on the sample receipt checklist. The samples in question will be logged into the LIMS, but the sample status will be put on "HOLD". Note: Samples must be stored in appropriate storage locations in order to maintain the integrity of the samples pending further instructions from the PM/Client. Waste samples that are stored in the Sample Login hood need to be clearly marked indicating that are to be returned to the client, disposed of or are waiting further PM/Client instructions.

Refer to TestAmerica Chicago's Sample Acceptance Policy (Attachment 9) 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. The corresponding job number is written on the air bill for reporting.

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, and 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 indicated on the Sample Handling Guide (Attachment 3).

NOTES:

- TestAmerica 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 an NCM to the appropriate Project Manager.

3.3 If the COC seals do not match, the difference(s) are noted on the Login Sample Receipt Checklist (Attachment 6) and on an NCM (Attachment 7).

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 LIMS from the COC. If a sampling time is not present on the COC, check the sample container for this documentation and enter it into LIMS. Note this in the NCM. If neither the COC nor the sample container document the 'sampling time', contact the PM and note in the NCM. Once resolved or if there are no discrepancies, review the sampling time entries entered into LIMS 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 sample. The date the trip blank is prepared by the Container Management group (documented on the sample label in the Collection Date/Time Field) will be entered into the 'Sample Comments' field in the job's Login/Samples Tab.

3.6 All water samples that require pH preservation and Method 608 samples will be checked for preservation by login personnel using pH paper (which is specific to the pH range being tested) and a disposable transfer pipette. Refer to the Sample Handling Guide for preservation requirements (Attachment 3). For samples that are acidified for preservation, pH paper strips with the range of 0.3 to 2.3 will be used for the verification. For samples that have base added for preservation, pH paper with the range of 0.0 to 13.0 will be used for the verification. 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 LIMS job login. The preservation check for each individual preserved container will be documented in the Job Login on the Receipt/Container Tab in the "Container pH" field.

The following documentation will be used:

< 2 – used for properly preserved acid samples
> 2 – improperly preserved – would require the addition of acid preservative
> 12 – used for properly preserved base samples
< 12 – improperly preserved – could require the addition of base preservative

3.7 If the samples are not preserved, an NCM 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 documentation.

3.8 If the samples are to be preserved by the login personnel, the information is recorded on the Preservative Lot Documentation for Sample Receipt Log (Attachment 13) and in the "preservation added" field in the Job Login on the Receipt/Container Tab. This information includes: date; client name; sample number; preservative; preservative lot number; volume of preservative added 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 24 hours".

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

3.11 **TestAmerica Chicago Residual Chlorine procedure.** TestAmerica 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, TestAmerica 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		Tested at bench after aliquot taken for analysis
GCMS BNA Method 625		Tested at login.
Drinking Water Inorganic:		
Phenol	...	BOD, TOX tested at bench
Cyanide		
Ammonia		

All unpreserved samples coming from a possible chlorinated waste stream as those identified above will be tested for the presence of Residual Chlorine at time of sample receipt prior to preparation and analysis either by log-in personnel directly or at the bench as described above. Potassium Iodide-Starch Paper test strips will be used for this purpose. 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. Samples for BOD and TOX are tested at the bench and treated accordingly. Documentation for tested samples at log-in will be done directly on the COC. Documentation for samples done at the bench will be completed at the bench appropriate for the test and parameter. For those samples received past hours or over the week-ends, notification will take place at the next possible time and the samples treated.

For GCMS 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 samples will be tested after the appropriate aliquot has been taken for analysis. Samples showing positive residual chlorine will be noted in a NCM and a note relayed to the appropriate Project Manager.

For GC/MS BNA samples for Method 625, if residual chlorine is present, the login personnel will be required to add 80 mg of sodium thiosulfate per liter of sample and mix well. The addition of the reagent will need to be documented on an NCM which is relayed to the project manager.

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 and NCM 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 NCM, 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 LIMS 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 include 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 COC is signed by a laboratory technician and the samples are placed in a walk-in cooler. 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.

For samples under the BP LaMP program, the temperature of the cooler will be checked upon receipt per the Sample Login: Cooler Receipt Temperature Recording Procedure posted in the Login area (Attachment 11). There are several 2nd shift personnel that are trained in this procedure for samples arriving after routine working hours. Please note that it is a requirement to record both the "uncorrected" and "corrected" temperature reading and the container type (P/G) on the Sample Receipt Cooler/Temperature Tracking Log (Attachment 12).

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 the Log-In Checklist and NCM (i.e., VOA vials are received, but an 8260 analysis does not appear on the COC). However, all analyses will be logged into LIMS 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 LIMS 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 LIMS Sample Receipt Checklist (Attachment 6). Any problems with sample condition will be noted on the COC and an NCM will be initiated and submitted to the PM. The Log-In NCM becomes a permanent part of the electronic record that becomes part of the final report generated in the Report Generation Department. 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 LIMS Job Number of 500-24357 for samples 1 through 10 is noted as 500-24357-1-A, 500-24357-2-B, 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 LIMS.

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 LIMS 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. Additionally, the Project Managers will identify 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 LIMS.

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.

5.9 After all the COC and sample information is reviewed to be in agreement, each sample bottle is labeled with their printed LIMS sample number. In the event that sample login receives only one (1) soil 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.

NPDES samples requiring North Carolina DEHNR/DEM certification must be maintained at between 1 and 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 the Internal Cooler Temperature Control Log (Attachment 8).

WIDNR samples temperatures must be verified at time of sample receipt to be </= 4.0 deg C. If the temperature is above the limit specified, an NCM must be completed, sent to the appropriate Project Manager and the client notified. The NCM will be included in the final report.

For samples under the BP LaMP program, samples will not be left out on the login counters for more than 20 minutes. If additional time is required to log the samples into TALs, the samples will be placed in the cooler to ensure that the sample temperatures are maintained at $\leq$ 6 °C. The Sample Receipt Checklist in TALs will be used to document that this criteria has been followed.

After samples for volatile method 5035 preserved with water are logged into TALs, they are placed in the MS VOA Sample freezer in the MS VOA laboratory by login personnel. The date and time the samples are placed in the freezer will be documented in the comment section of the prep batch in TALs.

Note: MS VOA samples that have a strong odor or are known to be hazardous will be stored in Cooler #8. A MS VOA Storage Blank will be created and stored with the samples. The storage blank will be analyzed at the same time as the samples.

Cooler No.	Contents
1	All workshare samples received from other TestAmerica Labs. Consecutive sequence of samples, based on unique lab ID# (i.e. San Francisco is 720-xxxxx, Savannah is 680-xxxxx). Special Contract Archived Samples Shelves are identified by lab name/number.
2 (ICOC)	Consecutive sequence of samples requiring Internal Chain of Custody (ICOC) (i.e. Special Project samples requiring ICOC are designated with the "Force ICOC" checked by the PM in the project setup). The sample custodians initiate the transfer of the samples to the storage location via the TALs ICOC module. This cooler is kept locked at all times. When samples are needed from this Cooler, the analysts obtain the samples directly from the locked cooler; then transport the samples to their lab. The analysts then electronically sign out the samples via the TALs ICOC module (Attachment 4). Upon completion of the analysis, the analysts electronically sign in the samples via the TALs ICOC module. 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).
3	Soils and Wastesludges only
4	Organic extraction water 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 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, TOX and Phenol. (ICOC N/A) Inorganic TCLP extracts MS VOA samples with a strong odor or those that are known to be hazardous
MS VOA Freezer	Samples for method 5035 preserved with water.

6.0 SAMPLE LOGIN

Within LIMS, the PMs create a Project that has the pre-selected test methods. The sample custodians pull up the project file in LIMS and use the information to assign to the sample numbers within the Job (set of project samples). A LIMS Training Brief is presented in Attachment 5. The LIMS Training Brief does not replace individualized training and is only provided as an example of the entire log-in process. The LIMS continues to be updated as new features are added and others revised.

7.0 SAMPLE TRACKING

7.1 All samples will remain in the appropriate coolers prior to and after analysis.

7.2 Internal Chain of Custody (ICOC) samples are placed in appropriately secured coolers and must be signed-out by the analyst when the samples are removed from the storage area. To document this, the analyst must electronically sign out each sample in TALs LIMs using the ICOC module each time the samples are removed from storage. When the samples are returned to the appropriate cooler, the analyst must electronically document that the samples have been returned to secure storage by using the ICOC module.

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 and documented on an NCM by the appropriate personnel.

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 TestAmerica Chicago's practice is to log subcontract work using a subbed job number in the LIMS. 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.

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 TestAmerica 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 TestAmerica, 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.

Note: Samples that require radiological analysis are **NOT** to be shipped on ice.

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 in an NCM and inform the PM of the incident.

The waste sample disposal is handled by waste management department (SOP UP-WM-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 LIMS.
- 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 UP-WM-001)
- All hazardous samples will be disposed of commercially or returned to the client.

10.0 SAMPLE BACKLOGS

1. All LIMS 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 LIMS paperwork are maintained in the Job's file folder that is maintained in the data management department. All COCs are scanned into TALs into the job's COC folder, making it readily available by all staff.

3. Every morning, the Project Manager reviews all of their LIMS 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: TestAmerica Chicago's Chain-of-Custody
Attachment 3: Sample Handling Guide (i.e., Hold Times; Preservation)
Attachment 4: Example TALS ICOC Sample History record
Attachment 5: LIMS Training Brief
Attachment 6: LIMS Sample Receipt Checklist
Attachment 7: Non-Conformance Memo (NCM)
Attachment 8: Internal Cooler Temperature Control Log
Attachment 9: TestAmerica Chicago Sample Acceptance Policy
Attachment 10: Flowchart for Internal Chain of Custody Procedures
Attachment 11: Sample Login: Cooler Receipt Temperature Recording Procedure
Attachment 12: Sample Receipt Cooler Temperature Tracking Log
Attachment 13 Preservative Lot Documentation for Sample Receipt

12.0 REVISION HISTORY

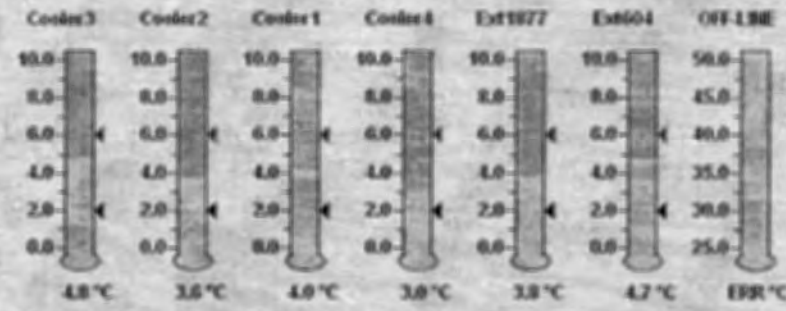
- Revision 28, was updated on 03/26/15
- Annual Review
- Section 4.0 was updated to clarify after hour sample receipt practices and removing the reference to the Shipment Receipt Custody Record.
- Section 7.1 was updated to remove the wet/dry board reference.
- Section 7.2 was updated to add details to the ICOC module text.

Attachment 1.

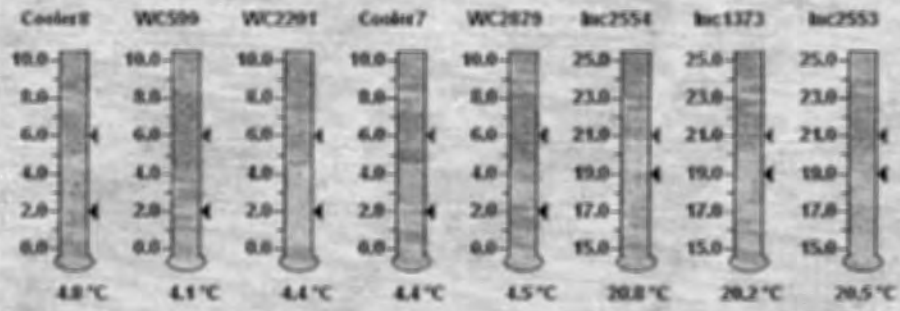
Example: Electronic Temperature Monitoring Diagram / Spreadsheet (012-001)

Temperature Monitoring System

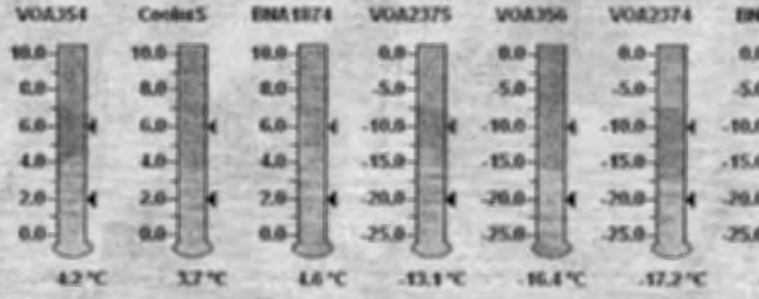
Area 1 - 2 - 3



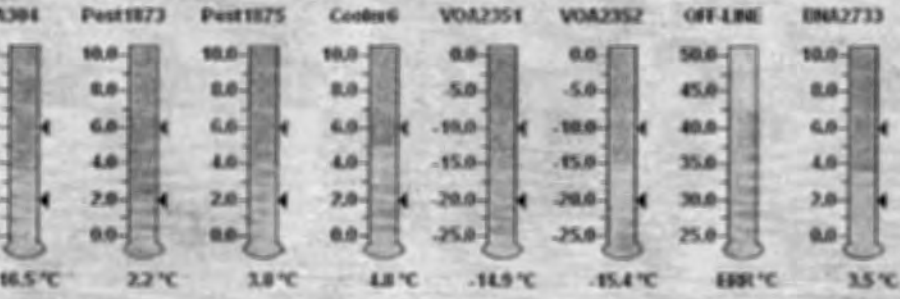
Area 1 - 2 - 3



Area 5



Area 5



Message Alert Window

3/31/2015, 5:16:41 AM, Pest219 temperature is 1
3/31/2015, 5:27:01 AM, Pest219 temperature is 1
3/31/2015, 5:37:21 AM, BNA304 temperature is -20
3/31/2015, 5:37:21 AM, Pest219 temperature is 1
3/31/2015, 5:47:41 AM, BNA304 temperature is -20
3/31/2015, 5:47:41 AM, Pest219 temperature is 1
3/31/2015, 6:08:21 AM, WC2879 temperature is E

E-Mail Notification

ON
Disabled

3/31/2015 7:01:02 AM

v.DGH-35

(100-210)

Attachment 2.

TestAmerica Chicago Chain-of-Custody
(013-001)

TestAmerica

THE LEADER IN ENVIRONMENTAL TESTING

2417 Bond Street, University Park, IL 60484
Phone: 708.534.5200 Fax: 708.534.5211

Chain of Custody Record

(optional)

Report To
Contact:
Company:
Address:
Address:
Phone:
Fax:
E-Mail:

(optional)

Bill To
Contact:
Company:
Address:
Address:
Phone:
Fax:
PO#/Reference#

Lab Job #:
Chain of Custody Number:
Page ____ of ____
Temperature °C of Cooler:

Client	Client Project #	Preservative
Project Name		Parameter
Project Location/State	Lab Project #	
Sampler	Lab PM	

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. NaHSO4
7. Cool to 4°
8. None
9. Other

Lab ID	MS/MSD	Sample ID	Sampling Date	Sampling Time	# of Containers	Matrix									Comments

Turnaround Time Required (Business Days)
___ 1 Day ___ 2 Days ___ 5 Days ___ 7 Days ___ 10 Days ___ 15 Days _____ Other
Requested Due Date _____

Sample Disposal
☐ Return to Client ☐ Disposal by Lab ☐ Archive for _____ Months (A fee may be assessed if samples are retained longer than 1 month)

Relinquished By	Company	Date	Time	Received By	Company	Date	Time
Relinquished By	Company	Date	Time	Received By	Company	Date	Time
Relinquished By	Company	Date	Time	Received By	Company	Date	Time

Lab Courier
Shipped
Hand Delivered

Matrix Key

WW – Wastewater	SE – Sediment
W – Water	SO – Soil
S – Soil	L – Leachate
SL – Sludge	WI – Wipe
MS – Miscellaneous	DW – Drinking Water
OL – Oil	O – Other
A – Air	

Client Comments

Lab Comments:

TAL-4124-500 (1209)

(100-310)

Attachment 3.

Sample Handling Guide (i.e., Hold Times; Preservation)
(014-001 to 014-002)

SAMPLE HANDLING GUIDE
Inorganic and Conventional Parameters

TestAmerica
THE LEADER IN ENVIRONMENTAL TESTING

Parameters	EPA Method*	Container	Rec. Quantity (mL)	Min. Quantity (mL)	Min. Quantity (g)	Preservative (Water)	Holding Time (Water)
Alkalinity	SM 2320B-97	P	250	50	1	≤ 6° C	14 days
Ammonia	SM4500 NH3 B-97 (prep); E350.1; SM4500-NH3G-97	P	250	100	1	≤ 6° C , H_2SO_4 to pH <2	28 days
Biochemical Oxygen Demand (BOD)	SM5210B-01	P	1000	370	5	≤ 6° C	48 hours
Bromide	300.0, 9056A	P	250	25	10	≤ 6° C	28 days
Chemical Oxygen Demand (COD)	SM5220C-97	P	100	10	1	≤ 6° C ; H_2SO_4 to pH <2	28 days
Chloride	300.0, 9056A, 9251, SM4500 Cl^-E-97	P	200	25	10	≤ 6° C	28 days
Chlorine, Residual	SM4500 Cl F-00	P	200	100	NA	≤ 6° C	Immediately
Chromium VI	7196A; SM3500CrB-09 (W) 7196A/3060A (S) 218.6; 7199 (W)*	P	250	100	2.5	≤ 6° C	24 hours (W) 30 days extr. (S) 7 days analyze *28 days w/pres.
Cyanide *	9010B/9014; 9010C/9014; SM4500 CN-E-99	P	100	50	1	≤ 6° C, NaOH to pH > 12, *Field test for Chlorine Residual & Sulfide: (Pos. hit req. Add'l Preserv.)	14 days
Ferrous Iron	SM3500_Fe_B-97	P	250	50	10	≤ 6° C	48 hours
Fluoride	300.0, 9056A, SM4500 F^-C-97	P	100	100	10	≤ 6° C	28 days
Hardness	SM 2340B-97	P	100	25	1	≤ 6° C; HNO_3 to pH < 2	6 months
Metals	6010B, 6010C, (W/S) 6020A, 200.7; 200.8 (W)	P	50	25	1	≤ 6° C; HNO_3 to pH < 2	6 months
Mercury	245.1, 7470A (W) 7471B (S)	P	25	25	1	≤ 6° C; HNO_3 to pH < 2	28 days
Nitrogen, Kjeldahl (TKN)	4500N_{ORG}C-97 (prep) and 4500NH3 G-97; E351.1	P	200	100	1	≤ 6° C, H_2SO_4 to pH < 2	28 days
Nitrate	300.0, 9056A, 353.2 rev 2.0; (SM4500 NO_3F-00 minus SM4500 NO_2B-00); Systea Easy (1-Reagent)	P	200	25	10	≤ 6° C	48 hours
Nitrite	300.0, 9056A, SM4500 NO_2 B-00	P	100	100	10	≤ 6° C	48 hours
Nitrate + Nitrite	353.2, SM4500 NO_3 F-00; Systea Easy (1-Reagent)	P	100	25	10	≤ 6° C , H_2SO_4 to pH < 2	28 days
Oil and Grease	1664A, 1664B (W) 9071B (S)	G	1000	1000	10	≤ 6° C, H_2SO_4 to pH < 2	28 days
Phenols	420.4, 9066	G	500	100	1	≤ 6° C , H_2SO_4 to pH < 2	28 days
Phosphorus, Total	SM4500P E-99	P	100	50	1	≤ 6° C , H_2SO_4 to pH < 2	28 days
Phosphate, Ortho	300.0, 9056A, SM4500 P E-99	P	100	50	10	≤ 6° C Filter within 15 min.	48 hours
pH	9040B, 9040C, 9041A (W) 9045C, 9045D (S) SM4500 H^+B-00 (W)	P	100	25	20	None	Immediately
Solids, Dissolved (TDS)	SM2540C-97	P	100	50	NA	≤ 6° C	7 days
Solids, Suspended (TSS)	SM2540D-07	P	500	200	NA	≤ 6° C	7 days
Solids, Volatile (TVS)	160.4, SM2540E-97	P	100	50	NA	≤ 6° C	7 days
Solids, Total (TS)	SM2540B-97	P	100	50	NA	≤ 6° C	7 days
Solids, Settleable	SM2540F-97	P	1000	1000	NA	≤ 6° C	48 Hours
Specific Conductance	120.1, 9050A, SM2510B-97	P	100	50	NA	≤ 6° C	28 days

(014-001)

SAMPLE HANDLING GUIDE
Inorganic and Conventional Parameters

TestAmerica
THE LEADER IN ENVIRONMENTAL TESTING

Inorganic Parameters	EPA Method*	Container	Rec. Quantity (mL)	Minimum Quantity (ml)	Minimum Quantity (g)	Preservative (Water)	Holding Time (Water)
Sulfate	300.0, 9056A (W/S) 9038, SM4500SO_4^{2} E-97 (W)	P	200	100	10	≤ 6° C	28 days
Sulfide	9030B/9034 (S) 9034; SM4500S^{2-} F-00 (W)	P	500	250	10	≤ 6° C , Zn acetate, NaOH to pH > 9; Zero Headspace	7 days
Thiocyanate	SM4500 CN-M	P	100	50	NA	≤ 6° C, H_2SO_4 or HNO_3 to pH < 2	14 days
Total Organic Carbon (TOC)	9060A, SM5310C-00 (W) Lloyd Kahn (S)	G-TLS	2 x 40	1 x 40	1	≤ 6° C, H_2SO_4 to pH < 2	28 days (W) 14 days (S)
Total Organic Halides (TOX)	9020B; SM5320B (W)	G-TLC (amber)	200	200	NA	≤ 6° C, H_2SO_4 to pH < 2 Zero Headspace	28 days
Total Petroleum Hydrocarbon (TPH)	1664A, 1664B (W) 9071B (S)	G-TLC (amber)	1000	1000	10	≤ 6° C , H_2SO_4 to pH < 2	28 days
Turbidity	180.1	P	100	100	NA	≤ 6° C	48 hours
Flaspoint / Ignitability	1010A; D93 (Closed Cup) D92 (Open Cup)		Vol. to fill 70 mL vessel	200	4 oz.	None	

Organic Parameters	EPA Method*	Container	Rec. Quantity (mL)	Min. Quantity (g)	Preservative (Water)	Holding Time (Water)
Diesel Range Organics (DRO)	8015B; 8015C	G-TLC (amber)	2 x 1000 2 x 250 (LVI)	15	≤ 6° C	7 days
Diesel Range Organics (DRO)	WI DRO (pre-weighed bottles)	G-TLC (amber)	2 x 1000 2 x 250 (LVI)	25	HCl to pH < 2	7 days
Gasoline Range Organics (GRO)	8015B; 8015C; WI GRO	G-TLS	3 x 40	State Specific	≤ 6° C, HCl to pH < 2	14 days
Volatile Organics	624, 8260B	G-TLS	3 x 40	State Specific	≤ 6° C , HCl to pH < 2 Zero Headspace	14 days 7 days (if not preserved)
Pesticides (Organochlorine) and PCBs	608, 8081A, 8081B, 8082, 8082A	G-TLC (amber)	2 x 1000 2 x 125 (LVI)	15	≤ 6° C , pH 5-8	7 days to extract 40 days to analyze
Chlorinated Herbicides	8151A	G-TLC (amber)	2 x 1000	30	≤ 6° C	7 days to extract 40 days to analyze
Semivolatile Organics (BNA), Polynuclear Aromatics	625, 610, 8270C, 8270D, 8310,	G-TLC (amber)	2 x 1000 2 x 250 (LVI)	15	≤ 6° C	7 days to extract 40 days to analyze

TCLP 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)	Minimum Sample Size (g)	Total Elapsed Time (days)
Volatiles	14	Not Applicable	14	25	28
Semivolatiles	14	7	40	100	61
Mercury	28	Not Applicable	28	100	56
Metals	180	Not Applicable	180	100	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 (20th 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 ≤ 6° 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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(014-002)

CHI-22-09-368/I-10/13

Attachment 4.

Example: TALs ICOC Sample History Record (015-001)

Historical Internal Chain of Custody

Login	Smp	Customer Sample ID	Matrix	Container ID	Lab Sample ID	Container Type	Location	Custody User	I/O	ICOC ID	ICOC Date
500-93845	1	CFVP2184L	Water	500-3196310	500-93845-A-1	Plastic 1 liter - unpreserved					
500-93845	1	CFVP2184L	Water	500-3196311	500-93845-B-1	Amber Glass 1 liter Wide -	Disposed	Yushinski, Charles	I	180-193883	03/28/15 11:16
500-93845	1	CFVP2184L	Water	500-3196311	500-93845-B-1	Amber Glass 1 liter Wide -	PEST/PCB	Lonzo, Michael A	I	180-193878	03/28/15 10:09
500-93845	1	CFVP2184L	Water	500-3196311	500-93845-B-1	Amber Glass 1 liter Wide -	Shared Samples In-Transi	Kelsey, Shawn M	O	500-60060	03/27/15 14:29
500-93845	1	CFVP2184L	Water	500-3196312	500-93845-C-1	Amber Glass 1 liter Wide -	26EE	Lonzo, Michael A	I	180-193879	03/28/15 10:10
500-93845	1	CFVP2184L	Water	500-3196312	500-93845-C-1	Amber Glass 1 liter Wide -	Shared Samples In-Transi	Kelsey, Shawn M	O	500-60060	03/27/15 14:29
500-93845	2	SV27769L	Water	500-3196313	500-93845-A-2	Plastic 1 liter - unpreserved					
500-93845	2	SV27769L	Water	500-3196314	500-93845-B-2	Amber Glass 1 liter Wide -	Disposed	Yushinski, Charles	I	180-193883	03/28/15 11:16
500-93845	2	SV27769L	Water	500-3196314	500-93845-B-2	Amber Glass 1 liter Wide -	26EE	Lonzo, Michael A	I	180-193879	03/28/15 10:10
500-93845	2	SV27769L	Water	500-3196314	500-93845-B-2	Amber Glass 1 liter Wide -	Shared Samples In-Transi	Kelsey, Shawn M	O	500-60060	03/27/15 14:29
500-93845	2	SV27769L	Water	500-3196315	500-93845-C-2	Amber Glass 1 liter Wide -	PEST/PCB	Lonzo, Michael A	I	180-193878	03/28/15 10:09
500-93845	2	SV27769L	Water	500-3196315	500-93845-C-2	Amber Glass 1 liter Wide -	Shared Samples In-Transi	Kelsey, Shawn M	O	500-60060	03/27/15 14:29

(015-001)

Attachment 5.

LIMS Training Brief

LOGIN

(For Project Managers, Project Manager Assistants, Client Service Managers, and Lab Managers)

Login

The Login application is divided into three different modules – Receipt, Login, and Job. Each of these modules performs a distinct function in the login process. The Receipt module allows the lab to receive samples, print sample container labels and note the condition of the samples and coolers upon receipt. If holding times or TAT are an issue, once the sample containers are labeled, the samples can be distributed to the lab without having to immediately complete the login process in TALS. The act of receiving the samples in TALS starts the login process.

In the Login module, sample information (client ids, sample matrix, sampling date/time) are entered for each of the received samples. A project that contains the client's requirements (created earlier by the project manager for that client) will be used to log the samples in. The project contains the method chains, project limits and reporting requirements for the samples. In the login module, methods are selected from the project in accordance with the chain of custody for each sample. By using the project as the client's template for login, the sample receiving group can log in jobs more efficiently. It is important to note that even though a job is logged in and samples may have been distributed to the lab for work, the data will not calculate until the login has been reviewed and approved by the project manager.

The last module in the Login application is the Job module. Each job is equivalent to a login when the login is first created. But a login can then be split into several jobs (each job with a separate set of reporting requirements) or several logins can be joined to create one job (a sample delivery group). Each job contains a separate set of deliverables, client contacts, pricing and turnaround times. The project manager for the project will review the login as a whole and the pricing and deliverables for each job separately.

In this User Guide, the user will learn how:

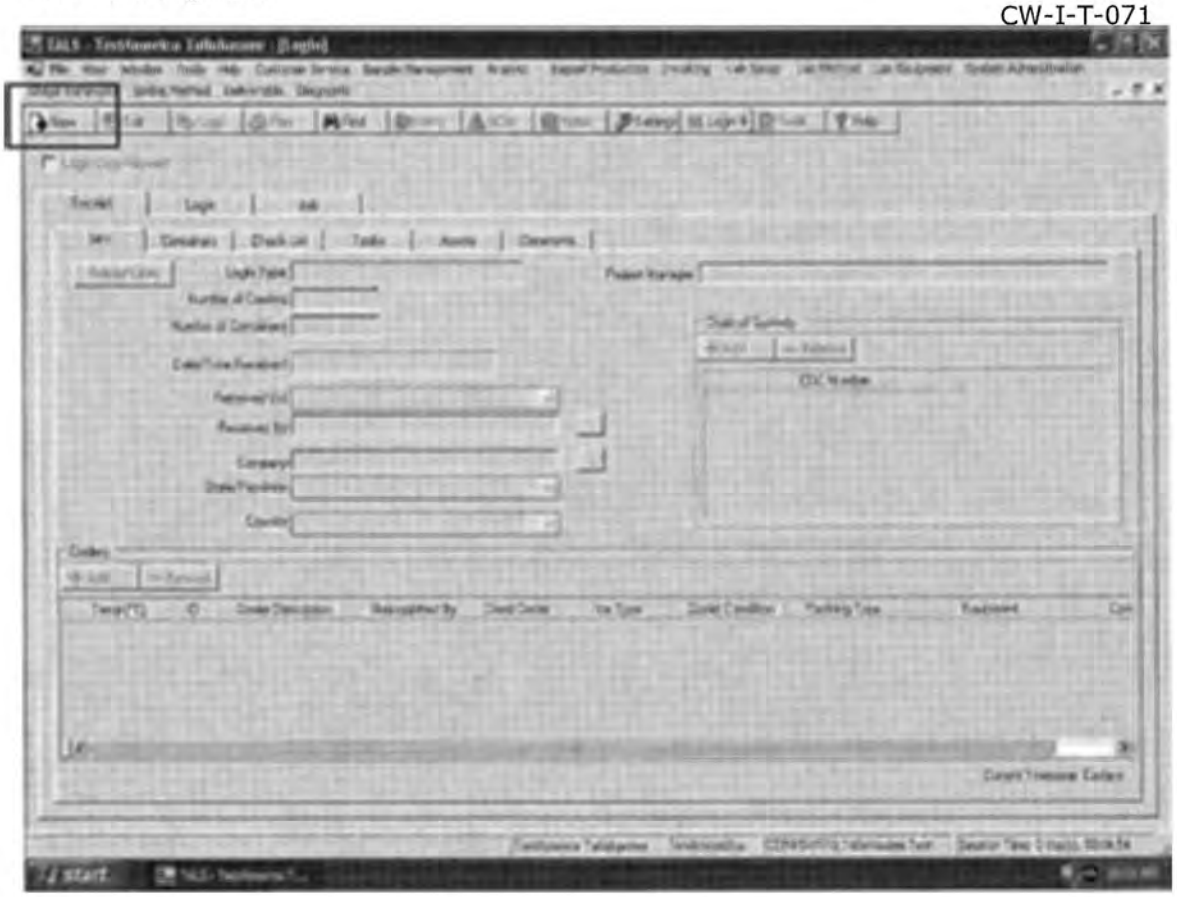
- To Receive Samples
- To Log-in Samples
- To Work with Jobs

To Start:

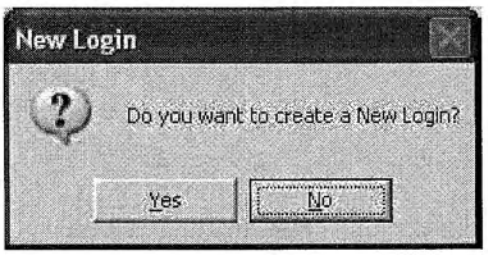
1. From the main menu, select *Sample Management* by clicking the application.
2. From the *Sample Management* menu, select *Login* by clicking the application.

To Receive Samples

1. Click the [**New**] button on the toolbar located at the top of the screen.



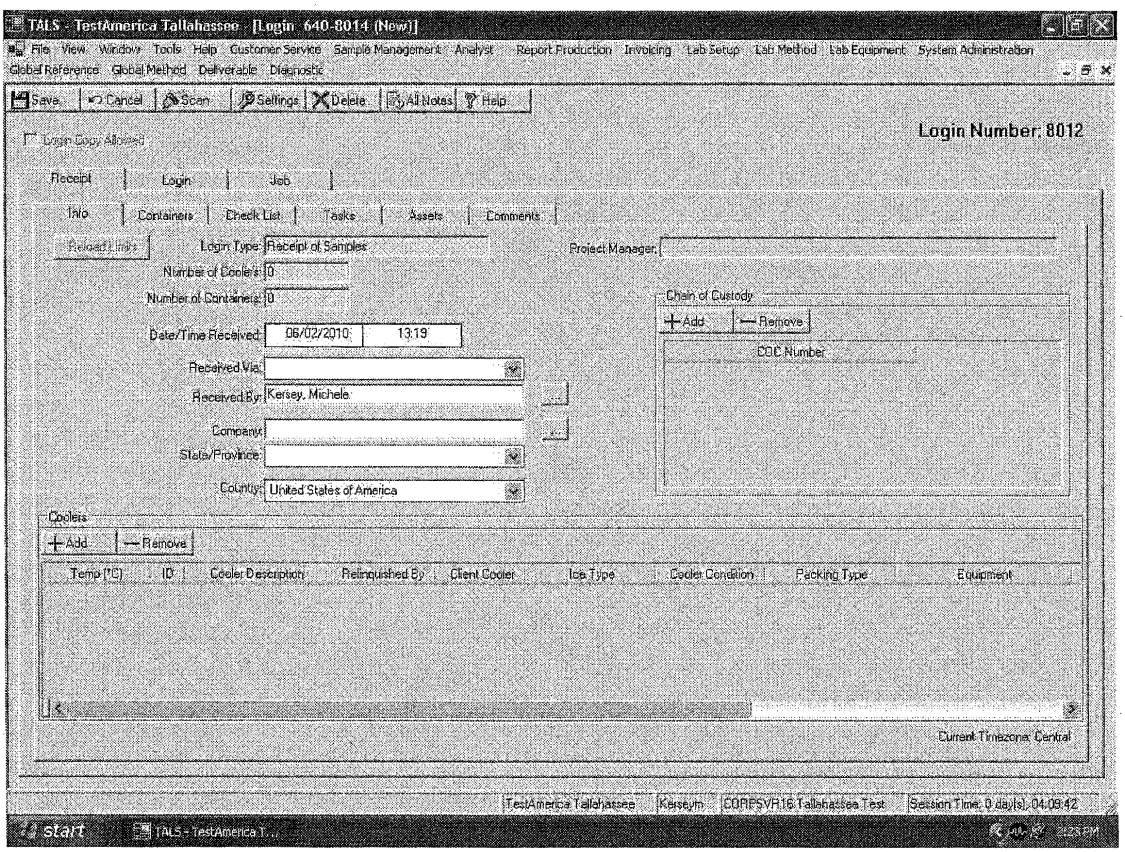
2. Click [**Yes**] to create a new Login.



Receipt - Info

When samples arrive at the laboratory, they must first be received in TALS. By keeping these modules separate, the sample receiving group can start the login, receive the samples and print the labels for the sample (s). Rush samples can then be moved directly into their respective areas to begin work before the samples are completely logged in. Until the sample login is complete, the samples will not relate to the login. All samples MUST be received before the login can be completed.

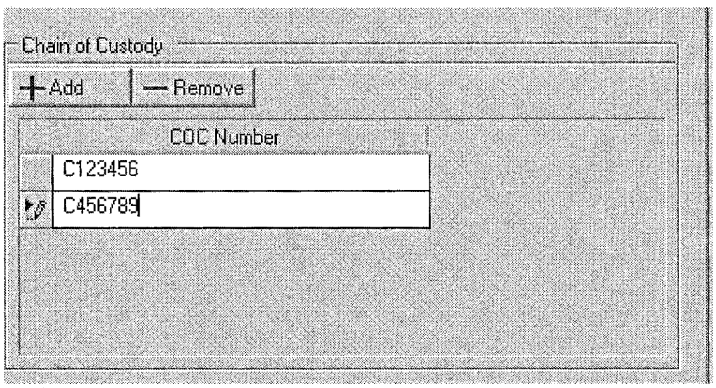
1. Grey shaded fields are non-editable. In each field below, enter the following:



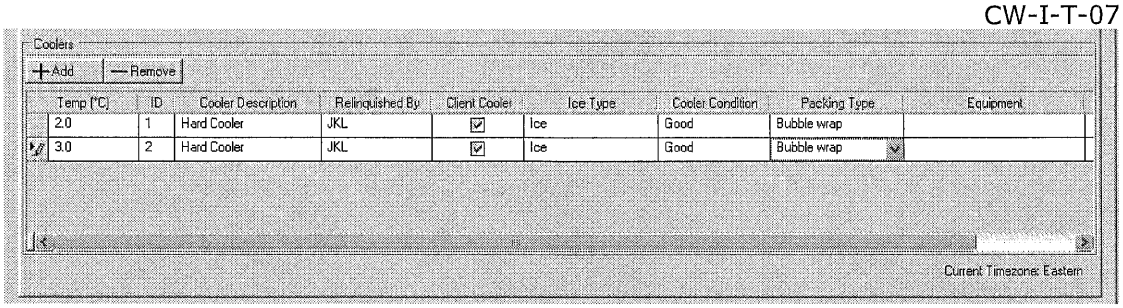
- **Login Type** – defaults to Receipt of Samples when a new login is chosen.
- **Project Manager** – automatically fills in when the project number is selected on the ***LOGIN*** tab.
- **Number of Coolers** – automatically filled in with the information supplied under the 'Coolers' grid at the bottom of the screen.

- **Number of Containers** – automatically filled in with the information supplied in the ***CONTAINERS*** tab.
- **Date/Time Received** – defaults to the time the login was created. To change the date/time to the actual receipt date/time noted on the COC, use the drop down/up arrows in each field.
- **Received Via** – mode of delivery of the samples (courier, client pickup, etc) selectable from dropdown menu.
- **Received By** – the individual that received the samples and signed the chain of custody. Defaults to the current user logged onto TALS. Change by clicking [...].
- **Company** – the client that sent the samples. This is an optional field. Select a client by clicking [...].
  - *Note: If a company is chosen in this field, the **PROJECT LOOKUP** on the **LOGIN** tab will automatically be filtered to display active projects from only that company.*
- **State/Province** - enter the State where samples were collected. This is a required field. Select from the dropdown menu.
- **Country** – enter the country where samples were collected. This is a required field. Select from the dropdown menu. The default is United States of America.

2. Click [Add] to enter the Client's Chain of Custody numbers.
   - Note*: Entering one (1) chain if custody will automatically add the COC number to all containers. If more than one COC is entered, a COC must be selected manually for each container. This may be done on the **CONTAINERS** tab.*



3. Click [Add] to enter the cooler information.
   - *Note: Entering one (1) cooler will automatically add the cooler number to all containers. If more than one cooler is entered, a cooler must be selected manually for each container. This may be done on the **CONTAINERS** tab.*

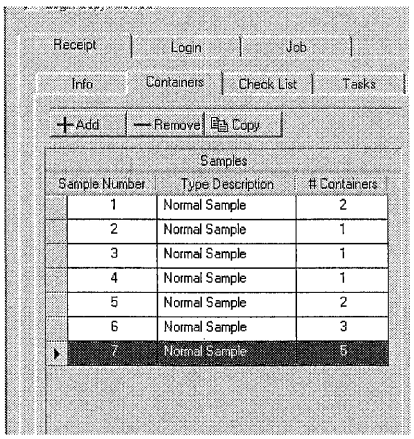


In the Cooler grid enter the following:

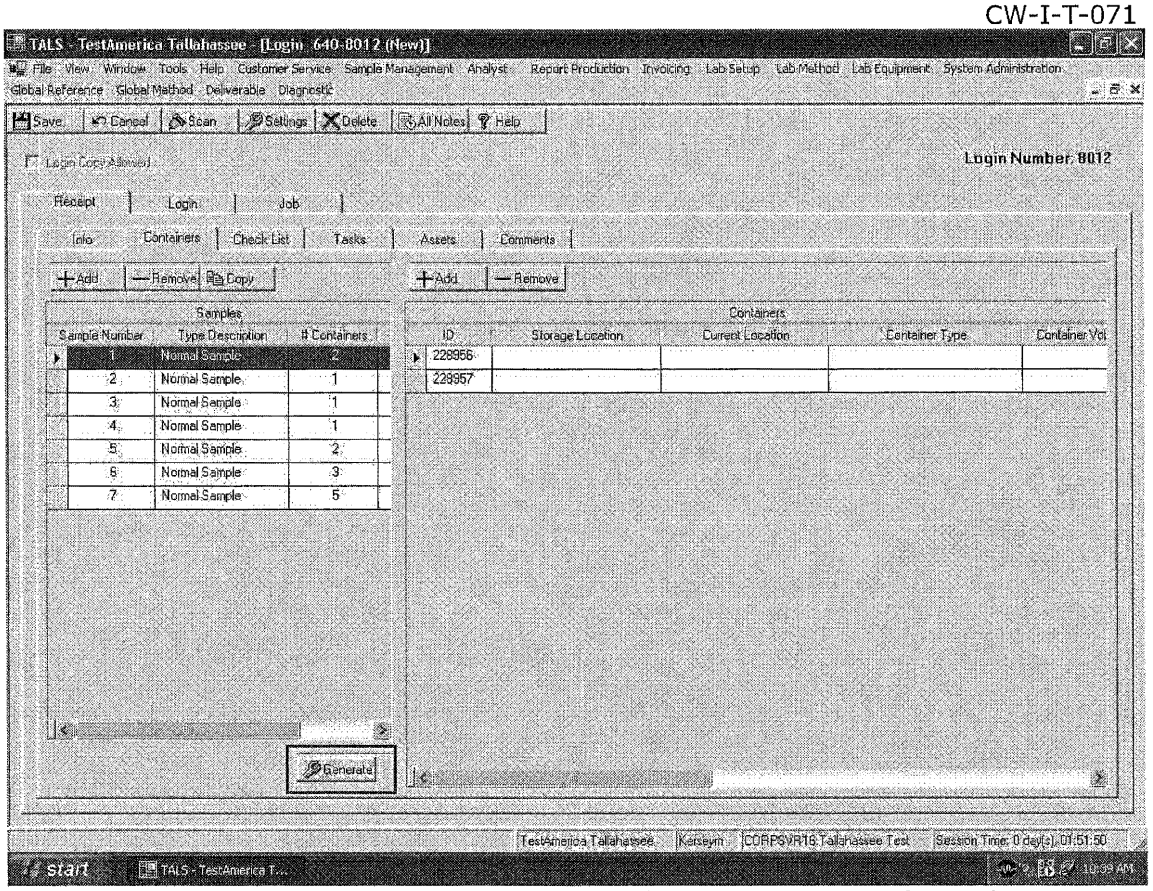
- **Temp (C)** - the internal temperature of the cooler upon receipt.
- **ID** – incremental number assigned to each cooler.
- **Cooler Description** –a description of the physical cooler, whether it is a thermal cooler, insulated box or other container.
- **Relinquished By** –the individual that signed the Chain of Custody and relinquished the custody of the samples to the laboratory sample custodian.
- **Client Cooler** – Check this box if this is a client provided cooler.
- **Ice Type** – type of ice that the samples were packed in (drop down list).
- **Cooler Condition** – the physical condition of the cooler upon arrival.
- **Packing Type** – the type of cushioning material used to keep the samples from breaking in transport (drop down list).
- **Equipment** – any equipment (sampling equipment, balances, palm pilots, etc) packaged with the samples.
- **Comments** – free form text field to enter any additional information about the cooler.

Receipt - Containers

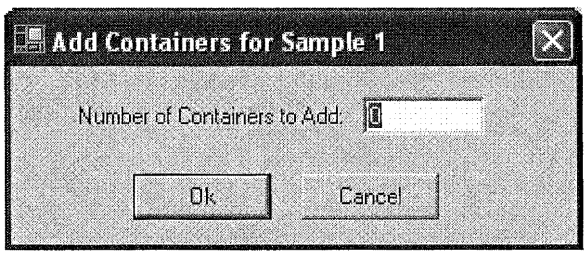
1. Click [Add] to enter the total number of samples received in the ***SAMPLES*** tab of the Login.
2. Enter the total number of containers received per sample in the space provided in the **#Containers** field.



3. Click **[Generate]** to create the container records in the CONTAINERS section of the Login automatically.



NOTE: Containers can also be added manually for each sample by selecting the sample in the Samples grid and then clicking the [Add] button on the Containers grid. The box below will be displayed to enter the number of containers desired for the sample.



4. After generating the CONTAINER records, fill in the following information in each of the fields in the container grid (fields are represented from left to right – scroll to the right to see the remaining fields)
   - **ID** – unique container ID assigned by TALS – no entry is allowed in this field.
   - **Storage Location** – storage locations are set up in the system defaults and populate the drop down list used in this field.
   - **Current Location** – where the container is currently being stored.
   - **Container Type** - use the drop-down arrow to select from a

valid values list of containers.

- **Container Volume** – auto-fill based on the selection in Container Type.
- **Container Units** – will auto-fill based on the selection in Container Type
- **Condition** – condition of container when received. Enter only exceptions to a condition of good.
- **COC** – use the drop down arrow to select available Chain of Custodies. If no COCs were entered on the ***RECEIPT INFO*** tab, this field will be blank. If there is only one COC, the field will be auto-filled.
- **Cooler** – select from the drop down list if more than one cooler was received or the field will be auto-filled if there is only one cooler. If no coolers were entered on the ***RECEIPT INFO*** tab, this field will be blank.
- **Assets** – pre-defined valid values in the *Assets* module.
- **Lab ID** – combination of the login location, the login number, the alpha occurrence of the container for that sample (A = 1, B = 2, etc.) and the sample number itself. The ID and Lab ID both indicate the same container and can be used interchangeably. No entry is allowed in this field.
- **TAG** – CLP sample containers may arrive with a tag. Enter the tag ID here.

Some shortcuts are available:

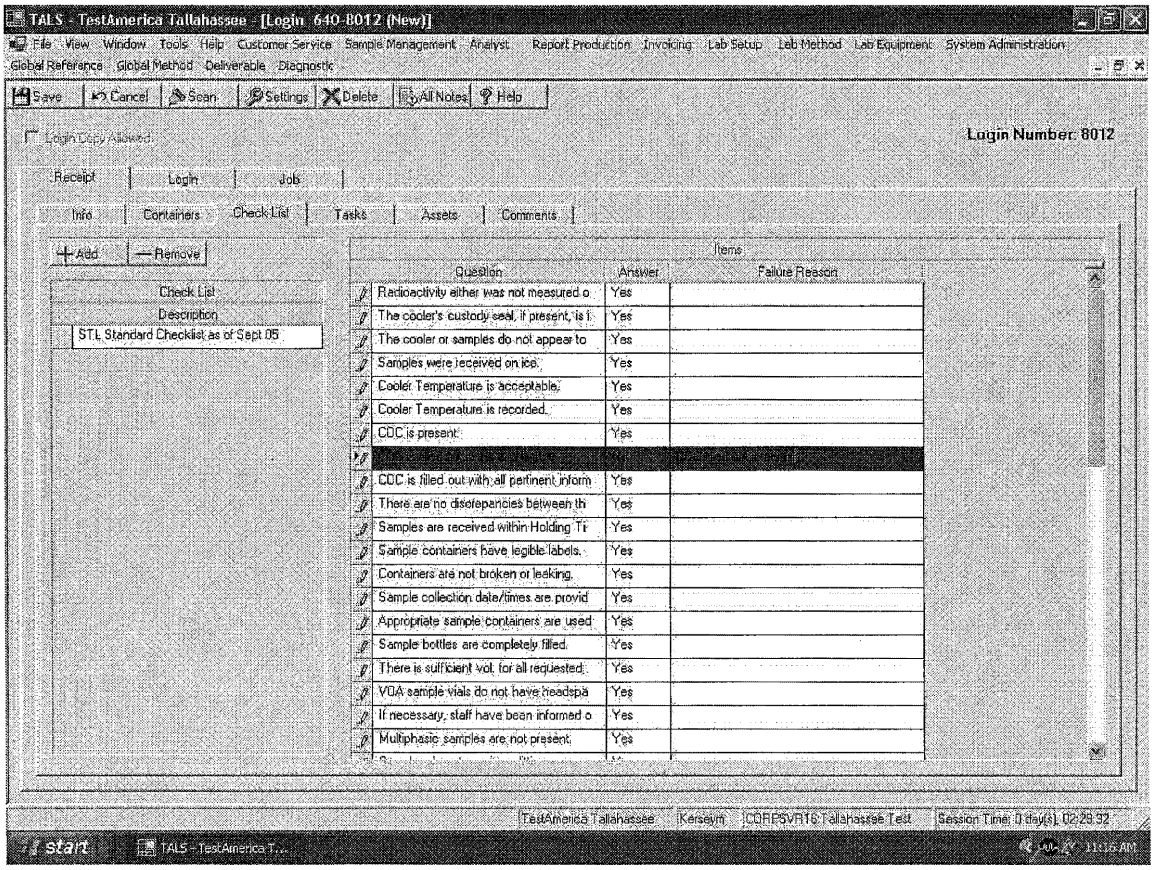
A) *Simultaneously pressing the **<CTRL>** key and the **<DOWN>** arrow will automatically fill in the field below the field the cursor is on (as long as the new field is blank).*

B) *Some fields allow filling in every row of a column automatically. Simply highlight the HEADER of a column (Storage Location for example). This turns the entire column BLUE. Right-Click in the blue and select a value from the valid values list. All fields in this column will now have the selected value.*

C) *Use the [**COPY**] button in the SAMPLES section. Simply fill out all of the information in the CONTAINERS section of the form for one sample. Click [**COPY**]. Choose the completed sample on the left-side of the form ("copy from") by clicking the left-most grey square (causing that row to be completely highlighted) and then choose the samples to "copy to" in the right-side of the form by clicking the left-most grey square of the row (causing that row to be completely highlighted). For more samples, simply hold the mouse button down when selecting the first sample and "drag" the highlight down the list of samples until all of the samples to copy are highlighted. Click [OK] and the container information will be copied to the new records.* **Note***: The copy function only works if the samples have the same number of containers.*

Receipt – Checklist

The Sample Receipt Checklist defines the condition of the samples when received.



1. Answer all questions.
2. A Failure Reason must be entered for all negative responses
3. If the question does not apply to this set of samples (i.e. a question pertaining to solid samples for a shipment of only waters) N/A may be used as a response. All 'No' answers will be highlighted in red.
4. To quickly enter the most common answer for all questions, highlight the HEADER of the ANSWER COLUMN. This will turn the entire column BLUE. Right-Click in the blue highlight and select an answer (Yes, No, N/A). All fields in this column will now have the selected response. Edit the answers that do not match the overall answer chosen.

Receipt – Tasks

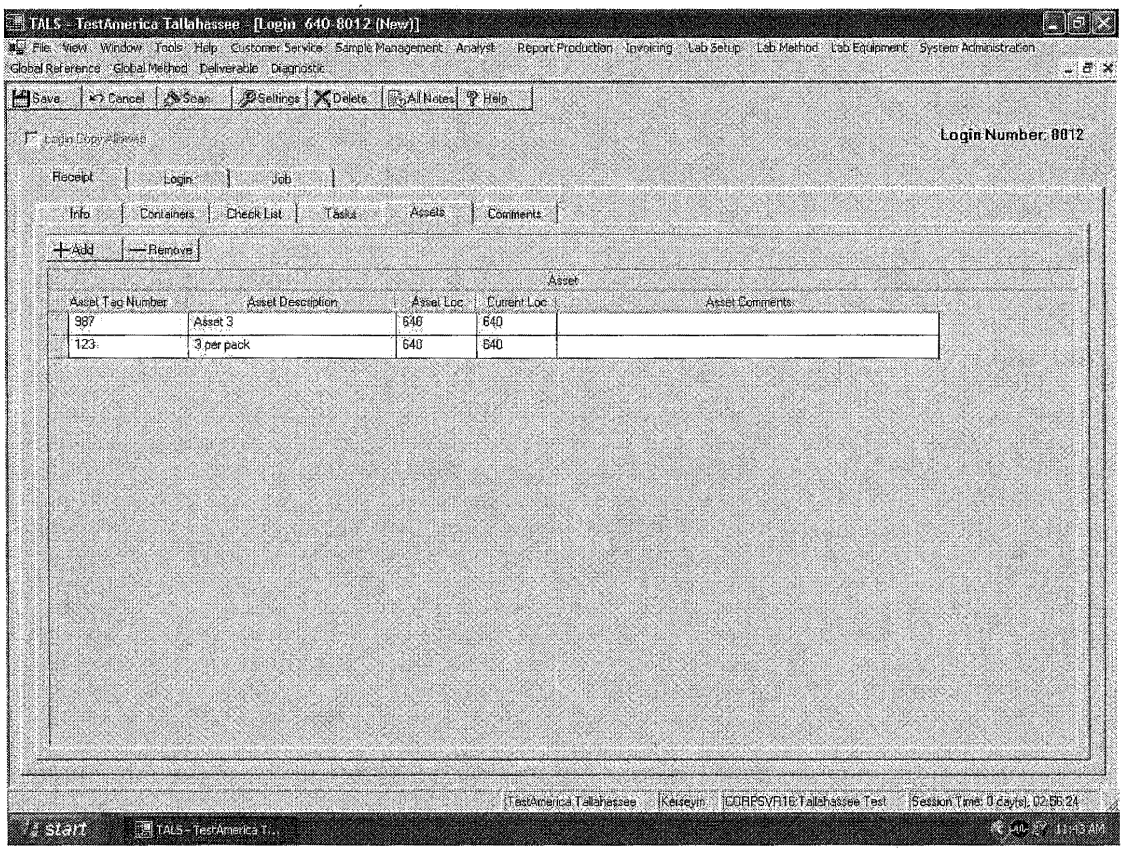
Tasks will be covered later in this document.

Receipt - Assets

The *Assets* tab is used to log company assets returned with a sample shipment. Assets are defined as anything a laboratory or service center will send out with a bottle order and expects to be returned to the lab. Tracking assets is optional but it is strongly suggested to track assets for any items that are either frequently never returned or exceedingly costly. The individual facility should use their judgment as to what items should be tracked as assets.

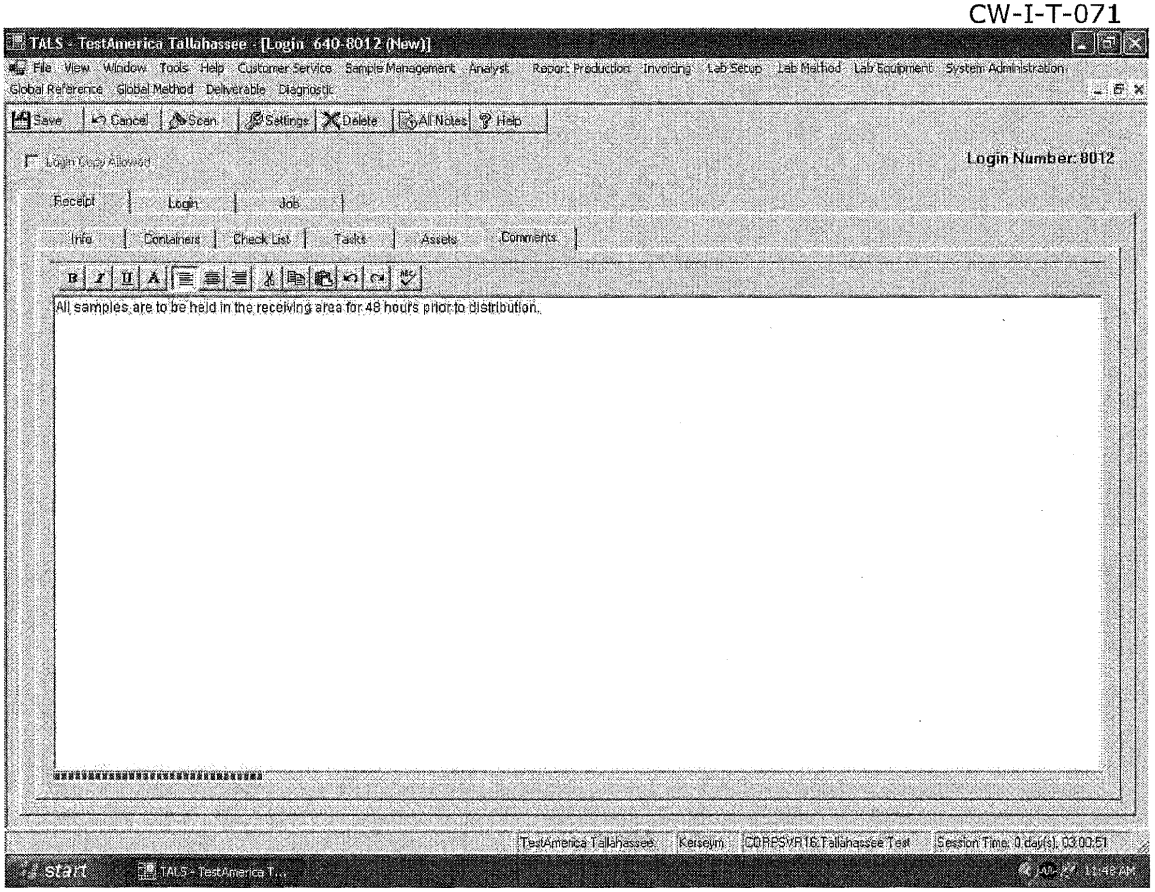
The basic premise for an asset is when an asset is included in a shipping order its asset number is stored with the shipping order. When shipped the item is marked as being out. Being associated with a shipping order, it can be determined when it was sent out and to whom. At the login receipt step, upon return to a facility, the asset can be signed back in whereby it will be marked as returned. The use of bar code scanners can be employed to speed the signing in and out of an asset.

Maintenance of a location's assets is found under *Sample Management*. See the *'Assets'* TALS training brief on the intranet for information on maintaining assets.



Receipt – Comments

Free-form area to enter any comments, notes, etc. These can appear on backlogs if desired.



Save

Click **[Save]** from the menu across the top of the screen to save the receipt information entered to this point.

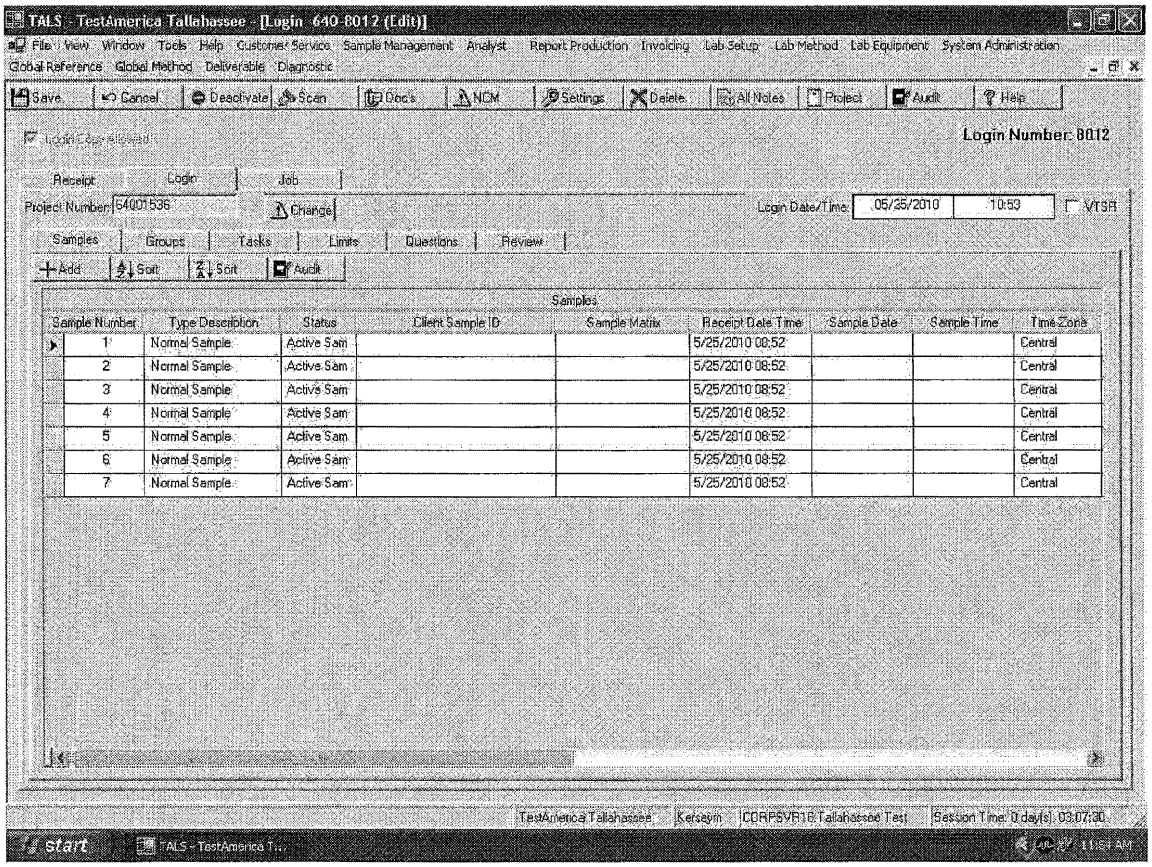
To Log-in Samples

1. Locate the Login by either clicking on the **[Find]** button (which will bring up a list of all the logins for this location or that have work for this location) or the [Login #] button where the login number can be entered directly. Either choice will load the login.
2. In the ***LOGIN*** tab, click the **[Add]** button next to the PROJECT NUMBER field. If a COMPANY was chosen on the ***RECEIPT*** tab, the list of projects will be filtered based on that company. Otherwise a complete list [of Active Projects] will be displayed. Filter the list based on the pop-up screen and select the appropriate project. Click **[OK].**

Note: If the wrong PROJECT is selected, click on the [Change] button and follow the instructions.

*Note: See **Sites and Events** documentation on the intranet for instructions for logging in samples that have Sites and Events assigned to the project.*

Login – Samples



1. Enter the information for each Client Sample. Some fields will be auto-filled based on information from the previous tabs and the Project.

Update the following information as needed.

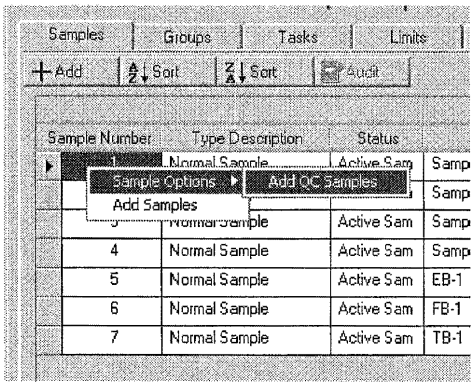
- **Sample Number**– assigned by the system as each sample is added. If samples are removed from the login, the sample number does not renumber.
- **Type Description** – default type will be normal sample (client sample). See how to add **Client Requested QC Samples** later in this user guide.
- **Status** – default is Active Sample. Select Hold or Inactive Sample from the dropdown list if applicable.
- **Client Sample ID** - Enter the Client Sample ID accurately, as it was entered on the COC. If the Project has "Sites and/or Events" associated with it, and the PM elected to verify against the site samples list, this field will NOT be free-form, but will be a drop-down list to select the appropriate Client Sample ID. See the *Sites and Events* documentation on the intranet for additional information on this feature.
- **Sample Matrix** - select the Sample Matrix from the drop-down list. This is the sample matrix that the lab will see on their backlogs and in Analyst Desktop. This is the matrix that is used in the reference data as well. If the client needs the matrix to be displayed on the report and EDD as something more specific such as 'ground water', use the alternate sample matrix field.

- **Receipt Date Time** – Date and Time samples were received at the laboratory. This field is copied from the date and time entered on the receipt tab.
- **Sample Date** – enter the sample date for each sample as it is entered on the COC. Click the down arrow to bring up a calendar to select the date.
- **Sample Time** – enter the sample time as entered on the COC. To enter the time in military time (24 hour clock), change the format by clicking on the [Settings] button at the top of the screen, then select 24 hour.
- **Time Zone** – change the time zone to the time zone where the samples were taken.
- **Hazard Level** – For laboratories with a Hazard Level Permit, enter the hazard level of the samples as received. The default will be Unconfirmed.
- **Alternate Sample Matrix** – as mentioned above, the client may require that a more descriptive sample matrix appear on their deliverables. Alternate Sample Matrices are added in the Site Sample module and are available as a drop down in the Login module.
- **Sample Types** – select the sample type from the drop-down list. (i.e. 'Field Blank, Trip Blank')
- **Action Limit Set** - If ONE Action Limit Set has been associated with this PROJECT, the list name will automatically fill in. If more than one exists, the appropriate list must be chosen from the drop-down choices. See Action Limit Sets documentation posted on the intranet for more information on this item.
- **Sample Comments** – enter any additional information about the sample that may need to be conveyed to the analytical group.
- **Login User** – copied from the **Receipt** tab.

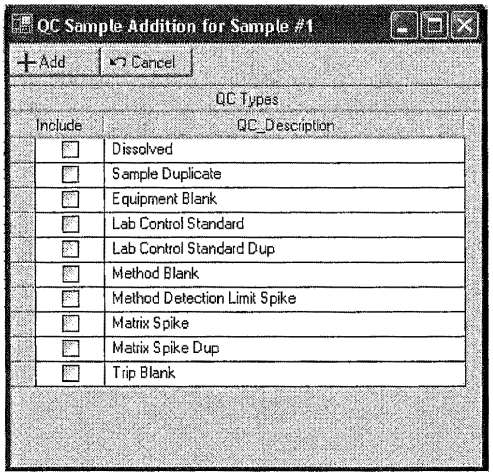
Client Requested QC Samples

If sample QC is received from the client and it is necessary to create this QC at login (MS/MSD/Duplicates, etc.) follow this procedure:

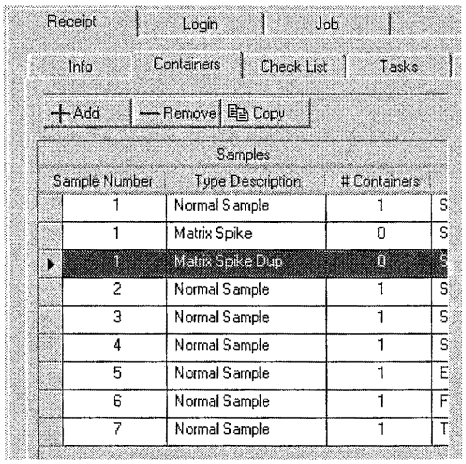
1. Select the sample by clicking on the sample Number or Type Description field.
2. Right Click to pull up the **Sample Options** menu and select **Add QC Samples.**



3. Choose the appropriate QC types from the popup box which will appear and click [**Add**].



4. Populate the appropriate information for the QC samples
5. Click back to the **RECIPT TAB – CONTAINERS SUBTAB** to add the appropriate information for the containers received specifically for the QC.



Login – Groups

The analyses associated with each sample are contained in LOGIN GROUPS. Sample Receiving will build these groups from the information contained in the PROJECT.

The groups can be created a variety of ways, ranging from easy to complex. The most straight-forward way is to organize your groups in relationship to the samples and the analyses requested on each sample.

For example:

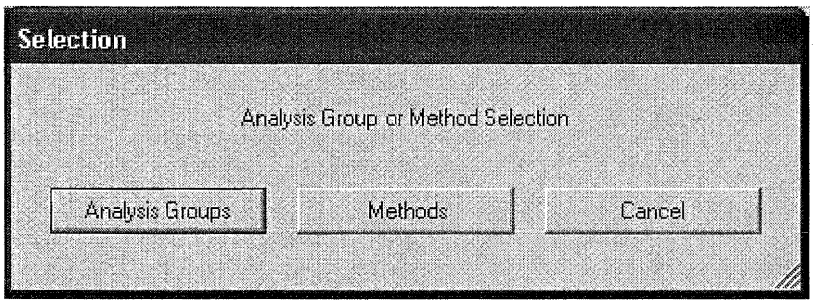
If sample #1 and #3 are both waters and are requesting GCMS VOA8260 Standard List and Lead … create a LOGIN GROUP with these two parameters and associate this group to these samples.

If sample #2 is only getting Lead … create another LOGIN GROUP that only contains Lead and associate this group to this sample.

In this example, you would have two login groups (with the correct analyses on each sample).

To CREATE A NEW LOGIN GROUP:

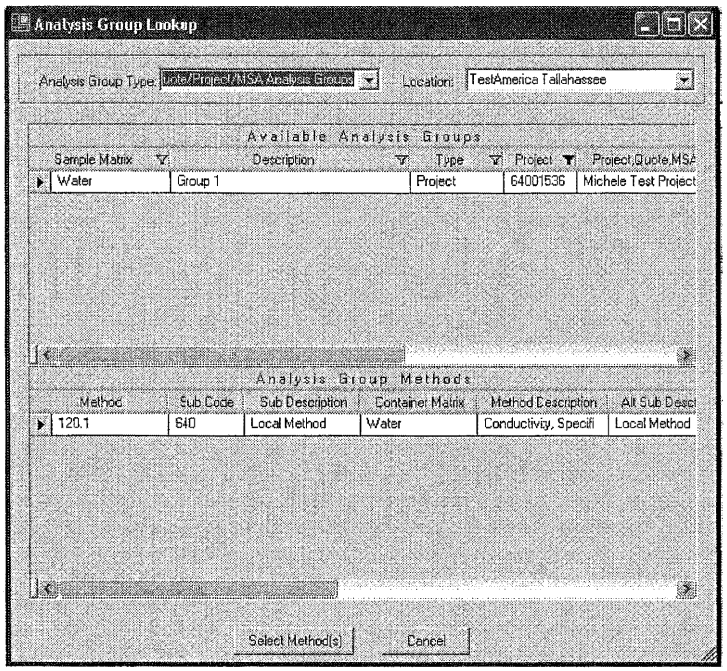
1. Click on the [**NEW**] Button.
2. From the pop-up menu choose from the following:



A. **[Analysis Groups]**

This shows all of the methods/groups the project manager has created in the PROJECT.

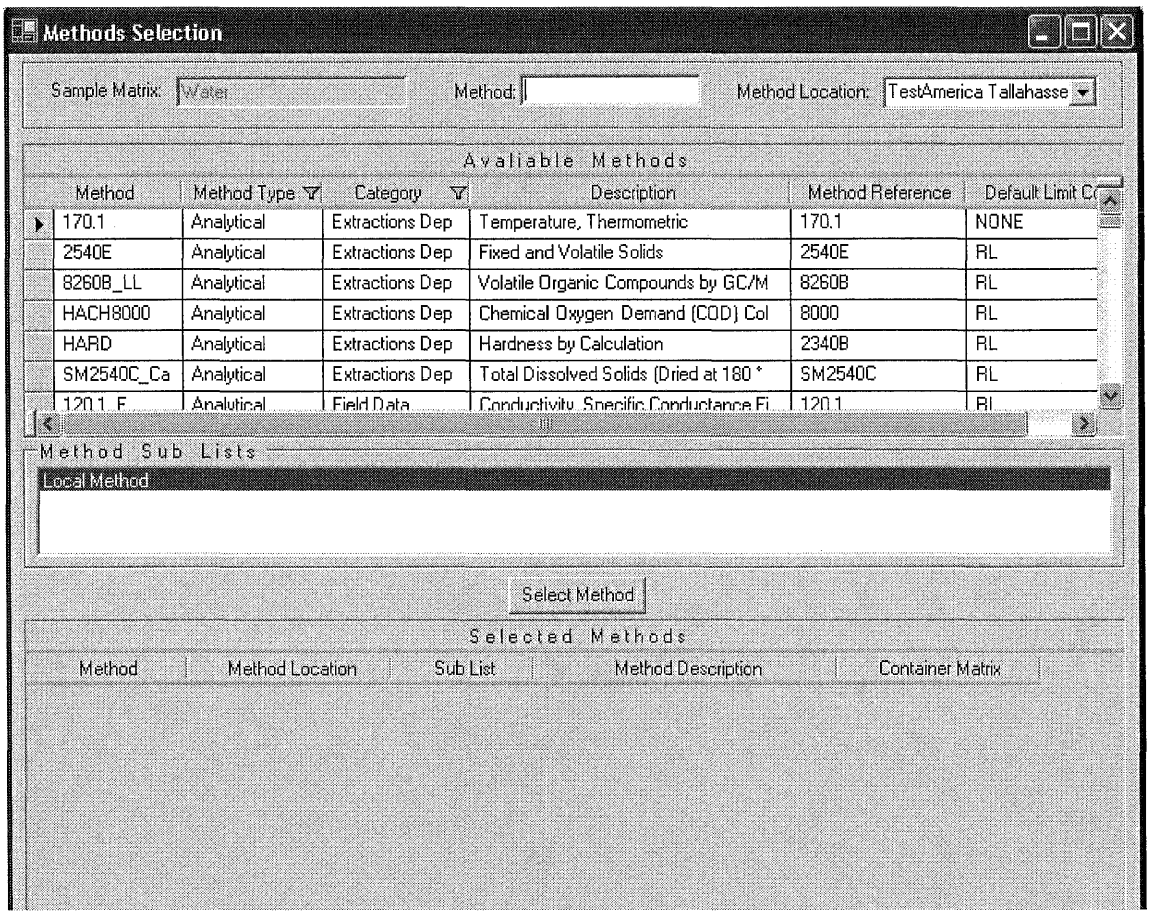
1. Paying attention to the Matrix and the Group Description, highlight the group that contains the methods of interest.
2. Once chosen, the associated METHODS will appear in the lower grid of this pop up.
3. Highlight the method(s) of choice and click on [Select Method]. A LOGIN GROUP with the chosen Methods and their corresponding preps is created.



A. **[METHODS]**

This shows all of the methods associated with the current lab location.

1. All available methods are shown.
2. Once a group is chosen, the associated METHODS will appear in the lower grid of this pop up.



A. Each LOGIN GROUP created may be viewed by highlighting the LOGIN GROUP in the "Login Groups" grid by clicking the left-most grey square and highlighting the entire row.
B. Once highlighted, the corresponding methods/preps will be displayed in the grid to the right.
C. The grid will be titled with the LOGIN GROUP NAME (which automatically comes from the PROJECT, but can be renamed by simply by typing over the title in the LOGIN GROUPS grid).
D. Columns in the LOGIN GROUP [DETAIL] GRID:
   - **Method Code** – the internal ID of the method
   - **Method Location** - the originating location of the method
   - **Method Description** – detailed description of the method
   - **Alt Sub-Desc** – this field is customized in the PROJECT to indicate to the lab if the list has been modified from its original state.
   - **Destination** – if method is being sent to another facility to be analyzed, this field will have the information of that location, otherwise, it will be your current location.
   - **TAT** – the Turnaround Time of the Method
   - **Condition** – the current condition of the method (Active, Cancelled, On Hold)
   - **Status** – the current status of the method (Ready, Batched, 1st Level Reviewed, etc.)
   - **Phase** – currently not used.
   - **Sub-Desc** – the default description of the sub-list chosen at creation.
   - **Holding Time Calculation** – method used to calculate holding time
   - **VTSR** – if checked, the holding time will be calculated off of the RECEIPT DATE and follow the holding time setup for this occurrence.
   - **Up Rpt Limit Type/Low Rpt Limit Type** – these fields work together to advise how the method will be reported. The "Upper Limit" is the value at which the sample will be reported (usually the RL), while the "Lower Limit" is the [lowest] value at which the system will report a value (usually the MDL).
   - **Container Matrix** – the matrix of the sample expected for this method
   - **Reporting Basis** – the basis of reporting this method such as a "Dissolved" Metal versus a "Total" Metal".
   - **Calibration Group** – currently not used
   - **Calibration Curve** – currently not used
   - **Dry Wt Adjust** – check if dry weight correction is needed for the method
   - **# Tics** – the max number of TICs to be reported. If TICs are required, the analyte Tentatively Identified Compound must be set to reportable. Do not enter TICs for logged in QC samples.
   - **Method Comments** – free flow comments
   - **Reporting Rules** – currently used for dual column analyses to indicate which rule to follow when reporting both columns.
   - **SAP** - Secondary Accounts Payable. The SAP field is a 10-character field available at the method and other charges level to support client required billing codes in Invoicing. The SAP field may be controlled by a Login Group, or through the pricing and Other Charges tabs of the Job in the Login Application. SAP information applied in the project will automatically populate in the login/job and may be modified accordingly.
   - **Do Not Report** – currently used to inhibit a method from printing on the final report, although the method is needed in the login for additional

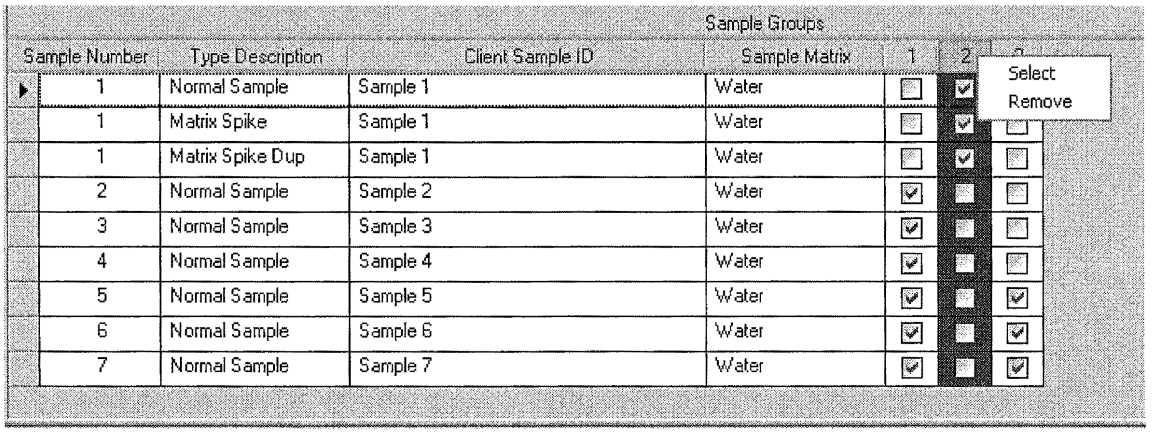
analyses (such as reporting the calculated result of HARDNESS, but not wanting to report the individual analyses required to achieve the hardness calculation)

- **Client Sub Description** – Client Sub List Description settings will copy down from project/quote module. Settings can be modified at the login level. See the *Client Sub List Description* documentation on the intranet for more information.

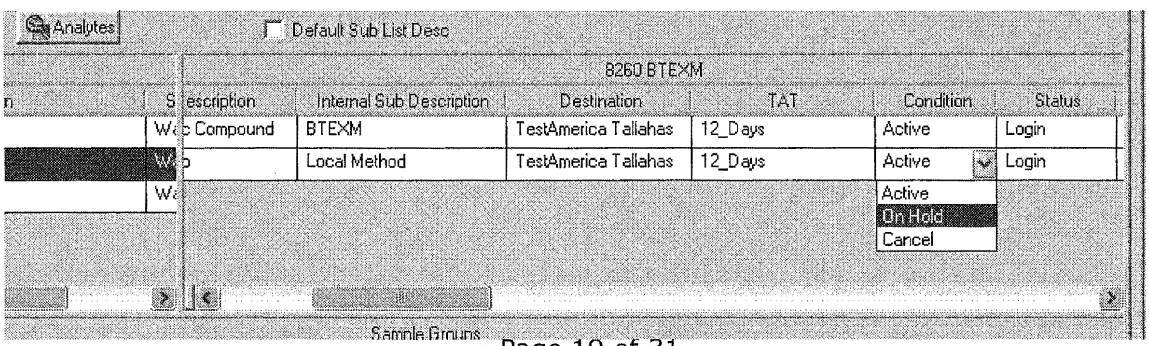
Note: *A "grey background" in the METHODS/PREP window indicates this login group is not associated to the sample highlighted in the "SAMPLE GROUPS" grid. A "white background" indicates this group is associated to the sample highlighted. Additionally, the LOGIN GROUP in the "Sample Groups" grid will turn blue to indicate which LOGIN GROUP has the focus.*

Once the LOGIN GROUP(S) has been created, associate the LOGIN GROUP to the correct sample(s).

A. Simply put a check-mark in the square corresponding to the correct sample and the correct login group.
B. A shortcut is available here as well. Simply highlight the HEADER of the LOGIN GROUP in the "Sample Group" grid (shown by the "1" and "2") by right-clicking and hit Select.



C. To **CANCEL** a method (or to place a method **ON HOLD**), highlight the appropriate SAMPLE in the sample grid, and the appropriate LOGIN Group.
D. Change the CONDITION of the method from ACTIVE to either CANCEL or ON HOLD as needed.
   - Changing the CONDITION of a prep method will automatically update the condition of the analytical method.



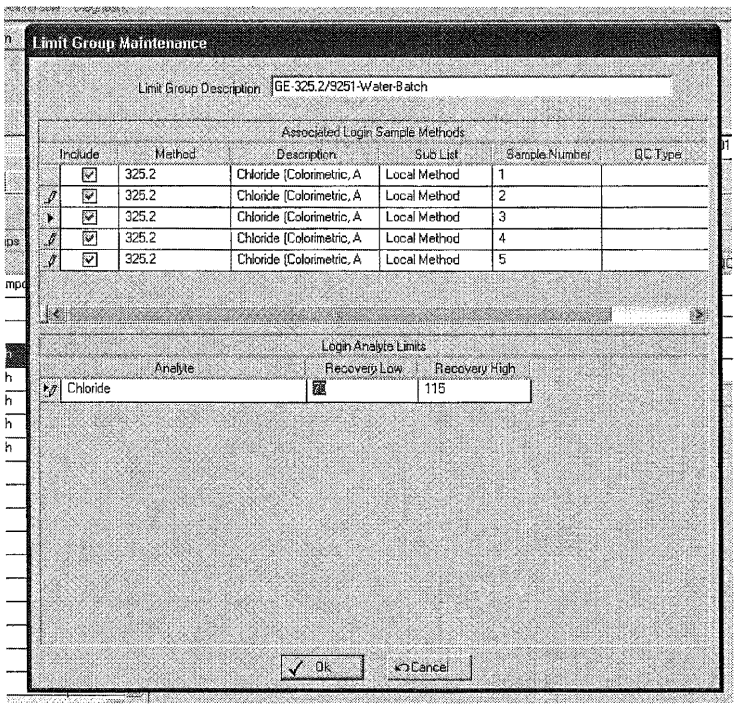
Login – Limits

This tab will be reviewed by the Project Manager to assure all of the appropriate limits have been loaded into the LOGIN.

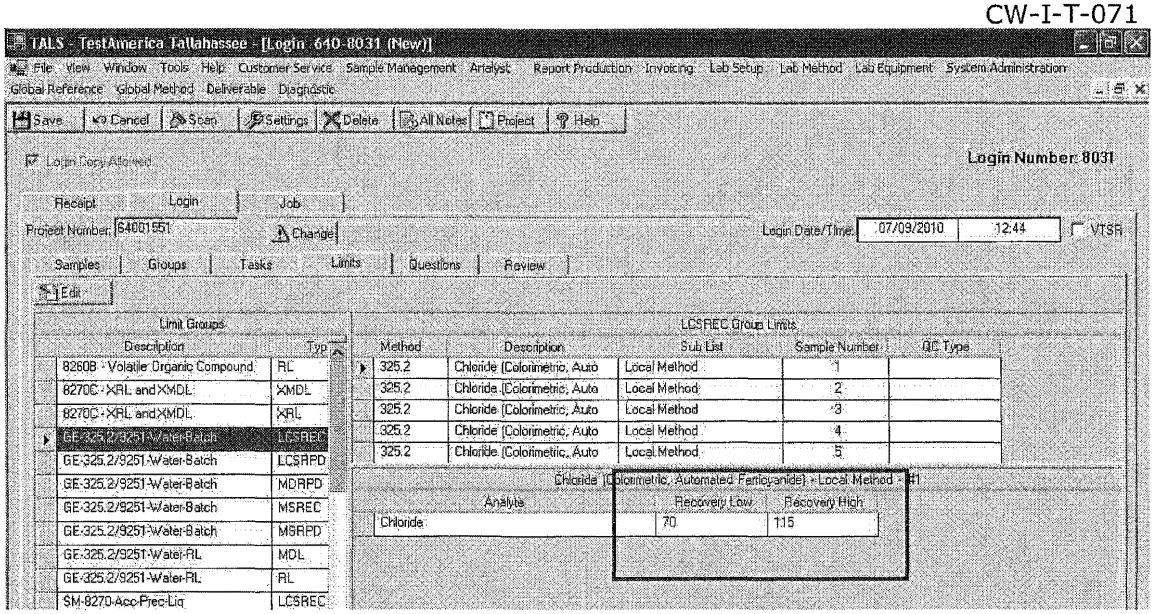
In general the limits pulled into the Login Limits are from the reference data built in the Method Limit Groups. Reference limits can be overridden if Project specific limits are defined in the Project. See *Projects* documentation posted on the intranet for more information.

Limits can also be overridden at the Login level.

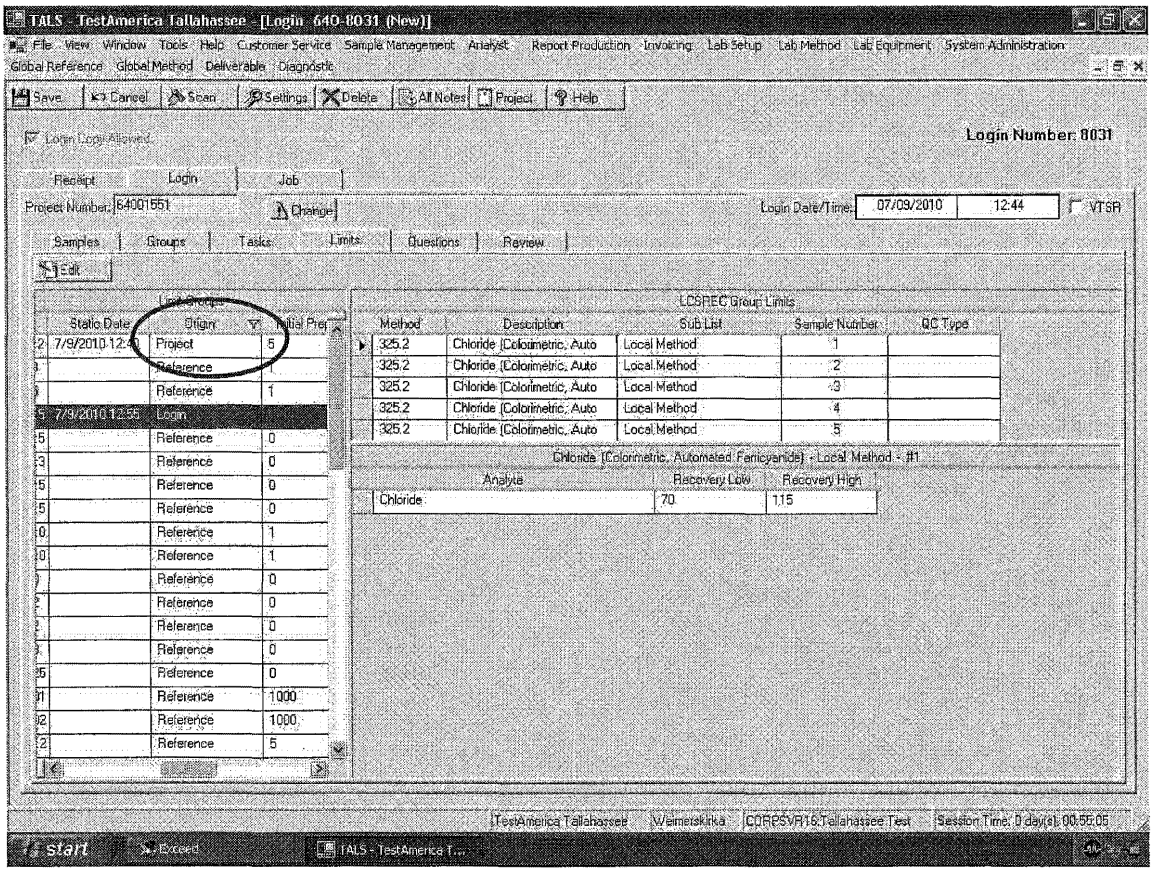
A. On the LOGIN LIMITS tab, highlight the limit to be changed and click [Edit].
B. Click [Yes] when asked to create a new limit group.
C. In the 'Limit Maintenance' pop up, select the samples the limit will be applied to (the default will have all samples checked to be included) and change the limit set in the lower grid. Click [OK].



D. The new limit will be entered into the login.



To tell which limits are applied to a limit type, scroll to the right in the Limit Groups grid to the **Origin** column.



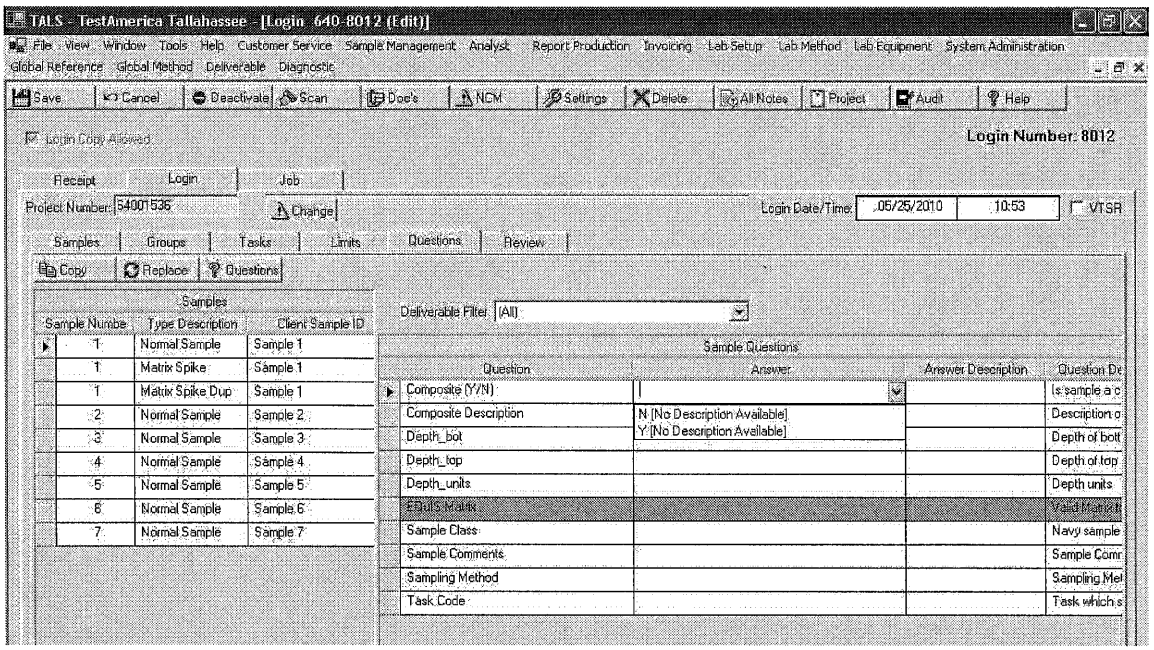
Login – Questions

This tab may have information that needs to be completed. If a client needs an EDD which requires information that is not otherwise available in TALS, **Questions** are used to accomplish this. Simply answer the question to store the answer. Login Questions are answered on a sample level basis. Questions are programmed into the EDD's and will pull into the login when an EDD that requires them is added.

Example:

Question: **What is the sample depth listed on the Chain of Custody?**
Answer: **4.5**

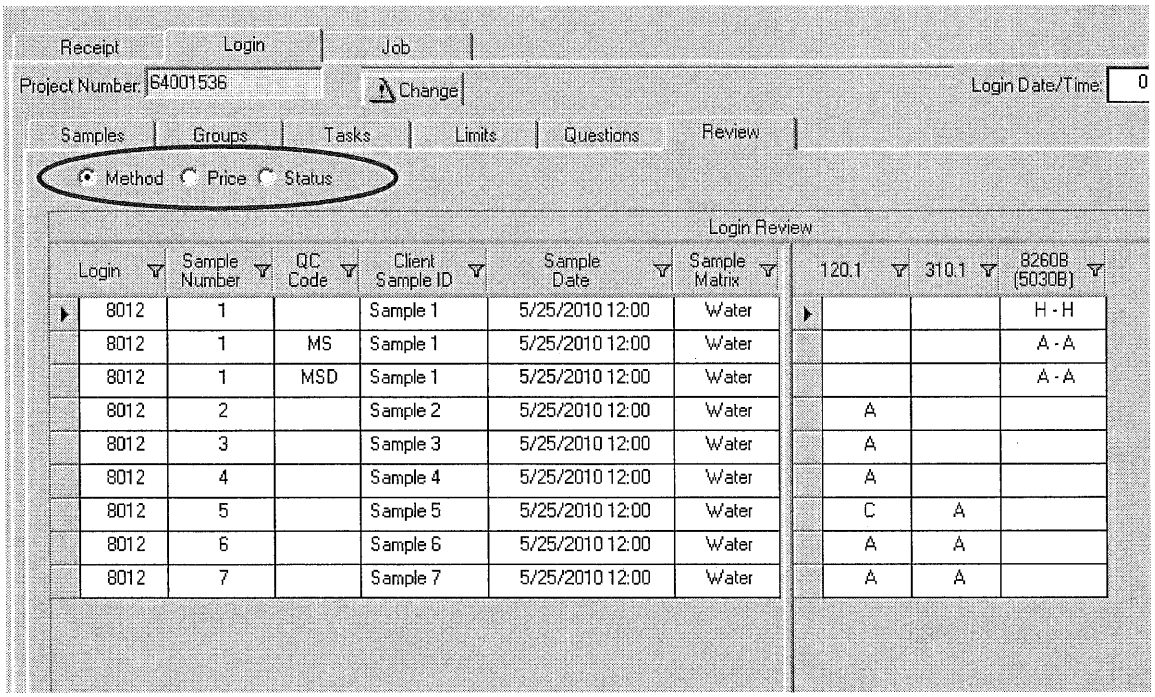
Note: *The answer to a particular question may be answered by the appropriate group, depending on the question. Just because this tab is in the LOGIN Module doesn't imply that the answer will come from Sample Receiving.*



Login – Review

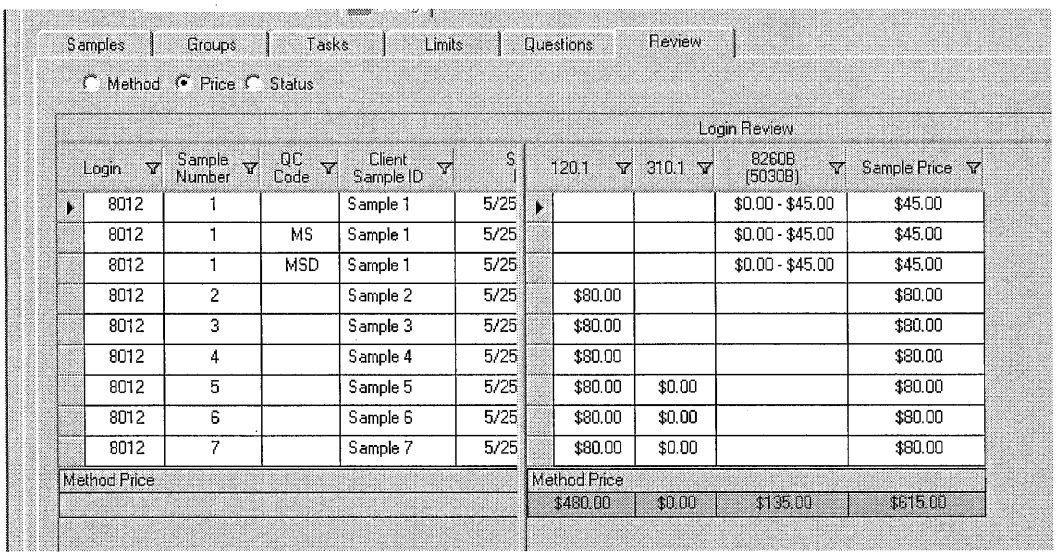
This tab shows methods, pricing and current statuses at a method chain level. Select the radio dials to toggle the views.

METHOD



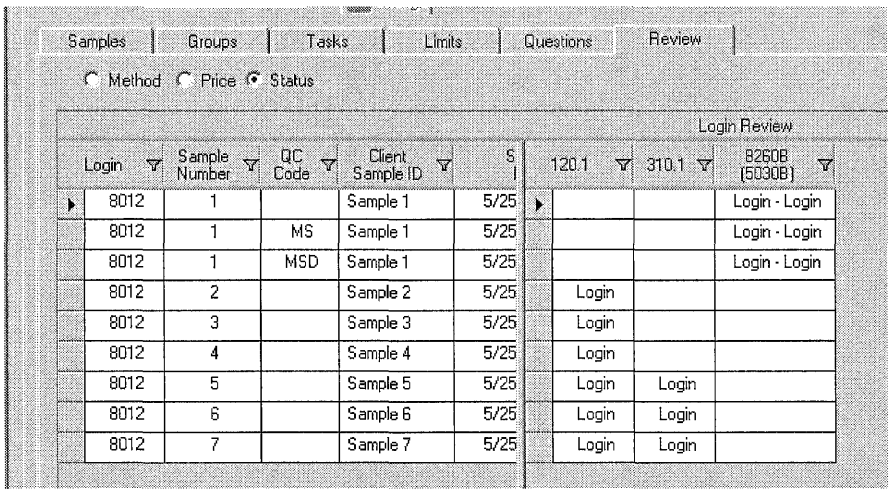
Login	Sample Number	QC Code	Client Sample ID	Sample Date	Sample Matrix	120.1	310.1	8260B (5030B)
8012	1		Sample 1	5/25/2010 12:00	Water			H - H
8012	1	MS	Sample 1	5/25/2010 12:00	Water			A - A
8012	1	MSD	Sample 1	5/25/2010 12:00	Water			A - A
8012	2		Sample 2	5/25/2010 12:00	Water	A		
8012	3		Sample 3	5/25/2010 12:00	Water	A		
8012	4		Sample 4	5/25/2010 12:00	Water	A		
8012	5		Sample 5	5/25/2010 12:00	Water	C	A	
8012	6		Sample 6	5/25/2010 12:00	Water	A	A	
8012	7		Sample 7	5/25/2010 12:00	Water	A	A	

PRICE



Login	Sample Number	QC Code	Client Sample ID	S	120.1	310.1	8260B (5030B)	Sample Price
8012	1		Sample 1	5/25			$0.00 - $45.00	$45.00
8012	1	MS	Sample 1	5/25			$0.00 - $45.00	$45.00
8012	1	MSD	Sample 1	5/25			$0.00 - $45.00	$45.00
8012	2		Sample 2	5/25	$80.00			$80.00
8012	3		Sample 3	5/25	$80.00			$80.00
8012	4		Sample 4	5/25	$80.00			$80.00
8012	5		Sample 5	5/25	$80.00	$0.00		$80.00
8012	6		Sample 6	5/25	$80.00	$0.00		$80.00
8012	7		Sample 7	5/25	$80.00	$0.00		$80.00
Method Price					Method Price			
					$480.00	$0.00	$135.00	$615.00

STATUS



Login	Sample Number	QC Code	Client Sample ID	S I	120.1	310.1	8260B (5030B)
8012	1		Sample 1	5/25			Login - Login
8012	1	MS	Sample 1	5/25			Login - Login
8012	1	MSD	Sample 1	5/25			Login - Login
8012	2		Sample 2	5/25	Login		
8012	3		Sample 3	5/25	Login		
8012	4		Sample 4	5/25	Login		
8012	5		Sample 5	5/25	Login	Login	
8012	6		Sample 6	5/25	Login	Login	
8012	7		Sample 7	5/25	Login	Login	

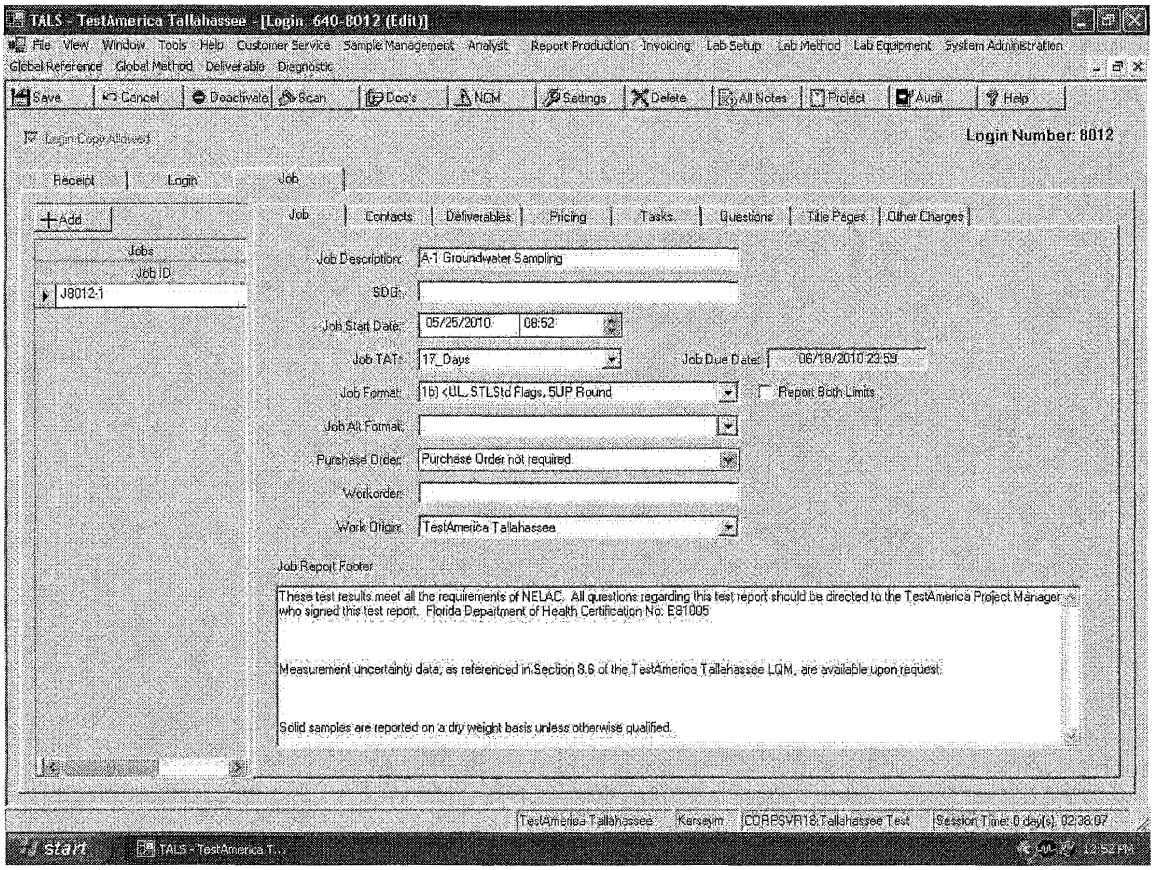
To Work with Jobs

Job – Job

A JOB is a set of samples from a single Login or multiple Logins. Samples are reported as JOBS.

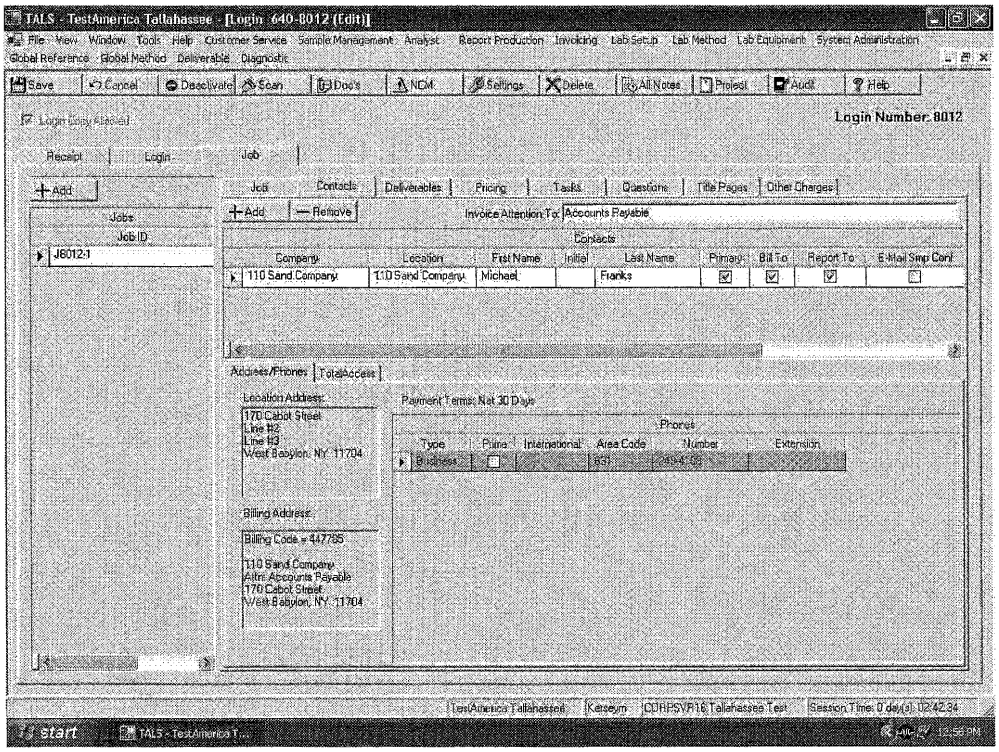
The JOB Tab is primarily a Project Manager function. This information is copied down from the associated Project.

1. **Job Description** – carried over from the Project
2. **SDG** – an alternate naming convention for joining multiple LOGINS into a JOB for reporting
3. **Job Start Date** – carried over from Receipt Date/Time
4. **Job TAT** – turnaround time defaulted from Project. Turnaround Time can be modified per Login and Job.
5. **Job Due Date** – calculated from turnaround time specified
6. **Job Format** – the default formatter from the Project. Formatter can be changed per Login and Job.
7. **Report Both Limits** – not used at this time
8. **Purchase Order** – carried over from the Project. PO can be changed per Login and Job.
9. **Workorder** - carried over from the Project. A work order can be changed per Login and Job.
10. **Work Origin** – location where samples arrived and were logged
11. **Job Report Footer** – carried over from the Project. Text can be edited at the login level.

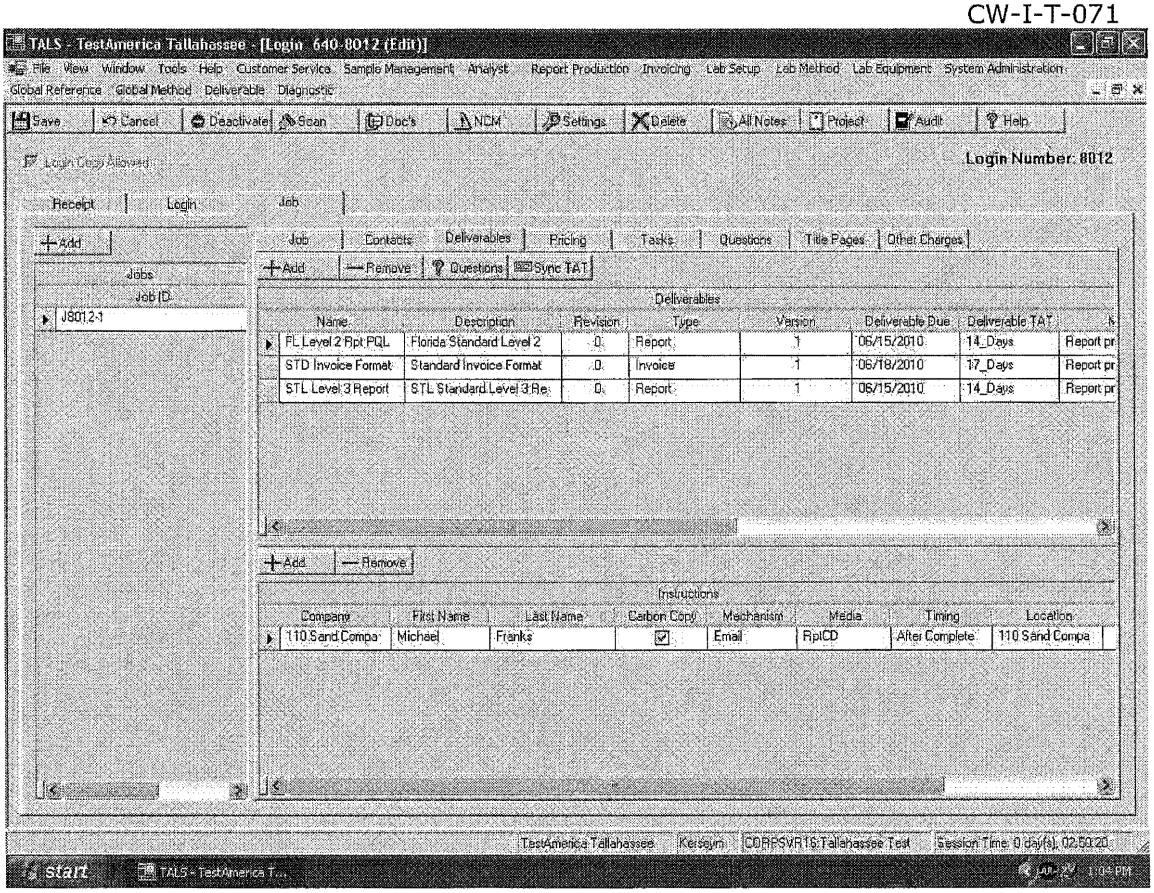


Job – Contacts

The Contact information is copied down from the Project exactly as built. The information can be modified per job. Additional contacts may be added as needed.

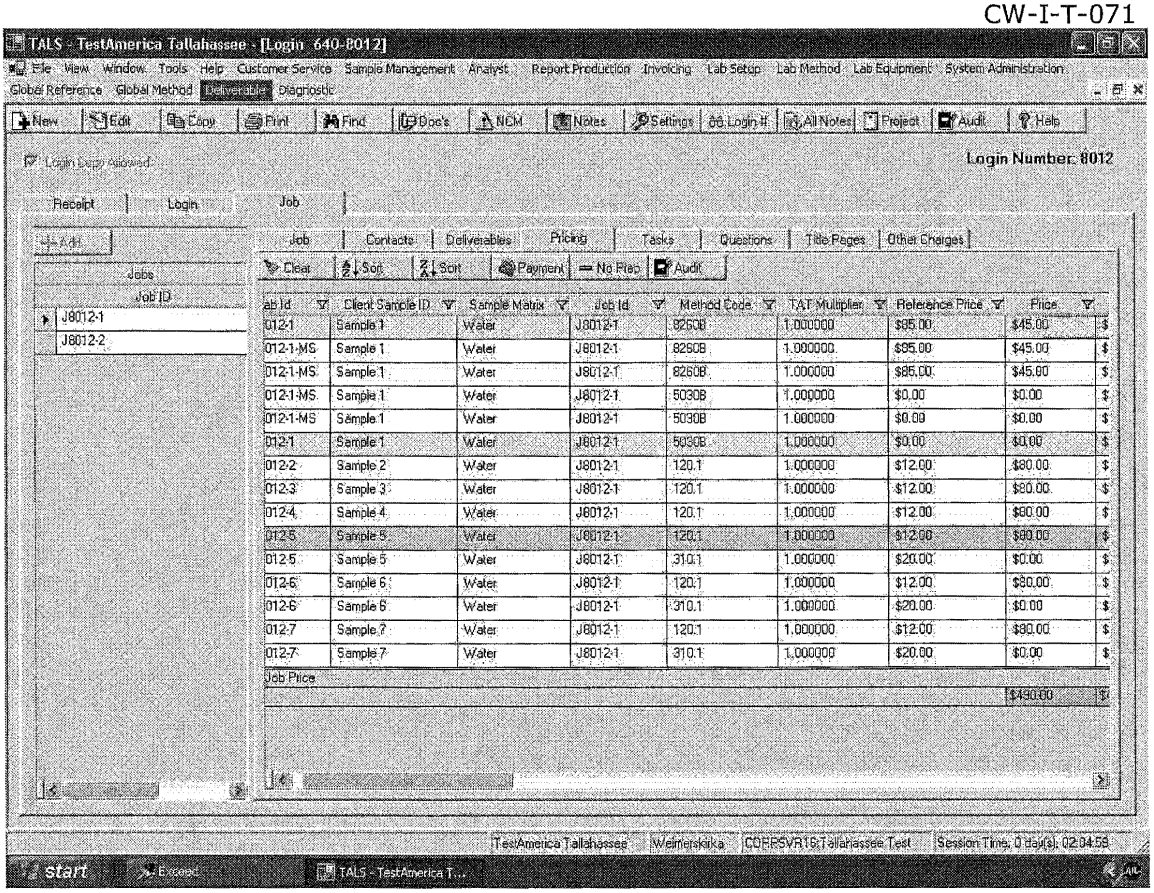


The Deliverable information is copied down from the Project exactly as built. The information can be modified per job. Additional Deliverables may be added as needed.



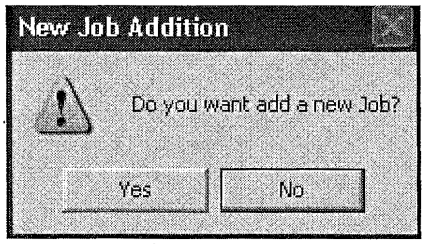
Job – Pricing

The Pricing is copied down from the Project. Pricing can be modified and additional JOBS can be added, if needed.

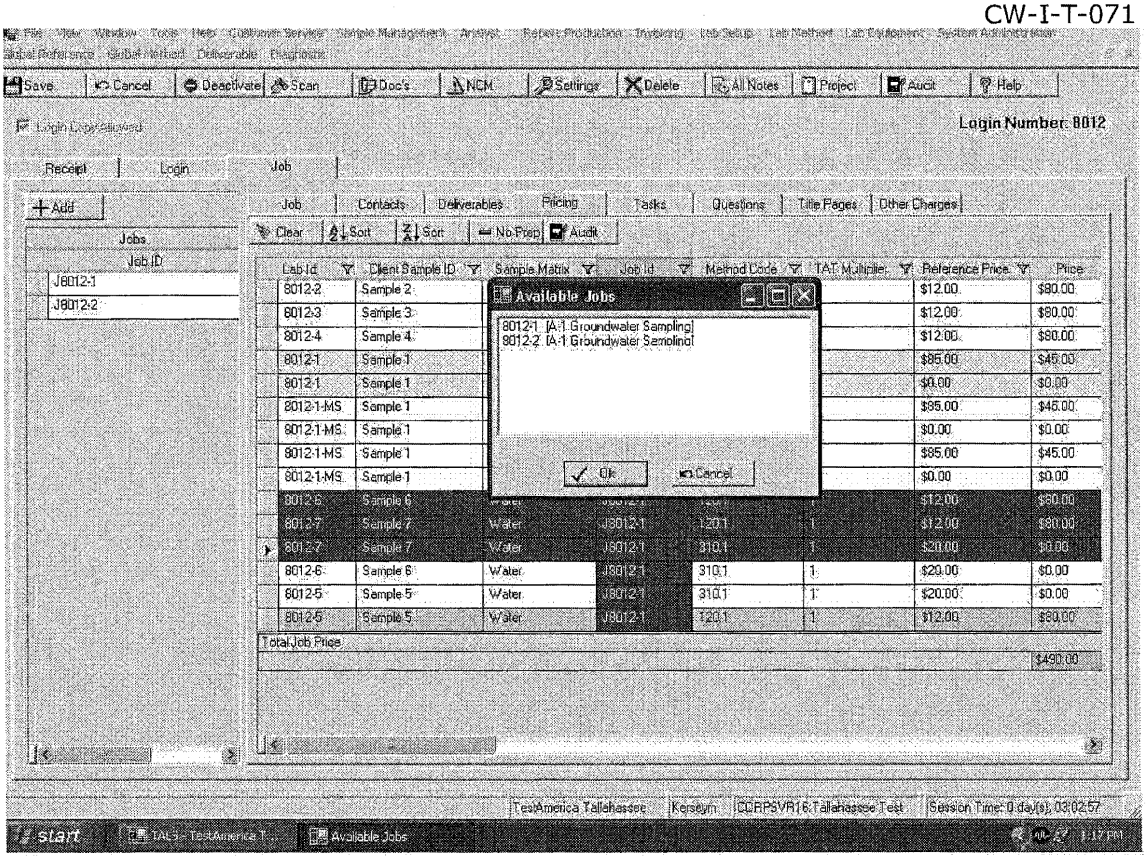


To add another JOB to a Login:

1. Click [Add]



2. A job can contain any combination of methods/samples/logins. You will assign these on the Pricing tab
3. Sort the Pricing grid by any combination of the headers. Sort and Filter the list to obtain the desired set of samples that you want on another job.
4. Left click the Job ID header to activate the column. Right-click the JOB ID header and choose the job # to assign desired job to samples/methods in the sorted grid.

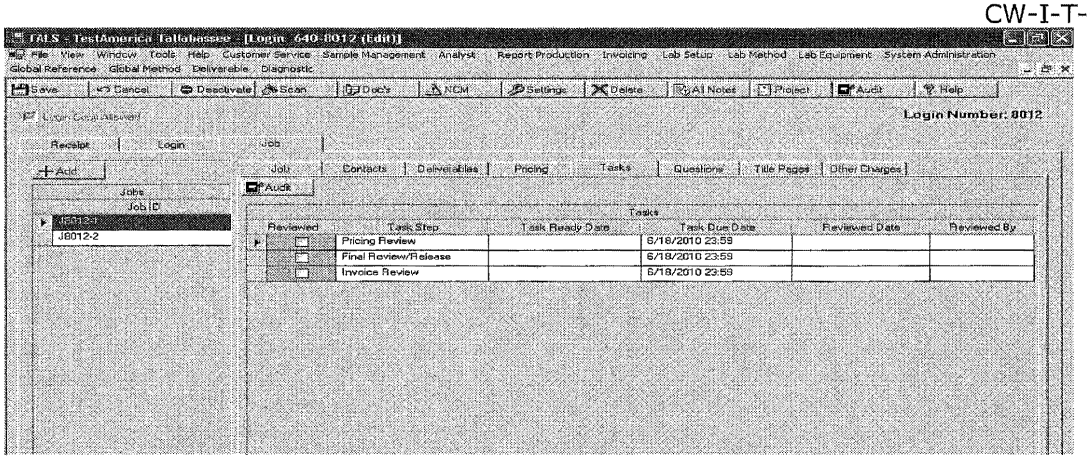


5. All the JOB information [tabs] can be customized per JOB.

Job – Task

1. As Tasks are completed (simply by placing a checkmark in the space provided next to the task), the date/time and user are recorded.
2. Tasks are hierarchal, and the previous Task must be completed before completing the next task.

*Note: The Login Review Task must be checked on the **Login – Task** tab prior to setting the review tasks in the **Job** tab.*



Job – Questions

This tab may have information that needs to be completed. If a client needs an EDD which requires information that is not otherwise available in TALS, **Questions** are used to accomplish this. Simply answer the question to store the answer. Job Questions are answered on a Job level basis. Questions are programmed into the EDD's and will pull into the login when an EDD that requires them is added.

Example:

Question: **What is the lab location?**
Answer: **TAL**

Note: *The answer to a particular question may be answered by the appropriate group, depending on the question. Just because this tab is in the LOGIN Module doesn't imply that the answer will come from Sample Receiving.*

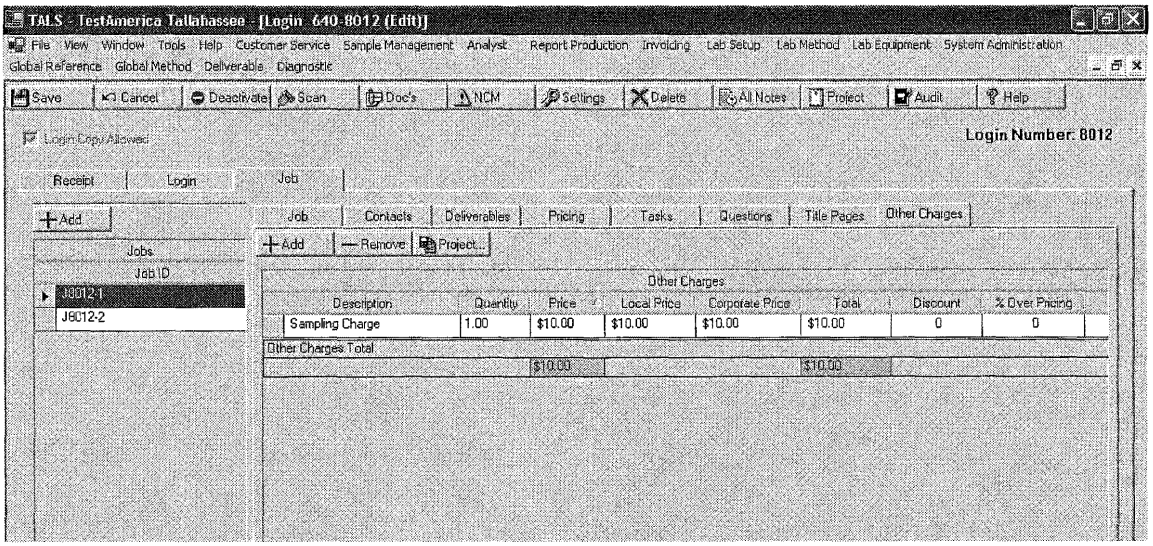
Job – Title Pages

Not used.

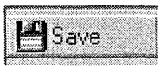
Job – Other Charges

Non-Analytical charges are copied from the Project. [**Add**] / [**Remove**] **Other Charges** as needed.

*Note: Certain Other Charges are automatically added based on the **Login** or **Project.** For example, EMF (Environmental Management Fees) are automatically added to all logins and are a percentage of the total price for the job.*



SAVE the Login.



Attachment 6.

LIMS Sample Receipt Checklist
(017-001)

Login Sample Receipt Checklist

Client: URS Corporation

Job Number: 500-93479-1

Login Number: 93479
List Number: 1
Creator: Lunt, Jeff T

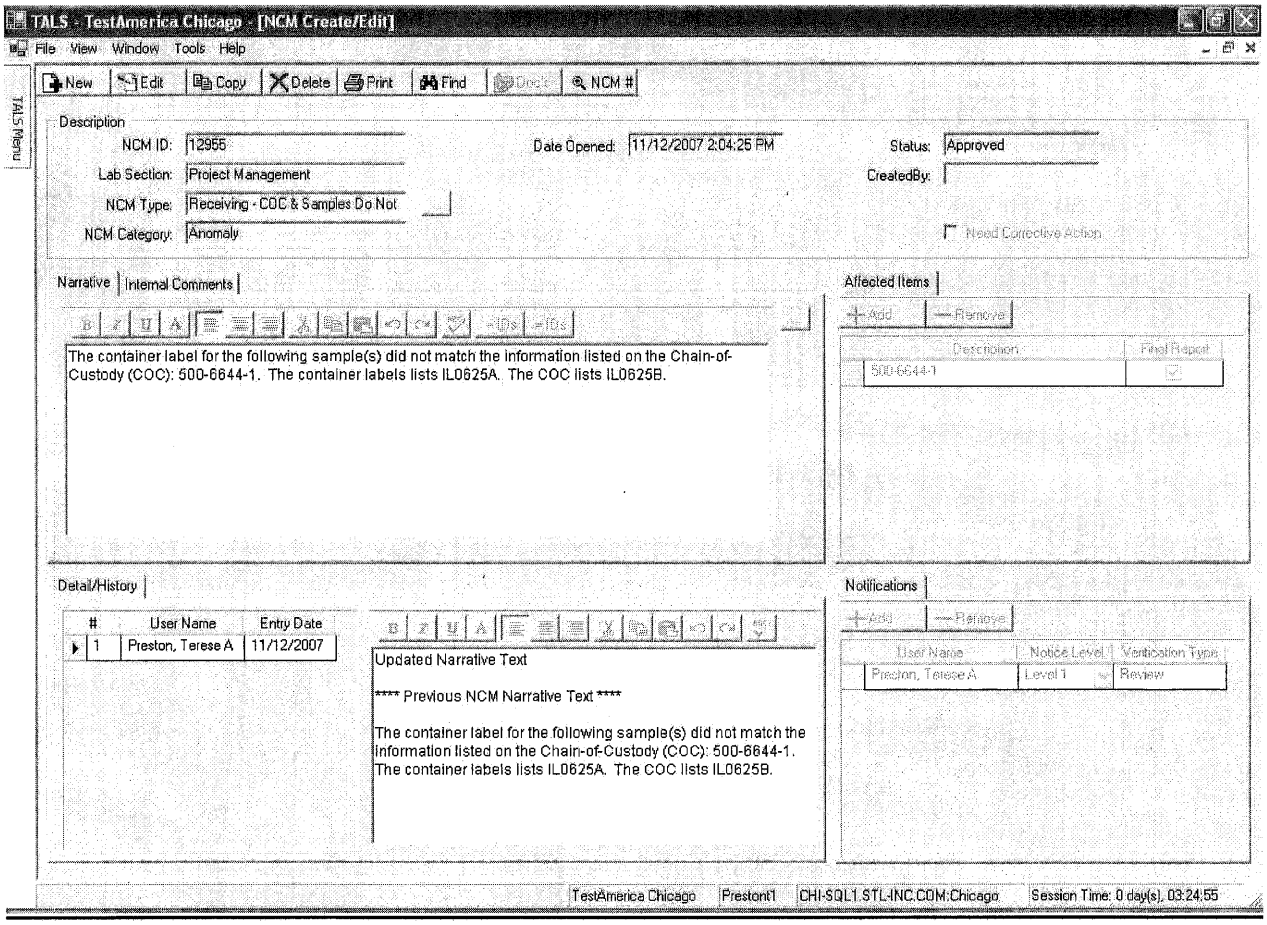
List Source: TestAmerica Chicago

Question	Answer	Comment
Radioactivity wasn't checked or is </= background as measured by a survey meter.	True	
The cooler's custody seal, if present, is intact.	True	
Sample custody seals, if present, are intact.	True	
The cooler or samples do not appear to have been compromised or tampered with.	True	
Samples were received on ice.	True	3.5,3.2
Cooler Temperature is acceptable.	True	BP LaMP temp checks per 3.1.3; Samples rec'd = 6C; Temp maintained during login.
Cooler Temperature is recorded.	True	
COC is present.	True	
COC is filled out in ink and legible.	True	
COC is filled out with all pertinent information.	True	
Is the Field Sampler's name present on COC?	True	
There are no discrepancies between the containers received and the COC.	True	
Samples are received within Holding Time.	True	
Sample containers have legible labels.	True	
Containers are not broken or leaking.	True	
Sample collection date/times are provided.	True	
Appropriate sample containers are used.	True	Verification of receipt of Trip Blank with volatile samples.
Sample bottles are completely filled.	True	
Sample Preservation Verified.	True	
There is sufficient vol. for all requested analyses, incl. any requested MS/MSDs	True	
Containers requiring zero headspace have no headspace or bubble is <6mm (1/4").	True	
Multiphasic samples are not present.	True	
Samples do not require splitting or compositing.	True	
Residual Chlorine Checked.	N/A	

(017-001)

Attachment 7.

Non-Conformance Memo (NCM)



Attachment 8.

Internal Cooler Temperature Control Log
(019-001)

TestAmerica 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:_____

CHI-22-11-020/B-07/07

(019-001)

Attachment 9.

TestAmerica Chicago Sample Acceptance Policy
(020-001)

TestAmerica

THE LEADER IN ENVIRONMENTAL TESTING

TestAmerica Chicago Sample Acceptance Policy

The following describes TestAmerica 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' with this policy will be formally addressed and conditions documented according to internal operating procedures. Subsequent analysis of such samples may or may not proceed and will be determined by discussion with the appropriate parties involved.

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.

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

Note:
BP LaMP requires samples are received $\leq$ 6 deg C and that temperature be maintained during the sample log-in process. Temperature verification checks are required at 20 minute intervals.
BP LaMP requires documentation that Trip Blanks are received with volatile samples.

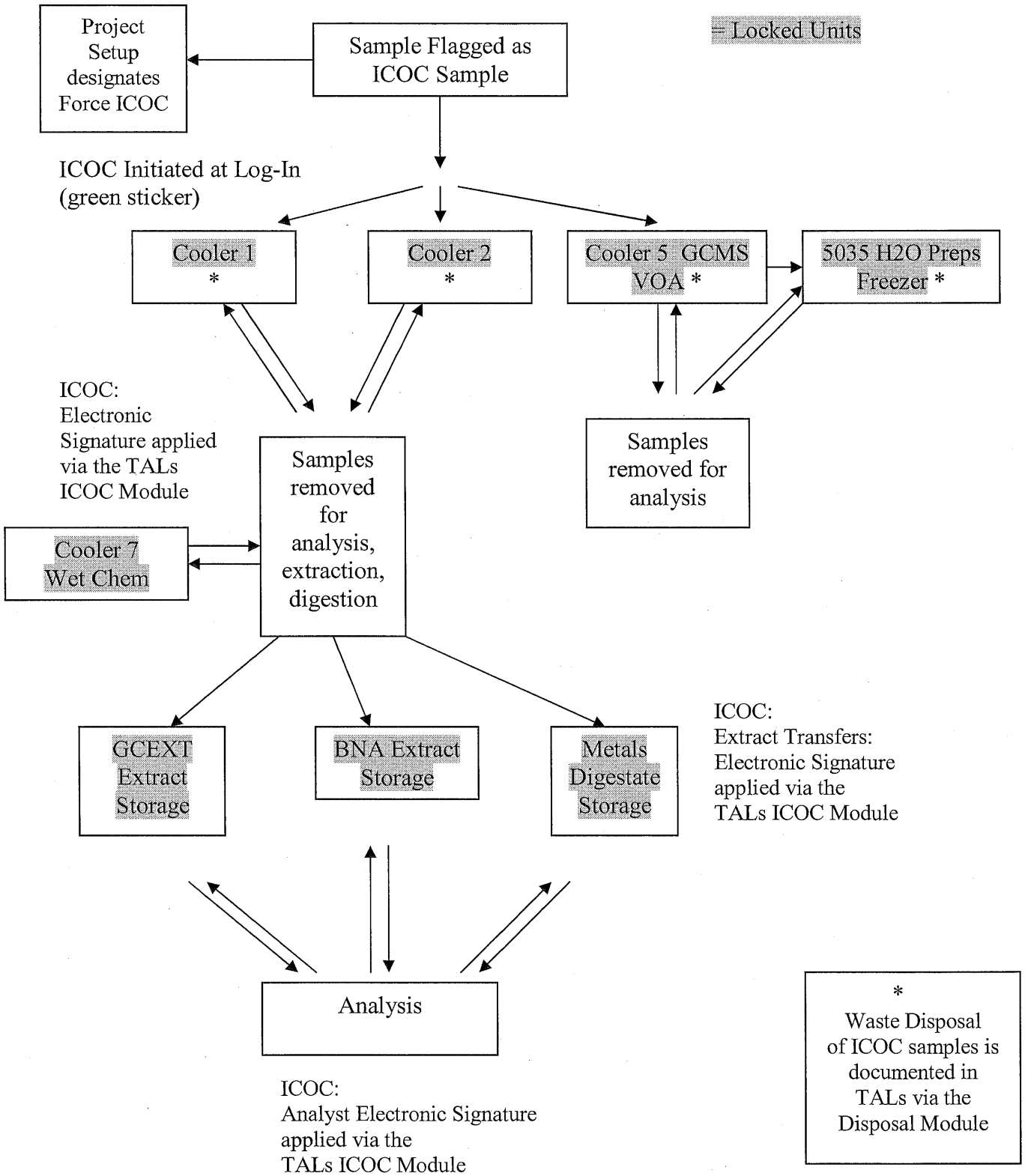
It is critical that the integrity of the samples be maintained during the sample login process. Samples need to be promptly stored in their designated sample coolers after the login process is completed and prior to sample homogenization, preparation or analysis.

(020-001)

Attachment 10.

Flowchart for Internal Chain of Custody Procedures (021-001)

Flowchart for Internal Chain of Custody



*All extracts and digestates enter appropriate waste streams refer to SOP *UP-WM-001* for details.

Attachment 11.

Sample Login: Cooler Receipt Temperature Recording Procedure (022-001)

TestAmerica Chicago

Sample Login: Cooler Receipt Temperature Recording Procedure

All samples coolers will have temperatures taken. Sample custodians will document sample receipt temperature readings on the designated client chain-of-custody and within LIMS on the Sample Receipt Checklist. These readings will be reported to the client in the final data report.

The IR Gun thermometer is used by the sample custodians to read sample temperatures through the containers in which they are received. There are certain precautions to follow with this thermometer. Do not directly touch the object to be measured with the probe, the unit should be positioned as close to the target as possible. Do not use the device where the probe temperature may exceed the -22°C to 110°C range. Refer to the manufacturer's instruction booklet for further care, operation and specifications. The factory emissivity has been set to 0.95, which will cover 90% of typical applications and should be sufficient for typical use in the laboratory and intended purpose.

A daily calibration check, prior to use, is performed by taking the reading of a sample bottle located in Cooler 8 which is labeled "For IR Gun Calibration". This measurement is documented on the IR Gun: Daily Calibration Verification Log.

1. Locate the thermometer in the marked/specified drawer in login.

2. Turn on the IR Thermometer (see ON/OFF button).

3. Hold the thermometer probe as close to the target/sample as possible without physically touching the sample with the end of the probe.

4. Take the reading and record the uncorrected and corrected temperatures as follows:

5. Calculate for CF (Correction Factor-a tag is attached to the cord of the instrument. Correction Factors are for Glass and Plastic). Use the corresponding CF to calculate the temperature ie. If the noted temperature is 2.0 degrees C for a sample housed in glass, and the CF for glass is +2.0 degrees C, then the calculated temp is 4.0 degrees C and should be recorded as such.

6. Record both the "Uncorrected" cooler temperature and "Corrected" cooler temperature, the sample container type (P/G) on the Sample Receipt Cooler/Temperature Tracking form located on the workstation bench in login. A temperature is required to be documented for each cooler received. The corrected temperatures will be transferred to the TALs Login Sample Receipt Checklist by the Sample Log-In personnel.

7. Put samples in Cooler 8

8. Leave COC on login bench (if no COC, leave a note for login that samples are located in Cooler 8 and no COC is present).

9. Turn OFF IR Thermometer and place it back in the mark/specified drawer.

(022-001)

Attachment 12.

Sample Receipt Cooler Temperature Tracking Log
(023-001)

TestAmerica Chicago
Sample Receipt Cooler/Temperature Tracking

Date:_____

Courier	Client Name	Container Type P / G	Uncorrected Temperature °C	Corrected Temperature °C

Reviewed by:_____ Date:_____

(023-001)

Attachment 13:

Preservative Lot Documentation for Sample Receipt (024-001)

TestAmerica Chicago

Preservative Lot Documentation for Sample Receipt

Date	Time	Client Name	Sample#	Preservative	Preservative Vol. Added (mls)	Lot#	Preserved by

Preservative: 1:1 H2SO4 20% HNO3 1:1 HCl 1:1 NaOH 1:1 NaOH/Zn Acetate

Reviewed by: _____ Date: _____

CHI-22-11-007/E-09/11 Page: _____

(100-024-001)

STATE OF ILLINOIS

ENVIRONMENTAL PROTECTION AGENCY
NELAP - RECOGNIZED
ENVIRONMENTAL LABORATORY ACCREDITATION

is hereby granted to

TESTAMERICA CHICAGO

2417 BOND STREET

UNIVERSITY PARK, IL 60484

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.

Celeste M. Crowley
Acting Manager
Environmental Laboratory Accreditation Program

Scott D. Siders
Accreditation Officer
Environmental Laboratory Accreditation Program

Certificate No.: 003642
Expiration Date: 04/30/2016
Issued On: 05/06/2015

State of Illinois
Environmental Protection Agency

Certificate No.: 003642

Awards the Certificate of Approval to:

TestAmerica Chicago
2417 Bond Street
University Park, IL 60484

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

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FOT Name: Drinking Water, Inorganic

Method: SM2320B,20Ed

Matrix Type: Potable Water

Alkalinity

Method: SM2340B,20Ed

Matrix Type: Potable Water

Hardness

Method: SM2510B,20Ed

Matrix Type: Potable Water

Conductivity

Method: SM2540C,20Ed

Matrix Type: Potable Water

Total Dissolved Solids

Method: SM4500Cl-F,20Ed

Matrix Type: Potable Water

Chlorine

Method: SM4500CN-E,20Ed

Matrix Type: Potable Water

Cyanide

Method: SM4500F-C,20Ed

Matrix Type: Potable Water

Fluoride

Method: SM4500H-B,20Ed

Matrix Type: Potable Water

Hydrogen Ion (pH)

Method: SM4500NO2-B,20Ed

Matrix Type: Potable Water

Nitrite

Method: SM4500NO3-F,20Ed

Matrix Type: Potable Water

TestAmerica Chicago
2417 Bond Street
University Park, IL 60484

FOT Name: Drinking Water, Inorganic

Matrix Type: Potable Water

Method: SM4500P-E,20Ed

Matrix Type: Potable Water

Orthophosphate

Method: SM4500SO4-E,20Ed

Matrix Type: Potable Water

Sulfate

Method: SM5310C,20Ed

Matrix Type: Potable Water

Dissolved Organic Carbon

Method: USEPA150.1

Matrix Type: Potable Water

Hydrogen ion (pH)

Method: USEPA200.7R4.4

Matrix Type: Potable Water

Aluminum
Arsenic
Barium
Beryllium
Cadmium
Calcium
Chromium
Copper
Iron
Magnesium
Manganese
Nickel
Silica
Silver
Sodium
Zinc

Method: USEPA200.8R5.4

Matrix Type: Potable Water

Aluminum
Antimony
Arsenic
Barium
Beryllium
Cadmium
Chromium
Copper
Lead
Manganese
Molybdenum
Nickel
Selenium
Silver
Thallium
Zinc

Method: USEPA245.1R3.0

Method: SM4500NO3-F,20Ed

Nitrate

Method: SM5310C,20Ed

Matrix Type: Potable Water

Total Organic Carbon (TOC)

TestAmerica Chicago
2417 Bond Street
University Park, IL 60484

FOT Name: Drinking Water, Inorganic

Method: USEPA245.1R3.0

Matrix Type: Potable Water

Mercury

Method: USEPA300.0R2.1

Matrix Type: Potable Water

Chloride

Fluoride

Nitrate

Nitrite

Orthophosphate

Sulfate

Method: USEPA353.2R2.0

Matrix Type: Potable Water

Nitrate

FOT Name: Non Potable Water, Inorganic

Method: Hach 10360

Matrix Type: NPW

Oxygen - Dissolved

Method: SM 4500 S2-F,2000

Matrix Type: NPW/SCM

Sulfide

Method: SM2320B,1997

Matrix Type: NPW/SCM

Alkalinity

Method: SM2340B,1997

Matrix Type: NPW

Hardness

Method: SM2510B,1997

Matrix Type: NPW/SCM

Specific Conductance

Method: SM2540B,1997

Matrix Type: NPW

Residue (Total)

Method: SM2540C,1997

Matrix Type: NPW

Residue (TDS)

Method: SM2540D,1997

Matrix Type: NPW

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FOT Name: Non Potable Water, Inorganic

Matrix Type: NPW

Method: SM2540E,1997

Matrix Type: NPW

Residue (% Volatile)

Method: SM2540F,1997

Matrix Type: NPW

Residue (settleable)

Method: SM3500Cr-B,2009

Matrix Type: NPW

Chromium VI

Method: SM4500CL-E,1997

Matrix Type: NPW/SCM

Chloride

Method: SM4500Cl-F,2000

Matrix Type: NPW

Chlorine, Total Residual

Method: SM4500Cl-G,2000

Matrix Type: NPW

Chlorine, Total Residual

Method: SM4500CN-E,1999

Matrix Type: NPW/SCM

Cyanide

Method: SM4500CN-G,1999

Matrix Type: NPW/SCM

Cyanide, Available

Method: SM4500F-C,1997

Matrix Type: NPW/SCM

Fluoride

Method: SM4500H-B,2000

Matrix Type: NPW

Hydrogen Ion (pH)

Method: SM4500NH3-G,1997

Matrix Type: NPW/SCM

Ammonia

Method: SM2540D,1997

Residue (TSS)

Total Kjeldahl Nitrogen

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University Park, IL 60484

FOT Name: Non Potable Water, Inorganic

Method: SM4500NO2-B,2000

Matrix Type: NPW/SCM

Nitrite

Method: SM4500NO3-F,2000

Matrix Type: NPW/SCM

Nitrate — Nitrate-nitrite (as N)

Method: SM4500O-G,2001

Matrix Type: NPW

Oxygen - Dissolved

Method: SM4500P-E,1999

Matrix Type: NPW/SCM

Orthophosphate (as P) — Phosphorus

Method: SM4500SO4-E,1997

Matrix Type: NPW

Sulfate

Method: SM5210B,2001

Matrix Type: NPW/SCM

Biochemical Oxygen Demand (BOD) — Carbonaceous Biochemical Oxygen Demand (CBOI

Method: SM5220C,1997

Matrix Type: NPW/SCM

Chemical Oxygen Demand (COD)

Method: SM5310C,2000

Matrix Type: NPW/SCM

Total organic carbon (TOC)

Method: USEPA120.1,1982

Matrix Type: NPW/SCM

Specific Conductance

Method: USEPA160.4,1971

Matrix Type: NPW/SCM

Residue (Volatile)

Method: USEPA1664A

Matrix Type: NPW/SCM

Oil and Grease

Method: USEPA1664B

Matrix Type: NPW/SCM

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FOT Name: Non Potable Water, Inorganic

Matrix Type: NPW/SCM

Method: USEPA180.1R2.0,1993

Matrix Type: NPW

- Turbidity

Method: USEPA200.7,1994

Matrix Type: NPW/SCM

- Aluminum
- Antimony
- Arsenic
- Barium
- Beryllium
- Boron
- Cadmium
- Calcium
- Chromium
- Cobalt
- Copper
- Iron
- Lead
- Magnesium
- Manganese
- Molybdenum
- Nickel
- Potassium
- Selenium
- Silica
- Silver
- Sodium
- Thallium
- Tin
- Titanium
- Vanadium
- Zinc

Method: USEPA200.8,1994

Matrix Type: NPW

- Aluminum
- Antimony
- Arsenic
- Barium
- Beryllium
- Boron
- Cadmium
- Calcium
- Chromium
- Cobalt
- Copper
- Iron
- Lead
- Magnesium
- Manganese
- Molybdenum
- Nickel
- Potassium
- Selenium
- Silver
- Sodium
- Thallium
- Tin
- Titanium

Method: USEPA1664B

- Oil & Grease

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FOT Name: Non Potable Water, Inorganic

Method: USEPA200.8,1994

Matrix Type: NPW

Vanadium

Zinc

Method: USEPA218.6R3.3,1994

Matrix Type: NPW

Chromium VI

Method: USEPA245.1R3.0,1994

Matrix Type: NPW

Mercury

Method: USEPA300.0R2.1,1993

Matrix Type: NPW

Bromide

Chloride

Fluoride

Nitrate

Nitrate-Nitrite (as N)

Nitrite

Orthophosphate (as P)

Sulfate

Method: USEPA350.1R2.0,1993

Matrix Type: NPW/SCM

Ammonia

Method: USEPA351.1,1978

Matrix Type: NPW/SCM

Total Kjeldahl Nitrogen

Method: USEPA353.2R2.0,1993

Matrix Type: NPW/SCM

Nitrate

Nitrate-nitrite (as N)

Method: USEPA420.4R1.0,1993

Matrix Type: NPW/SCM

Phenolics

FOT Name: Non Potable Water, Organic

Method: USEPA608

Matrix Type: NPW

4,4'-DDD

4,4'-DDE

4,4'-DDT

Aldrin

alpha-BHC

beta-BHC

Chlordane

delta-BHC

Dieldrin

Endosulfan I

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FOT Name: Non Potable Water, Organic

Method: USEPA608

Matrix Type: NPW

Endosulfan sulfate	Endosulfan II
Endrin aldehyde	Endrin
Heptachlor	gamma-BHC (Lindane)
Methoxychlor	Heptachlor epoxide
PCB-1221	PCB-1016
PCB-1242	PCB-1232
PCB-1254	PCB-1248
Toxaphene	PCB-1260

Method: USEPA610

Matrix Type: NPW

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

Method: USEPA624

Matrix Type: NPW

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 (Propenal)
Acrylonitrile	Benzene
Bromodichloromethane	Bromoform
Bromomethane	Carbon tetrachloride
Chlorobenzene	Chloroethane
Chloroform	Chloromethane
cis-1,3-Dichloropropene	Dibromochloromethane
Dichloromethane (Methylene chloride)	Ethylbenzene

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FOT Name: Non Potable Water, Organic

Method: USEPA624

Matrix Type: NPW

- Methyl tert-butyl ether (MTBE)
- Tetrachloroethene
- Toluene
- trans-1,2-Dichloroethene
- trans-1,3-Dichloropropene
- Trichloroethene
- Trichlorofluoromethane
- Vinyl chloride
- Xylenes (total)

Method: USEPA625

Matrix Type: NPW

- 1,2,4-Trichlorobenzene
- 1,2-Dichlorobenzene
- 1,3-Dichlorobenzene
- 1,4-Dichlorobenzene
- 2,2-Oxybis (1-chloropropane)
- 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-Dinitrotoluene (2,6-DNT)
- 2-Chloronaphthalene
- 2-Chlorophenol
- 2-Methyl-4,6-dinitrophenol
- 2-Nitrophenol
- 3,3'-Dichlorobenzidine
- 4-Bromophenyl phenyl ether
- 4-Chloro-3-methylphenol
- 4-Chlorophenyl phenyl ether
- 4-Nitrophenol
- Acenaphthene
- Acenaphthylene
- Anthracene
- Benzidine
- Benzo(a)anthracene
- Benzo(a)pyrene
- Benzo(b)fluoranthene
- Benzo(g,h,i)perylene
- Benzo(k)fluoranthene
- Benzyl butyl phthalate
- Bis(2-chloroethoxy) methane
- Bis(2-chloroethyl) ether
- Bis(2-ethylhexyl) phthalate
- Chrysene
- Dibenz(a,h)anthracene
- Diethyl phthalate
- Dimethyl phthalate
- Di-n-butyl phthalate
- Di-n-octyl phthalate
- Fluoranthene
- Fluorene
- Hexachlorobenzene
- Hexachlorobutadiene
- Hexachlorocyclopentadiene
- Hexachloroethane
- Indeno(1,2,3-cd) pyrene
- Isophorone
- Naphthalene
- Nitrobenzene
- N-Nitrosodimethylamine
- N-Nitrosodi-n-propylamine
- N-Nitrosodiphenylamine
- Pentachlorophenol
- Phenanthrene
- Phenol

TestAmerica Chicago
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FOT Name: Non Potable Water, Organic

Method: USEPA625

Matrix Type: NPW

Pyrene

FOT Name: Solid and Chemical Materials, Inorganic

Method: 1010A

Matrix Type: NPW/SCM

Ignitability

Method: 1311

Matrix Type: NPW/SCM

TCLP (Organic and Inorganic)

Method: 1312

Matrix Type: NPW/SCM

Synthetic Precipitation Leaching Procedure

Method: 6010B

Matrix Type: NPW/SCM

Aluminum
Antimony
Arsenic
Barium
Beryllium
Boron
Cadmium
Calcium
Chromium
Cobalt
Copper
Iron
Lead
Lithium
Magnesium
Manganese
Molybdenum
Nickel
Potassium
Selenium
Silica
Silver
Sodium
Strontium
Thallium
Tin
Titanium
Vanadium
Zinc

Method: 6010C

Matrix Type: NPW/SCM

Aluminum
Antimony
Arsenic
Barium
Beryllium
Boron
Cadmium
Calcium

TestAmerica Chicago
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University Park, IL 60484

FOT Name: Solid and Chemical Materials, Inorganic

Method: 6010C

Matrix Type: NPW/SCM

- Cobalt
- Iron
- Lithium
- Manganese
- Nickel
- Selenium
- Silver
- Strontium
- Tin
- Vanadium
- Chromium
- Copper
- Lead
- Magnesium
- Molybdenum
- Potassium
- Silica
- Sodium
- Thallium
- Titanium
- Zinc

Method: 6020A

Matrix Type: NPW

- Aluminum
- Arsenic
- Beryllium
- Cadmium
- Chromium
- Copper
- Lead
- Manganese
- Nickel
- Selenium
- Sodium
- Vanadium
- Antimony
- Barium
- Boron
- Calcium
- Cobalt
- Iron
- Magnesium
- Molybdenum
- Potassium
- Silver
- Thallium
- Zinc

Method: 7196A

Matrix Type: NPW/SCM

- Chromium VI

Method: 7199

Matrix Type: NPW

- Chromium VI

Method: 7470A

Matrix Type: NPW

- Mercury

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2417 Bond Street
University Park, IL 60484

FOT Name: Solid and Chemical Materials, Inorganic **Method: 7471B**

Matrix Type: SCM

Mercury

Method: 9014

Matrix Type: NPW/SCM

Cyanide

Method: 9020B

Matrix Type: NPW/SCM

TOX (Total Organic Halides)

Method: 9034

Matrix Type: NPW/SCM

Sulfides

Method: 9038

Matrix Type: NPW

Sulfate

Method: 9040B

Matrix Type: NPW

Hydrogen Ion (pH)

Method: 9040C

Matrix Type: NPW

Hydrogen Ion (pH)

Method: 9045C

Matrix Type: SCM

Hydrogen Ion (pH)

Method: 9045D

Matrix Type: SCM

Hydrogen Ion (pH)

Method: 9050A

Matrix Type: NPW/SCM

Specific Conductance

Method: 9056A

Matrix Type: NPW/SCM

Bromide
Chloride
Fluoride
Nitrate
Nitrite
Phosphate

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University Park, IL 60484

FOT Name: Solid and Chemical Materials, Inorganic

Method: 9056A

Matrix Type: NPW/SCM

Sulfate

Method: 9060A

Matrix Type: NPW/SCM

Total Organic Carbon (TOC)

Method: 9066

Matrix Type: NPW/SCM

Phenolics

Method: 9071B

Matrix Type: NPW/SCM

Oil and Grease Extractable

Method: 9095A

Matrix Type: NPW/SCM

Paint Filter

Method: 9095B

Matrix Type: NPW/SCM

Paint Filter

Method: 9251

Matrix Type: NPW/SCM

Chloride

FOT Name: Solid and Chemical Materials, Organic

Method: 8015B

Matrix Type: NPW/SCM

Diesel range organics (DRO)

Gasoline range organics (GRO)

Method: 8015C

Matrix Type: NPW/SCM

Diesel range organics (DRO)

Gasoline range organics (GRO)

Method: 8015D

Matrix Type: NPW/SCM

Diesel range organics (DRO)

Gasoline range organics (GRO)

Method: 8081A

Matrix Type: NPW/SCM

4,4'-DDD

4,4'-DDE

4,4'-DDT

Alachlor

Aldrin

alpha-BHC

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2417 Bond Street
University Park, IL 60484

FOT Name: Solid and Chemical Materials, Organic

Method: 8081A

Matrix Type: NPW/SCM

- Atrazine
- Chlordane - not otherwise specified
- Dieldrin
- Endosulfan II
- Endrin
- Endrin ketone
- gamma-Chlordane
- Heptachlor epoxide
- Kepone
- Simazine
- alpha-Chlordane
- beta-BHC
- delta-BHC
- Endosulfan I
- Endosulfan sulfate
- Endrin aldehyde
- gamma-BHC (Lindane)
- Heptachlor
- Isodrin
- Methoxychlor
- Toxaphene

Method: 8081B

Matrix Type: NPW/SCM

- 4,4'-DDD
- 4,4'-DDT
- Aldrin
- alpha-Chlordane
- beta-BHC
- delta-BHC
- Endosulfan I
- Endosulfan sulfate
- Endrin aldehyde
- gamma-BHC (Lindane)
- Heptachlor
- Isodrin
- Methoxychlor
- Toxaphene
- 4,4'-DDE
- Alachlor
- alpha-BHC
- Atrazine
- Chlordane - not otherwise specified
- Dieldrin
- Endosulfan II
- Endrin
- Endrin ketone
- gamma-Chlordane
- Heptachlor epoxide
- Kepone
- Simazine

Method: 8082

Matrix Type: NPW/SCM

- PCB-1016
- PCB-1232
- PCB-1248
- PCB-1260
- PCB-1221
- PCB-1242
- PCB-1254

Method: 8082A

TestAmerica Chicago
2417 Bond Street
University Park, IL 60484

FOT Name: Solid and Chemical Materials, Organic **Method: 8082A**

Matrix Type: NPW/SCM

PCB-1016	PCB-1221
PCB-1232	PCB-1242
PCB-1248	PCB-1254
PCB-1260	

Method: 8151A

Matrix Type: NPW

4-Nitrophenol

Matrix Type: NPW/SCM

2,4,5-T	2,4,5-TP (Silvex)
2,4-D	2,4-DB
Dalapon	Dicamba
Dichloroprop	Dinoseb
Pentachlorophenol	Picloram

Method: 8260B

Matrix Type: NPW/SCM

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
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, MIBK	Acetone
Acetonitrile	Acrolein (Propenal)

TestAmerica Chicago
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University Park, IL 60484

FOT Name: Solid and Chemical Materials, Organic

Method: 8260B

Matrix Type: NPW/SCM

- Acrylonitrile
- Allyl chloride
- Benzene
- Benzyl chloride
- Bromobenzene
- Bromochloromethane
- Bromodichloromethane
- Bromoform
- Bromomethane
- Carbon disulfide
- Carbon tetrachloride
- Chlorobenzene
- Chlorodibromomethane (Dibromochloromethane)
- Chloroethane
- Chloroform
- Chloromethane
- Chloroprene
- cis-1,2-Dichloroethene
- cis-1,3-Dichloropropene
- Dibromomethane
- Dichlorodifluoromethane
- Dichloromethane (Methylene chloride)
- Diethyl ether
- Ethanol
- Ethyl acetate
- Ethyl ether
- Ethyl methacrylate
- Ethylbenzene
- Hexachlorobutadiene
- Isopropyl ether
- Isopropylbenzene
- Methacrylonitrile
- Methyl ethyl ketone
- Methyl iodide (Iodmethane)
- Methyl isobutyl ketone
- Methyl methacrylate
- Methyl-t-butyl ether
- m-Xylene
- Naphthalene
- n-Butanol
- n-Butylbenzene
- n-Propylbenzene
- o-Xylene
- Pentachloroethane
- p-Isopropyltoluene
- Propionitrile (Ethyl cyanide)
- p-Xylene
- sec-Butylbenzene
- Styrene
- t-Butyl alcohol
- 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)

Method: 8270C

Matrix Type: NPW/SCM

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FOT Name: Solid and Chemical Materials, Organic **Method: 8270C**

Matrix Type: NPW/SCM

1,2,4-Trichlorobenzene
1,2-Diphenylhydrazine
1,3-Dichlorobenzene
1,4-Dichlorobenzene
1,4-Dioxane
1,4-Phenylenediamine
1-Methylnaphthalene
2,3,4,6-Tetrachlorophenol
2,4,6-Trichlorophenol
2,4-Dimethylphenol
2,4-Dinitrotoluene (2,4-DNT)
2,6-Dinitrotoluene (2,6-DNT)
2-Chloronaphthalene
2-Methylnaphthalene
2-Naphthylamine
2-Nitrophenol
3,3'-Dimethylbenzidine
3-Nitroaniline
4-Aminobiphenyl
4-Chloro-3-methylphenol
4-Chlorophenyl phenyl ether
4-Nitrophenol
5-Nitro-o-toluidine
Acenaphthene
Acetophenone
Aniline
Aramite
Benzo(a)anthracene
Benzo(b)fluoranthene
Benzo(k)fluoranthene
Benzyl alcohol
Bis(2-chloroethyl) ether
Bis(2-ethylhexyl) phthalate
Carbazole
1,2,4,5-Tetrachlorobenzene
1,2-Dichlorobenzene
1,3,5-Trinitrobenzene (1,3,5-TNB)
1,3-Dinitrobenzene (1,3-DNB)
1,4-Dinitrobenzene
1,4-Naphthoquinone
1-Chloronaphthalene
1-Naphthylamine
2,4,5-Trichlorophenol
2,4-Dichlorophenol
2,4-Dinitrophenol
2,6-Dichlorophenol
2-Acetylaminofluorene
2-Chlorophenol
2-Methylpyridine (2-Picoline)
2-Nitroaniline
3,3'-Dichlorobenzidine
3-Methylcholanthrene
4,6-Dinitro-2-methylphenol
4-Bromophenyl phenyl ether
4-Chloroaniline
4-Nitroaniline
4-Nitroquinoline-1-oxide
7,12-Dimethylbenz(a)anthracene
Acenaphthylene
alpha,alpha-Dimethylphenethylamine
Anthracene
Benzidine
Benzo(a)pyrene
Benzo(g,h,i)perlyene
Benzoic acid
Bis(2-chloroethoxy) methane
Bis(2-chloroisopropyl) ether
Butyl benzyl phthalate
Carbofuran (Furaden)

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FOT Name: Solid and Chemical Materials, Organic

Method: 8270C

Matrix Type: NPW/SCM

- Chrysene
- Chlorobenzilate
- Dibenz(a,h)anthracene
- Diallate
- Dibenzofuran
- Dibenz(a,j)acridine
- Dimethoate
- Diethyl phthalate
- Di-n-butyl phthalate
- Dimethyl phthalate
- Dinoseb
- Di-n-octyl phthalate
- Ethyl methanesulfonate
- Diphenylamine
- Fluoranthene
- Famphur
- Hexachlorobenzene
- Fluorene
- Hexachlorocyclopentadiene
- Hexachlorobutadiene
- Hexachlorophene
- Hexachloroethane
- Indeno(1,2,3-cd) pyrene
- Hexachloropropene
- Isophorone
- Isodrin
- Kepone
- Isosafrole
- m-Dinitrobenzene
- m-Cresol (3-Methylphenol)
- Methyl methanesulfonate
- Methapyrilene
- Naphthalene
- Methyl parathion
- N-Nitrosodiethylamine
- Nitrobenzene
- N-Nitrosodi-n-butylamine (N-Nitrosodibutylamine)
- N-Nitrosodimethylamine
- N-Nitrosodiphenylamine
- N-Nitrosodi-n-propylamine
- N-Nitrosomorpholine
- N-Nitrosomethylethylamine
- N-Nitrosopyrrolidine
- N-Nitrosopiperidine
- o-Cresol (2-Methylphenol)
- O,O,O-Triethyl phosphorothioate
- Parathion
- o-Toluidine
- p-Dimethylaminoazobenzene
- p-Cresol (4-Methylphenol)
- Pentachloronitrobenzene
- Pentachlorobenzene
- Phenacetin
- Pentachlorophenol
- Phenol
- Phenanthrene
- p-Phenylenediame
- Phorate
- Pyrene
- Pronamide
- Safrole
- Pyridine
- Thionazine (Zinophos)

Method: 8270D

Matrix Type: NPW/SCM

- 1,2,4,5-Tetrachlorobenzene
- 1,2,4-Trichlorobenzene

Awards the Certificate of Approval

TestAmerica Chicago
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FOT Name: Solid and Chemical Materials, Organic **Method: 8270D**

Matrix Type: NPW/SCM

1,2-Diphenylhydrazine
1,3-Dichlorobenzene
1,4-Dichlorobenzene
1,4-Dioxane
1,4-Phenylenediamine
1-Methylnaphthalene
2,3,4,6-Tetrachlorophenol
2,4,6-Trichlorophenol
2,4-Dimethylphenol
2,4-Dinitrotoluene (2,4-DNT)
2,6-Dinitrotoluene (2,6-DNT)
2-Chloronaphthalene
2-Methylnaphthalene
2-Methylpyridine (2-Picoline)
2-Nitroaniline
3,3'-Dichlorobenzidine
3-Methylcholanthrene
3-Nitroaniline
4-Aminobiphenyl
4-Chloro-3-methylphenol
4-Chlorophenyl phenyl ether
4-Nitroaniline
4-Nitroquinoline-1-oxide
7,12-Dimethylbenz(a)anthracene
Acenaphthylene
alpha,alpha-Dimethylphenethylamine
Anthracene
Benzidine
Benzo(a)pyrene
Benzo(g,h,i)perlyene
Benzoic acid
Bis(2-chloroethoxy) methane
Bis(2-chloroisopropyl) ether
Butyl benzyl phthalate
1,2-Dichlorobenzene
1,3,5-Trinitrobenzene (1,3,5-TNB)
1,3-Dinitrobenzene (1,3-DNB)
1,4-Dinitrobenzene
1,4-Naphthoquinone
1-Chloronaphthalene
1-Naphthylamine
2,4,5-Trichlorophenol
2,4-Dichlorophenol
2,4-Dinitrophenol
2,6-Dichlorophenol
2-Acetylaminofluorene
2-Chlorophenol
2-Methylphenol (o-Cresol)
2-Naphthylamine
2-Nitrophenol
3,3'-Dimethylbenzidine
3-Methylphenol (m-Cresol)
4,6-Dinitro-2-methylphenol
4-Bromophenyl phenyl ether
4-Chloroaniline
4-Methylphenol (p-Cresol)
4-Nitrophenol
5-Nitro-o-toluidine
Acenaphthene
Acetophenone
Aniline
Aramite
Benzo(a)anthracene
Benzo(b)fluoranthene
Benzo(k)fluoranthene
Benzyl alcohol
Bis(2-chloroethyl) ether
Bis(2-ethylhexyl) phthalate
Carbazole

TestAmerica Chicago
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University Park, IL 60484

FOT Name: Solid and Chemical Materials, Organic

Matrix Type: NPW/SCM

Method: 8270D

- Chlorobenzilate
- Diallate
- Dibenz(a,j)acridine
- Diethyl phthalate
- Dimethyl phthalate
- Di-n-octyl phthalate
- Diphenylamine
- Famphur
- Fluorene
- Hexachlorobutadiene
- Hexachloroethane
- Hexachloropropene
- Isodrin
- Isosafrole
- m-Cresol (3-Methylphenol)
- Methapyrilene
- Methyl parathion
- Nitrobenzene
- N-Nitrosodimethylamine
- N-Nitrosodi-n-propylamine
- N-Nitrosomethylethylamine
- N-Nitrosopiperidine
- O,O,O-Triethyl phosphorothioate
- o-Toluidine
- p-Cresol (4-Methylphenol)
- Pentachlorobenzene
- Pentachlorophenol
- Phenanthrene
- Phorate
- Pronamide
- Pyridine
- Thionazine (Zinophos)
- Carbofuran (Furaden)
- Chrysene
- Dibenz(a,h)anthracene
- Dibenzofuran
- Dimethoate
- Di-n-butyl phthalate
- Dinoseb
- Ethyl methanesulfonate
- Fluoranthene
- Hexachlorobenzene
- Hexachlorocyclopentadiene
- Hexachlorophene
- Indeno(1,2,3-cd) pyrene
- Isophorone
- Kepone
- m-Dinitrobenzene
- Methyl methanesulfonate
- Naphthalene
- N-Nitrosodiethylamine
- N-Nitrosodi-n-butylamine (N-Nitrosodibutylamine)
- N-Nitrosodiphenylamine
- N-Nitrosomorpholine
- N-Nitrosopyrrolidine
- o-Cresol (2-Methylphenol)
- Parathion
- p-Dimethylaminoazobenzene
- Pentachloronitrobenzene
- Phenacetin
- Phenol
- p-Phenylenediame
- Pyrene
- Safrole

Method: 8310

Matrix Type: NPW

State of Illinois
Environmental Protection Agency

Certificate No.: 003642

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FOT Name: Solid and Chemical Materials, Organic

Method: 8310

Matrix Type: NPW

- Acenaphthylene
- Acenaphthene
- 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

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