ARCADIS BBL
Infrastructure, environment, facilities

Imagine the result

Chevron Environmental Management Company

Quality Assurance Project Plan

Former Tappan Terminal Site
Hastings on Hudson, New York

November 2007

Table of Contents

Tables

Attachments

Distribution List

Organization	Individual
ARCADIS of New York (ARCADIS BBL)	William McCune, Dennis K. Capria, Jo Ann Robertson
New York State Department of Environmental Conservation	William Ports
Test America	Jim Stellrecht, Verl Preston

FORMER TAPPAN TERMINAL SITE
HASTINGS ON HUDSON

QUALITY ASSURANCE PROJECT PLAN

Prepared By: ARCADIS BBL

Approved: Mark R Hendrickson
Project Manager – Mark Stella/Mark Hendrickson
Chevron Environmental Management Company

Approved: _____
Project Manager – William T. McCune
ARCADIS BBL

Approved: _____
Quality Assurance Coordinator – Dennis Capria
ARCADIS BBL

Approved: Tony Bogolin
Project Manager – Tony Bogolin
Test America

Approved: Verl O Preston
Quality Assurance Manager – Verl Preston
Test America

Preface

This *Quality Assurance Project Plan* (QAPP) was prepared for the former Tappan Terminal Site (the Site) located in Hastings on Hudson, New York. This QAPP will support future work plans for the Site.

This QAPP was prepared in a manner consistent with the following reference and guidance documents:

- United States Environmental Protection Agency (USEPA) guidance document entitled *EPA Requirements for Quality Assurance Project Plans*, EPA-QA/R-5 (USEPA, 2001), which replaces QAMS-005/80, *Interim Guidance and Specifications for Preparing Quality Assurance Project Plans* (USEPA, 1980)

- USEPA *Guidance for Quality Assurance Project Plans* (USEPA, 2002)

Information contained in this QAPP has been organized into the following sections:

Section	Content
Project Management	
1	Project Organization
2	Project Background
3	Project Description
4	Quality Objectives and Criteria for Measurement Data
5	Special Training Requirements/Certification
6	Documentation and Records
Measurement/Data Acquisition	
7	Sampling Process Design
8	Sampling Method Requirements
9	Sample Handling and Custody Requirements
10	Analytical Method Requirements
11	Quality Control Requirements
12	Instrument/Equipment Testing, Inspection, and Maintenance Requirements
13	Instrument Calibration and Frequency
14	Inspection/Acceptance Requirements for Supplies and Consumables
15	Data Acquisition Requirements for Non-Direct Measurements
16	Data Management

Section	Content
Assessment/Oversight	
17	Assessment and Response Actions
18	Reports to Management
Data Validation and Usability	
19	Data Reduction and Review
20	Data Validation and Verification
21	Reconciliation with User Requirements

Details on each of the subjects listed above are provided in the subsequent sections. This document also contains pertinent information from the Pre-Design Work Plans related to measuring and evaluating the analytical data.

Acronyms and Abbreviations

ASP	Analytical Services Protocol
CLP	Contract Laboratory Program
COC	Chain-of-Custody
DQOs	Data Quality Objectives
EDD	Electronic Data Deliverable
GC	Gas Chromatography
GC/MS	Gas Chromatography/Mass Spectrometry
GIS	Geographic Information System
MS	Matrix Spike
MSD	Matrix Spike Duplicate
NYSDEC	New York State Department of Environmental Conservation
OSHA	Occupational Safety and Health Administration
PCB	Polychlorinated biphenyl
QAC	Quality Assurance Coordinator
QAPP	Quality Assurance Project Plan
QA/QC	Quality Assurance/Quality Control
RPD	Relative percent difference
SDG	Sample Delivery Group
SOP	Standard Operating Procedure

SVOC	Semi-Volatile Organic Compound
TOC	Total Organic Carbon
USEPA	United States Environmental Protection Agency
VOC	Volatile Organic Compound

1. Project Organization

1.1 Project Organization

Investigations performed as part of the Pre-Design Investigation for the Former Tappan Terminal, located in Hastings on Hudson, New York, will require integration of personnel from the organizations identified below, collectively referred to as the "project team." A detailed description of the responsibilities of each member of the project team is presented below.

1.1.1 Overall Project Management

On behalf of Chevron Environmental Management Company (Chevron), ARCADIS BBL has overall responsibility for the Pre-Design Investigation activities. ARCADIS BBL personnel will perform related sampling activities, evaluate data, and prepare the deliverables as specified in the Pre-Design Work Plans. Project direction will be provided by Chevron, with oversight by the New York State Department of Environmental Conservation (NYSDEC). A list of key project management personnel is provided below.

Company/Organization	Title	Name	Phone Number
NYSDEC	Project Manager	William Ports	518.402.9667
	Quality Assurance Manager	TBD	TBD
Chevron	Project Coordinators	Mark Stella Mark Hendrickson	713.432.2643 713.432.2634
ARCADIS BBL	Project Officer	John Vogeley	925.274.1100
	Project Manager	William McCune	315.671.9172
	Field Manager	Jo Ann Robertson	315.671.9143
	Quality Assurance Coordinator	Dennis K. Capria	315.671.9299
Lab – Test America	Project Manager	Tony Bogolin	716.504.9833
	Quality Assurance Manager	Ms. Verl Preston	716.691.2600

1.1.2 Task Managers

The staff performing the investigations and site activities will be directed by representatives of the project team. The personnel responsible for each of the site activities are listed below.

Company/Organization	Title	Name	Phone Number
ARCADIS BBL	Field Task Manager	Jo Ann Robertson	315.671.9143
	Health and Safety Officer	Jay D. Keough, Certified Safety Professional	609.860.0590
	Database Administrator	TBA	
	Data Validator	TBA	

1.2 Team Member Responsibilities

The responsibilities of the various team members are summarized below by organization.

1.2.1 Chevron Environmental Management Company

Project Coordinator

Responsibilities and duties include:

- Provide overall direction of site actions.
- Direct ARCADIS BBL.
- Review ARCADIS BBL work products, including data, memoranda, letters, reports, and all other documents transmitted to the NYSDEC.

1.2.2 ARCADIS BBL

Project Officer

Responsibilities and duties include:

- Oversee ARCADIS BBL work products.
- Provide ARCADIS BBL approval for major project deliverables.

Project Manager

Responsibilities and duties include:

- Manage and coordinate the project as defined in the Pre-Design Investigation Work Plans, with an emphasis on adhering to the objectives of the site activities.
- Review documents prepared by ARCADIS BBL.
- Ensure that corrective actions are taken for deficiencies cited during any audits of site activities.

Task Managers

The Pre-Design Investigation components will be managed by various Task Managers, as set forth in Section 1.1.2. Duties of each Task Manager include, as appropriate:

- Manage relevant day-to-day activities.
- Develop, establish, and maintain files on relevant site activities.
- Review data reductions from the relevant site activities.
- Perform final data review of field data reductions and reports on relevant site activities.
- Ensure that corrective actions are taken for deficiencies cited during audits of relevant site activities.
- Perform overall quality assurance/quality control (QA/QC) of the relevant portions of the site activities.
- Review relevant field records and logs.
- Instruct personnel working on relevant site activities.
- Coordinate field and laboratory schedules pertaining to relevant site activities.
- Request sample bottles from laboratory.

- Review field instrumentation, maintenance, and calibration to meet quality objectives.
- Prepare reports pertaining to relevant site activities.
- Maintain field and laboratory files of notebooks/logs, data reductions, and calculations and transmit originals to the Project Manager.

Field Personnel

Responsibilities and duties include:

- Perform field procedures associated with the investigations as set forth in the Pre-Design Investigation Work Plans.
- Perform field analyses and collect quality assurance samples.
- Calibrate, operate, and maintain field equipment.
- Reduce field data.
- Maintain sample custody.
- Prepare field records and logs.

Quality Assurance Coordinator (QAC)

Responsibilities and duties include:

- Review laboratory data packages.
- Oversee and interface with the analytical laboratory.
- Coordinate field QA/QC procedures with Task Managers (including audits of field activities) concentrating on field analytical measurements and practices to meet data quality objectives (DQOs).
- Review field reports.
- Perform and review audit reports.

- Prepare interim QA/QC compliance reports.
- Prepare a QA/QC report in accordance with United States Environmental Protection (USEPA) Region II guidelines, which includes an evaluation of field and laboratory data and data usability reports.

1.2.3 Analytical Laboratories

General responsibilities and duties of the analytical laboratories include:

- Perform sample analyses and associated laboratory QA/QC procedures.
- Supply sampling containers and shipping cartons.
- Maintain laboratory custody of sample.
- Strictly adhere to all protocols in the QAPP.

Project Manager

Responsibilities and duties include:

- Serve as primary communication link between ARCADIS BBL and laboratory technical staff.
- Monitor workloads and ensure availability of resources.
- Oversee preparation of analytical reports.
- Supervise in-house chain-of-custody (COC).

Quality Assurance Manager

Responsibilities and duties include:

- Supervise personnel reviewing and inspecting all project-related laboratory activities.
- Conduct audits of all laboratory activities.

1.2.4 NYSDEC

Project Manager

Responsibilities and duties include:

- Provide NYSDEC review and approval of the Pre-Design Investigation Work Plans, supporting documents, and future deliverables.
- Monitor progress of site activities.

Quality Assurance Manager

Responsibilities and duties include:

- Review and approval of the QAPP.
- Review of the QA/QC portion of any submitted report.
- Monitor progress of the Pre-Design Investigation.
- Ensure that all activities are performed in compliance with applicable federal, state, and regional requirements.
- Perform field and laboratory audits, if necessary.

2. Project Background

2.1 Site Location and Description

The Tappan Terminal site is located on 15 acres along the Hudson River waterfront in the Village of Hastings-on-Hudson, Westchester County, New York. The site comprises two properties, the Exxon/Mobil property, which is located adjacent to the Hudson River, and the Uhlich Color Company, which is located along the railroad tracks that define the eastern boundary of the site. The Uhlich property is a former pigment manufacturing facility, and the Exxon/Mobil property was most recently used as a petroleum distribution terminal. The Uhlich Color Company was recently acquired by the Magruder Color Company, and has discontinued operations at the site. A small portion of the southern end of the Exxon/Mobil property is leased to the Pioneer Boat Club for use as a marina.

Limited access to the site is from Railroad Avenue at the southeast corner of the site and over the Zinsser Bridge that crosses the railroad tracks. Both portions of the site are surrounded by a chain link fence that is in good repair. This bridge has fallen into disrepair, and is no longer open to vehicular traffic.

2.2 Site History and Summary of Activities

The Uhlich property has been used for manufacturing and chemical use by several owners and occupants from 1897 to 2002. The property was created by disposal of manmade fill into the Hudson River between 1868 and 1970. The uses of the property have included: manufactured dye, pigments, and photographic chemicals, storage of trucks and materials, and the first floor of Building 50 has been used as a laboratory from 1962 to 1972. By 2003, all the buildings on the Uhlich property were demolished.

In 1961, the Tappan Tanker Terminal purchased the western section of the property and begun operating a petroleum distribution facility. In 1975, Mobil Oil Company purchased the terminal and continued operations until 1985.

2.3 Current Status

NYSDEC issued a Record of Decision for Former Tappan Terminal Site that included both the Uhlich and the Tappan Terminal Site in September 2006.

3. Project Description

This section presents a description of the investigation activities to be conducted during the Pre-Design Investigation. The soil investigation on the Uhlich Property is discussed in the Pre-Design Investigation Work Plan for the Uhlich Property and includes delineation of grossly contaminated soils. The Pre-Design Investigation for the chlorobenzene source area and the bench scale pilot studies is discussed in the Groundwater Pre-Design Investigation Work Plans.

Sampling activities associated with the Pre-Design Investigation will be conducted under the following tasks:

- Soil investigation
- Groundwater investigation
- Bench Scale and Pilot Studies

Sampling protocols to be followed during the investigation activities are detailed in the Pre-Design Investigation Work Plans. Samples collected during the investigation will be analyzed in accordance with USEPA SW-846 Test Methods for Evaluating Solid Waste, with NYSDEC Analytical Services Protocol (ASP) Revision 2005. Table 2 presents a list of the constituents that will be analyzed for samples collected as part of the Pre-Design Investigation. Health and safety protocols to be followed by field personnel during completion of the investigation activities are discussed in the Health and Safety Plan.

A brief description of the objectives for each task associated with the Pre-Design Investigation is presented below. A more detailed description can be found in the associated Pre-Design Investigation Work Plans.

3.1 Soil Investigation

The objectives of the soil investigation are to:

- Define the extent of the grossly containments soils on the Uhlich property.
- Collect soil samples from the chlorobenzene source area for characterization.

In addition to the objectives outlined above, the subsurface information collected as part of this investigation will be used to characterize chlorobenzene source area.

3.2 Groundwater Investigation

The objectives of the groundwater investigation are to:

- Define the vertical and horizontal extent of the chlorobenzene plume.
- Collect groundwater samples to evaluate the natural attention of the chlorobenzene plume.
- Collect groundwater samples to evaluate the effectiveness of the remedial measures.

3.3 Project Schedule

The project schedule is presented in the Pre-Design Investigation Work Plans.

4. Quality Objectives and Criteria for Measurement Data

The DQO process, as described in the USEPA EPA QA/G-4 QAPP (2006) instructions document, is intended to provide a "logical framework" for planning field investigations. The following section addresses, in turn, each of the seven sequential steps in the EPA QA/G-4 QAPP DQO process.

Step 1: State the Problem

The Pre-Design Investigation will be conducted at the Former Tappan Terminal Site and the Uhlich Property in three phases, including source investigations, bench scale, and pilot testing. The sampling and analysis program is intended to generate data evaluate remedial measures for treatment the source areas.

Step 2: Identify the Goal of the Study

The initial use of the data is descriptive (distribution and concentration) and there is no decision point for this descriptive application. Subsequent to review of the descriptive information, an exposure evaluation will be performed based on the findings of the Site investigation.

Step 3: Identify Information Inputs

Decision inputs incorporate both concentration and distribution of constitutes of concern in site media. A fundamental basis for decision-making is that a sufficient number of data points of acceptable quality are available from the investigation to support the decision. Thus, the necessary inputs for the decision are: 1) the proportion of non-rejected (usable) data points; and 2) the quantity of data needed to evaluate remedial measures.

The data will be evaluated for completeness, general conformance with requirements of this QAPP, and consistency among data sets and with historical data, as appropriate.

Step 4: Define the Boundaries of the Study

The Tappan Terminal site is located on 15 acres along the Hudson River waterfront in the Village of Hastings-on-Hudson, Westchester County, New York. The site comprises two properties, the Exxon/Mobil property, which is located adjacent to the

Hudson River, and the Uhlich Color Company, which is located along the railroad tracks that define the eastern boundary of the site.

Step 5: Developing the Analytical Approach

The decision on whether data can be used in the exposure evaluation will be based on the validation results. Following validation, the data will be flagged, as appropriate, and any use restrictions noted. The sampling plan has been devised so that the loss of any single data point will not hinder description of the distribution of constitutes of concern or the development of a risk assessment. Given this, a reasonable decision rule would be that 90% of the data points not be rejected and deemed unusable for exposure evaluation purposes. Applicable actions would be evaluated, if needed based on the results of the exposure evaluation.

Step 6: Specify Performance or Acceptance Criteria

Specifications for this step call for: 1) giving forethought to corrective actions to improve data usability; and 2) understanding the representative nature of the sampling design. This QAPP has been designed to meet both specifications for this step. The sampling and analysis program has been developed based on a review of previous site data and knowledge of present Site conditions. Corrective actions are described elsewhere in the document and in the appended documents. The representative nature of the sampling design has been assured by discussions among professionals familiar with the Site and the appropriate government agencies.

Step 7: Develop the Plan for Obtaining Data

The overall quality assurance objective is to develop and implement procedures for field sampling; COC, laboratory analysis, and reporting that will provide results to support the evaluation of the site data consistent with National Contingency Plan requirements. Specific procedures for sampling, COC, laboratory instrument calibration, laboratory analysis, data reporting, internal quality control, audits, preventive.

A DQO summary for the sampling investigation efforts is presented in the subsequent section. The summary consists of stated DQOs relative to data uses, data types, data quantity, sampling and analytical methods, and data measurement performance criteria.

4.1 Data Categories

Three data categories have been defined to address various analytical data uses and the associated QA/QC effort and methods required to achieve the desired levels of quality. These categories are:

Screening Data: Screening data affords a quick assessment of site characteristics or conditions. This DQO is applicable to data collection activities that involve rapid, non-rigorous methods of analysis and quality assurance. This objective is generally applied to physical and/or chemical properties of samples, degree of contamination relative to concentration differences, and preliminary health and safety assessment.

Screening Data with Definitive Confirmation: Screening data allows rapid identification and quantitation, although the quantitation can be relatively imprecise. This DQO is available for data collection activities that require qualitative and/or quantitative verification of a select portion of sample findings (10% or more). This objective can also be used to verify less rigorous laboratory-based methods.

Definitive Data: Definitive data are generated using analytical methods such as approved USEPA reference methods. Data are analyte-specific, with confirmation of analyte identity and concentration. Methods produce raw data (e.g., chromatograms, spectra, digital values) in the form of paper printouts or computer-generated electronic files.

It is anticipated that both screening and definitive data categories will be used during the investigation. Field parameters (e.g., turbidity, conductivity, temperature, and pH) which will be obtained during water column sampling for use in qualitatively interpreting other site data will be determined using screening techniques. All remaining parameters will be determined using definitive techniques.

For this project, three levels of data reporting have been defined. They are as follows:

Level 1 – Minimal Reporting: Minimal or "results only" reporting is used for analyses that, either due to their nature (i.e., field monitoring) or the intended data use (i.e., preliminary screening), do not generate or require extensive supporting documentation.

Level 2 – Modified Reporting: Modified reporting is used for analyses that are performed following standard USEPA-approved methods and QA/QC protocols and

that, based on the intended data use, require some supporting documentation but not, however, full "Contract Laboratory Program (CLP)-type" reporting.

Level 3 – Full Reporting: Full "CLP-type" reporting is used for those analyses that, based on intended data use, require full documentation. This reporting level would include ASP Superfund and Category B reporting.

The analytical methods to be used during the Pre-Design Investigation will be USEPA SW-846 methods with NYSDEC ASP Revision 2005, QA/QC requirement, and Category B reporting deliverables.

4.2 Field Investigations

As part of the Pre-Design Investigation, field investigations will be conducted to support the DQOs. Details of the field sampling investigations are described in the Pre-Design Investigation Work Plans.

5. Special Training Requirements/Certification

In compliance with the Occupational Safety and Health Administration's (OSHA) final rule, "Hazardous Waste

Operations and Emergency Response," 29CFR§1910.120(e), all personnel performing Pre-Design Investigation activities at the Site will have completed the requirements for OSHA 40-Hour Hazardous Waste Operations and Emergency Response training. Persons in field supervisory positions will have also completed the additional OSHA 8-Hour Supervisory Training.

6. Documentation and Records

6.1 General

Samples of the various media will be collected as described in the Pre-Design Investigation Work Plans. Detailed descriptions of the documentation and reporting requirements are presented below.

6.2 Sample Designation System

6.2.1 Sample Codes

Samples will be identified with a unique designation system that will facilitate sample tracking. The sample designation system to be employed during the sampling activities will be consistent, yet flexible enough to accommodate unforeseen sampling events and conditions. An alpha-numeric system is considered appropriate and will be used by field personnel to assign each sample with a unique sample identification number. The sample identification number will begin with a two-letter prefix indicating the sample location Uhlich Color Property – “UC” and the Former Tappan Terminal Property – “TT” , followed by two letters indicating the sample type and two digits indicating the sequential sample number collected from the location.

The samples types will be designated using the following codes:

- Surface Soil – “SS”
- Soil Boring – “SB”
- Groundwater – “MW”
- Trip Blank – “TB”
- Equipment Blank – “EB”

The two-digit sample number beginning with “01” will be assigned in the field and incremented by one as samples are collected from one to the next.

- Where necessary, the code system will be supplemented to accommodate additional sample identification information. For example, the code for soil samples will include a qualifier to identify the section increment (e.g., 0 to 0.5 feet).

Additional sample volumes collected for matrix spike (MS) and matrix spike duplicate (MSD) analysis will be noted on the COC forms, and the associated additional sample containers will be labeled with the appropriate suffix (MS or MSD). Rinse blanks will use to same coding scheme noted above, substituting the location code with the prefix "RB" (e.g., the first rinse blank associated with soil collection would be named RBSD01). Field duplicates will be labeled as ordinary field samples with a unique identification number (e.g., the first field duplicate associated collection would be named DUPSB01). Duplicate samples will not be identified and the laboratory will analyze them as "blind" quality control samples.

6.2.2 Field Documentation

Field personnel will provide comprehensive documentation covering all aspects of field sampling, field analysis, and sample COC. This documentation constitutes of a record that allows reconstruction of all field events to aid in the data review and interpretation process. All documents, records, and information relating to the performance of the field work will be retained in the project file.

The various forms of documentation to be maintained throughout the action include:

- Daily Production Documentation - A field notebook consisting of a waterproof, bound notebook that will contain a record of all activities performed at the Site.

- Sampling Information - Detailed notes will be made as to the exact sampling location, physical observations, and weather conditions (as appropriate).

- Sample COC – COC forms will provide the record of responsibility for sample collection, transport, and submittal to the laboratory. COC forms will be filled out at each sampling site, at a group of sampling sites, or at the end of each day of sampling by ARCADIS BBL's field personnel designated to be responsible for sample custody. In the event the samples are relinquished by the designated sampling person to other sampling or field personnel, the COC form will be signed and dated by the appropriate personnel to document the sample transfer. The original COC form will accompany the samples to the laboratory, and copies will be forwarded to the project files. A sample COC form is included in Attachment 3.

Persons will have custody of samples when the samples are in their physical possession, in their view after being in their possession, or in their physical possession and secured so they cannot be tampered with. In addition, when samples are secured in a restricted area accessible only to authorized personnel, they will be deemed to be in the custody of such authorized personnel.

- Field Equipment, Calibration, and Maintenance Logs - To document the calibration and maintenance of field instrumentation, calibration and maintenance logs will be maintained for each piece of field equipment that is not factory-calibrated.

6.3 Laboratory Documentation Files

6.3.1 Laboratory Project Files

The laboratory will establish a file for all pertinent data. The file will include all correspondence, faxed information, phone logs, and COC forms. The laboratory will retain all project files and data packages for a period of 5 years.

6.3.2 Laboratory Logbooks

Workbooks, bench sheets, instrument logbooks, and instrument printouts will be used to trace the history of samples through the analytical process and document important aspects of the work, including the associated quality controls. As such, logbooks, bench sheets, instrument logs, and instrument printouts will be part of the permanent record of the laboratory.

Each page or entry will be dated and initialed by the analyst at the time of entry. Errors in entry will be crossed out in indelible ink with a single stroke, corrected without the use of white-out or by obliterating or writing directly over the erroneous entry, and initialed and dated by the individual making the correction. Pages of logbooks that are not used will be completed by lining out unused portions.

Information regarding the sample, analytical procedures performed, and the results of the testing will be recorded on laboratory forms or personal notebook pages by the analyst. These notes will be dated and will also identify the analyst, the instrument used, and the instrument conditions.

Laboratory notebooks will be periodically reviewed by the laboratory group leaders for accuracy, completeness, and compliance to this QAPP. All entries and calculations will

be verified by the laboratory group leader. If all entries on the pages are correct, then the laboratory group leader will initial and date the pages. Corrective action will be taken for incorrect entries before the laboratory group leader signs.

6.3.3 Computer Tape and Hard Copy Storage

All electronic files and deliverables will be retained by the laboratory for not less than 5 years; hard copy data packages (or electronic copies) will also be retained for not less than 5 years.

6.4 Data Reporting Requirements

Data will be reported both in the field and by the analytical laboratory, as described below.

6.4.1 Field Data Reporting

Information collected in the field through visual observation, manual measurement, and/or field instrumentation will be recorded in field notebooks or data sheets and/or on forms. Such data will be reviewed by the appropriate Task Manager for adherence to the Pre-Design Investigation Work Plans and for consistency. Concerns identified as a result of this review will be discussed with the field personnel, corrected if possible, and, as necessary, incorporated into the data evaluation process.

If applicable, field data forms and calculations will be processed and included in appendices to the appropriate reports (when generated). The original field logs, documents, and data reductions will be kept in the project file at the ARCADIS BBL office in Syracuse, New York.

6.4.2 Laboratory Data Reporting

The laboratory is responsible for preparing ASP Category B data packages for all volatile organic compounds (VOCs), semi-volatile organic compounds (SVOCs), inorganics, total cyanide and total organic carbon (TOC) data, reduced data packages, and case narratives for all other analyses.

All data reports for all parameters will include, at a minimum, the following items:

Narrative: Summary of activities that took place during the course of sample analysis, including the following information:

- laboratory name and address
- date of sample receipt
- cross reference of laboratory identification number to contractor sample identification
- analytical methods used
- deviations from specified protocol
- corrective actions taken

Analytical Results: Reported according to analysis type and including the following information, as acceptable:

- sample ID
- laboratory ID
- date of collection
- date of receipt
- date of extraction
- date of analysis
- detection limits

Sample results on the report forms will be collected for dilutions. Soil samples will be reported on a dry weight basis. Unless otherwise specified, results will be reported uncorrected for blank contamination.

The data for VOCs, SVOCs, inorganics, and TOC analyses will be expanded to include all supporting documentation necessary to provide a Category B package. This additional documentation will include, but is not limited to, all raw data required to recalculate any result, including printouts, chromatograms, and quantitation reports. The report also will include standards used in calibration and calculation of analytical results; sample extraction, digestion, and other preparation logs; standard preparation logs, instrument run logs; and moisture content calculations.

6.5 Project File

Project documentation will be placed in project files according to ARCADIS BBL requirements identified in the corporate quality procedure (QP 1.02) for document management. Project files typically consist of the following components:

1. Agreements/Proposals (filed chronologically)
2. Change Orders/Purchase Orders (filed chronologically)
3. Invoices (filed chronologically)
4. Project Management (filed by topic)
5. Correspondence (filed chronologically)
6. Notes and Data (filed by topic)
7. Public Relations Information (filed by topic)
8. Regulatory Documents (filed chronologically)
9. Marketing Documents (filed chronologically)
10. Final Reports/Presentations (filed chronologically)
11. Draft Reports/Presentations (filed chronologically)
12. Documents Prepared by Others (filed chronologically)

Final reports (including QAPPs and quality assurance reports) are filed in folder #10 – Final Reports/Presentations. Analytical laboratory documentation (when received) and field data are filed in folder #6 – Notes and Data. Filed materials may be removed and signed out by authorized personnel on a temporary basis only.

7. Sampling Process Design

Information regarding the sampling design and rationale and associated sampling locations can be found in the Pre-Design Investigation Work Plans.

8. Sampling Method Requirements

Source materials, groundwater and soil samples will be collected as described in the Pre-Design Investigation Work Plans. The Pre-Design Investigation Work Plans also contain procedures that will be followed to drill and sample soil borings; install and develop monitoring wells; measure water levels; collect groundwater samples; perform field measurements; and handle, package, and ship collected samples.

9. Sample Handling and Custody Requirements

9.1 Sample Containers and Preservation

Appropriate sample containers, preservation methods, and laboratory holding times for Pre-Design Investigation samples are shown in Table 4.

The analytical laboratory will supply appropriate sample containers and preservatives, as necessary. The bottles will be purchased pre-cleaned according to USEPA Office of Solid Waste and Emergency Response Directive 9240.05A requirements. The field personnel will be responsible for properly labeling containers and preserving samples (as appropriate). Sample labeling procedures are discussed in Section 9.2.2.

9.2 Field Custody Procedures

The objective of field sample custody is to assure that samples are not tampered with from the time of sample collection through time of transport to the analytical laboratory. Persons will have "custody of samples" when the samples are in their physical possession, in their view after being in their possession, or in their physical possession and secured so they cannot be tampered with. In addition, when samples are secured in a restricted area accessible only to authorized personnel, they will be deemed to be in the custody of such authorized personnel.

Field custody documentation consists of both field logbooks and field COC forms.

9.2.1 Field Logbooks

Field logbooks will provide the means of recording data collecting activities performed. As such, entries will be described in as much detail as possible so that persons going to the Site could reconstruct a particular situation without reliance on memory.

Field logbooks will be bound field survey books or notebooks. Logbooks will be assigned to field personnel, but will be stored in a secure location when not in use. Each logbook will be identified by the project-specific document number. The title page of each logbook will contain the following:

- person to whom the logbook is assigned
- logbook number

- project name
- project start date
- end date

Entries into the logbook will contain a variety of information. At the beginning of each entry, the date, start time, weather, names of all sampling team members present, level of personal protection being used, and the signature of the person making the entry will be entered. The names of visitors to the Site, field sampling or investigation team personnel, and the purpose of their visit will also be recorded in the field logbook.

Measurements made and samples collected will be recorded. Entries will be made in ink, and no erasures will be made. If an incorrect entry is made, the information will be crossed out with a single strike mark. Whenever a sample is collected or a measurement is made, a detailed description of the location of the station shall be recorded. The number of the photographs taken of the station, if any, will also be noted. All equipment used to make measurements will be identified, along with the date of calibration.

Samples will be collected following the sampling procedures documented in the Pre-Design Investigation Work Plans. The equipment used to collect samples will be noted, along with the time of sampling, sample description, depth at which the sample was collected, volume, and number of containers. Sample identification numbers will be assigned prior to sample collection. Field duplicate samples, which will receive an entirely separate sample identification number, will be noted under sample description.

9.2.2 Sample Labeling

Preprinted sample labels will be affixed to sample bottles prior to delivery at the sampling site. The following information is required on each sample label:

- project
- date collected
- time collected
- location

- sampler
- analysis to be performed
- preservative
- sample number

9.2.3 Field COC Forms

Completed COC forms will be required for all samples to be analyzed. COC forms will be initiated by the sampling crew in the field. The COC forms will contain the unique sample identification number, sample date and time, sample description, sample type, preservation (if any), and analyses required. The original COC form will accompany the samples to the laboratory. Copies of the COC will be made prior to shipment (or multiple copy forms used) for field documentation. The COC forms will remain with the samples at all times. The samples and signed COC forms will remain in the possession of the sampling crew until the samples are delivered to the express carrier (e.g., Federal Express) or hand delivered to a mobile or permanent laboratory, or placed in secure storage.

Sample labels will be completed for each sample using waterproof ink. The labels will include sample information such as: sample number and location, type of sample, date and time of sampling, sampler's name or initials, preservation, and analyses to be performed. The completed sample labels will be affixed to each sample bottle and covered with clear tape.

Whenever samples are split with a government agency or other party, a separate COC will be prepared for those samples and marked to indicate with whom the samples are being split. The person relinquishing the samples to the facility or agency should request the representative's signature acknowledging sample receipt. If the representative is unavailable or refuses, this is noted in the "Received By" space.

9.3 Management of Investigation Derived Materials and Wastes

Management of investigation-derived materials and wastes will be performed consistent with the USEPA guidance *Guide to Management of Investigation – Derived Wastes,* 9345.3-03FS, dated January 1992. Disposable equipment (including personal protective equipment) and debris will be containerized and appropriately labeled during the sampling events, and will be disposed of accordingly. All purged groundwater and

water generated during equipment decontamination will be containerized and temporally staged onsite in a 55-gallon drum, and will be disposed of appropriately based on analytical results. Equipment will be decontaminated, as appropriate, as discussed in the Pre-Design Investigation Work Plans. All soil cuttings associated with drilling of soil borings will also be collected and temporally stored onsite in a 55-gallon drum(s), and disposed of properly following receipt of analytical results.

9.4 Packing, Handling, and Shipping Requirements

Sample packaging and shipment procedures are designed to insure that the samples will arrive at the laboratory, with the COC, intact.

Samples will be packaged for shipment as outlined below:

- Ensure that sample containers have the sample labels securely affixed to the container with clear packing tape.
- Check the caps on the sample containers to ensure that they are properly sealed.
- Wrap the sample container cap with clear packing tape to prevent it from becoming loose.
- Complete the COC form with the required sampling information and ensure that the recorded information matches the sample labels. NOTE: If the designated sampler relinquishes the samples to other sampling or field personnel for packing or other purposes, the sampler will complete the COC prior to this transfer. The appropriate personnel will sign and date the COC form to document the sample custody transfer.
- Using duct tape, secure the outside drain plug at the bottom of the cooler.
- Wrap sample containers in bubble wrap or other cushioning material.
- Place 1 to 2 inches of cushioning material at the bottom of the cooler.
- Place the sealed sample containers into the cooler.
- Place ice in plastic bags and seal. Place loosely in the cooler.
- Fill the remaining space in the cooler with cushioning material.

- Place COC forms in a plastic bag and seal. Tape the forms to the inside of the cooler lid.

- Close the lid of the cooler, lock, and secure with duct tape.

- Wrap strapping tape around both ends of the cooler at least twice.

- Mark the cooler on the outside with the following information: shipping address, return address, “Fragile” labels, and arrows indicating “this side up.” Cover the labels with clear plastic tape. Place a signed custody seal over the sample cooler lid.

Samples will be hand-delivered or delivered by an express carrier within 48 hours of the time of collection. Shipments will be accompanied by the COC form identifying the contents. The original form will accompany the shipment; copies will be retained by the sampler for the sampling office records. If the samples are sent by common carrier, a bill of lading will be used. Receipts or bills of lading will be retained as part of the permanent project documentation. Commercial carriers are not required to sign off on the COC form as long as the forms are sealed inside the sample cooler and the custody seals remain intact.

Sample custody seals and packing materials for filled sample containers will be provided by the analytical laboratory. The filled, labeled, and sealed containers will be placed in a cooler on ice and carefully packed to eliminate the possibility of container breakage.

Additional procedures for packing, handling, and shipping environmental samples are presented in the Pre-Design Investigation Work Plans.

9.5 Laboratory Custody Procedures

9.5.1 General

Upon sample receipt, laboratory personnel will be responsible for sample custody. The original field COC form will accompany all samples requiring laboratory analysis. The laboratory will use COC guidelines described in the USEPA guidance documents. Samples will be kept secured in the laboratory until all stages of analysis are complete. All laboratory personnel having samples in their custody will be responsible for documenting and maintaining sample integrity.

9.5.2 Sample Receipt and Storage

Immediately upon sample receipt, the laboratory sample custodian will verify the cooler seal, open the cooler, and compare the contents against the field COC. If a sample container is missing, a sample container is received broken, the sample is in an inappropriate container, or has not been preserved by appropriate means, ARCADIS BBL will be notified. The laboratory sample custodian will be responsible for logging the samples in, assigning a unique laboratory identification number to each sample, labeling the sample bottle with the laboratory identification number, and moving the sample to an appropriate storage location to await analysis. The project name, field sample code, date sampled, date received, analysis required, storage location and date, and action for final disposition will be recorded in the laboratory tracking system. Relevant custody documentation will be placed in the project file.

9.5.3 Sample Analysis

Analysis of an acceptable sample will be initiated by worksheets that contain all pertinent information for analysis.

Samples will be organized into sample delivery groups (SDGs) by the laboratory. A SDG may contain up to 20 field samples (field duplicates, trip blanks, and rinse blanks are considered field samples for the purposes of SDG assignment). All field samples assigned to a single SDG shall be received by the laboratory over a maximum of 7 calendar days and must be processed through the laboratory (preparation, analysis, and reporting) as a group. Every SDG must include a minimum of one site-specific MS/MSD pair, which shall be received by the laboratory at the start of the SDG assignment.

9.5.4 Sample Storage Following Analysis

Samples will be maintained by the laboratory for at least one month after the final report is delivered to ARCADIS BBL. The laboratory will be responsible for the eventual and appropriate disposal of the samples. The analytical laboratory will inform ARCADIS BBL before any samples are disposed. Unused portions of the samples, sample extracts and associated wastes will be disposed of by the laboratory in accordance with applicable rules and regulations as specified in their standard operating procedure (SOP) for waste disposal.

10. Analytical Method Requirements

10.1 Field Parameters and Methods

Field analytical procedures will include the measurement of pH, turbidity, temperature, conductivity, and groundwater levels. Specific field measurement protocols are provided in the Pre-Design Investigation Work Plans.

10.2 Laboratory Parameters and Methods

The methods listed below include the range of analyses expected to be performed. The associated laboratory SOPs can be found in Attachment 1.

Laboratory analytical requirements presented in the sub-sections below include a general summary of requirements, specifics related to each sample medium to be analyzed, and details of the methods to be used for this project. SW-846 methods with NYSDEC ASP 2005 Revision, QA/QC, and reporting deliverables requirements will be used for all analytes.

EPA Method 8270 has been modified to include analysis of the following SVOCs: 9,10-anthracenedione, 1,4-dihydroxy-9,10-anthracenedione, 1-hydroxy-9,10-anthracenedione, 0-chloroaniline, (z)-9-octadecenamide, 2-methyl-benzenamine and p-aminotoluene. The calibration curves, purity of standard documentation, and the demonstration of capability for the additional SVOCs is included in Attachment 1 along with the SOP for EPA Method 8270.

10.2.1 General

The following tables summarize general analytical requirements:

Table	Title
Table 1	Environmental and Quality Control Sample Analyses
Table 3	Parameters, Methods, and Quantitation Limits
Table 4	Sample Containers, Preservation Methods, and Holding Times Requirements

10.2.2 Pre-Design Investigation Sample Matrices

10.2.2.1 Groundwater

Analyses will be performed following the methods listed in Table 1. Analytical results for all analyses will be reported in units identified in Table 3.

10.2.2.2 Soil

Analyses in this category will relate to soil samples. Analyses will be performed following the methods listed in Table 1. Results will be reported as dry weight, in units presented in Table 3. Moisture content will be reported separately.

10.3 Analytical Requirements

The primary sources to describe the analytical methods to be used during the investigation are provided in USEPA SW-846 Test Methods for Evaluating Solid Waste, Third Edition, and USEPA Methods for Chemical Analysis of Water and Waste with NYSDEC ASP 2005 Revision, QA/QC, and reporting deliverables requirements. Detailed information regarding QA/QC is provided in NYSDEC ASP 2005 Revision, Exhibits D and E.

11. Quality Control Requirements

11.1 Quality Assurance Indictors

The overall quality assurance objective for this QAPP is to develop and implement procedures for sampling, COC, laboratory analysis, instrument calibration, data reduction and reporting, internal quality control, audits, preventive maintenance, and corrective action, such that valid data will be generated. These procedures are presented or referenced in the following sections of the QAPP. Specific quality control checks are discussed in Section 11.2.

Quality assurance indicators are generally defined in terms of five parameters:

1. Representativeness
2. Comparability
3. Completeness
4. Precision
5. Accuracy

Each parameter is defined below. Specific objectives for the site actions are set forth in other sections of this QAPP as referenced below.

11.1.1 Representativeness

Representativeness is the degree to which sampling data accurately and precisely represent site conditions, and is dependent on sampling and analytical variability and the variability of environmental media at the Site. The actions have been designed to assess the presence of the chemical constituents at the time of sampling. The Pre-Design Investigation Work Plans present the rationale for sample quantities and location. This QAPP presents field sampling and laboratory analytical methodologies. The use of the prescribed field and laboratory analytical methods with associated holding times and preservation requirements are intended to provide representative data.

11.1.2 Comparability

Comparability is the degree of confidence with which one data set can be compared to another. Comparability between this investigation, and to the extent possible, with existing data will be maintained through consistent sampling and analytical methodology set forth in the Pre-Design Investigation Work Plans and this QAPP, SW-846 analytical methods with NYSDEC ASP Revision 2005, QA/QC requirements, and Category B reporting deliverables, and through use of QA/QC procedures and appropriately trained personnel.

11.1.3 Completeness

Completeness is defined as a measure of the amount of valid data obtained from an event and/or investigation compared to the total amount that was obtained. This will be determined upon final assessment of the analytical results, as discussed in Section 11.6.

11.1.4 Precision

Precision is the measure of reproducibility of sample results. The goal is to maintain a level of analytical precision consistent with the project objectives. To maximize precision, sampling and analytical procedures will be followed. All work for this investigation will adhere to established protocols presented in the Pre-Design Investigation Work Plans. Checks for analytical precision will include the analysis of MSDs, laboratory duplicates, and field duplicates. Checks for field measurement precision will include obtaining duplicate field measurements. Further discussion of precision quality control checks is provided in Section 11.4.

11.1.5 Accuracy

Accuracy is the deviation of a measurement from the true value of a known standard. Both field and analytical accuracy will be monitored through initial and continuing calibration of instruments. In addition, internal standards, MSs, blank spikes, and surrogates (system monitoring compounds) will be used to assess the accuracy of the laboratory analytical data. Further discussion of these quality control samples is provided in Section 11.5.

11.2 Field Quality Control Checks

11.2.1 Field Measurements

To verify the quality of data using field instrumentation, duplicate measurements will be obtained and reported for all field measurements. A duplicate measurement will involve obtaining measurements a second time at the same sampling location.

11.2.2 Sample Containers

Certified-clean sample containers in accordance with Exhibit I of the NYSDEC ASP Revision 2005 (Eagle Picher pre-cleaned containers or equivalent) will be supplied by the laboratory.

11.2.3 Field Duplicates

Field duplicates will be collected from the different site materials to verify the reproducibility of the sampling methods. Field duplicates will be prepared by placing well homogenized aliquots (except samples for VOC analysis) from the same sample location into individual sample containers, which are submitted blind to the laboratory. Field duplicate water samples and soil samples for VOC analysis will constitute co-located samples rather than homogenized aliquots. In general, field duplicates will be analyzed at a 5% frequency (every 20 samples) for the chemical constituents. Table 1 provides an estimated number of field duplicates to be prepared for each applicable parameter and matrix.

11.2.4 Rinse Blanks

Rinse blanks are used to monitor the cleanliness of the sampling equipment and the effectiveness of the cleaning procedures. Rinse blanks will be prepared and submitted for analysis once per day per matrix. Rinse blanks will be prepared by filling sample containers with analyte-free water (supplied by the laboratory) which has been routed through a cleaned sampling device. When dedicated sampling devices or sample containers are used to collect the samples, rinse blanks will not be necessary. Table 1 provides an estimated number of rinse blanks for environmental media samples to be collected during the Pre-Design Investigation.

11.2.5 Trip Blanks

Trip blanks will be used to assess whether site samples have been exposed to non-site-related volatile constituents during storage and transport. Trip blanks will be analyzed at a frequency of once per day, per cooler containing samples to be analyzed for volatile organic constituents. A trip blank will consist of a container filled with analyte-free water (supplied by the laboratory) which remains unopened with field samples throughout the sampling event. Trip blanks will only be analyzed for VOCs. Table 1 provides an estimated number of trip blanks collected for each matrix and parameter during the Pre-Design Investigation.

11.3 Analytical Laboratory Quality Control Checks

11.3.1 General

Internal laboratory quality control checks will be used to monitor data integrity. These checks will include method blanks, MS/MSDs, spike blanks, internal standards, surrogate samples, calibration standards, and reference standards. Project quality control limits for duplicates and MSs are identified in Table 2. Laboratory control charts will be used to determine long-term instrument trends.

11.3.2 Method Blanks

Sources of contamination in the analytical process, whether specific analyses or interferences, need to be identified, isolated, and corrected. The method blank is useful in identifying possible sources of contamination within the analytical process. For this reason, it is necessary that the method blank is initiated at the beginning of the analytical process and encompasses all aspects of the analytical work. As such, the method blank would assist in accounting for any potential contamination attributable to glassware, reagents, instrumentation, or other sources which could affect sample analysis. One method blank will be analyzed with each analytical series associated with no more than 20 samples.

11.3.3 MS/MSDs

MS/MSDs will be used to measure the accuracy of analyte recovery from the sample matrices and will be site-specific. MS/MSD pairs will be analyzed at a 5% frequency (every 20 samples or once every week, whichever comes first).

When MS recoveries are outside quality control limits, associated control sample and surrogate spike recoveries will be evaluated, as applicable, to attempt to verify the reason for the deviation and determine the effect on the reported sample results. Table 1 presents an estimated number of MS and MSD analyses for each applicable parameter.

11.3.4 Surrogate Spikes

Surrogates are compounds which are unlikely to occur under natural conditions that have properties similar to the analytes of interest. This type of control is primarily used for organic samples analyzed by gas chromatography/mass spectrometry (GC/MS) and GC methods and is added to the samples prior to purging or extraction. The surrogate spike is utilized to provide broader insight into the proficiency and efficiency of an analytical method on a sample-specific basis. This control reflects analytical conditions that may not be attributable to sample matrix.

If surrogate spike recoveries exceed specified quality control limits, the analytical results need to be evaluated thoroughly in conjunction with other control measures. In the absence of other control measures, the integrity of the data may not be verifiable and reanalysis of the samples with additional control may be necessary.

Surrogate spike compounds will be selected utilizing the guidance provided in the analytical methods.

11.3.5 Laboratory Duplicates

For inorganics, laboratory duplicates will be analyzed to assess laboratory precision. Laboratory duplicates are defined as a separate aliquot of an individual sample that is analyzed as a separate sample. Table 1 presents an estimated number of laboratory duplicates for each applicable parameter.

11.3.6 Calibration Standards

Calibration check standards analyzed within a particular analytical series provide insight regarding the instruments' stability. A calibration check standard will be analyzed at the beginning and end of an analytical series, or periodically throughout a series containing a large number of samples.

In general, calibration check standards will be analyzed after every 12 hours, or more frequently, as specified in the applicable analytical method. In analyses where internal standards are used, a calibration check standard will only be analyzed in the beginning of an analytical series. If results of the calibration check standard exceed specified tolerances, then all samples analyzed since the last acceptable calibration check standard will be reanalyzed.

Laboratory instrument calibration standards will be selected utilizing the guidance provided in the analytical methods, as summarized in Section 13.

11.3.7 Internal Standards

Internal standard areas and retention times will be monitored for organic analyses performed by GC/MS methods. Method-specified internal standard compounds will be spiked into all field samples, calibration standards, and quality control samples after preparation and prior to analysis. If internal standard areas in one or more samples exceed the specified tolerances, the cause will be investigated, the instrument will be recalibrated if necessary, and all affected samples will be reanalyzed.

The acceptability of internal standard performance will be determined using the guidance provided within the analytical methods.

11.3.8 Reference Standards/Control Samples

Reference standards are standards of known concentration and independent in origin from the calibration standards. The intent of reference standard analysis is to provide insight into the analytical proficiency within an analytical series. This includes preparation of calibration standards, validity of calibration, sample preparation, instrument set-up, and the premises inherent in quantitation. Reference standards will be analyzed at the frequencies specified within the analytical methods.

11.4 Data Precision Assessment Procedures

Field precision is difficult to measure because of temporal variations in field parameters. However, precision will be controlled through the use of experienced field personnel, properly calibrated meters, and duplicate field measurements. Field duplicates will be used to assess precision for the entire measurement system including sampling, handling, shipping, storage, preparation, and analysis.

Laboratory data precision for organic analyses will be monitored through the use of MS/MSD and laboratory duplicates as identified in Table 1.

The precision of data will be measured by calculation of the relative percent difference (RPD) by the following equation:

$$RPD = \frac{(A-B)}{(A+B)/2} \times 100$$

Where:

A = Analytical result from one of two duplicate measurements
B = Analytical result from the second measurement

Precision objectives for MSD and laboratory duplicate analyses are identified in the NYSDEC ASP Revision 2005 and contained in Table 2.

11.5 Data Accuracy Assessment Procedures

The accuracy of field measurements will be controlled by experienced field personnel, properly calibrated field meters, and adherence to established protocols. The accuracy of field meters will be assessed by review of calibration and maintenance logs.

Laboratory accuracy will be assessed via the use of MSs, surrogate spikes, internal standards, and reference standards. Where available and appropriate, quality assurance Performance Standards will be analyzed periodically to assess laboratory accuracy. Accuracy will be calculated in terms of percent recovery as follows:

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

Where:

A = Value measured in spiked sample or standard
X = Value measured in original sample
B = True value of amount added to sample or true value of standard

This formula is derived under the assumption of constant accuracy over the original and spiked measurements. If any accuracy calculated by this formula is outside of the

acceptable levels, data will be evaluated to determine whether the deviation represents unacceptable accuracy, or variable, but acceptable accuracy. Accuracy objectives for MS recoveries and surrogate recovery objectives are identified in the NYSDEC ASP 2005 Revision and contained in Table2.

11.6 Data Completeness Assessment Procedures

Completeness of a field or laboratory data set will be calculated by comparing the number of valid sample results generated to the total number of results generated.

$$\text{Completeness} = \frac{\text{Number valid results}}{\text{Total number of results generated}} \times 100$$

As a general guideline, overall project completeness is expected to be at least 90%. The assessment of completeness will require professional judgment to determine data usability for intended purposes.

12. Instrument/Equipment Testing, Inspection, and Maintenance Requirements

12.1 General

Testing and maintenance schedules have been developed for both field and laboratory instruments. A summary of the testing and maintenance activities to be performed is presented below.

12.2 Field Instruments and Equipment

Prior to field sampling, each piece of field equipment will be inspected to ensure that it is operational. If the equipment is not operational, it will be serviced prior to its use. All meters which require charging or batteries will be fully charged and have fresh batteries. If instrument servicing is required, it is the responsibility of the appropriate Task Manager or field personnel to follow the maintenance schedule and arrange for timely service. Field instruments will be maintained according to the manufacturers' instructions.

Logbooks will be kept for each field instrument. Each logbook will contain records of operation, maintenance, calibration, and any problems and repairs. Logbooks for each piece of equipment shall be maintained in project records. The Task Managers will review calibration and maintenance logs.

12.2.1 Equipment Maintenance

All measuring and test equipment to be used in support of the Pre-Design Investigation activities that directly affect the quality of the analytical data shall be subject to preventative maintenance measures that minimize equipment downtime. Equipment will be examined to certify that it is in operating condition. This includes checking the manufacturer's operating manual to ensure that all maintenance requirements are being observed. Field notes from previous sampling events will be reviewed to ensure that any prior equipment problems are not overlooked and that any necessary repairs to equipment have been carried out.

Field equipment returned from a site will be inspected to confirm that it is in working order. The inspection will be recorded in the logbook or field notebooks, as appropriate. It will also be the obligation of the last user to record any equipment problems in the logbook. Non-operational field equipment will either be repaired or replaced. Appropriate spare parts will be made available for field meters.

Consultant-/subcontractor-owned or leased equipment maintenance shall be in accordance with the manufacturer's instructions.

12.3 Laboratory Instruments and Equipment

12.3.1 General

Laboratory instrument and equipment documentation procedures include details of any observed problems, corrective measure(s), routine maintenance, and instrument repair (which will include information regarding the repair and the individual who performed the repair).

Preventive maintenance of laboratory equipment generally will follow the guidelines recommended by the manufacturer. A malfunctioning instrument will be repaired immediately by in-house staff or through a service call from the manufacturer.

12.3.2 Instrument Maintenance

Maintenance schedules for laboratory equipment adhere to the manufacturer's recommendations. Records reflect the complete history of each instrument and specify the time frame for future maintenance. Major repairs or maintenance procedures are performed through service contracts with manufacturer or qualified contractors. Paperwork associated with service calls and preventative maintenance calls will be kept on file by the laboratory.

Laboratory Systems Managers are responsible for the routine maintenance of instruments used in the particular laboratory. Any routine preventative maintenance carried out is logged into the appropriate logbooks. The frequency of routine maintenance is dictated by the nature of samples being analyzed, the requirements of the method used, and/or the judgment of the Laboratory Systems Manager.

All major instruments are backed up by comparable (if not equivalent) instrument systems in the event of unscheduled downtime. An inventory of spare parts is also available to minimize equipment/instrument downtime.

12.3.3 Equipment Monitoring

On a daily basis, the operation of balances, incubators, ovens, refrigerators, and water purification systems will be checked and documented. Any discrepancies will be immediately reported to the appropriate laboratory personnel for resolution.

13. Instrument Calibration and Frequency

13.1 Field Instruments and Equipment

The calibration of field instruments is governed by specific SOPs documented in the Pre-Design Investigation Work Plans for the applicable field analysis method, and such procedures take precedence over the following discussion.

Field personnel are responsible for ensuring that a master calibration/maintenance log is maintained following the procedures specified for each measuring device. Where applicable, each log will include, at a minimum, the following information:

- name of device and/or instrument calibrated
- device/instrument serial/identification numbers
- calibration method
- tolerance
- calibration standard used
- frequency of calibration
- date(s) of calibration(s)
- name of person(s) performing calibration(s)

Instruments and equipment used to gather, generate, or measure environmental data will be calibrated at the intervals specified by the manufacturer or more frequently, and in such a manner that accuracy and reproducibility of results are consistent with the manufacturer's specifications. In the event that an internally calibrated field instrument fails to meet calibration/checkout procedures, it will be returned to the manufacturer for service. Equipment found to be out of tolerance during the period of use shall be removed from the field and measuring and testing activities performed using the equipment shall be addressed via the corrective action system described in Section 17.4 of this QAPP.

13.2 Laboratory Instrument and Equipment

Instrument calibration will follow the specifications provided by the instrument manufacturer or specific analytical method used. The analytical methods for target constituents are identified separately below.

VOCs

Equipment calibration procedures will follow guidelines presented in NYSDEC ASP 2005 Revision, Exhibits D and E, Part II Section 2.

SVOCs

Equipment calibration procedures will follow guidelines presented in NYSDEC ASP 2005 Revision, Exhibits D and E, Part II Section 3

Metals

Equipment calibration procedures will follow guidelines presented in NYSDEC ASP 2005 Revision, Exhibits D and E, Part III Sections 1, 3, and 5.

TOC

Equipment calibration procedures will follow guidelines presented in Lloyd Kahn Method.

The equipment calibration procedures for following parameter groups/methods: Methane, Chloride, Nitrogen as ammonia, Nitrate, Sulfate, Sulfide, Phosphorous as orthophosphate, Total dissolved organic carbon, pH, Oxidation-reduction potential, Alkalinity, Alkalinity-Bicarbonate, Carbon Dioxide, Iron (filtered and unfiltered), Manganese (filtered and unfiltered) will follow guidelines presented in NYSDEC ASP 2005 Revision, Exhibit D.

When analyses are conducted according to the USEPA SW-846 methods, the calibration procedures and frequencies specified in the applicable method will be followed, as noted in the attached SOPs (Attachment 1). For analyses governed by SOPs, see the appropriate SOP for the required calibration procedures and frequencies. Records of calibrations will be filed and maintained by the laboratory. These records will be subject to quality assurance audit. For all instruments, the

laboratory will maintain trained repair staff with in-house spare parts or will maintain service contracts with vendors.

All standards used in the calibration of equipment are traceable, directly or indirectly, to National Institute of Standards and Technology. All standards received shall be logged into standard receipt logs maintained by the individual analytical groups. Each group shall maintain a standards log which tracks the preparation of standards used for calibration and quality control purposes.

14. Inspection/Acceptance Requirements for Supplies and Consumables

All supplies to be used in the field and laboratory will be available when needed. They will be free of target chemicals and interferences. All reagents will be tested prior to use with site samples. All standards will be verified against a second source standard. The laboratory will follow a “first in first out” procedure for the storage and use of all consumables to minimize the risk of contamination and degradation. The various supplies and consumables required on-site are noted in the various field SOPs included in the Pre-Design Investigation Work Plans.

15. Data Acquisition Requirements for Non-Direct Measurements

At this point in time, historical data generated by outside parties is not anticipated to be used directly in completing the Pre-Design Investigation. However, historical data will be used as guidance in determining sampling locations for the Pre-Design Investigation.

Prior to their use, historic data sets will be reviewed according to the procedures identified in subsequent sections of this QAPP to determine the appropriate uses of such data. The extent to which these data can be validated will be determined by the analytical level and QC data available. The evaluation of historic data for Pre-Design Investigation purposes requires the following:

- identification of analytical levels
- evaluation of QC data, when available
- development of conclusions regarding the acceptability of the data for intended uses

Acceptability of historic data for intended uses will be determined by application of these procedures and professional judgment. If the historic data quality cannot be determined, its use will be limited to general trend evaluations.

16. Data Management

The purpose of the data management is to ensure that all of the necessary data are accurate and readily accessible to meet the analytical and reporting objectives of the project. The field investigations will encompass a large number of samples and analytes from a large geographic area. Due to the large amount of resulting data, the need arises for a structured, comprehensive, and efficient program for management of data.

The data management program established for the project includes field documentation and sample QA/QC procedures, methods for tracking and managing the data, and a system for filing all site-related information. More specifically, data management procedures will be employed to efficiently process the information collected such that the data are readily accessible and accurate. These procedures are described in detail in the following section.

The data management plan has five elements: 1) sample designation system; 2) field activities; 3) sample tracking and management; 4) data management system; and 5) document control and inventory.

16.1 Sample Designation System

A concise and easily understandable sample designation system is an important part of the project sampling activities. It provides a unique sample number that will facilitate both sample tracking and easy re-sampling of select locations to evaluate data gaps, if necessary. The sample designation system to be employed during the sampling activities will be consistent, yet flexible enough to accommodate unforeseen sampling events or conditions. A combination of letters and numbers will be used to yield a unique sample number for each field sampled collected, as outlined in Section 6.2.1.

16.2 Field Activities

Field activities designed to gather the information necessary to make decisions during the Pre-Design Investigation process require consistent documentation and accurate record keeping. During site activities, standardized procedures will be used for documentation of field activities, data security, and quality assurance. These procedures are described in further detail in the following subsections.

16.2.1 Field Documentation

Complete and accurate record keeping is a critical component of the field investigation activities. When interpreting analytical results and identifying data trends, investigators realize that field notes are an important part of the review and validation process. To ensure that the field investigation is thoroughly documented, several different information records, each with its own specific reporting requirements, will be maintained, including:

- field logs
- COC forms

A description of each of these types of field documentation is provided below.

Field Logs

The personnel performing the field activities will keep field logs that detail all observations and measurements made during the Pre-Design Investigation. Data will be recorded directly into site-dedicated, bound notebooks, with each entry dated and signed. To ensure at any future date that notebook pages are not missing, each page will be sequentially numbered. Erroneous entries will be corrected by crossing out the original entry, initialing it, and then documenting the proper information. In addition, certain media sampling locations will be surveyed to accurately record their locations. The survey crew will use their own field logs and will supply the sampling location coordinates to the Database Administrator.

COC Forms

COC forms are used as a means of documenting and tracking sample possession from time of collection to the time of disposal. A COC form will accompany each field sample collected, and one copy of the form will be filed in the field office. All field personnel will be briefed on the proper use of the COC procedure. COC procedures and a sample form are included in the Pre-Design Investigation Work Plans.

Instrument Calibration Records

As part of data quality assurance procedures, field monitoring and detection equipment will be routinely calibrated. Instrument calibration ensures that equipment used is of the

proper type, range, accuracy, and precision to provide data compatible with the specified requirements and desired results. Calibration procedures for the various types of field instrumentation are described in Section 13.1. In order to demonstrate that established calibration procedures have been followed, calibration records will be prepared and maintained to include, as appropriate, the following:

- calibration date and time
- type and identification number of equipment
- calibration frequency and acceptable tolerances
- identification of individual(s) performing calibration
- reference standards used
- calibration data
- information on calibration success or failure

The calibration record will serve as a written account of monitoring or detection equipment QA. All erratic behavior or failures of field equipment will be subsequently recorded in the calibration log.

16.2.2 Data Security

Measures will be taken during the field investigation to ensure that samples and records are not lost, damaged, or altered. When not in use, all field notebooks will be stored at the field office or locked in the field vehicle. Access to these files will be limited to the field personnel who utilize them.

16.3 Sample Management and Tracking

A record of all field documentation will be maintained to ensure the validity of data used in the site analysis. To effectively execute such documentation, specific sample tracking and data management procedures will be used throughout the sampling program.

Sample tracking will begin with the completion of COC forms as summarized in Section 9.2.3. The completed COC forms associated with samples collected will be faxed to the QAC. Copies of all completed COC forms will be maintained in the field office. The laboratory shall verify receipt of the samples electronically (via email) on the following day.

When analytical data are received from the laboratory, the QAC will review the incoming analytical data packages against the information on the COCs to confirm that the correct analyses were performed for each sample and that results for all samples submitted for analysis were received. Any discrepancies noted will be promptly followed-up by the QAC.

16.4 Data Management System

In addition to the sample tracking system, a data management system will be implemented. The central focus of the data management system will be the development of a personal computer-based project database. The project database, to be maintained by the Database Administrator, will combine pertinent geographical, field, and analytical data. Information that will be used to populate the database will be derived from three primary sources: surveying of sampling locations, field observations, and analytical results. Each of these sources is discussed in the following sections.

16.4.1 Computer Hardware

The database will be constructed on Pentium®-based personal computer work stations connected through a Novell network server. The Novell network will provide access to various hardware peripherals, such as laser printers, backup storage devices, image scanners, modems, etc. Computer hardware will be upgraded to industrial and corporate standards, as necessary, in the future.

16.4.2 Computer Software

The database will be written in Microsoft Access, running in a Windows operating system. Custom applets, such as diskette importing programs, will be written in either Microsoft VBA or Microsoft Visual Basic. Geographic Information System (GIS) applications will be developed in ESRI ArcGIS, with additional customization performed with Visual Basic. Tables and other database reports will be generated through Access in conjunction with Microsoft Excel, Microsoft Word, and/or Seagate Crystal Reports.

These software products will be upgraded to current industrial standards, as necessary.

16.4.3 Survey Information

In general, each location sampled as part of the Pre-Design Investigation will be surveyed to ensure accurate documentation of sample locations for mapping and GIS purposes (if appropriate), to facilitate the re-sampling of select sample locations during future monitoring programs and remediation activities. The surveying activities that will occur in the field will consist of the collection of information that will be used to compute a northing and easting in state plane coordinates for each sample location and the collection of information to compute elevations relative to the National Geodetic Vertical Datum of 1988 for select sample locations, as appropriate. All field books associated with the surveying activities will be stored as a record of the project activities.

16.4.4 Field Observations

An important part of the information that will ultimately reside in the data management system for use during the project will originate in the observations that are recorded in the field.

Following each sampling event, a status memorandum may be prepared by the field personnel who performed the sampling activities. The purpose of the status memo is to present a summary and a record of the sampling event. Topics to be discussed include the locations sampled, the sampling methodologies used, QA/QC procedures, blind duplicate and MS/MSD sample identification numbers, equipment decontamination procedures, personnel involved in the activity, and any other noteworthy events that occurred.

Tables are typically attached to the memorandum and are used to summarize measurements that were recorded in the field books. It is anticipated that these tables will be developed using a personal computer spreadsheet program to reduce possible transcription error and to facilitate the transfer of information to the data management system. For example, for soil samples, the table would present the sampling date and time, water depth, soil depth, depth of soil recovered in a given core, the depth increment submitted for analysis, and a description of the lithology.

Status memos are valuable tools to keep project personnel informed on the details of the field activities and are also invaluable during the development of the final report.

Each status memo will be reviewed for accuracy and completeness by the respective sampling activity manager. Following the approval and finalization of each memo, the status memo will be used to transfer field observations into the data management system.

All pertinent field data will be manually entered into the appropriate database tables from the COC forms and field notebooks.

16.4.5 Analytical Results

Analytical results will be provided by the laboratory in both a digital and a hard copy format. The data packages will be examined to ensure that the correct analyses were performed for each sample submitted and that all of the analyses requested on the COC form were performed. If discrepancies are noted, the QAC will be notified and will promptly follow up with the laboratory to resolve any issues.

Each data package will be validated in accordance with the procedures presented in Section 20. Any data that does not meet the specified standards will be flagged pending resolution of the issue. The flag will not be removed from the data until the issue associated with the sample results is resolved. Although flags may remain for certain data, the use of that data may not necessarily be restricted.

Following completion of the data validation, the digital files will be used to populate the appropriate database tables. An example of the format of electronic data deliverable (EDD) format is included in Table 5. This format specifies one data record for each constituent for each sample analyzed. Specific fields include:

- sample identification number.
- date sampled.
- date analyzed.
- parameter name.
- analytical result.
- units.

- detection limit.
- qualifier(s).

The individual EDDs, supplied by the laboratory in either an ASCII comma separated value format or in a Microsoft Excel worksheet, will be loaded into the appropriate database table via a custom-designed user interface Visual Basic program. Any analytical data that cannot be provided by the laboratory in electronic format will be entered manually. After entry into the database, the EDD data will be compared to the field information previously entered into the database to confirm that all requested analytical data have been received.

16.4.6 Data Analysis and Reporting

The database management system will have several functions to facilitate the review and analysis of the Pre-Design Investigation data. Data entry screens will be developed to assist in the keypunching of field observations. Routines will also be developed to permit the user to scan analytical data from a given site for a given media. Several output functions that have been developed by ARCADIS BBL will be appropriately modified for use in the data management system.

A valuable function of the data management system will be the generation of tables of analytical results from the project databases. The capability of the data management system to directly produce tables reduces the redundant manual entry of analytical results during report preparation and precludes transcription errors that may occur otherwise. This data management system function creates a digital comma-delimited ASCII file of analytical results and qualifiers for a given media. The ASCII file is then processed through a spreadsheet, which transforms the comma-delimited file into a table of rows and columns. Tables of analytical data will be produced as part of data interpretation tasks, the reporting of data, and the generation of the Pre-Design Investigation Report.

Another function of the data management system will be to create digital files of analytical results and qualifiers suitable for transfer to mapping/presentation software. A function has been created by ARCADIS BBL that creates a digital file consisting of sample location number, state plane coordinates, sampling date, and detected constituents and associated concentrations and analytical qualifiers. The file is then transferred to an AutoCAD work station, where another program has been developed to plot a location’s analytical data in a "box" format at the sample location (represented by the state plane coordinates). This routine greatly reduces the redundant

keypunching of analytical results and facilitates the efficient production of interpretative and presentation graphics.

The data management system also has the capability of producing a digital file of select parameters that exists in one or more of the databases. This type of custom function is accomplished on an interactive basis and is best used for transferring select information into a number of analysis tools, such as statistical or graphing programs.

16.5 Document Control and Inventory

ARCADIS BBL maintains project files at its Syracuse, New York office. Each client project is assigned a file/job number. Each file is then broken down into the following subfiles:

1. Agreements/Proposals (filed chronologically)
2. Change Orders/Purchase Orders (filed chronologically)
3. Invoices (filed chronologically)
4. Project Management (filed by topic)
5. Correspondence (filed chronologically)
6. Notes and Data (filed by topic)
7. Public Relations Information (filed by topic)
8. Regulatory Documents (filed chronologically)
9. Marketing Documents (filed chronologically)
10. Final Reports/Presentations (filed chronologically)
11. Draft Reports/Presentations (filed chronologically)
12. Documents Prepared by Others (filed chronologically)

Originals, when possible, are placed in the files. These are the central files and will serve as the site-specific files for the Pre-Design Investigation.

17. Assessment and Response Actions

17.1 General

Performance and systems audits will be completed in the field and laboratory during the Pre-Design Investigation as described below.

17.2 Field Audits

The following field performance and systems audits will be completed during this project.

The appropriate Task Manager will monitor field performance. Field performance audit summaries will contain an evaluation of field activities to verify that activities are performed according to established protocols. The ARCADIS BBL QAC will review field reports and communicate concerns to the ARCADIS BBL Project Manager and/or Task Managers, as appropriate. In addition, the ARCADIS BBL QAC will review the rinse and trip blank data to identify potential deficiencies in field sampling and cleaning procedures. In addition, systems audits comparing scheduled QA/QC activities from this document with actual QA/QC activities completed will be performed. The appropriate Task Manager and QAC will periodically confirm that work is being performed consistent with this QAPP, the Pre-Design Investigation Work Plans.

17.3 Laboratory Audits

The laboratory will perform internal audits consistent with NYSDEC ASP 2005 Revision, Exhibits D and E.

Internal laboratory audits are conducted by the laboratory QAC. As part of the audit, the overall performance of the laboratory staff is evaluated and compared to the performance criteria outlined in the laboratory quality assurance manual and SOPs. The results of the audits are summarized and issued to each department supervisor, the Laboratory Manager, and the Laboratory Director. A systems audit of each laboratory is also performed by the QAC to determine if the procedures implemented by each laboratory are in compliance with the quality assurance manual and SOPs.

In addition to the laboratory's internal audits, as participants in state and federal certification programs, the laboratory is audited by representatives of the regulatory agency issuing certification. Audits are usually conducted on an annual basis and focus

on laboratory conformance to the specific program protocols for which the laboratory is seeking certification. The auditor reviews sample handling and tracking documentation, analytical methodologies, analytical supportive documentation, and final reports. The audit findings are formally documented and submitted to the laboratory for corrective action, if necessary.

ARCADIS BBL reserves the right to conduct an on-site audit of the laboratory prior to the start of analyses for the project. Additional audits may be performed during the course of the project, as deemed necessary.

17.4 Corrective Action

Corrective actions are required when field or analytical data are not within the objectives specified in this QAPP or the Pre-Design Investigation Work Plans. Corrective actions include procedures to promptly investigate, document, evaluate, and correct data collection and/or analytical procedures. Field and laboratory corrective action procedures for the actions are described below.

17.4.1 Field Procedures

When conducting the action field work, if a condition is noted by the field crew that would have an adverse effect on data quality, corrective action will be taken so as not to repeat this condition. Condition identification, cause, and corrective action implemented by the Field Manager or a designee, will be documented on a Corrective Action Form and reported to the appropriate ARCADIS BBL Task Manager, QAC, and Project Manager.

Examples of situations that would require corrective actions are provided below:

- Protocols as defined by the QAPP and the Pre-Design Investigation Work Plans have not been followed.
- Equipment is not in proper working order or is not properly calibrated.
- QC requirements have not been met.
- Issues resulting from performance or systems audits have not been resolved.

Project personnel will continuously monitor ongoing work performance in the normal course of daily responsibilities.

17.4.2 Laboratory Procedures

In the laboratory, when a condition is noted to have an adverse effect on data quality, corrective action will be taken so as not to repeat this condition. Condition identification, cause, and corrective action taken will be documented and reported to the appropriate Project Manager and QAC.

Corrective action may be initiated, at a minimum, under the following conditions:

- Specific laboratory analytical protocols have not been followed.
- Protocols as defined by this QAPP have not been followed.
- Predetermined data acceptance standards are not obtained.
- Equipment is not in proper working order or calibrated.
- Sample and test results are not completely traceable.
- QC requirements have not been met.
- Issues resulting from performance or systems audits have not been resolved.

Laboratory personnel will continuously monitor ongoing work performance in the normal course of daily responsibilities. Corrective action is initiated at a point were the problem has been identified. At whatever level this occurs (analyst, supervisor, data review, or quality control), it is brought to the attention of the laboratory QAC and, ultimately, the Laboratory Director. Final approval of any action deemed necessary is subject to the approval of the Laboratory Director.

Any corrective action deemed necessary based on system or performance audits or the results of data review will be implemented. The corrective action may include sample re-extraction, re-preparation, re-analysis, cleanup, dilutions, matrix modifications, or other activities.

18. Reports to Management

18.1 Internal Reporting

The analytical laboratory will submit analytical reports to ARCADIS BBL for review. If required, ARCADIS BBL will, in turn, submit the reports to the data validator for review. Supporting data (i.e., historic data, related field or laboratory data) will also be reviewed to evaluate data quality, as appropriate. The ARCADIS BBL Quality Assurance Manager will incorporate results of the data validation reports (if required) and assessments of data usability into a summary report (if required) that will be submitted to the ARCADIS BBL Project Manager and appropriate Task Managers. If required, this report will be filed in the project file at ARCADIS BBL's office and will include the following:

1. Assessment of data accuracy, precision, and completeness for both field and laboratory data

2. Results of the performance and systems audits

3. Significant QA/QC problems, solutions, corrections, and potential consequences

4. Analytical data validation report

18.2 Pre-Design Investigation Reporting

Upon sample transport to the laboratory, a copy of the chain-of-custody will be forwarded to ARCADIS BBL's Project Manager. Upon receipt of the ASP - Category B Data Package from the laboratory, the ARCADIS BBL Quality Assurance Manager will determine if the data package has met the required data quality objectives. The analytical data package will be submitted to the ARCADIS BBL Project Manager and the analytical data will be incorporated into the Pre-Design Investigation Report in a tabulated format.

19. Data Reduction and Review

19.1 General

After field and laboratory data are obtained, the data will be subject to the following:

1. Reduction, or manipulation mathematically, or otherwise into meaningful and useful forms

2. Review

3. Organization, interpretation, and reporting

4. Data validation

19.2 Field Data Reduction and Review

19.2.1 Field Data Reduction

Information collected in the field through visual observation, manual measurement, and/or field instrumentation will be recorded in field notebooks or data sheets, and/or on forms. Such data will be reviewed by the appropriate Task Manager for adherence to the Pre-Design Investigation Work Plans and this QAPP and for consistency. Concerns identified as a result of this review will be discussed with the field personnel, corrected if possible, and, as necessary, incorporated into the data evaluation process.

19.2.2 Field Data Review

Field data calculations, transfers, and interpretations will be conducted by the field personnel and reviewed for accuracy by the appropriate Task Manager and the QAC. Logs and documents will be checked for:

1. General completeness

2. Readability

3. Usage of appropriate procedures

4. Appropriate instrument calibration and maintenance

5. Reasonableness in comparison to present and past data collected

6. Correct sample locations

7. Correct calculations and interpretations

19.3 Laboratory Data Reduction and Review

19.3.1 Laboratory Data Reduction

The calculations used for data reduction will be specified in each of the analytical methods referenced previously. Whenever possible, analytical data will be transferred directly from the instrument to a computerized data system. Raw data will be entered into permanently bound laboratory notebooks. The data entered are sufficient to document all factors used to arrive at the reported value.

Concentration calculations for chromatographic analyses will be based on response factors. Quantitation will be performed using either internal or external standards.

Inorganic analyses will be based on regression analysis. Regression analysis is used to fit a curve through the calibration standard data. The sample concentrations will be calculated using the resulting regression equations.

Non-aqueous values will be reported on a dry-weight basis. Unless otherwise specified, all values will be reported uncorrected for blank contamination.

19.3.2 Laboratory Data Review

Data will be subject to multi-level review by the laboratory. The group leader will review all data reports prior to release for final data report generation. The QAC will review the final data reports, and the QA Manager will review a cross-section of the final data reports prior to shipment to ARCADIS BBL.

If discrepancies or deficiencies exist in the analytical results, then corrective action will be taken, as discussed in Section 17. Deficiencies discovered as a result of internal data review, as well as the corrective actions to be used to rectify the situation, will be documented on a Corrective Action Form. This form will be submitted to the ARCADIS BBL Project Manager.

19.4 Data Validation and Verification

All data generated for health and safety and engineering design/control purposes will be subjected to the data validation and verification procedures outlined in Section 20. Data generated for disposal purposes will not be reviewed.

20. Data Validation and Verification

Data validation entails a review of the quality control data and the raw data to verify that the laboratory was operating within required limits, the analytical results were correctly transcribed from the instrument read outs, and which, if any, environmental samples were related to any out-of-control quality control samples. The objective of data validation is to identify any questionable or invalid laboratory measurements.

ARCADIS BBL will validate all data generated producing a NYSDEC data usability summary report for each individual SDG using the most recent versions of the USEPA's Function Guidelines (USEPA, 1999; 2002) and USEPA Region II SOPs for data validation available at the time of project initiation, where appropriate. These procedures and criteria may be modified as necessary to address project-specific and method-specific criteria, control limits, and procedures. Data validation will consist of data screening, checking, reviewing, editing, and interpretation to document analytical data quality and to determine whether the quality is sufficient to meet the DQOs.

The data validator will verify that reduction of laboratory measurements and laboratory reporting of analytical parameters is in accordance with the procedures specified for each analytical method and/or as specified in this QAPP. Any deviations from the analytical method or any special reporting requirements apart from that specified in this QAPP will be detailed on COC forms.

Upon receipt of laboratory data, the following procedures will be executed by the data validator:

- Evaluate completeness of data package.
- Verify that field COC forms were completed and that samples were handled properly.
- Verify that holding times were met for each parameter. Holding time exceedences, should they occur, will be documented. Data for all samples exceeding holding time requirements will be flagged as either estimated or rejected. The decision as to which qualifier is more appropriate will be made on a case-by-case basis.
- Verify that parameters were analyzed according to the methods specified.

- Review QA/QC data (i.e., make sure duplicates, blanks, and spikes were analyzed on the required number of samples, as specified in the method; verify that duplicate and MS recoveries are acceptable).

- Investigate anomalies identified during review. When anomalies are identified, they will be discussed with the Project Manager and/or Laboratory Manager, as appropriate.

- If data appears suspect, investigate the specific data of concern. Calculations will be traced back to raw data; if calculations do not agree, the cause will be determined and corrected.

Deficiencies discovered as a result of the data review, as well as the corrective actions implemented in response, will be documented and submitted in the form of a written report addressing the following topics as applicable to each method:

- assessment of the data package

- description of any protocol deviations

- failures to reconcile reported and/or raw data

- assessment of any compromised data

- overall appraisal of the analytical data

- table of site name, sample quantities, matrix, and fractions analyzed

It should be noted that qualified results do not necessarily invalidate data. The goal to produce the best possible data does not necessarily mean producing data without quality control qualifiers. Qualified data can provide useful information.

Resolution of any issues regarding laboratory performance or deliverables will be handled between the laboratory and the data validator. Suggestions for reanalysis may be made by the ARCADIS BBL QAC at this point.

Data validation reports will be kept in the project file at the ARCADIS BBL office in Syracuse, New York.

21. Reconciliation with User Requirements

The data results will be examined to determine the performance that was achieved for each data usability criteria. The performance will then be compared with the project objectives and DQOs. Deviations from objectives will be noted. Additional action may be warranted when performance does not meet performance objectives for critical data. Options for corrective action relating to incomplete information, questionable results or inconsistent data, may include any or all of the following:

- retrieval of missing information
- request for additional explanation or clarification
- reanalysis of sample from extract (when appropriate)
- recalculation or reinterpretation of results by the laboratory

These actions may improve the data quality, reduce uncertainty, and may eliminate the need to qualify or reject data.

If these actions do not improve the data quality to an acceptable level, the following additional actions may be taken:

- extrapolation of missing data from existing data points
- use of historical data
- evaluation of the critical/non-critical nature of the sample

If the data gap cannot be resolved by these actions, an evaluation of the data bias and potential for false negatives and positives can be performed. If the resultant uncertainty level is unacceptable, the following action must be taken:

- additional sample collection and analysis

22. References

United States Environmental Protection Agency (USEPA). *Interim Guidance and Specifications for Preparing Quality Assurance Project Plans*. QAMS-005/80. Office of Research and Development. (December 1980).

USEPA. *Guide to Management of Investigation-Derived Wastes*. 9345.3-03FS. (January, 1992).

United States Environmental Protection Agency. *Contract Laboratory Program National Functional Guidelines for Inorganic Data Review*. EPA-540/R-94-013. (February 1994a).

USEPA. *Contract Laboratory Program National Functional Guidelines for Organic Data Review*. EPA-540/R-99-008 (October 1999).

USEPA. *EPA Requirements for Quality Assurance Project Plans for Environmental Operations*. EPA-QA/R-5. Office of Environmental Information. (March, 2001).

USEPA. *Contract Laboratory Program National Functional Guidelines for Inorganic Data Review*. EPA-540/R-01-008 (July 2002).

USEPA. *Guidance for Quality Assurance Project Plans*. EPA-QA/G-5. Office of Environmental Information. (December, 2002).

USEPA. *Test Methods for Evaluating Solid Waste*. SW-846 3rd Edition, Update 3. Office of Solid Waste (December 1996).

TABLES

Table 1. Sample Quantities and Quality Control Frequencies, Quality Assurance Project Plan, Chevron Environmental Management Company, Former Tappan Terminal Site, Hastings-on-Hudson, New York

Parameter	Estimated Environmental Sample Quality	Field QC Analyses						Laboratory QC Sample						Total
		Trip Blank		Rinse Blank		Field Duplicate		Matrix Spike		trix Spike Duplica		Lab Duplicate		
		Freq.	No.	Freq.	No.	Freq.	No.	Freq.	No.	Freq.	No.	Freq.	No.	
Soil														
Volatile Organic Compounds (SW-846 8260-TCL)	30	NA	2	1/day	2	NA	2	1/20	2	1/20	2	NA	--	40
Semivolatile Organic Compounds plus TICs (SW-846 8270-TCL)	25	NA	--	1/day	3	1/20	2	1/20	2	1/20	2	NA	--	34
Ferrous Iron (SM3500-Fe-D)	30	NA	--	1/day	2	NA	2	1/20	2	1/20	2	NA	--	38
Total Organic Carbon (Lloyd Kahn)	30	NA	--	1/day	2	NA	2	1/20	2	1/20	2	NA	--	38
Soil (Leachate- TCLP)														
TCLP-Volatiles (SW-846 1311/8260-Benzene)	4	NA	--	NA	--	NA	--	NA	--	NA	--	NA	--	4
Reactivity	4	NA	--	NA	--	NA	--	NA	--	NA	--	NA	--	4
Flash point (Ignitability)	4	NA	--	NA	--	NA	--	NA	--	NA	--	NA	--	4
Groundwater														
Volatile Organic Compounds (SW-846 8260- TCL)	160	NA	20	1/day	8	NA	8	1/20	8	1/20	8	NA	--	212
Semivolatile Organic Compounds plus TICs (SW-846 8270-TCL)	110	NA	--	1/day	6	NA	6	1/20	6	1/20	6	NA	--	134
Alkalinity (EPA Method 310.1)	15	NA	--	1/day	1	NA	1	1/20	1	1/20	1	NA	--	19
Alkalinity-Bicarbonate (EPA Method 310.1)	15	NA	--	1/day	1	NA	1	1/20	1	1/20	1	NA	--	19
Carbon Dioxide (RSK175 modified))	15	NA	--	1/day	1	NA	1	1/20	1	1/20	1	NA	--	19
Chloride (EPA Method 300.0)	15	NA	--	1/day	1	NA	1	1/20	1	1/20	1	NA	--	19
Ferrous Iron (SM3500-Fe-D)	15	NA	--	1/day	1	NA	1	1/20	1	1/20	1	NA	--	19
Manganese (EPA Method 6010)	15	NA	--	1/day	1	NA	1	1/20	1	1/20	1	NA	--	19
Methane (RSK175)	15	NA	--	1/day	1	NA	1	1/20	1	1/20	1	NA	--	19
Nitrate (EPA Method 353.2)	15	NA	--	1/day	1	NA	1	1/20	1	1/20	1	NA	--	19
Nitrogen as ammonia (350.1)	15	NA	--	1/day	1	NA	1	1/20	1	1/20	1	NA	--	19
Phosphorous as orthophosphate (365.2)	15	NA	--	1/day	1	NA	1	1/20	1	1/20	1	NA	--	19
Sulfate (EPA 375.4)	15	NA	--	1/day	1	NA	1	1/20	1	1/20	1	NA	--	19
Sulfide (SM4500-S-2D)	15	NA	--	1/day	1	NA	1	1/20	1	1/20	1	NA	--	19
Total Dissolved Organic Carbon (SW-9060)	15	NA	--	1/day	1	NA	1	1/20	1	1/20	1	NA	--	19

Notes:

Sample counts are an approximation.

1/day One rinse blank per day or one per 20 samples, whichever is more frequent. Rinse blanks not required when dedicated sampling equipment is used.

Freq Frequency

NA Not Applicable

No. Number

QC Quality Control

Table 2. Analytical Quality Control Limits 1, Quality Assurance Project Plan, Chevron Environmental Management Company, Former Tappan Terminal Site, Hastings-on-Hudson, New York

Parameter	Accuracy - % Recovery			Precision - RPD		
	Surrogate	MS/MSD	LCS	MS/MSD	Lab Duplicate	Field Duplicate
Soil						
Volatile Organics	60-140	60-140	70-140	25	--	50
Semivolatile Organics	20-140	20-140	40-120	40	--	50
Metals	--	80-120	80-120	--	20	50
Total Organic Carbon	--	70-130	70-130	--	30	50
Soil (leachates)						
Volatile Organics	75-115	60-145	70-140	20	--	50
Reactivity	--	70-130	70-130	--	30	50
Corrosivity	--	--	70-130	--	30	50
Groundwater						
Volatile Organics	75-115	60-145	70-140	20	--	30
Semivolatile Organics	20-140	20-130	40-120	40	--	30
Metals	--	80-120	80-120	--	30	30
Wet Chemistry and Miscellaneous	--	70-130	70-130	--	30	30

Note:

[1] The listed QC limits are based on SW-846 guidance and are advisory. The actual limits are determined based on laboratory performance. Frequent failure to meet the QC limits; however, warrant investigation of the laboratory.

Table 3. Parameters, Methods, and Target Reporting Limits, Quality Assurance Project Plan,
Chevron Environmental Management Company, Former Tappan Terminal Site, Hastings-on-Hudson, New York

	Water (ug/L)			Soil (ug/kg)		
Analyte	NYS GW STD./G.V.[3]	Laboratory MDL	Laboratory RL	TAGM G.V.[4]	Laboratory MDL	Laboratory RL
Volatile Organic Compounds 8260 [1]						
Dichlorodifluoromethane	5	0.285	1	NA	0.413	5
Chloromethane	5	0.346	1	NA	0.302	5
Bromomethane	5	0.282	1	NA	0.459	5
Vinyl chloride	2	0.243	1	200	0.204	10
Chloroethane	5	0.324	1	1,900	0.361	5
Trichlorofluoromethane	5	0.152	1	NA	0.550	5
Methylene chloride	5	0.438	1	100	2.200	5
1,1,2-Trichloro-1,2,2-trifluoroethane	5	0.309	1	6,000	0.530	5
Acetone	50	1.345	5	200	1.097	25
Carbon disulfide	60	0.232	1	2,700	0.429	5
Methyl acetate	NA	0.450	1	NA	0.998	5
1,1-Dichloroethene	5	0.293	1	400	0.612	5
1,1-Dichloroethane	5	0.273	1	200	0.581	5
trans-1,2-Dichloroethene	5	0.333	1	300	0.516	5
Methyl tert-butyl ether	10	0.284	1	NA	0.491	5
Chloroform	7	0.336	1	300	0.309	5
1,2-Dichloroethane	0.6	0.458	1	100	0.251	5
cis-1,2-Dichloroethene	5	0.366	1	NA	0.246	5
2-Butanone	50	1.318	5	300	0.812	25
1,1,1-Trichloroethane	5	0.265	1	800	0.363	5
Cyclohexane	NA	0.220	1	NA	0.230	5
Carbon tetrachloride	5	0.267	1	600	0.681	5
Bromodichloromethane	50	0.386	1	NA	0.257	5
1,2-Dichloropropane	1	0.332	1	NA	0.256	5
cis-1,3-Dichloropropene	0.4	0.355	1	NA	0.285	5
Trichloroethene	5	0.324	1	700	0.345	5
Methylcyclohexane	NA	0.221	1	NA	0.324	5
Dibromochloromethane	50	0.322	1	NA	0.276	5
1,2-Dibromoethane	0.0006	0.416	1	NA	0.190	5
1,1,2-Trichloroethane	1	0.419	1	NA	0.251	5
Benzene	1	0.350	1	60	0.547	5
trans-1,3-Dichloropropene	0.4	0.368	1	NA	0.642	5
Bromoform	50	0.257	1	NA	0.461	5
Isopropylbenzene	5	0.319	1	NA	0.328	5
4-Methyl-2-pentanone	NA	1.346	5	1,000	6.250	25
2-Hexanone	50	1.251	5	NA	6.250	25
Tetrachloroethene	5	0.365	1	1,400	0.299	5
Toluene	5	0.510	1	1,500	0.848	5
1,1,2,2-Tetrachloroethane	5	0.485	1	600	0.333	5
Chlorobenzene	5	0.317	1	1,700	0.514	5
Ethylbenzene	5	0.344	1	5,500	0.345	5
Styrene	5	0.314	1	NA	0.250	5
Xylenes (total)	5	0.930	3	1,200	2.937	15
1,3-Dichlorobenzene	3	0.331	1	1,600	0.297	5
1,4-Dichlorobenzene	3	0.369	1	8,500	0.229	5
1,2-Dichlorobenzene	3	0.401	1	7,900	0.316	5
1,2-Dibromo-3-chloropropane	0.04	0.467	1	NA	0.366	5
1,2,4-Trichlorobenzene	5	0.408	1	3,400	0.304	5
Methyl t-butyl ether (MTBE)	10	0.284	1	NA	0.491	5
Semivolatile Organic Compounds 8270 [2]						
Benzaldehyde	NA	0.268	5	NA	18.512	170
Phenol	1	0.446	5	330	17.768	170
bis(2-Chloroethyl)ether	NA	0.180	5	NA	14.574	170
2-Chlorophenol	NA	0.505	5	800	8.593	170
2-Methylphenol	NA	0.228	5	330	5.191	170
2,2'-oxybis(1-Chloropropane)	5	0.424	5	NA	17.637	170
Acetophenone	NA	0.104	5	NA	8.663	170
4-Methylphenol	NA	0.353	5	900	9.403	170

See Notes on Page 3.

Table 3. Parameters, Methods, and Target Reporting Limits, Quality Assurance Project Plan,
Chevron Environmental Management Company, Former Tappan Terminal Site, Hastings-on-Hudson, New York

Analyte	Water (ug/L)			Soil (ug/kg)		
	NYS GW STD./G.V.[3]	Laboratory MDL	Laboratory RL	TAGM G.V.[4]	Laboratory MDL	Laboratory RL
Semivolatile Organic Compounds 8270 [2] (Cont'd.)						
N-Nitrosos-di-n-propylamine	50	0.452	5	NA	13.370	170
Hexachloroethane	5	2.824	5	NA	13.064	170
Nitrobenzene	0.4	0.538	5	330	7.483	170
Isophorone	50	0.320	5	4,400	8.436	170
2-Nitrophenol	NA	0.603	5	330	7.716	170
2,4-Dimethylphenol	50	0.961	5	NA	45.596	170
bis(2-Chloroethoxy)methane	5	0.376	5	NA	9.183	170
2,4-Dichlorophenol	5	0.787	5	400	8.850	170
Naphthalene	10	0.116	5	13,000	2.810	170
4-Chloroaniline	5	0.331	5	330	49.545	170
Hexachlorobutadiene	0.5	2.595	5	NA	8.638	170
Caprolactam	NA	4.590	5	NA	73.024	170
4-Chloro-3-methylphenol	NA	0.596	5	330	6.944	170
2-Methylnaphthalene	NA	0.082	5	36,400	2.045	170
Hexachlorocyclopentadiene	5	2.500	5	NA	51.037	170
2,4,6-Trichlorophenol	NA	0.994	5	NA	11.137	170
2,4,5-Trichlorophenol	NA	0.988	5	100	36.815	170
1,1'-Biphenyl	5	0.065	5	NA	10.514	170
2-Chloronaphthalene	10	0.084	5	NA	11.326	170
2-Nitroaniline	5	0.498	10	800	54.145	330
Dimethylphthalate	50	0.300	5	2,000	4.404	170
Acenaphthylene	NA	0.047	5	41,000	1.381	170
2,6-Dinitrotoluene	5	0.509	5	1,000	41.304	170
3-Nitroaniline	5	1.549	10	800	38.813	330
Acenaphthene	20	0.112	5	50,000	1.984	170
2,4-Dinitrophenol	10	2.224	10	800	59.061	330
4-Nitrophenol	NA	1.525	10	800	40.917	330
Dibenzofuran	NA	0.098	5	6,200	1.757	170
2,4-Dinitrotoluene	5	0.447	5	NA	26.134	170
Diethylphthalate	50	0.110	5	7,100	5.100	170
4-Chlorophenyl-phenylether	NA	0.167	5	NA	3.598	170
Fluorene	50	0.074	5	50,000	3.889	170
4-Nitroaniline	5	0.455	10	NA	18.856	330
4,6-Dinitro-2-methylphenol	NA	2.274	10	NA	58.290	330
N-Nitrosodiphenylamine	50	0.260	5	NA	9.228	170
4-Bromophenyl-phenylether	NA	0.900	5	NA	53.705	170
Hexachlorobenzene	0.04	0.445	5	410	8.386	170
Atrazine	7.5	1.087	5	NA	7.511	170
Pentachlorophenol	1	5.144	10	1,000	57.896	330
Phenanthrene	50	0.113	5	50,000	3.542	170
Anthracene	50	0.056	5	50,000	4.322	170
Carbazole	NA	0.089	5	NA	1.953	170
Di-n-butyl phthalate	50	0.299	5	8,100	58.349	170
Fluoranthene	50	0.098	5	50,000	2.446	170
Pyrene	50	0.068	5	50,000	1.093	170
Butylbenzylphthalate	50	1.740	5	50,000	45.328	170
3,3'-Dichlorobenzidine	5	0.375	5	NA	148.000	170
Benzo(a)anthracene	0.002	0.257	5	330	2.914	170
Chrysene	0.002	0.273	5	400	1.688	170
bis(2-Ethylhexyl)phthalate	5	4.760	5	50,000	54.386	170
Di-n-octyl phthalate	50	0.241	5	50,000	3.948	170
Benzo(b)fluoranthene	0.002	0.385	5	1,100	3.275	170
Benzo(k)fluoranthene	0.002	0.066	5	1,100	1.858	170
Benzo(a)pyrene	ND	0.091	5	330	4.069	170
Indeno(1,2,3-cd)pyrene	0.002	0.153	5	3,200	4.669	170
Dibenz(a,h)anthracene	NA	0.200	5	330	1.985	170
Benzo(g,h,i)perylene	NA	0.362	5	50,000	2.026	170
9,10-anthracenedione	NA	5*	10	NA	150*	330

See Notes on Page 3.

Table 3. Parameters, Methods, and Target Reporting Limits, Quality Assurance Project Plan, Chevron Environmental Management Company, Former Tappan Terminal Site, Hastings-on-Hudson, New York

Analyte	Water (ug/L)			Soil (ug/kg)		
	NYS GW STD./G.V.[3]	Laboratory MDL	Laboratory RL	TAGM G.V.[4]	Laboratory MDL	Laboratory RL
Semivolatile Organic Compounds 8270 [2] (Cont'd.)						
1,4-dihydroxy-9,10-anthracendione	NA	11.8	40	NA	112	660
1-hydroxy-9,10-anthracenedione	NA	8.0*	20	NA	250*	660
0-chloroaniline	NA	1.17	10	NA	29.5	330
(z)-9-octadecenamide	NA	31.2	100	NA	817	3300
2-methyl-benzenamine	NA	1.48	10	NA	68.9	330
p-aminotoluene	NA	1.08	10	NA	165**	330
Wet Chemistry and Miscellaneous						
Alkalinity (EPA Method 310.1)	NA	790	5000	--	--	--
Alkalinity-Bicarbonate (EPA Method 310.1)	NA	5000	10000	--	--	--
Carbon Dioxide (RSK175)	NA	210	1000	--	--	--
Chloride (EPA Method 300.0)	250000	282	500	--	--	--
Ferrous Iron (SM 3500 FeD)	300	15	100	--	--	--
Manganese (EPA Method 6010)	300	0.16	3	--	--	--
Methane (RSK175)	NA	0.2219	1	--	--	--
Nitrate (EPA Method 353.2)	10000	11	50	--	--	--
Nitrogen as ammonia (350.1)	2000	9	20	--	--	--
Phosphorous as orthophosphate (365.2)	20	6	20	--	--	--
Sulfate (EPA 375.4)	250000	580	5000	--	--	--
Sulfide (SM4500-S-2D)	50	22	100	--	--	--
Total Dissolved Organic Carbon (SW-9060)	NA	360	1000	--	--	--
Total Organic Carbon (SW-9060)	NA	360	1000	--	7,200	500,000

Notes:

1 USEPA. Office of Solid Waste and Emergency Response. *Test Methods for Evaluating Solid Waste SW-846 3rd ed. Washington, D.C. 1996.*

2 The target reporting limits are based on wet weight. The actual reporting limits will vary based on sample weight and moisture content.

3 Water guidance values (GV) are as presented in the NYSDEC, Division of Water, Technical and Operation Guidance Series (TOGS) document titled, *Ambient Water Quality Standards and Guidance Values and Groundwater Effluent Limitations* (TOGS 1.1.1), dated June 1998, last revised April 2000.

4 Soil/Sediment guidance values (GV) are as presented in the NYSDEC Technical and Administrative Guidance Memorandum (TAGM) titled, *Determination of Soil Cleanup Objectives and Cleanup Levels*, HWR-94-4046 (TAGM 4046) dated January 24, 1994.

* Based on response to MDL Verification due to non-linearity compound

** MDL Verification spike did not yield any recovery at calculated MDL. MDL adjusted to reflect concentration which can be detected (spike level of replicates).

Table 4. Sample Containers, Preservation, and Holding Times, Quality Assurance Project Plan, Chevron Environmental Management Company, Former Tappan Terminal Site, Hastings-on-Hudson, New York

Parameter	Method[1]	Bottle Type	Preservation	Holding Time[2]
Soil				
Volatile Organic Compounds	8260	1 - 4 oz glass jar with Teflon®-lined lid	Cool to 4°C	12 days to analysis
Semivolatile Organic Compounds	8270	1 - 4 oz glass jar with Teflon®-lined lid	Cool to 4°C	10 days to extraction 40 days to analysis
TCLP-Volatiles	SW-846 1311/8260-Benzene	1 - 4 oz glass jar with Teflon®-lined lid	Cool to 4°C	7 days to TCLP extraction 7 days to analysis
Reactivity	(SW846 Sect. 7.3 withdrawn)	1 - 8 oz glass jar with Teflon®-lined lid	Cool to 4°C	180 days to analysis
Flash point (Ignitability)	1010	"	"	180 days to analysis
Ferrous Iron	SM3500-Fe-D	"	"	immediately after extraction
Total Organic Carbon	SW-9060	1 - 2 oz glass jar with Teflon®-lined lid	Cool to 4°C	14 days to analysis
Water				
Volatile Organic Compounds	8260	2 - 40 ml glass vials with Teflon®-lined lid	HCl to pH<2 Cool to 4°C	12 days to analysis
Semivolatile Organic Compounds	8270	2 - 1 liter amber glass bottle with Teflon®-lined lid	Cool to 4°C	5 days to extraction 40 days to analysis
Alkalinity	(EPA Method 310.1)	1 - 1 L poly container, no headspace	Cool to 4°C	12 days to analysis
Alkalinity-Bicarbonate	(EPA Method 310.1)	"	"	12 days to analysis
Carbon Dioxide	(ASTM Method D1946)	2 - 40 ml glass vials with Teflon®-lined lid	HCl to pH<2	12 days to analysis
Chloride	(EPA Method 300.0)	1 - 8 oz plastic	Cool to 4°C	26 days to analysis
Ferrous Iron	(SM3500-Fe-D)	1 - 4 oz plastic	Cool to 4°C	immediately
Manganese	(EPA Method 6010)	1 - 8 oz plastic	HNO3 to pH<2	180 days to analysis
Methane	(RSK 175)	2 - 40 ml glass vials with Teflon®-lined lid	HCl to pH<2	12 days to analysis
Nitrate	(EPA Method 353.2)	1 - 8 oz plastic	Cool to 4°C	24 hours to analysis
Nitrogen as ammonia	(EPA Method 350.1)	1 - 8oz plastic	H2SO4 to pH<2	26 days to analysis
Phosphorous as orthophosphate	(EPA Method 365.2)	"	Cool to 4°C	24 hours to analysis
Sulfate	(ASTM D516)	1 - 8 oz plastic	Cool to 4°C	26 days to analysis
Sulfide	(SM4500-S-2D)	1 - 8 oz plastic	Zinc Acetate + NaOH to pH>9	5 days to analysis
Total Dissolved Organic Carbon	(SW-9060)	2 - 40 ml glass vials with Teflon®-lined lid	HCl to pH<2, filtered	26 days to analysis

Notes:

1 USEPA. Office of Solid Waste and Emergency Response. *Test Methods for Evaluating Solid Waste. SW-846 3rd ed. Washington, D.C. 1996.*
USEPA. Methods for Chemical Analysis of Water and Waste. EMSL-Cincinnati. 1983:
APHA. *Standard Methods for the Examination of Water and Wastewater.* Washington, DC. 1998.
ASTM International. 2003. *Annual Book of ASTM Standards 2003 Section 4 Construction*, Volume 04.08. West Conshohocken, PA. ASTM International.
Department of the Army. 1986. *Engineering Manual Laboratory Soils Testing*. Washington, D.C. Department of the Army, Office of the Chief of Engineers.

2 All holding times are measured from date of collection.

3 VTSR = Verified Time of Sample Receipt (ASP 2005 hold times expressed as VTSR).

Table 5. Electronic Data Deliverable (EDD) Format, Quality Assurance Project Plan, Chevron Environmental Management Company, Former Tappan Terminal Site, Hastings-on-Hudson, New York

Field Name	Maximum Length	Data Type	Comments
FIELD SAMPLE ID	50	TEXT	From the chain of custody. Add "RE" or "DL" to differentiate reanalyses and dilutions.
SDG	50	TEXT	
LAB SAMPLE ID	50	TEXT	
MATRIX	10	TEXT	SOIL, WATER, SEDIMENT, etc.
SAMPLE TYPE	10	TEXT	FB, RB, TB, FD, FS for Field Blank, Rinse Blank, Trip Blank, Field Duplicate and Field Sample, respectively. DEFAULT TO FS
DATE COLLECTED	--	DATE/TIME	MM/DD/YY
TIME COLLECTED*	--	DATE/TIME	Military time
DEPTH START	--	NUMBER	
DEPTH END	--	NUMBER	
DEPTH UNITS	25	TEXT	FEET, INCHES, METERS, etc.
ANALYTICAL METHOD	50	TEXT	
CAS NUMBER	25	TEXT	
ANALYTE	100	TEXT	
RESULT VALUE	--	NUMBER	For non-detected results, enter Reporting Limit ("U" must be present in Lab Qualifier field).
LAB QUALIFIER	10	TEXT	"U" for non-detected, others as defined by laboratory.
REPORTING LIMIT	--	NUMBER	
RESULT UNIT	25	TEXT	
DILUTION FACTOR	--	NUMBER	
REPORTABLE RESULT	--	YES/NO	DEFAULT TO YES
FILTERED?	--	YES/NO	
DATE ANALYZED	--	DATE/TIME	MM/DD/YY
TIME ANALYZED*	--	DATE/TIME	Military time
DATE EXTRACTED*	--	DATE/TIME	MM/DD/YY
LABORATORY NAME*	50	TEXT	

Notes:

1 This definition is for an "Excel-type" spreadsheet. Fields flagged with an "*" are optional and may be left blank if not available electronically from the laboratory.

2 Depth-related fields may be left blank for samples and matrices for which they are not applicable.

ATTACHMENTS

ARCADIS BBL

Attachment 1

Laboratory Standard Operating Procedures

STL Buffalo
LABORATORY STANDARD OPERATING PROCEDURES

SOP No.	Revision No.	Effective Date	Page
AGV-RSK-05	1	October 27, 2005	1 of 17

TITLE: Dissolved Gases – Modified Method RSK – 175

Supersedes: Revision 0

REVIEWED & APPROVED BY:	SIGNATURE	DATE
Gary T. Rudz, Supervisor		
Verl D. Preston, Quality Manager		
Christopher A. Spencer, Laboratory Director		

1.0 IDENTIFICATION OF TEST METHOD

1.1. This method is a modification of RSK SOP-175 from the USEPA RS Kerr Laboratory.

2.0 APPLICABLE MATRIX

2.1. Groundwater, drinking water, surface water and other aqueous samples.

3.0 REPORTING LIMITS

Compound	µg/L
Methane	1
Ethane	2
Ethane	2

4.0 SCOPE AND APPLICATION

4.1. This method is used to qualify and quantify aliphatic and olefinic hydrocarbons normally found in the gas phase at room temperature in water. The method is applicable to the preparation of water samples for the analysis of the headspace through introduction into a capillary column equipped with gas chromatography. This method is restricted to use by or under the supervision of analysts experienced in the use of gas chromatography and the integration of gas chromatography.

TITLE: Dissolved Gases – Modified Method RSK – 175

Supersedes: Revision 0

5.0 SUMMARY OF THE TEST METHOD

5.1. A water sample is collected in the field in a 44mL VOA vial with no headspace. Prior to analysis the sample is transferred into a 22mL serum vial with a crimp cap. Headspace is generated using UHP helium. The sample is loaded onto the headspace autosampler and analyzed by gas chromatography equipped with an FID detector. The headspace concentration is related to the starting water concentration through the use of Henry's Law.

6.0 DEFINITIONS

6.1. *Henry's Law*: Henry's Law states that the ration of the partial pressure of a gas in a closed system and molar concentration in solution is a constant. This constant varies with temperature. The constant is compound specific. (See Attachment A).

6.2. Definitions of other terms used in this SOP may be found in the STL Buffalo Laboratory Quality Manual (LQM).

7.0 INTERFERENCES

7.1. Method interference may be caused by contaminants in solvents, reagents, glassware and other processing apparatus that lead to discreet artifacts. All of these materials must be routinely demonstrated to be free from interference under conditions of the analysis by running laboratory method blanks as described in the Quality Control section. Specific selection of reagents may be required to avoid introduction of contaminants.

7.2. Background atmospheric methane is commonly present at levels of 5-10 ppm in the atmosphere. This may cause small amounts of methane to be present in laboratory blanks (typically less than 0.25 ug/L).

7.3. Carry-over contamination is routinely not a problem with this analysis due to the volatile nature of the gases being tested. The characteristics of the column and the GC temperature program are hot enough and long enough to prevent carryover. Each sample has its own new VOA vial and is directly injected on the GC. The syringe is flushed between samples. The analyst must be familiar with the characteristics of the system to determine when carryover may have occurred. Reanalysis of suspected carryover samples must be done as soon as possible.

8.0 SAFETY

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

TITLE: Dissolved Gases – Modified Method RSK – 175

Supersedes: Revision 0

8.2. The toxicity or carcinogenicity of each reagent used in this SOP has not been precisely defined. Additional health and safety information can be obtained from the applicable Material Safety Data Sheets (MSDSs) maintained in the laboratory and on-line in the EH&S section of the STL IntraNet (Oasis).

8.3. Each sample is treated as a potential health hazard. Exposure to each sample is reduced to the lowest possible level by whatever means available.

8.4. Personal protective equipment, including but not necessarily limited to eye protection, lab coats and gloves must be worn and used as specified in the CSM. This includes all personnel, visitors and contractors that are in a laboratory area unless that area has been designated by the EH&S Coordinator as an exclusion area.

8.5. All standard making is preformed in the hood, while wearing proper personal protective equipment.

8.6. PRIMARY MATERIALS USED

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

Material (1)	Hazards	Exposure Limit (2)	Signs and symptoms of exposure
Hydrochloric Acid	Corrosive Poison	5 ppm-Ceiling	Inhalation of vapors can cause coughing, choking, inflammation of the nose, throat, and upper respiratory tract, and in severe cases, pulmonary edema, circulatory failure, and death. Can cause redness, pain, and severe skin burns. Vapors are irritating and may cause damage to the eyes. Contact may cause severe burns and permanent eye damage.
1 – Always add acid to water to prevent violent reactions.			
2 – Exposure limit refers to the OSHA regulatory exposure limit.			

9.0 EQUIPMENT AND SUPPLIES

9.1. Gas Chromatograph – Analytical system complete with gas chromatograph and a headspace autosampler and all required accessories, including FID, column supplies, recorder, gases and syringes. A data system for measuring peak heights and/or areas is recommended.

TITLE: Dissolved Gases – Modified Method RSK – 175

Supersedes: Revision 0

9.2. Column A: RTX-QPLOT (Restek #19718) – column temperature maximum 250°C fused silicon 0.32 min ID x 30 meter.

9.3. Column B: RTX-UPLOT (Restek #19724) – column temperature maximum 250°C fused silicon 0.32 min ID x 30 meter.

9.3.1. Second column confirmation is optional for most protocols and procedures but is required for AFCEE samples

9.4. Sample Containers: 44mL VOA vials, 22mL crimp cap vials.

9.5. Tekmar® 7000 headspace autosampler

9.6. Syringes: various sizes from 10uL to 5mL gastight syringes.

9.7. Various sample "loops"

9.8. Tedlar bags: 1 Liter

10.0 REAGENTS AND STANDARDS

10.1. Gas cylinders of ultrahigh purity helium, and nitrogen.

10.2. Calibration Standards: The standard is composed of 1% (molar basis) or 10,000 ppmv Methane, Ethane, Ethene and Acetylene.

10.2.1. A true second source material is not available for these calibration standards. STL Buffalo attempts to obtain two different lot numbers in order to perform calibration verification.

10.3 The calibration levels are achieved by injecting different amounts of the primary source calibration standard into a 22-ml vial that contains 17 ml of deionized water and 5 ml of headspace.

10.4 Laboratory Control Sample (LCS of MSB) and Initial Calibration Verification Samples: The LCS/MSB and ICV are prepared using the secondary source calibration standard and are fortified to the concentration of the middle calibration standard.

11.0 SAMPLE COLLECTION, PRESERVATION, SHIPMENT AND STORAGE

11.1. Samples are collected in the field in a 44mL VOA vial. The samples are preserved with 1:1 HCL to a pH of less than 2.

11.2. Care should be taken that no headspace is present when capping the vials.

TITLE: Dissolved Gases – Modified Method RSK – 175

Supersedes: Revision 0

11.3. Samples are maintained at a temperature of 4+/-2°C and must be analyzed within 14 days of collection.

11.4. If dissolved CO2 and CO are being determined sample should be collected in 44mLVoa vial with no preservative. If acid is added, dissolved carbonates will converted to CO2 and bias the results.

12.0 QUALITY CONTROL

12.1. Initial Demonstration of Capability:

12.1.1. For the standard analyte list, the initial demonstration of capability and method detection limits (MDL) studies described in Section 16 must be acceptable before analysis of samples may begin.

12.1.2. For non-standard analytes a MDL study should be performed and calibration curve generated before analyzing any samples. In any event the minimum initial demonstration required is analysis of an extracted standard at the reporting limit and a single point calibration.

12.2. In–house historical control limits should be determined for matrix spikes and laboratory control samples. These limits are reviewed annually.

12.3. Batch definition: The batch is set up of up to 20 samples of the same matrix processed using the same procedures and reagents within the same time period. The batch should contain a method blank, a laboratory control sample (LCS/MSB) and a matrix spike/matrix spike duplicate (MS/MSD). If insufficient sample is available for a MS/MSD, a LCSD (MSBD) may be substituted.

12.4. Matrix Spike / Matrix Spike Duplicates – MS and MSD samples are to be prepared at a frequency of at least 5% (1 MS per 20 samples and 1 MSD per 20 samples). Batch MS/SD (i.e., 1/20 over a number of job/cases) is an option to fulfilling the requirements, but at least one (1) set of MS/SD should be run per day of instrument operation.

12.4.1. The MS and MSD are fortified at a concentration equal to Level C of the calibration curve.

12.5. Laboratory Control Sample / Matrix Spike Blank – a minimum of one (1) matrix spike blank is required every 20 samples or 24 hours, whichever comes first. The LCS/MSB is prepared using the secondary source calibration standard.

12.5.1. Spiking levels for LCS are the same as for MS/MSD (Level C of curve).

12.5.2. The LCS/MSB must provide a recovery that is within $\pm$50% of its theoretical value (until internal limits are established). In the event of LCS failure, re-analysis is required. If re-

analysis continues to indicate LCS failure, all sample analyses completed relative to that LCS are subject to re- analysis.

12.5.3. If the LCS recoveries are biased high and the associated samples are ND for the parameter of interest, the sample data is acceptable and may be processed for reporting.

12.6. Method Blank- A method blank must be analyzed once every 20 samples or 24 hours, whichever comes first. The method blank consists of reagent water containing all reagents specific to the method and is carried through the entire analytical procedure.

12.6.1. If a method blank exhibits contamination above the Laboratory Quantitation Limit, all related samples results must be evaluated.

12.6.1.1. Positive method blank results slightly below the reporting limit should still be evaluated by the analyst for potential impact on sample results at or near the reporting limit.

12.6.1.2. The common lab contaminant for this method is Methane. Methane can to be present up to 5 times the reporting limit.

12.6.2. Samples containing the same analytes found in the method blank must be re-analyzed.

12.0 CALIBRATION AND STANDARDIZATION

12.1. Using the primary source calibration standard, a five-point curve is established for each compound of interest using the concentrations noted in the table below:

Compound	Cal Level A	Cal Level B	Cal Level C	Cal Level D	Cal Level E
Volume Injected (ul)	**2**	**5**	**10**	**20**	**50**
Volume Diluent (ml)	**16.998**	**16.995**	**16.990**	**16.980**	**16.950**
Methane (ug/L)	0.772	1.93	3.86	7.72	19.29
Ethane (ug/L)	1.33	3.38	6.76	13.52	33.76
Ethene (ug/L)	1.45	3.62	7.24	14.48	36.17

13.1.1. For the initial calibration curve to be acceptable it must meet the appropriate criteria of either average response factor or correlation coefficient curve fit.

13.1.1.1. The average calibration factor may be used if the average percent Relative Standard Deviation (%RSD) of the response factors is $\leq$30%.

13.1.1.2. The correlation coefficient may be used if it is $\geq$0.995.

13.1.2. Removal or replacement of levels from the middle of a calibration is not permitted unless an injection or instrument problem confined to that point can be clearly documented. Removal of points for individual analytes from levels other than the highest and lowest is not permitted in any event.

13.2. Initial Calibration Verification (ICV): A check of the initial calibration curve must be made after the initial calibration. The ICV consists of the injection of a second source standard prepared at Level D of the calibration curve.

13.2.1. The ICV must meet ±15% of its theoretical value.

13.3. Continuing Calibration Verification (CCV): For calibration verification (i.e. continuing calibration) of the analytical curve, a standard prepared at Level D of the curve (20 ul in 16.98 ml VOA free water) must be analyzed every 20 samples or 24 hours (whichever comes first) and at the end of each analysis sequence.

13.3.1. The CCV response factor must be within ±30% of the calibration average response factor.

13.4. Retention time windows (RTW): To obtain retention windows, three levels from an initial calibration are used to calculate standard deviation and the mean for all target compounds. The width of the retention time window is ±3X the standard deviation of the mean absolute retention time from the 3 levels in the initial calibration. The retention times are updated based on the analyte retention time in the Daily CCV.

14.0 PROCEDURE

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

14.2. Any unauthorized deviations from this procedure must also be documented as a job exception, with a cause and corrective action described.

14.3. Sample Analysis

14.3.1. Remove samples from the refrigerator and allow them to come to room temperature.

14.3.2. Transfer the sample into a 22mL vial with a crimp cap.

14.3.3. Place the vial upside down, and then insert a 22-gauge needle into the septum.

14.3.4. Using an additional 5-mL gastight syringe, inject 5 mL of UHP helium into the sample. The helium forces out an equal amount of sample through the 5-mL gastight syringe to create a headspace volume of 5mL.

14.3.5. Withdraw the syringes from the vial and load the sample onto the Tekmar® headspace autosampler. The autosampler allows the sample's water and headspace phases to equilibrate at 60°C.

14.3.6. 100uL of the sample headspace are injected directly onto the GC column where target compounds, if present, are detected by the FID. The instrument operating conditions are outlined below.

14.4. Sample Dilutions:

14.4.1. Measure 20-40 mls of VOA free water into a 50-ml Class-A volumetric flask

14.4.2. Using a gas-tight syringe, measure a set amount of sample and introduce into the volumetric.

14.4.3. Finish filling the volumetric to the meniscus with VOA free water

14.4.4. Stopper and invert several times to complete the mixing.

14.4.5. Transfer sample to a 22ml vial with crimp cap

14.4.6. Proceed with analysis as indicated in sections 14.3.3-14.3.6.

14.4.7. Example: For a 10 fold dilution

14.4.7.1. Use 40 mls VOA free water in a 50-ml volumetric

14.4.7.2. Introduce 5 mls of sample

14.4.7.3. Bring to final volume of 50-ml with VOA free water

14.5. Instrument Performance Specifications – The gas chromatograph is set up to reflect the following operating conditions:

14.5.1. Injection B Temp. 200°C

14.5.2. Detector B 250°C

14.5.3. Oven Maximum 250°C

14.5.4. Range 2 0

14.5.5. Signal 1 Att. 0

14.5.6. Signal 2 Att. 0

14.6. Column conditions reflect the constituents of interest in a particular analysis.

14.6.1. Initial Temperature 45°C

14.6.2. Initial Time 2 min.

14.6.3. Rate 20°C/min.

14.6.4. Final Temperature 220°C

14.6.5. Final Time 0 min.

14.7. Tekmar® 7000 autosampler conditions:

14.7.1. Platen- 65°C

14.7.2. Platen Equilibrium Time- 1.0

14.7.3. Sample Equilibrium Time- 3.0

14.7.4. Vial Size- 20ml

14.7.5. Mix – On

14.7.6. Mix Power- 3

14.7.7. Stabilize- 2.0

14.7.8. Pressure- .50

14.7.9. Pressure Equilibrium Time- .20

14.7.10. Loop- 0.3

14.7.11. Loop Equilibrium- 0.05

14.7.12. Injection- 0.5

14.7.13. Valve- 120°C

14.7.14. Line - 120°C

14.7.15. Cycle Time- 12

14.8. Upon establishment of or verification of established instrumental operating conditions, the following instrument maintenance procedures are performed daily:

14.8.1. Analyze blanks(s) before samples and determine system to be clean and free of interference.

14.8.2. If interference is present: (1) bake out column at 230°C, and/or (2) cut capillary column at injector end about 6-12 inches.

14.8.3. Log maintenance activities into the instrument maintenance log.

14.9. Analytical Documentation

14.9.1. Record all analytical information in the analytical logbook, including the analytical data from standards, blanks, LCS/MSBs, MS/MSDs and any corrective actions or modifications to the method.

14.9.2. All standards are logged into the department standard logbook. All standards are assigned a unique number for identification.

14.9.3. Documentation such as all associated instrument printouts and daily calibration data corresponding to all final runs is available for each data file.

14.9.4. Sample results and associated QC are reviewed by the primary analyst and entered into the laboratory LIMs system. A secondary technical review and evaluation of the LIMs data is performed and documented prior to release of data for reporting.

15.0 CALCULATIONS

15.1. Calibration Factor for GC-FID

$$\mathbf{CF\chi = Ax/C\chi}$$

where:

$CF\chi$ = Calibration factor of compound χ
$A\chi$ = Peak height or area
$C\chi$ = Calibration amount

15.2. Percent Difference for Calibration Factors

$$\%D = (CF_{ave} - CF_c / CF_{ave}) \times 100$$

where:
CF_{ave} = Average CF for an analyte from the initial calibration
CF_c = CF for an analyte from current check standard

15.3. Relative Standard Deviation

$$\%RSD = (SD/CF_{ave}) \times 100$$

where:
CF_{ave} = Average CF for an analyte for the initial calibration
SD = Standard Deviation of average CFs for a compound

15.4. Sample concentration in water

$$C\chi = (A\chi/CF_{ave}) \times DF$$

where:
$C\chi$ = Concentration of target analyte χ in sample (ug/L)
$A\chi$ = Peak area of analyte χ
CF_{ave} = Average calibration factor for an analyte for the calibration
DF = Dilution Factor

16.0 METHOD PERFORMANCE

16.1. Each analyst, prior to sample analysis, must analyze an Initial Demonstration of Capability.

16.1.1. Four replicate QC check standards composed of 20ul of Scotty Specialty Gases at 1% (mole basis) analyzed with a mean recovery of 80-120% provides an acceptable IDOC.

16.2 A Method Detection Limit Study (MDL) is performed on an annual basis in accordance with the current specifications described in 40CFR part 136, Appendix B. The final MDL for each analyte should be at least less than ½ of the laboratory quantitation limit (but preferably less than 1/3).

TITLE: Dissolved Gases – Modified Method RSK – 175

Supersedes: Revision 0

17.0 DATA ASSESSMENT AND ACCEPTANCE CRITERIA FOR QUALITY CONTROL MEASURES

17.1. Initial Calibration Curve (ICAL):

17.1.1. Average Response Factor (RF) $\leq$30%
or
17.1.2. Correlation coefficient $\geq$0.995

17.2 Initial Calibration Verification (ICV) $\pm$15% of theoretical value (This is the 2nd source standard analyzed immediately after the initial calibration curve)

17.3 Continuing Calibration Verification (CCV) $\pm$30% of average response factor

17.4 Laboratory Control Sample (LCS/MSB) $\pm$50% of theoretical value

17.5 Matrix Spike/Matrix Spike Duplicate (MS/MSD) – within historically based statistical limits.

17.5.1 If MS/MSD fall outside QC limits but LCS/MSB is provides acceptable results, no corrective action is required.

17.6 Method Blank (MB) – less than laboratory quantitation limit

18.0 CORRECTIVE ACTIONS FOR OUT-OF-CONTROL DATA

18.1. ICAL: Analysis can not begin without an acceptable calibration. Instrument maintenance may be required. Refer to STL Corporate Policy P-T-001, R3 for information on the proper selection of calibration points.

18.2. ICV: Reanalyze calibration curve if unacceptable ICV is obtained.

18.3. CCV:

18.3.1. Reanalyze the CCV

18.3.1.1. If the 2nd analysis is acceptable, analytical sequence may continue, however the previous 10 samples must be re-analyzed.

18.3.1.2. If the 2nd analysis is unacceptable, analyze a new ICAL

18.3.2. If the CCV is out high and there are no positives in the samples, the results may be reported. This situation must however be noted in the logbook and on the Job Summary.

18.3.3 GVOA Decision Tree - running 2 consecutive CCVs

18.3.3.1 Due to the use of auto-samplers, two CCVs may be analyzed after each 10 samples. The following decision tree must be used to evaluate the use of the corresponding sample data.

IF: CCV1 Passes
CCV2 Passes
Continue analysis sequence; sample data acceptable before and after CCV pair.

IF: CCV1 Passes
CCV2 Fails
Re-analyze all samples injected after failed CCV.

IF: CCV1 Fails
CCV2 Passes
Continue analysis sequence; sample data acceptable before and after CCV pair.

IF: CCV1 Fails
CCV2 Fails
Perform maintenance and/or re-calibration. Re-analyze samples injected before and after the failed CCVs.

18.4. Method Blank: Reanalyze all samples associated with an unacceptable method blank.

18.5. MSB (LCS):

18.5.1. If below limits: Reanalyze all samples associated with an unacceptable MSB

18.5.2. If above limits: Reanalyze all samples with detections. Reanalysis is *not required* if samples are ND.

18.6. MS/MSD:

18.6.1. Matrix interference can be assumed and corrective action is not required if both of the following conditions are met:

18.6.1.1. MSB recovery is acceptable

18.6.1.2. Recoveries in both the MS and MSD are consistent (RPD<30%)

18.6.1.2.1. If sample appearance indicates that the MS/MSD pair may not provide reproducible results, the poor results may be accepted but this should be noted in the job summary and case narrative.

18.6.2. If MSB is unacceptable – reanalysis of batch is required.

18.6.3. If recoveries in MS/MSD are different (i.e.: one high, one low) further evaluation should be made. Matrix interference can not be assumed in this case. Discussion with the department supervisor, operations manager or QA manager should be included in the final decision process prior to releasing data.

19.0 CONTINGENCIES FOR HANDLING OUT-OF-CONTROL OR UNACCEPTABLE DATA

19.1. The analyst will communicate any non-correctable out-of-control data to the Project Manager using the laboratory Job Exception Form. Issues which require generation of a Job Exception may be, but are not limited to, the following items.

19.1.1. Holding time exceedances

19.1.2. Unacceptable LCS/MSB recoveries

19.1.3. Matrix-related interferences which impact data quality

19.1.4. Insufficient sample volume provided for analysis

19.2. The Project Manager will notify the client of any holding time exceedances or necessity for reporting data with unacceptable quality control. This notification will be done as soon as possible to allow for scheduling of resampling events if required.

20.0 WASTE MANAGEMENT/ POLLUTION PREVENTION

20.1. 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."

20.2. Proper disposal of liquid volatile analysis waste is based upon the type of solvent used. The solvent used is water.

20.3. Disposal of liquid volatile waste is broken down into two categories: Aqueous waste and solvent waste.

20.3.1. Aqueous waste is temporarily stored in a laboratory approved waste receptacle and labeled "A" waste.

20.3.2. Solvent waste is stored in laboratory approved metal waste receptacle and labeled "C" waste.

20.4 Waste receptacles are taken to sample control where they are disposal of.

20.5 Glass waste such as pipettes and vials are rinsed and disposed of in approved glass receptacles.

21.0 REFERENCE

21.1. Method D 4128-89, "Standard Practice for Identification of Organic Compounds in Water by Combined Gas Chromatograph and Electron Impact Mass Spectrometry", American Society for Testing and Materials.

21.2. USEPA RSK SOP-175, Revision 0, August 11, 1994

21.3. "STL Buffalo Laboratory Quality Manual", Current revision

21.4. "Chemical Hygiene Plan", January 1991, Severn Trent Laboratories.

22.0 TABLES, DIAGRAMS, FLOWCHARTS AND VALIDATION DATA

22.1 Attachment 1: Table of Compound Constants

22.2 Attachment 2: Example Calculation

23.0 CHANGES FROM PREVIOUS REVISION

23.1. Laboratory Director change, updated signature

23.2. Section 8.1: Included Corporate EH&S statement.

23.3. Section 10.2.1: Removed reference to two separate vendors. Stated that 2nd lot# was used for calibration verification.

23.4. Section 18.0: Specific Corrective Action information included for out-of-control QC indicators. Included dual CCV decision tree.

23.5. Section 20.1: Included Corporate EH&S statement related to waste disposal regulations.

TITLE: **Dissolved Gases – Modified Method RSK – 175**

Supersedes: **Revision 0**

Attachment 1:

Table of Compound Constants

Compound	Molecular Weight (g)
Methane	16
Ethane	30
Ethene	28
Propane	44
Propene	42

TITLE: **Dissolved Gases – Modified Method RSK – 175**

Supersedes: **Revision 0**

Attachment 2:

Example Calculation

(Based on 22°C at 754 mmHg – Molar Equivalent 0.04099)

(Level D) 20ul of 10,000 ppmv – 17 ml H2O

$$\frac{10{,}000\ ul CH_4}{L N_2} \times \frac{1 L CH_4}{1{,}000{,}000\ ul CH_4} \times \frac{0.04099\ moles CH_4}{1 L CH_4} \times \frac{16\ g CH_4}{1 mole CH_4} \times \frac{0.00002\ L N_2}{0.017\ L H_2} \times$$

$$\frac{1{,}000{,}000\ ug CH_4}{1 g CH_4} = \frac{0.1312}{0.017} = \frac{7.72\ ug CH_4}{L H_2O}$$

STL BUFFALO
LABORATORY STANDARD OPERATING PROCEDURES

SOP No.	Revision No.	Effective Date	Page
AMB-8270C-66	6	August 30, 2005	1 of 46

TITLE: ANALYTICAL METHODS FOR GC/MS SEMIVOLATILE SAMPLES BY SW846 3RD EDITION METHOD 8270C

SUPERCEDES: Revision 5

REVIEWED & APPROVED BY:	SIGNATURE	DATE
Verl Preston, Quality Manager		
Christopher A. Spencer, Laboratory Director		
P. McNamara, Supervisor		

1.0 IDENTIFICATION OF TEST METHODS

1.1 Method 8270C, SW846, 3RD Edition, update III.

2.0 APPLICABLE MATRIX

2.1 Waters, soils, sludges and TCLP.

3.0 REPORTING LIMIT

3.1 See Table 1 for a listing of laboratory quantitation limits.

4.0 SCOPE AND APPLICATION:

4.1 The analytical method is utilized for the analysis of water, air sampling media, sediment and soil from hazardous waste sites for the organic compounds listed in Table 1. Table 1 includes CAS numbers and estimated quantitation limits for each analyte. Typical sample size should be 30 grams for soils and 1 liter for waters. The method begins with the extraction of the sample aliquot either by sonication (soils) or separatory funnel extraction (waters), into 1:1 methylene chloride/ acetone mixture. The extraction volume is then concentrated to 1.0ml final volume for waters and soils. The extracts are prepared for analysis with the addition of internal standard to each vial. One microliter of each extract is then directly injected into a gas chromatograph and the compounds are separated by

mass using a capillary column and analyzed using a mass spectrometer. A summary of the analysis procedure is provided in Attachment A.

5.0 SUMMARY OF TEST METHOD

5.1 See Scope and Application.

6.0 DEFINITIONS

6.1 All definitions are in parenthesis. Additional definitions scan be found in the STL Buffalo Laboratory Quality Manual (LQM).

7.0 INTERFERENCES

7.1 Some of the possible interferences that arise during GCMS Semivolatile analysis include, but are not limited to:

1. Glassware contamination
2. Matrix interference
3. Aldol condensation
4. System air leaks
5. Injection port/liner contamination
6. Warped filament, and/or dirty source and rods
7. APIX analytes Methapyrilene and Phentermine split at all concentrations and require manual integration in calibration standard.

7.2 Raw GC/MS data from all blanks, samples, and spikes must be evaluated for interferences. Determine if the source of interference is in the preparation and/or cleanup of the samples and take corrective action to eliminate the problem.

7.3 See section 1.4 and 3.0 of method 8270c for other interferences, with the exception that there is no carryover in direct injection GCMS.

8.0 SAFETY:

All STL employees are required to comply with STL's Corporate Safety Manual, which has been prepared in accordance with OSHA requirements. This includes wearing the proper laboratory clothing such as lab coat, gloves and safety glasses.

The health and safety hazards of many of the chemicals used in this procedure have not been fully defined. Additional health and safety information can be obtained from the MSDS files maintained in the laboratory. The following specific hazards are known:

Chemicals that have been classified as carcinogens or potential carcinogens, under OSHA include: Benzo(a)anthracene, benzidine, 3,3'-dichlorobenzidine, benzo(a)pyrene, dibenzo(a,h)anthracene, and n-nitrosodimethylamine. Primary standards should be purchased in solution. If neat materials must be obtained, they shall be handled in a hood.

Exposure to chemicals must be maintained as low as reasonably achievable; therefore, unless they are known to be non-hazardous, all samples should be opened, transferred, and prepared in a fume hood, or under other means of mechanical ventilation. Solvent and waste containers should be kept closed unless transfers are being made.

8.1 Analysts are expected to use caution and common sense while working in a laboratory environment. Each employee is required to read the companies' Corporate Safety Manual. All of the samples to be analyzed have the potential to contain hazardous substances. Most standards also contain hazardous chemicals and many do contain known carcinogens. Employees must use protective equipment when handling standards, samples and extracts including gloves, lab coats and safety glasses. It is the analyst's responsibility to read and familiarize themselves with the MSDS of each chemical and/or reagent involved in this method.

8.2 Samples, standards and/or extracts should never be opened or transferred outside of a fume hood.

8.3 Waste disposal is all C waste with the exception of some acids used in the cleaning of equipment which is disposed of in AN waste.

8.4 Spills should be cleaned up promptly and waste should be disposed of as per the Chemical Hygiene Plan.

8.5 There is also the danger of burns while doing repair or maintenance on a gas chromatograph. One must use caution while working on or near the injection port or transfer line.

8.6 PRIMARY MATERIALS USED

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

Material (1)	Hazards	Exposure Limit (2)	Signs and symptoms of exposure
Methanol	Flammable Poison Irritant	200 ppm-TWA	A slight irritant to the mucous membranes. Toxic effects exerted upon nervous system, particularly the optic nerve. Symptoms of overexposure may include headache, drowsiness and dizziness. Methyl alcohol is a defatting agent and may cause skin to become dry and cracked. Skin absorption can occur; symptoms may parallel inhalation exposure. Irritant to the eyes.
Methylene Chloride	Carcinogen Irritant	25 ppm-TWA 125 ppm-STEL	Causes irritation to respiratory tract. Has a strong narcotic effect with symptoms of mental confusion, light-headedness, fatigue, nausea, vomiting and headache. Causes irritation, redness and pain to the skin and eyes. Prolonged contact can cause burns. Liquid degreases the skin. May be absorbed through skin.
Sodium Hydroxide	Corrosive	2 Mg/M3-Ceiling	Severe irritant. Effects from inhalation of dust or mist vary from mild irritation to serious damage of the upper respiratory tract, depending on severity of exposure. Symptoms may include sneezing, sore throat or runny nose. Contact with skin can cause irritation or severe burns and scarring with greater exposures. Causes irritation of eyes, and with greater exposures it can cause burns that may result in permanent impairment of vision, even blindness.
Sulfuric Acid	Corrosive Oxidizer Dehydrator Poison Carcinogen	1 Mg/M3-TWA	Inhalation produces damaging effects on the mucous membranes and upper respiratory tract. Symptoms may include irritation of the nose and throat, and labored breathing. Symptoms of redness, pain, and severe burn can occur. Contact can cause blurred vision, redness, pain and severe tissue burns. Can cause blindness.
1 – Always add acid to water to prevent violent reactions.			
2 – Exposure limit refers to the OSHA regulatory exposure limit.			

9.0 EQUIPMENT AND SUPPLIES:

9.1 Calibrated micro syringes 10, 25, 50, 100, 500, 1,000 microliter.

9.2 2ml amber vials and caps.

TITLE: ANALYTICAL METHODS FOR GC/MS SEMIVOLATILE SAMPLES BY SW846 3RD EDITION METHOD 8270C

SUPERCEDES: Revision 5

9.3 Disposable pipets and pipet bulbs.

9.4 Volumetric flasks.

9.5 Gas Chromatograph/Mass Spectrometer (GC/MS) System

9.5.1 Gas Chromatograph –

- Hewlett Packard 6890
- Carrier gas Helium UPC grade or equivalent

9.5.2 Gas Chromatography Column

- Analysis: ZB-5 or ZB-5MS (Crossbond 5% diphenyl-95% dimethyl polysiloxane) 30 meter 0.25mm ID.25 or 0.50 µmdf, or an equivalent alternative.

9.5.3 Deactivated Guard Column installed between injection port and chromatograghic column with deactivated union.

9.5.4. Mass Spectrometer

- HP5973 and HP5973 inert
- Tuning compound PFTBA
- Scan Range 35-500 AMU/second

9.5.5 Data System

- HP Chemstation
- Teknivent and HP enviroquant software

10.0 REAGENTS AND STANDARDS:

10.1 Methylene Chloride – high purity

10.2 Standards:

10.2.1 Stock Standards

Calibration Mix #1 1000µg/ml
Calibration Mix #2 2000µg/ml
Benzidines Mix 2000 µg/ml
N-Nitrosodiphenylamines 5000 µg/ml

OLM Mix 2000 µg/ml
Benzoic Acid 2000 µg/ml
BN/AP Surrogate Mix 4000 µg/ml
DFTPP mix 50µg/ml or equivalent
Internal Standard Mix 2.0 mg/ml

All Certificates of Analysis received from the manufacturer are maintained in a laboratory notebook. Stock standards are prepared every twelve months or sooner, if necessary.

10.2.2 Initial and Continuing Calibration Solutions

8270 Stock Solution

Standard	Solvent	Stock Conc.	Initial Wt/Vol.	Final Vol.	Final Conc.	Final Conc. In Samples
Calibration Mix # 1	$MECL_2$	1000 ng/ul	400µl	2000ul	200 ng/ul	200 ug/L
Calibration Mix # 4	$MECL_2$	2000 ng/ul	200µl	2000ul	200 ng/ul	200 ug/L
Benzidines Mix	$MECL_2$	2000 ng/ul	200µl	2000ul	200 ng/ul	200 ug/L
N-Nitrosodiphenylamine Mix	$MECL_2$	5000 ng/ul	80µl	2000ul	200 ng/ul	200 ug/L
BN/AP Mix	$MECL_2$	4000 ng/ul	100µl	2000ul	200 ng/ul	200 ug/L
OLM Mix	$MECL_2$	2000 ng/ul	200µl	2000ul	200 ng/ul	200 ug/L
Benzoic Acid	$MECL_2$	2000 ng/ul	400µl	2000ul	200 ng/ul	400 ug/L

10.2.3 Working Standards

10.2.3.1. Surrogate Standard Spiking Solution (A00001-AIMS® Code)
Surrogate Standard Spiking solution is prepared that contains nitrobenzene-d5, terphenyl1-d14, 2-fluorobiphenyl, and 1,2-dichlorobenzene-d4 at a concentration of 100µg/ml; phenol-d5, 2,4,6-tribromophenol, 2-fluorophenol and 2-chlorophenol-d4 at a concentration of 150µg/ml. Surrogate standards are added to all samples and calibration solutions. Additional surrogates may be added at the laboratory's discretion.

Standard	Solvent	Stock Conc.	Initial Wt/Vol.	Final Vol.	Final Conc. In Samples
Semivolatile Acid Surrogate Phenol-d5 2,4,6-Tribromophenol 2-Fluorophenol 2-Chlorophenol-d4	MEOH	10,000ng/ul	1,500ul	100,000ul	150ug/L
Semivolatile B/N Surrogate Nitrobenzene-d5 Terphenyl-d14 2-Fluorobiphenyl 1,2-Dichlorobenzene-d4	MEOH	5000ng/ul	2,000ul	100,000ul	100ug/L

10.2.3.2. Matrix Spiking Solution (11 compound) (A00055-AIMS® Code)
The 11 compound matrix spiking solution consists of the following:

Bases/Neutrals	Acids
1,2,4-Trichlorobenzene	Pentachlorophenol
Acenaphthene	Phenol
2,4-Dinitrotoluene	2-Chlorophenol
Pyrene	4-Chloro-3-methylphenol
N-Nitroso-di-n-propylamine	4-Nitrophenol
1,4-Dichlorobenzene	

a. Using the Intermediate Acid and BN Standards, the Matrix Spike solution is prepared that contains each of the base-neutral compounds above at 100µg/ml in methanol and the acid compounds at 100µg/ml in methanol.

Standard	Solvent	Stock Conc.	Initial Wt/Vol.	Final Vol.	Final Conc. in Solution	Final Conc. In Aqueous Samples
Acid Matrix Spike Intermediate	MeOH	10000ng/ul	5000uls	500mls	100 ug/ml	100 µg/L
BN Matrix Spike Intermediate	MeOH	5000ng/ul	10000uls	500mls	100 ug/ml	100 ug/L

10.2.3.3 Matrix Spiking Solution (all compound) (A00193- AIMS® Code)

The all compound matrix spiking solution contains each of the following SVOA target analytes at 100µg/ml in methanol. Additional compounds may be included in the spike mixture if required for a specific project.

Ancenaphthene	Dibenzo(a,h)anthracene	Indeno(1,2,3-cd)pyrene
Acenaphthylene	Dibenzofuran	Isophorone
Anthracene	di-n-butyl phthalate	2-Methylnaphthalene
Benzo(a)anthracene	1,2-Dichlorobenzene	2-Methylphenol
Benzo(b)fluoranthene	1,3-Dichlorobenzene	4-Methylphenol
Benzo(k)fluoranthene	1,4-Dichlorobenzene	Naphthalene
Benzo(ghi)perylene	3,3'Dichlorobenzidine	2-Nitroaniline
Benzo(a)pyrene	2,4-Dichlorophenol	3- Nitroaniline
Benzoic acid	Diethyl phthalate	4- Nitroaniline
Benzyl alcohol	2,4-Dimethylphenol	Nitrobenzene
Bis(2-chloroethoxy)methane	Dimethyl phthalate	2-Nitrophenol
Bis(2-chloroethyl)ether	4,6-Dinitro-2-methylphenol	4-Nitrophenol
2,2'-oxybix(1-Chloropropane)	2,4-Dinitrophenol	N-nitrosodiphenylamine
Bis(2-ethylhexyl)phthalate	2,4-Dinitrotoluene	N-Nitroso-Di-n-propylamine
4-Bromophenyl phenyl ether	2,6-Dinitrotoluene	Pentachlorophenol
Butyl benzyl phthalate	Di-n-octyl phthalate	Phenanthrene
2-Chloroaniline	Fluoranthene	Phenol
4-Chloro-3-methylphenol	Fluorene	Pyrene
2-Chloronaphthalene	Hexachlorobenzene	1,2,4-Trichlorobenzene
2-Chlorophenol	Hexachlorobutadiene	2,4,5-Trichlorophenol
4-Chlorophenyl phenyl ether	Hexachlorocyclopentadiene	2,4,6-Trichlorophenol
Chrysene	Hexachloroethane	

10.2.3.4. Instrument Performance Check Solution (DFTPP)

A solution of Decafluorotriphenylphosphine (DFTPP) at a concentration of 50 ng/µL in methylene chloride is prepared such that a 1µL injection will result in a final concentration of 50ng on column. The instrument performance check solution also contains Benzidine, Pentachlorophenol and 4,4-DDT for use in evaluating chromatographic performance.

DFTPP Check Solution

Standard	Solvent	Final Conc.
DFTPP	$MECL_2$	50 µg/ml
Benzidine	$MECL_2$	50 µg/ml
Pentachlorophenol	$MECl_2$	50 µg/ml
4,4'-DDT	$MECL_2$	50 µg/ml

10.2.3.5. Initial and Continuing Calibration Solutions

Calibration standards are prepared at a minimum of five concentration levels (10, 50, 80, 120, and 160 total ng per 1µL). Each calibration standard should contain each compound of interest and each surrogate.

Standard	Solvent	Stock Conc.	Initial Wt/Vol.	Final Vol.	Final Conc,	Final Conc. In Samples
50ppm SSTD 050 Internal Standard Custom Mix	$MECL_2$	200 ng/ul 2000ng/ul 500ng/ul	250µl 20µl 60ul	1000ul	50 ng/ul 40ng/ul 80ng/ul	50ug/l
10ppm SSTD 010 Internal Standard Custom Mix	$MECL_2$	200 ng/ul 2000 ng/ul 500 ng/ul	50µl 20µl 80ul	1000ul	10ng/ul 40ng/ul 50ng/ul	10ug/l
80ppm SSTD 080 Internal Standard Custom Mix	$MECL_2$	200 ng/ul 2000 ng/ul 500 ng/ul	400µl 20µl 40ul	1000ul	80 ng/ul 40ng/ul 100ng/ul	80ug/l
120ppm SSTD 120 Internal Standard	$MECL_2$	200 ng/ul 2000 ng/ul	300µl 10µl	500ul	120 ng/ul 40ng/ul	120ug/l
160ppm SSTD 160 Internal Standard	$MECL_2$	200 ng/ul 2000 ng/ul	400µl 10µl	500ul	160 ng/ul 40ng/ul	160ug/l

10.2.3.6. Internal Standard Solution

Internal Standard solution is prepared at a concentration to maintain a 40ng level per 1µl. The Internal Standard solution contains the following compounds at a concentration of 40ng/µl, Acenaphthene-d10, Chrysene-d12, 1,4-Dichlorobenzene-d4, Napthalene-d8, Perylene-d12 and Phenanthrene-d10.

10.2.3.7. Storage of Standard Solutions

Stock, secondary dilution, and working standards are stored at 4°C or less in teflon-lined crimp-cap amber bottles or vials. These standards are prepared every twelve months or sooner, if necessary.

The continuing calibration standard (50ng) is stored at 4°C or less in teflon-lined crimp-cap amber vials. This standard may be prepared weekly, but may be continued to be used if no degradation and/or evaporation has occurred.

Samples, sample extracts and standards are stored in separate refrigerators.

10.2.3.8 Expiration of Standard Solutions.

Before any analyst uses a standard solution he or she must consult the "Standards Chemical History Log" to determine when or if the standard has expired.

11.0 SAMPLE COLLECTION, PRESERVATION AND STORAGE

11.1 Water samples may be collected in 1L (or more) amber glass containers with teflon-lined, screw-caps.

11.2 Soil/Sediment Samples may be collected in glass containers fitted with teflon-lined screw-caps or closed end tubes.

11.3 All samples are stored at 4 C (+/-2C) from the time of collection until extraction

11.4 Aqueous samples must be extracted within 7 days of collection and analyzed within 40 days of extraction.

11.5 Soil samples must be extracted within 14 days of collection and analyzed within 40 days of extraction.

TITLE: ANALYTICAL METHODS FOR GC/MS SEMIVOLATILE SAMPLES BY SW846 3RD EDITION METHOD 8270C

SUPERCEDES: Revision 5

12.0 QUALITY CONTROL

12.1 Method Blanks - A method blank is a volume of a clean reference matrix (reagent water for water samples, or purified sodium sulfate/clean sand for soil/sediment samples) that is carried through the entire analytical procedure. The volume or weight of the reference matrix must be approximately equal to the volume or weight of samples associated with the blank. The purpose of a method blank is to determine the levels of contamination associated with the processing and analysis of samples.

12.1.1 A method blank must be prepared once for the following, whichever is more frequent:

- Each prep batch

- Each 20 samples in a batch, including matrix spikes/matrix spike duplicates, that are of a similar matrix (water, soil/sediment or similar concentration (soil/sediment only), or

- Whenever samples are extracted by the same procedure (separatory funnel extraction or sonication).

12.1.2 For semivolatile analysis, a method blank for water samples consists of 1 L volume of reagent water spiked with 1.0mL of the surrogate spiking solution. For medium or low level soil/sediment samples, a method blank consists of 1g or 30g of sodium sulfate/clean sand spiked with 1.0mL of the surrogate spiking solution, respectively. Extract, concentrate, cleanup and analyze the blank according to procedures for water and soil samples.

12.1.3 Acceptance Criteria – levels of target analytes in the method blank must be less than the required reporting limit or less than one-tenth the concentration of the respective analyte in the associated samples. For USACE all target analytes must be less than one half of the MRL (Method Reporting Limit) and common laboratory contaminants must be less than the MRL. The MRL is set at either the MDL or the MDL Check.

12.1.4 Corrective Actions for Method Blank Analyses - If the acceptance criteria for method blank analysis are not met, the analytical system may be assumed to be out of control. The following corrective actions may be taken:

- If contamination is the problem, then the source of the contamination must be investigated and appropriate corrective measures must be taken and documented before further sample analysis proceeds. It is the laboratory's responsibility to ensure that method interferences caused by contaminants in solvent, reagents, glassware, and sample storage and processing hardware that lead to discrete artifacts and/or elevated baselines in the GC/MS be eliminated. Samples associated with the contaminated blank must be re-extracted and re-analyzed.

- If surrogate recoveries in the method blank do not meet the acceptance criteria, first reanalyze the method blank. If the surrogate recoveries do not meet the acceptance criteria after reanalysis, re-extract and re-analyze the blank and all associated samples OR the samples may be reported as estimated, and noted in the case narrative.

- If the method blank does not meet internal standard response requirements, check calculations, the internal standard spiking solutions, and the instrument operation. If the calculations were incorrect, correct the calculations and verify that the internal standard responses meet their acceptance criteria. If the internal standard compound spiking solution was improperly prepared, concentrated, or degraded, re-prepare solutions and re-extract/reanalyze samples. If the instrument malfunctioned, correct the instrument problem and reanalyze the method blank. If the instrument malfunction affected the calibration, recalibrate the instrument before reanalyzing the blank

12.2 Matrix Spike Blank/Matrix Spike/Matrix Spike Duplicate(MSB/MS/MSD)

12.2.1 A matrix spike blank, matrix spike and matrix spike duplicate are analyzed to evaluate the analytical system and the effects of sample matrix on the methods used for semivolatile analysis.

12.2.2 The matrix spike blank, matrix spike, and matrix spike duplicate are spiked with the compounds of interest (at concentrations noted in the standard preparation section).

12.2.3 A matrix spike blank, matrix spike and matrix spike duplicate are extracted and analyzed for every batch of 20 samples of a similar matrix. Matrix spike and matrix spike duplicates are not performed for field QC samples such as rinsates, or field/trip blanks

12.2.4 If insufficient sample amount is received to perform matrix spike and matrix spike duplicate analysis, duplicate matrix spike blanks may be processed.

12.2.5 Dilutions

Dilutions of MS/MSD samples are performed only if the unspiked sample requires a dilution in order to maintain any target compound concentrations in the upper half of the calibration. MS/MSD samples will not be diluted to get spiked or non-spiked compounds below the highest calibration standard.

12.2.6 Calculations for MS/MSD

The concentrations of spiked compounds are determined using equations described for sample analysis. After determining the compound concentrations, the percent recovery is calculated using Equation 1.

Equation 1

$$\text{Matrix Spike Recovery} = \frac{\text{SSR - SR}}{\text{SA}} x100$$

Where,
SSR= Spike Sample Result
SR = Sample Result
SA = Spike Added

The relative percent difference between the matrix spike and matrix spike duplicate is calculated using Equation 2.

Equation 2

$$\text{RPD} = \frac{[\text{MSR - MSDR}]}{1/2\,(\text{MSR} + \text{MSDR})} \text{ x } 100$$

Where,
RPD = Relative Percent Difference
MSR = Matrix Spike Recovery
MSDR = Matrix Spike Duplicate Recovery

The vertical bars in the formula above indicate the absolute value of the difference, hence RPD is always expressed as a positive value

12.2.7. Technical Acceptance Criteria for MS/MSD

The acceptance criteria for sample analysis (retention time, surrogate and IS recovery) must be met for matrix spike and matrix spike duplicate analysis also.

The matrix spike recovery limits are based on historical data and are updated annually.

The matrix spike recovery limits are advisory. If the recovery limits are not met, no further corrective action will be necessary. However, frequent occurrences of this nature should be investigated.

Re-extraction and re-analysis of the matrix spike and matrix spike duplicate may be necessary if, in the technical judgment of the analyst and/or supervisors, an error was made during the extraction procedure

12.3 Technical Acceptance Criteria for MSB:

The acceptance criteria for sample analysis (retention time, surrogate and IS recovery) must be met for the matrix spike blank analysis also.

The matrix spike blank recovery limits are based on historical data and are updated annually.

If the Matrix Spike Blank was found to be unacceptable all samples in the associated batch must be re-extracted and re-analyzed. If the sample was not within extraction hold time, a job exception must be filed and both analysis must be included with the report.

12.4 Surrogate Recoveries

The surrogate compound concentrations are determined using calculations found in Section 9. The recoveries are then determined using Equation 3

Equation 3

$$\%\ Recovery = \frac{Concentration\ (\vee\ amount)\ found}{Concentration\ (\vee\ amount)\ spiked}$$

Recovery limits for surrogate compounds are based on historical data and are updated annually.

12.5 QC Acceptance Criteria for AFCEE or USACE projects are provided in Attachments B and C respectively.

13.0 CALIBRATION AND STANDARDIZATION

13.1 Instrument Operating Conditions

- Gas Chromatograph; The following are recommended GC conditions that may vary slightly depending on the compound list and the column film thickness.

Initial Temperature: 40-50°C
Initial Hold Time: 3 minutes (hold time may vary to ensure proper chromatographic separation).

Temperature program 40-50°C to 70°C at 20°C/min to 195 at
16°C/min to 325 at 30°C/min
Final Temperature: 325°C
Final Hold Time: As necessary for TCL compound identification
Injector Temperature: 250°C
Source Temperature: 230°C
Transfer Line Temperature: 310°C
Injector: splitless
Front Inlet Pressure: 7.00 psi
Purge Flow: 15.0 mL/min
Purge Time: 0.50 min
Total flow: 19.2 mL/min
Injection Volume: 1µl
Carrier Gas: Helium
Carrier Flow: 36 cm/sec

- Mass Spectrometer

Electron Energy: 70 volts (nominal)
Mass Range: 35 to 500 amu
Scan Time: Not to exceed 1 second per scan

13.2 Instrument Performance Check

The GC/MS system is tuned using Perfluorotributylamine (PFTBA) such that an injection of 50ng of DFTPP will meet the abundance criteria listed in Table 2.

Prior to the analysis of standards or samples, the mass calibration and resolution of the GC/MS system is verified by the analysis of DFTPP. This analysis will verify the proper tuning of the system for 12 hours. After 12 hours, the instrument performance must be verified before standard and sample analysis may continue.

The mass spectrum of DFTPP may be background subtracted to eliminate column bleed or instrument background ions.

Breakdown of 4,4'-DDT into 4,4'-DDD and 4,4'-DDE may be used to assess GC column performance and injection port inertness and must be less than 20%.

The compounds Benzidine and Pentachlorophenol should be present and at their normal responses for this concentration. Peak tailing should not be visible (PCP tailing factor <5 and Benzidine <3). If responses are poor and excessive peak tailing is present, corrective actions for the GC/MS instrument performance check solution may be required.

All subsequent standards and samples must be acquired under the same GC/MS tuning conditions that were used for the analysis of the instrument performance check solution.

13.2.1 Technical Acceptance Criteria for the GC/MS Instrument Performance Check (DFTPP) is listed in Table 2.

13.2.2 Corrective Actions for the GC/MS Instrument Performance Check
If any of the acceptance criteria are not met, the DFTPP should be re-injected to insure that the injection made was not a cause for failure. If, after reinjection, acceptance criteria have not been met, one or more of the following corrective actions may be taken:

1. Retune the GC/MS
2. Clean the source; replace parts, etc...
3. Cut the column at the injector end
4. Replace the column
5. Replace the septum in the injector
6. Replace the injector liner
7. Clean injection port with $MeCl_2$
8. Change injection port seal
9. An instrument service call may be placed.

13.3 Initial Calibration

After the instrument performance check criteria has been met and prior to the analysis of samples, the GC/MS system is calibrated at a minimum of five concentration levels in order to establish instrument sensitivity and linearity.

The initial calibration shall be performed when major instrument maintenance has been performed or if continuing calibration criteria cannot be met.

Major instrument maintenance may consist of source cleaning, column changing, or quadrapole rod adjustment. Preventative maintenance such as septum changes, injector liner changes or column cutting may not require an initial calibration to be performed.

13.3.1 Procedure

Five calibration standards are prepared which contain all target and surrogate compounds. A 20µl aliquot of internal standard solution is added to a 1mL aliquot of each calibration standard solution. The resulting concentration of internal standards is 40ng. A 1µl injection would result in a final concentration of 40ng on column. The internal standards used are given in Table 3.

The relative response factors (RRF) for each target and surrogate compound is determined using equation 4. The characteristic ions for a given compound are listed in Tables 3 and 6. Internal standard assignments are listed in Table 4.

Equation 4

$$RRF = \frac{A_x}{A_{is}} x \frac{C_{is}}{C_x}$$

Where,
A_x = Area of the characteristic ion for the compound to be measured (see Table 4)
A_{is} = Area of the characteristic ion for specific internal standard (see Table 3)
C_{is} = Amount of the internal standard injected (ng)
C_x = Amount of the compound to be measured injected (ng)

The mean relative response factor (RRF) must be calculated for all compounds. Calculate the % Relative Standard Deviation (%RSD) of the RRF values for the initial calibration using the following equation:

Equation 5

$$\%RDS = \frac{Standard\ Deviation}{Mean} x\ 100$$

Where,

$$Standard\ Deviation = \sqrt{\frac{\sum_{i+1}^{n} (X_i - \overline{X})^2}{(n-1)}}$$

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

13.3.2 Acceptance Criteria for Initial Calibration

The average response factor (RRF) for each System Performance Check Compound (listed in Table 5) must be greater than or equal to the compound's minimum acceptable relative response factor of 0.050.

The %RSD over the initial calibration range for relative response factor for each Calibration Check (Table 5) compound %RSD must be less than or equal to the 30%.

The %RSD over the initial calibration range for the relative response factor for all other compounds must be less than or equal to 15%.

OR

The mean %RSD for all compounds must be less than or equal to 15% .

OR

A least squares regression correlation coefficient of greater than 0.990 for all compounds greater than 15% RSD.

OR

A non-linear coefficient of determination of greater than 0.990 for all compounds greater than 15% RSD. For a 2nd order non-linear regression, 6 calibration points must be used and for a 3rd order non-linear regression, 7 calibration points must be used.

13.3.3. Corrective Actions for Initial Calibration

If any of the acceptance criteria for initial calibration are not met, it may be necessary to reanalyze one or more of the calibration standards. If after reanalysis, the acceptance criteria have not been met, it may be necessary to take further corrective actions.

The following corrective actions may be taken if the acceptance criteria for initial calibration cannot be met.

1. Prepare fresh standards and reanalyze the initial calibration.
2. Replace the septum on the injector
3. Replace the injector liner
4. Cut the column at the injector end
5. Retune the GC/MS system and reanalyze the instrument performance check
6. Clean the source
7. An instrument service call may be placed

The acceptance criteria must be met before sample analysis may proceed.

13.4 Initial Calibration Verification

To verify the accuracy of the initial calibration, a standard is obtained from a source different from the calibration standards.

Immediately following analysis of an acceptable initial calibration curve, a 80ng/µl aliquot of this independent standard is injected.

Recoveries of all compounds shall fall within ±25% of the expected value, however, recoveries of up to 40% are allowable for up to four compounds.

13.5 Continuing Calibration

If there is no time left in the 12-hour time period after initial calibration, the instrument performance check may be analyzed and a 50ng/1µl standard may be analyzed to verify the calibration of the instrument.

The continuing calibration check must be analyzed once every 12-hour time period of operation. This check must be analyzed prior to the analysis of samples for a given 12-hour time period.

13.5.1 Procedure for Continuing Calibration

The 50ng/µl standard is used for the continuing calibration. The relative response factor is calculated using procedures described for initial calibration.

If quantitation is performed using response factor, calculate the percent difference between the mean relative response factor from the most recent initial calibration and the continuing calibration relative response factor for each semivolatile target and surrogate compound using Equation 6.

Equation 6

$$\%\ Difference_{RRF} = \frac{RRF_c - \overline{RRF_i}}{\overline{RRF_i}} \times 100$$

Where,

$\overline{RRF_i}$ = Mean relative response factor from the most recent initial calibration meeting technical acceptance criteria

RRF_c = Relative response factor from continuing calibration standard

If quantitation is performed using a least squares regression or a non-linear model, calculate the concentration of all analytes and surrogates in the continuing calibration as described in section 8.3.2 of this SOP. Calculate the percent drift using Equation 7.

Equation 7:

$$\%\text{Drift} = \frac{\text{Conc}_E - \text{Conc}_A}{\text{Conc}_E} x100$$

Where:

Conc_E = Expected Concentration
Conc_A = Actual Concentration

13.5.2 Acceptance Criteria for Continuing Calibration

The relative response factor (RRF) for each System Performance Check Compound must be greater than or equal 0.050.

The RRF of percent drift for Calibration Check Compounds must be less than 20%. The RRF percent difference or percent drift for all other compounds must be within ±25%, with up to four compounds within ±40%D. For expanded list and additional compounds not on the EPA TCL list a percent drift of 40% is allowed. Any analyte may have an elevated response >40%D if it is not detected in the associated samples.

Internal Standard retention times and responses are evaluated after acquisition of the continuing calibration check. If the retention time of any internal standard shifts by more than 30 seconds or the response of any internal standard is outside of the-50%to +100% range, the system shall be inspected and corrected as needed. The CCV will be reanalyzed after inspection. If the problem is not resolved, a new initial calibration must be performed.

13.5.3 Corrective Actions for Continuing Calibration

If any of the technical acceptance criteria for continuing calibration are not met, it may be necessary to reanalyze the continuing calibration standard. If after reanalysis the acceptance criteria cannot be met, further corrective actions may be required.

The following corrective actions may be taken if the acceptance criteria for continuing calibration cannot be met.

1. Replace the septum on the injector
2. Replace the injector liner
3. Replace injection port seal
4. Cut the column at the injector end

5. Retune the GC/MS system and reanalyze the instrument performance check
6. Prepare fresh standards
7. Reanalyze the initial calibration

13.6 Calibration acceptance criteria for AFCEE and USACE projects are provided in Attachments B and C respectively.

14.0 PROCEDURE:

14.1 Sample extracts shall be analyzed only after the GC/MS system has met the instrument performance check, initial calibration, continuing calibration and second source calibration verification requirements. The same instrument conditions must be employed for the analysis of samples as were used for calibration.

Internal standard solution is added to each sample extract. 20µL of internal standard solution is added to each accurately measured 1.0mL of water sample extract. For soil/sediment samples and water samples subjected to GPC, 10µL of internal standard solution is added to each accurately measured 0.5mL of sample extract. This will result in a concentration of 40ng/µL of each internal standard.

Necessary dilutions are made prior to adding internal standard solution. The internal standard solution must be added so that the concentration of each internal standard is 40ng/µL.

14.2. Dilutions

Dilutions of sample extracts are required if any target compound exceeds the initial calibration range. The dilution chosen should keep the response of the largest target compound within the calibration range.

14.3. Qualitative Identification

14.3.1 Target Compounds

Target compound identification is done by comparing the sample mass spectrum to that of the standard. The following criteria must be satisfied in order to verify identifications. Elution of the sample analyte within GC relative retention time unit window established from the 12-hour calibration standard.

Correspondence of the sample analyte and calibration standard component mass spectra.

To establish correspondence of the GC relative retention time (RRT), the sample component RRT must compare with $\pm$0.06 RRT units of that of the standard RRT. If samples are

analyzed within the same 12-hour period as the initial calibration, the 50ng standard is used to verify relative retention times.

To establish correspondence of the sample component mass spectra to that of the standard, the following criteria must be met:

- All ions present in the standard mass spectrum at a relative intensity greater than 10.0 percent (most abundant ion in the spectrum equals 100.0 percent) must be present in the sample spectrum.

- The relative intensities of ions specified in the paragraph above must agree within $\pm$20.0 percent between the standard and sample spectrum. (Example: For an ion with an abundance of 50.0 percent in the standard spectrum, the corresponding sample ion abundance must be between 30.0 and 70.0 percent).

- Ions greater than 10.0 percent in the sample spectrum but not present in the standard spectrum must be considered and accounted for by the analyst making the comparison. The verification process should favor false positives. All compounds meeting the identification criteria must be reported with their spectra. When target compounds are below contract required quantitation limits (CRQL) but the spectrum meets the identification criteria, report the concentration with a "J".

If a compound does not meet all of the above criteria, but in the technical judgment of the mass spectral interpretation specialist the identification is correct, the compound will be identified. Documentation of such by the specialist on the raw data is required.

14.3.2 Non-Target Compounds

A library search may be executed for non-target sample components for the purpose of tentative identification. For this purpose, the NIST/EPA/NIH mass spectral library is used to identify non-target compounds of greatest apparent concentration by a forward search of the library. The following compounds will not be identified by a library search routine:

a. Internal standard compounds
b. Surrogate compounds
c. Volatile target compounds

Peaks that are suspected to be aldol-condensation reaction products (i.e., 4-methyl-4-hydroxy-7-pentanone and 4-methyl-3-pentene-2-one) are searched and reported as part of the 30 tentatively identified compounds.

14.3.3 Guidelines for Making Tentative Identifications

Major ions in the reference spectrum (ions greater than 10 percent of the most abundant ion) should be present in the sample spectrum.

The relative intensities of the major ions should agree within ±20 percent.
Molecular ions present in reference spectrum should be present in sample spectrum.

Ions present in the sample spectrum but not in the reference spectrum should be reviewed for possible background contamination or presence of co-eluting compounds.

Ions present in the reference spectrum but not in the sample spectrum should be reviewed for possible subtraction from the sample spectrum because of background contamination or co-eluting compounds.

If, in the technical judgment of the mass spectral interpretation specialist, no tentative identification can be made the compound will be reported as unknown. Further identification may be possible, such as molecular weights or classifications (i.e., unknown hydrocarbon, unknown acid, etc.)

Pesticide target compounds may be tentatively identified by a library search.

14.4 Technical Acceptance Criteria For Sample Analysis

The samples must be analyzed on a GC/MS system meeting the DFTPP initial calibration, continuing calibration, and blank technical acceptance criteria. The sample must undergo cleanup procedures, when required, on a GPC meeting the acceptance criteria for GPC calibration.

The sample must be extracted and analyzed within the holding times.

The sample must have an associated method blank meeting the blank acceptance criteria.
All Matrix Spike Blank recoveries must fall within the laboratory derived limits. Recoveries above the upper control limit are acceptable as long as the analyte was not detected in the associated samples above the quantitation limit.

All surrogates should fall within the laboratory derived limits (Up to one BN and/or one AP surrogate may fall outside the control limit as long as the recovery is greater than 10%).

The relative retention time of each surrogate must be within ±0.06 RRT units of its relative retention time in the continuing calibration standard.

The instrumental response (EICP area) for each of the internal standards must be within the inclusive range of -50.0 percent and +100.0 percent of the response of the internal standards in the most recent continuing calibration analysis.

The retention time shift for each of the internal standards must be within ±0.50 minutes (30 seconds) between the sample and the most recent continuing calibration standard analysis.

Excluding those ions in the solvent front, no ion may saturate the detector. No target compound concentration may exceed the upper limit of the 12-hour standard calibration range unless a more dilute aliquot of the sample extract is also analyzed.

14.5 Corrective Actions for Sample Analysis

The technical acceptance criteria must be met before data are reported. Contamination from laboratory sources requires re-extraction and reanalysis.

14.5.1 Surrogate Compounds

If the technical acceptance criteria for surrogate compound recoveries are not met, the following corrective actions are taken in the given order:

a. Calculations, injection volumes, preparation volumes are checked to insure that an error was not made; if all calculations, volumes, etc., were correct the analyst will proceed to the next step in the corrective action process.

b. The sample is re-injected to insure that an error during injection was not made. If after re-injection, surrogate recoveries are outside of the acceptance criteria, the analysis will proceed to the next step in the corrective action process.

c. The sample is re-extracted. Exceptions: (1) in the case where the recoveries in a sample, MS/MSD agree (i.e., all samples exhibited recoveries outside of criteria limits) it will be noted in the Case narrative. (2) Insufficient sample remains for re-extraction. In this instance, the client will be contacted in order to determine the next procedure to follow. If this situation should arise, it will be documented in the Case narrative. (see form B: Re-extraction request form).

d. After re-extraction, the sample is re-injected. If after re-analysis surrogate recoveries are within criteria limits, this extract is considered the first because the original problem may have been due to a laboratory error. If, after re-analysis surrogate recoveries are not within criteria limits, a matrix effect may be assumed. If this should occur, both analyses may be reported. The instance will be documented in the Case Narrative.

14.5.2 Internal Standard Compounds

If the technical acceptance criteria for internal standard recoveries are not met, the following corrective actions are taken in the given order:

a. Calculations, internal standard solution volumes and injected volumes are checked to insure that an error was not made. If all calculations and volumes were correct the analyst will proceed to the next step in the corrective action process.

b. The sample is re-injected to insure that the instrument was working properly. If after re-analysis, the internal standard recoveries are with criteria limits, the second analysis will be reported only. If after re-analysis the internal standard recoveries are outside of criteria limits, both analyses will be reported and it may be assumed that a matrix effect was involved. If this instance should arise, it will be documented in the Case Narrative.

Exception: If internal standard recoveries of a sample, MS/MSD agree (i.e., recoveries are outside of criteria limits for all three samples, it may be assumed that a matrix effect is involved and no corrective action is necessary. The instance will be documented in the Case Narrative.

14.5.3 Relative Retention Times

If the technical acceptance criteria for the relative retention times of the internal standard compounds or surrogate compounds are not met, the following corrective actions are taken in the given order:

a. Carrier gas, zone temperatures and instrument temperature programs are checked to insure that an error was not made or that the gas tank was not dry or clogged. If no errors are found the analyst will proceed to the next step in the corrective action process.

b. The sample is re-analyzed to insure that an error was not made during the first injection. If, after reanalysis, the relative retention times are not within the technical acceptance criteria, it may be assumed that a matrix effect was involved. Both analyses will be reported and the instance will be documented in the Case Narrative. If, after re-analysis, the relative retention times are within the technical acceptance criteria, the second analysis will be reported only.

Exception: If the relative retention times of a sample, MS/MSD agree (i.e., relative retention times are outside of criteria limits for the sample, MS and MSD, it may be assumed that a matrix effect was involved and further corrective action is not necessary.

14.5.4 Matrix Spike Blanks.

If the Matrix Spike Blank was found to be unacceptable all samples in the associated batch must be re-extracted and re-analyzed. If the sample was not within extraction hold time, a job exception must be filed and both analysis must be included with the report.

14.6 Injection Logs: Injection Logs must contain the following information:

a. Date, time, and analyst initials
b. File number (FRN), sample ID, vial #, and job #
c. Injection volume, final volume, initial volume and dilution factor
d. Indicate if tailing of degradation was present in the tune
e. References for the standard, tune mix, IS mix
f. Daily maintenance performed
g. Any non-conformances with the samples

15.0 CALCULATIONS:

15.1. Target Compounds

Target compounds identified shall be quantitated by the internal standard method. The internal standard used shall be the one assigned to that analyte for quantitation (see Table 4). The EICP area of primary characteristic ions of analytes listed in Tables 3 and 6 are used for quantitation.

In instances where manual quantitation is necessary due to co-elution baseline noise or matrix interferences, all instances will be initialed and dated by the analyst. The quantitation report is documented as such by an "m" next to the compound that has been edited. In all instances of manual integration, a hardcopy of the EICP for that compound will be supplied with the raw data, this applies to all target compounds, internal standards and surrogate compounds.

The average response factor (RRF) from the initial calibration analysis (linear model) is used to calculate the concentration in the sample. Secondary ion quantitation is allowed ONLY when there are sample interferences with the primary ion. If secondary ion quantitation is performed, the reason is then documented in the case Narrative. The area of a secondary ion cannot be used for the area of a primary ion unless a relative factor is calculated using the secondary ion.

15.2 Water Samples

The following Equation (Eq. 8) is used to determine the concentration of target compounds identified in water samples:

Equation 8

$$Concentration\ \mu g/L = \frac{(A_x)(I_s)(V_c)(Df)(GPC)}{(A_{is})(RRF)(V_o)(V_i)}$$

Where,

A_x = Area of the characteristic ion for the compound to be measured
A_{is} = Area of the characteristic ion for the internal standard
I_s = Amount of internal standard injected in nanograms (ng)
V_o = Volume of water extracted in milliliters (mL)
V_i = Volume of extract injected in microliters (µL)
V_t = Volume of the concentrated extract in microliters (µL) (V_t = 1,000 µL if sample was not subjected to GPC; V_t = 500 µL if sample was subjected to GPC)
RRF= Relative response factor determined from the 12-hour calibration standard
GPC= GPC factor.
GPC= 1.0 if water sample was not subjected to GPC;
Df = Dilution factor. The dilution factor for analysis of water samples for semivolatiles by this method is defined as follows:

$$\frac{\text{µL most conc. extract used to make dilution + µL clean solvent}}{\text{µL most conc. extract used to make dilution}}$$

If no dilution is performed, Df = 1.0

15.3 Soil/Sediment Samples

The following Equation (Eq. 9) is used to determine the concentration of target compounds in soil/sediment samples:

Equation 9

$$Concentration\ \mu g/Kg\ (Dry\ weight\ basis) = \frac{(A_x)(I_3)(V_t)(Df)(GPC)}{(A_{is})(RRF)(V_i)(W_s)(D)}$$

Where,

A_x, I_s, A_{is} are as given for water, above.
V_t = Volume of the concentrated extract in microliters (µL) (V_t = 500 µL)

V_i = Volume of the extract injected in microliters (µL)

D = $\frac{100 - \% \text{ moisture}}{100}$

W_s = Weight of sample extracted in grams (g)

GPC= GPC factor (GPC = 2.0 to account for GCP cleanup)

RRF= Relative response factor determined from the 12-hour calibration standard.

Df = Dilution factor. The dilution factor for analysis of soil/sediment samples for semivolatile by this method is defined as follows:

$$\frac{\mu L \text{ most conc. extract used to make dilution} + \mu L \text{ clean solvent}}{\mu L \text{ most conc. extract used to make dilution}}$$

If no dilution is performed, Df = 1.0.

The factor of 2.0 in the numerator is used to account for the amount of extract not recovered from the use of GPC cleanup. Concentrating the extract collected after GPC to 0.5mL maintains the sensitivity of the soil/sediment method.

15.4 Tentatively Identified Compounds

Non-Target Compounds

An estimated concentration for non-target compounds tentatively identified is quantitated by the internal standard method. For quantitation, the nearest internal standard free of interferences is to be used. The equations for calculating concentrations are the same as equations 8 and 9. Total area counts (or peak heights) from the total ion chromatograms are used for both the compounds to be measured and the internal standard. A relative response factor (RRF) of one (1) is assumed. The resulting concentration is to be qualified as "J" (estimated, due to lack of a compound specific response factor), and "N" (Presumptive evidence of presence), indicating the quantitative and qualitative uncertainties is calculated for all tentatively identified compounds as well as those identified as unknowns.

15.5 Rounding

For rounding off numbers to the appropriate level of precision, observe the following common rules. If the figure following those to be retained is less than 5, drop is (round down). If the figure is greater than 5, drop it and increase the last digit to be retained by 1 (round up). If the figure following the last digit to be retained equals 5, round up if the digit to be retained is off, and round down if that digit is even.

SOP No.	Revision No.	Effective Date	Page
AMB-8270C-66	6	August 30, 2005	29 of 46

TITLE: ANALYTICAL METHODS FOR GC/MS SEMIVOLATILE SAMPLES BY SW846 3RD EDITION METHOD 8270C

SUPERCEDES: Revision 5

15.6 Organic Significant Figures

For volatile and semivolatile results, report analytical results to one significant figure if the value is less than 10, and two significant figures if the value is above 10.

16.0 METHOD PERFORMANCE:

16.1 Method Detection Limits (MDLs)
MDL's are seven blank samples spiked with 20ng/mic of all compounds of interest. These are extracted and analyzed as both waters and soils along with a blank.

16.2 Initial Demonstration of Capability (IDOC): The initial demonstration with each sample preparation and determinative method combination utilized must be performed by generating data of acceptable accuracy and precision for target analytes in a clean matrix. This is also done for new staff or when significant changes in instrumentation are made as stated in section 8.0 of Method 8000.

17.0 DATA ASSESSMENT AND ACCEPTANCE CRITERIA FOR QUALITY CONTROL MEASURES:

17.1 When internal standards are out of range a re-injection is required unless the problem can be determined to be a result of excessive matrix interference.

17.2 When surrogates are out of range, a re-extraction is required unless excessive visible chromatographic matrix interference is present. In this case, the Project Manager should be consulted to decide how to proceed.

17.3 When a positive hit for an analyte is above the calibration range a dilution must be performed to bring the value within calibration range .

17.4 When there are low spike recoveries in the matrix spike blank the entire extraction batch needs to be re-extracted. If there are high spike recoveries the associated sample data needs to be examined to assess if it may be biased.

18.0 CORRECTIVE ACTIONS FOR OUT OF CONTROL DATA:

18.1 Corrective actions for out of control data require Project Manager, Laboratory Director and/or QA Officer notification. This can be accomplished either verbally, written using a Job Exception Report or both.

SOP No.	Revision No.	Effective Date	Page
AMB-8270C-66	6	August 30, 2005	30 of 46

TITLE: ANALYTICAL METHODS FOR GC/MS SEMIVOLATILE SAMPLES BY SW846 3RD EDITION METHOD 8270C

SUPERCEDES: Revision 5

19.0 CONTINGIENCIES FOR HANDLING OUT –OF- CONTROL OR UNACCEPTABLE DATA:

19.1 Contingency measures for handling out of control or unacceptable data requires The Project Manager to notify the client for input.

20.0 WASTE MANAGEMENT AND POLLUTION PREVENTION

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

20.1 There are two types of aqueous waste generated in the lab:

1. A-Waste: All non-nitric acid and alkaline aqueous waste.

2. AN-Waste: All aqueous waste containing nitric acid.

These types of waste are to be disposed of into appropriately market plastic containers.

The following are the other types of lab waste and where to dispose of:

1. C-Waste: all solvent waste gets dumped into appropriately marked metal cans. These cans need to be grounded whenever they are emptied to reduce explosion hazards. Discarded standards will also be dumped into C-waste cans.

2. Solid Waste: all contaminated paper, solid sample waste, sodium sulfate and all other non-glass material that has been contaminated is to be wrapped in foil and gathered to be dumped into 55 gallon drums.

3. Glass: contaminated glass needs to be rinsed off with methylene chloride and disposed of with all other glass in glass specific containers with special extra thick polypropylene liners. These containers are for glass only.

4. Extract Vials: extract vials are to be archived after they have been shot. After archival period, vials are to be crushed into a 55 gallon drum.

21.0 REFERENCE

21.1 USEPA Methods for Evaluating Solid Waste; SW-846, Third Edition, Update III, Method 8270C, 12/96.

TITLE: ANALYTICAL METHODS FOR GC/MS SEMIVOLATILE SAMPLES BY SW846 3RD EDITION METHOD 8270C

SUPERCEDES: Revision 5

22.0 TABLES, DIAGRAMS, FLOWCHARTS AND VALIDATION DATA:

22.1 Attachment A: SOP Procedure Summary

22.2 Attachment B: AFCEE Summary

22.3 Attachment C: USACE Summary

22.4 Table 1: Target Compound List and EQLs

22.5 Table 2: Ion Abundance Criteria

22.6 Table 3: Characteristic Ions for Internal Standards

22.7 Table 4: Internal Standards and Corresponding Target Compounds Assigned for Quantitation

22.8 Table 5: Relative Response Factor Criteria for ICV and CCV

22.9 Table 6: Characteristic Ions for Target Compounds and Surrogates

22.10 Attachment D: Job Summary Checklist

23.0 CHANGES FROM PREVIOUS REVISION

23.1 Section 10.0: Provided calibration concentrations in units of measure comparable to final sample concentrations.

23.2 Added Job Summary Checklist as Attachment D.

23.3 Added corporate safety and waste management sections.

23.4 Updated appropriate sections to reflect the change over to new instrumentation.

23.5 Where necessary, changed the word ‘should’ to ‘shall’.

SOP No.	Revision No.	Effective Date	Page
AMB-8270C-66	6	August 30, 2005	32 of 46

TITLE: ANALYTICAL METHODS FOR GC/MS SEMIVOLATILE SAMPLES BY SW846 3RD EDITION METHOD 8270C

SUPERCEDES: Revision 5

ATTACHMENT A - SOP PROCEDURE SUMMARY

I. Preparing the instrument;
   1. Cut column, change liner and septa, inject conditioning solution
   2. Ramp GC oven temp. to 325°C and ramp GC inj. Port pressure to 80 psi to see if pressure holds.

II. Shoot DFTPP tune mix
   1. Shoot 1 ul of the dftpp tune mix
   2. Evaluate the DFTPP peak using the 3rd Edition or criteria
   3. Evaluate the tailing factors of pentachlorophenol and benzidine.
   4. Evaluate the degradation of 4,4'-DDT to 4,4'-DDD and 4,4'-DDE.

III. Shoot single or 5pt. calibration;
   1. Shoot 1 ul of the 50ng continuing standard (CCC)
   2. Evaluate the continuing; 4pts may be out but none over 40%d.
   3. If CCC does not pass criteria, then a 5pt. curve (ICC) must be shot.

IV. Load Samples;
   1. Load blanks and MSBs in the beginning and dark samples toward the end.
   2. Very thick samples may be diluted.
   3. All samples must be shot within 12 hours of the tune injection.

V. Analyze data;
   1. Quantitate all samples; need raw and enhanced spectra for positive and negative hits and 20 TICs .
   2. Shoot dilutions on any samples with positive hits over 160ng.
   3. Shoot reinjections (RI's) on any sample that has internal standards out, unless there is severe matrix interference that accounts for the low recovery.
   4. Samples with more than one BN or AP surrogate out needs to be re-extracted (RE).

VI. AIMS Entry;
   1. Enter tunes, ICC's and CCC's.
   2. Enter all samples to be included with the job.
   3. Identify and enter all TIC's
   4. Calculate, close and run data validator.

VII. Review Data;
   1. Correct or explain any errors on the data validator.
   2. Make copies of logbooks, tunes, curves and standards and include them with the report.
   3. Check that all calculations have been made correctly.
   4. Turn in job for validation.

SOP No.	Revision No.	Effective Date	Page
AMB-8270C-66	6	August 30, 2005	33 of 46

TITLE: ANALYTICAL METHODS FOR GC/MS SEMIVOLATILE SAMPLES BY SW846 3RD EDITION METHOD 8270C

SUPERCEDES: Revision 5

Attachment B

Semivolatile organics (also known as base/neutral and acid extractables) in water and soil samples are analyzed using method SW8270C. This technique determines quantitatively the concentration of a number of SVOCs. Samples are extracted and both base/neutral and acid extracts are then concentrated through evaporation. Compounds of interest are separated and quantified using a capillary column GC/mass spectrometer. The RLs are listed in Table 7.2.10-1.

The mass spectrometer is tuned every 12 hours to give an acceptable spectrum for decafluorotriphenylphosphine (DFTPP). The tuning acceptance criteria are given in the following list as an ion abundance for each specified mass:

- mass 51 30 percent to 60 percent of mass 198
- mass 68 less than 2 percent of mass 69
- mass 70 less than 2 percent of mass 69
- mass 127 40 percent to 60 percent of mass 198
- mass 197 less than 1 percent of mass 198
- mass 198 base peak, 100 percent relative abundance
- mass 199 5 percent to 9 percent of mass 198
- mass 275 10 percent to 30 percent of mass 198
- mass 365 greater than 1 percent of mass 198
- mass 441 present, but less then mass 443
- mass 442 greater than 40 percent of mass 198
- mass 443 17 percent to 23 percent of mass 442

The IS method is used for quantitation of analytes of interest. For quantitation, RFs are calculated from the base ion peak of a specific IS that is added to each calibration standard, blank, QC sample, and sample.

Parameter/Method	Analyte	Water		Soil	
		RL	Unit	RL	Unit
Semivolatile organics Base/Neutral Extractables SW8270C	1,2,4-Trichlorobenzene	10.0	µg/L	0.7	mg/kg
	1,2-DCB	10.0	µg/L	0.7	mg/kg
	1,3-DCB	10.0	µg/L	0.7	mg/kg
	1,4-DCB	10.0	µg/L	0.7	mg/kg
	2,4-DNT	10.0	µg/L	0.7	mg/kg
	2,6-DNT	10.0	µg/L	0.7	mg/kg
	2-Chloronaphthalene	10.0	µg/L	0.7	mg/kg
	2-Methylnaphthalene	10.0	µg/L	0.7	mg/kg
	2-Nitroaniline	50.0	µg/L	3.3	mg/kg
	3-Nitroaniline	50.0	µg/L	3.3	mg/kg
	3,3'-Dichlorobenzidine	20.0	µg/L	1.3	mg/kg
	4-Bromophenyl phenyl ether	10.0	µg/L	0.7	mg/kg
	4-Chloroaniline	20.0	µg/L	1.3	mg/kg
	4-Chlorophenyl phenyl ether	10.0	µg/L	0.7	mg/kg
	4-Nitroaniline	50.0	µg/L	3.3	mg/kg
	Acenaphthylene	10.0	µg/L	0.7	mg/kg
	Acenaphthene	10.0	µg/L	0.7	mg/kg
	Anthracene	10.0	µg/L	0.7	mg/kg
	Benz (a) anthracene	10.0	µg/L	0.7	mg/kg
	Benzo (a) pyrene	10.0	µg/L	0.7	mg/kg
	Benzo (k) fluoranthene	10.0	µg/L	0.7	mg/kg
	Benzo (b) fluoranthene	10.0	µg/L	0.7	mg/kg
	Benzo (g,h,i) perylene	10.0	µg/L	0.7	mg/kg
	Benzyl alcohol	20.0	µg/L	1.3	mg/kg
	Bis (2-chloroethoxy) methane	10.0	µg/L	0.7	mg/kg
	Bis (2-chloroethyl) ether	10.0	µg/L	0.7	mg/kg
	Bis (2-chloroisopropyl) ether	10.0	µg/L	0.7	mg/kg
	Bis (2-ethylhexyl) phthalate	10.0	µg/L	0.7	mg/kg
	Butyl benzylphthalate	10.0	µg/L	0.7	mg/kg
	Chrysene	10.0	µg/L	0.7	mg/kg
	Di-n-butylphthalate	10.0	µg/L	0.7	mg/kg
	Di-n-octylphthalate	10.0	µg/L	0.7	mg/kg
	Dibenz (a,h) anthracene	10.0	µg/L	0.7	mg/kg
	Dibenzofuran	10.0	µg/L	0.7	mg/kg
	Diethyl phthalate	10.0	µg/L	0.7	mg/kg
	Dimethly phthalate	10.0	µg/L	0.7	mg/kg
	Fluoranthene	10.0	µg/L	0.7	mg/kg
	Fluorene	10.0	µg/L	0.7	mg/kg
	Hexachlorobenzene	10.0	µg/L	0.7	mg/kg
	Hexachlorobutadiene	10.0	µg/L	0.7	mg/kg
	Hexachloroethane	10.0	µg/L	0.7	mg/kg
	Indeno (1,2,3-cd) pyrene	10.0	µg/L	0.7	mg/kg
	Isophorone	10.0	µg/L	0.7	mg/kg

Parameter/Method	Analyte	Water		Soil	
		RL	Unit	RL	Unit
Semivolatile organics Base/Neutral Extractables SW8270C (concluded)	n-Nitrosodiphenylamine	10.0	µg/L	0.7	mg/kg
	n-Nitrosodi-n-propylamine	10.0	µg/L	0.7	mg/kg
	Naphthalene	10.0	µg/L	0.7	mg/kg
	Nitrobenzene	10.0	µg/L	0.7	mg/kg
	Phenanthrene	10.0	µg/L	0.7	mg/kg
	Pyrene	10.0	µg/L	0.7	mg/kg
Semivolatile organics Acid Extractables SW8270C	2,4,5-Trichlorophenol	50.0	µg/L	3.3	mg/kg
	2,4,6-Trichlorophenol	10.0	µg/L	0.3	mg/kg
	2,4-Dichlorophenol	10.0	µg/L	0.3	mg/kg
	2,4-Dimethylphenol	10.0	µg/L	0.3	mg/kg
	2,4-Dinitrophenol	50.0	µg/L	3.3	mg/kg
	2-Chlorophenol	10.0	µg/L	0.3	mg/kg
	2-Methylphenol	10.0	µg/L	0.3	mg/kg
	2-Nitrophenol	10.0	µg/L	0.3	mg/kg
	4,6-Dinitro-2-methylphenol	50.0	µg/L	3.3	mg/kg
	4-Chloro-3-methylphenol	20.0	µg/L	1.3	mg/kg
	4-Methylphenol	50.0	µg/L	2.0	mg/kg
	4-Nitrophenol	50.0	µg/L	1.6	mg/kg
	Benzoic acid	100	µg/L	5.0	mg/kg
	Pentachlorophenol	50.0	µg/L	3.3	mg/kg
	Phenol	10.0	µg/L	0.3	mg/kg

SOP No.	Revision No.	Effective Date	Page
AMB-8270C-66	6	August 30, 2005	34 of 46

TITLE: ANALYTICAL METHODS FOR GC/MS SEMIVOLATILE SAMPLES BY SW846 3RD EDITION METHOD 8270C

SUPERCEDES: Revision 5

Table 7.2.10-2. QC Acceptance Criteria for Method SW8270C

Method	Analyte	Accuracy Water (% R)	Precision Water (% RPD)	Accuracy Soil (% R)	Precision Soil (% RPD)	Assoc. IS	Assoc. Sur.
SW8270C	1,2,4-Trichlorobenzene	37–120	≤20	44–125	≤30	2	4
	1,2-DCB	33–120	≤20	45–125	≤30	1	3
	1,3-DCB	32–120	≤20	39–125	≤30	1	3
	1,4-DCB	32–120	≤20	35–125	≤30	1	3
	2,4-DNT	51–120	≤20	48–125	≤30	3	4
	2,6-DNT	49–120	≤20	48–125	≤30	3	4
	2-Chloronaphthalene	49–120	≤20	45–125	≤30	3	4
	2-Methylnaphthalene	46–120	≤20	47–125	≤30	2	5
	2-Nitroaniline	48–120	≤20	44–125	≤30	3	2
	3,3'-Dichlorobenzidine	20–120	≤20	25–128	≤30	5	6
	3-Nitroaniline	20–126	≤20	27–125	≤30	3	2
	4-Bromophenyl phenyl ether	52–120	≤20	46–125	≤30	4	1
	4-Chloroaniline	20–120	≤20	25–125	≤30	2	5
	4-Chlorophenyl phenyl ether	50–120	≤20	47–125	≤30	3	4
	4-Nitroaniline	36–120	≤20	34–125	≤30	3	2
	Acenaphthylene	50–120	≤20	44–125	≤30	3	4
	Acenaphthene	47–120	≤20	46–125	≤30	3	4
	Anthracene	54–120	≤20	53–125	≤30	4	1
	Benz (a) anthracene	56–100	≤20	52–125	≤30	5	6
	Benzo (a) pyrene	53–120	≤20	50–125	≤30	6	6
	Benzo (b) fluoranthene	45–124	≤20	45–125	≤30	6	6
	Benzo (g,h,i) perylene	38–123	≤20	38–126	≤30	6	6
	Benzo (k) fluoranthene	45-124	≤20	45-125	≤30	6	6
	Benzyl alcohol	30–120	≤20	25–125	≤30	1	3
	Bis (2-chloroethoxy) methane	46–120	≤20	43–125	≤30	2	5
	Bis (2-chloroethyl) ether	37–120	≤20	38–125	≤30	1	3
	Bis (2-chloroisopropyl) ether	26–131	≤20	25–125	≤30	1	3
	Bis (2-ethylhexyl) phthalate	42–126	≤20	47–127	≤30	5	6
	Butyl benzyl phthalate	46–120	≤20	49–125	≤30	5	6
	Chrysene	55–120	≤20	53–125	≤30	5	6
	Di-n-butyl phthalate	54–120	≤20	56–125	≤30	4	1
	Di-n-octyl phthalate	37–137	≤20	41–132	≤30	5	6
	Dibenz (a,h) anthracene	42–127	≤20	41–125	≤30	6	6
	Dibenzofuran	54–120	≤20	51–125	≤30	3	4
	Diethyl phthalate	41–120	≤20	50–125	≤30	3	4
	Dimethyl phthalate	25–127	≤20	49–125	≤30	3	4
	Fluoranthene	54–120	≤20	54–125	≤30	4	1
	Fluorene	50–120	≤20	49–125	≤30	3	2
SW8270C (Continued)	Hexachlorobenzene	52–120	≤20	47–125	≤30	4	1
	Hexachlorobutadiene	27–120	≤20	40–125	≤30	2	5
	Hexachloroethane	28–120	≤20	34–125	≤30	1	3
	Indeno (1,2,3-c,d) pyrene	43–125	≤20	38–125	≤30	5	6
	Isophorone	50–120	≤20	43–125	≤30	2	5
	n-Nitrosodi-n-propylamine	34–128	≤20	40–125	≤30	1	3
	n-Nitrosodiphenylamine	48–120	≤20	49–125	≤30	4	1
	Naphthalene	39–120	≤20	40–125	≤30	2	5
	Nitrobenzene	44–120	≤20	41–125	≤30	2	4
	Phenanthrene	51–120	≤20	50–125	≤30	4	1
	Pyrene	49–128	≤20	46–125	≤30	5	6
	2,4,5-Trichlorophenol	49–120	≤20	49–125	≤30	3	1
	2,4,6-Trichlorophenol	49–126	≤20	43–125	≤30	3	1
	2,4-Dichlorophenol	48–120	≤20	45–125	≤30	2	5
	2,4-Dimethylphenol	28–120	≤20	32–125	≤30	2	5
	2,4-Dinitrophenol	25–130	≤20	25–132	≤30	3	4
	2-Chlorophenol	37–120	≤20	44–125	≤30	1	3
	2-Methylphenol	38–120	≤20	40–125	≤30	1	3
	2-Nitrophenol	39–123	≤20	42–125	≤30	2	4
	4,6-Dinitro-2-Methyl Phenol	40–130	≤20	29–137	≤30	4	1
	4-Chloro-3-Methyl Phenol	47–120	≤20	46–125	≤30	2	5
	4-Methylphenol	32–120	≤20	41–125	≤30	1	3
	4-Nitrophenol	20–120	≤20	25–138	≤30	3	2
	Benzoic Acid	20-120	≤20	25-125	≤30	2	5
	Pentachlorophenol	38–120	≤20	25–125	≤30	4	1
	Phenol	20–120	≤20	39–125	≤30	1	5

Method	Analyte	Accuracy Water (% R)	Precision Water (% RPD)	Accuracy Soil (% R)	Precision Soil (% RPD)	Number
SW8270C (Concluded)	*Surrogates:*					
	2,4,6-Tribromophenol	42–124		36–126		1
	2-Fluorobiphenyl	48–120		43–125		2
	2-Fluorophenol	20–120		37–125		3
	Nitrobenzene-D5	41–120		37–125		4
	Phenol-D5	20–120		40–125		5
	Terphenyl-D14	51–135		32–125		6
	Internal Standards:					
	1,4-Dichlorobenzene-D4					1
	Naphthalene-D8					2
	Acenaphthene-D10					3
	Phenanthrene-D10					4
	Chrysene-D12					5
	Perylene-D12					6

SOP No.	Revision No.	Effective Date	Page
AMB-8270C-66	6	August 30, 2005	35 of 46

TITLE: ANALYTICAL METHODS FOR GC/MS SEMIVOLATILE SAMPLES BY SW846 3RD EDITION METHOD 8270C

SUPERCEDES: Revision 5

Summary of Calibration and QC Procedures for Method SW8270C

Method	Applicable Parameter	QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action[a]	Flagging Criteria[b]
SW8270C	Semi-Volatile Organics	Five-point initial calibration for all analytes	Initial calibration prior to sample analysis	SPCCs average RF ≥ 0.050 and %RSD for RFs for CCCs ≤ 30% and one option below	Correct problem then repeat initial calibration	Apply R to all results for all samples associated with the calibration
				option 1 linear-mean RSD for all analytes ≤15% with no individual analyte RSD >30%		Apply R to all results for specific analyte(s) for all samples associated with the calibration
				option 2 linear - linear least squares regression r ≥ 0.995 for each analyte		
				option 3 non-linear - COD ≥ 0.990 (6 points shall be used for second order, 7 points shall be used for third order)		
		Second-source calibration verification	Once per five-point initial calibration	All analytes within ±25% of expected value	Correct problem then repeat initial calibration	Apply R to all results for specific analyte(s) for all samples associated with the calibration
		Retention time window calculated for each analyte	Each sample	Relative retention time (RRT) of the analyte within ± 0.06 RRT units of the RRT	Correct problem then reanalyze all samples analyzed since the last retention time check	Apply R to all results for the specific analyte(s) in the sample
		Continuing Calibration verification	Daily, before sample analysis and every 12 hours of analysis time	SPCCs average RF ≥ 0.050; and CCCs ≤ 20% difference (when using RFs)or drift (when using least squares regression or non-linear calibration)	Correct problem then repeat initial calibration	Apply R to all results for all samples associated with the calibration verification
				All calibration analytes within ±20% of expected value		Apply R to all results for specific analyte(s) for all samples associated with the calibration verification

SOP No.	Revision No.	Effective Date	Page
AMB-8270C-66	6	August 30, 2005	36 of 46

TITLE: ANALYTICAL METHODS FOR GC/MS SEMIVOLATILE SAMPLES BY SW846 3RD EDITION METHOD 8270C

SUPERCEDES: Revision 5

Summary of Calibration and QC Procedures for Method SW8270C

Method	Applicable Parameter	QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action[a]	Flagging Criteria[b]
SW8270C	Semi-Volatile Organics	Five-point initial calibration for all analytes	Initial calibration prior to sample analysis	SPCCs average RF ≥ 0.050 and %RSD for RFs for CCCs ≤ 30% and one option below	Correct problem then repeat initial calibration	Apply R to all results for all samples associated with the calibration
				option 1 linear-mean RSD for all analytes ≤15% with no individual analyte RSD >30%		Apply R to all results for specific analyte(s) for all samples associated with the calibration
				option 2 linear - linear least squares regression r ≥ 0.995 for each analyte		
				option 3 non-linear - COD ≥ 0.990 (6 points shall be used for second order, 7 points shall be used for third order)		
		Second-source calibration verification	Once per five-point initial calibration	All analytes within ±25% of expected value	Correct problem then repeat initial calibration	Apply R to all results for specific analyte(s) for all samples associated with the calibration
		Retention time window calculated for each analyte	Each sample	Relative retention time (RRT) of the analyte within ± 0.06 RRT units of the RRT	Correct problem then reanalyze all samples analyzed since the last retention time check	Apply R to all results for the specific analyte(s) in the sample
		Continuing Calibration verification	Daily, before sample analysis and every 12 hours of analysis time	SPCCs average RF ≥ 0.050; and CCCs ≤ 20% difference (when using RFs)or drift (when using least squares regression or non-linear calibration)	Correct problem then repeat initial calibration	Apply R to all results for all samples associated with the calibration verification
				All calibration analytes within ±20% of expected value		Apply R to all results for specific analyte(s) for all samples associated with the calibration verification

SOP No.	Revision No.	Effective Date	Page
AMB-8270C-66	6	August 30, 2005	37 of 46

TITLE: ANALYTICAL METHODS FOR GC/MS SEMIVOLATILE SAMPLES BY SW846 3RD EDITION METHOD 8270C

SUPERCEDES: Revision 5

Summary of Calibration and QC Procedures for Method SW8270C

Method	Applicable Parameter	QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action[a]	Flagging Criteria[b]
SW8270C	Semi-Volatile Organics	Five-point initial calibration for all analytes	Initial calibration prior to sample analysis	SPCCs average RF ≥ 0.050 and %RSD for RFs for CCCs ≤ 30% and one option below	Correct problem then repeat initial calibration	Apply R to all results for all samples associated with the calibration
				option 1 linear-mean RSD for all analytes ≤15% with no individual analyte RSD >30%		Apply R to all results for specific analyte(s) for all samples associated with the calibration
				option 2 linear - linear least squares regression r ≥ 0.995 for each analyte		
				option 3 non-linear - COD ≥ 0.990 (6 points shall be used for second order, 7 points shall be used for third order)		
		Second-source calibration verification	Once per five-point initial calibration	All analytes within ±25% of expected value	Correct problem then repeat initial calibration	Apply R to all results for specific analyte(s) for all samples associated with the calibration
		Retention time window calculated for each analyte	Each sample	Relative retention time (RRT) of the analyte within ± 0.06 RRT units of the RRT	Correct problem then reanalyze all samples analyzed since the last retention time check	Apply R to all results for the specific analyte(s) in the sample
		Continuing Calibration verification	Daily, before sample analysis and every 12 hours of analysis time	SPCCs average RF ≥ 0.050; and CCCs ≤ 20% difference (when using RFs)or drift (when using least squares regression or non-linear calibration)	Correct problem then repeat initial calibration	Apply R to all results for all samples associated with the calibration verification
				All calibration analytes within ±20% of expected value		Apply R to all results for specific analyte(s) for all samples associated with the calibration verification

SOP No.	Revision No.	Effective Date	Page
AMB-8270C-66	6	August 30, 2005	38 of 46

TITLE: ANALYTICAL METHODS FOR GC/MS SEMIVOLATILE SAMPLES BY SW846 3RD EDITION METHOD 8270C

SUPERCEDES: Revision 5

Attachment C

Summary of Measurement quality objectives for Method 8270 Semivolatiles

QC Element	Target Analyte/Surrogate	Poor Performers/Sporadic Marginal Failures[1]
Initial Calibration (I.9.2.2.7)	Instrument Evaluation: SPCCs: minimum RF values per method requirements CCCs: verify %RSD • 30%	No allowance
	Primary Evaluation (all target analytes) : r • 0.995, %RSD • 15%, r^2 • 0.990	No allowance
	Alternative Evaluation: Mean %RSD for all target analytes • 15%, with maximum allowable restriction noted at right for individual analytes.	Alternative Evaluation: Maximum allowable %RSD for each individual target analyte • 40%
ICV (I.9.3)	%Rec = 70% - 130%	No allowance
CCV (I.9.5 / I.9.5.2 / I.9.5.2.4)	Instrument Evaluation: SPCCs: minimum RF values per method requirements	No allowance
	Primary Evaluation (CCCs): %Drift • 20%, %D • 20%	No allowance
MB (I.10.2.1 / I.11.4.1)	Target Analytes: Analytes < one-half MRL	Common Lab Contaminants: Analytes < MRL
LCS (I.10.2.2 / I.11.4.2)	Water: %Rec = 60% - 120% (~15 analytes) = 45% - 135% (~30 analytes) = 20% - 150% (~15 analytes) Solids: %Rec = 60% - 120% (~20 analytes) = 45% - 135% (~25 analytes) = 30% - 150% (~15 analytes)	Sporadic Marginal Failures[1]: Water: %Rec = 15% - 150% Solids: %Rec = 25% - 150%
MS (I.10.2.3 / I.11.4.3 / I.11.4.3.2)	Water: %Rec = 45% - 135% Solids: %Rec = 45% - 135%	Sporadic Marginal Failures[1]: Water: %Rec = 15% - 150% Solids: %Rec = 20% - 150%
MSD/MD (I.10.2.4 / I.11.4.4)	Water: RPD • 50% Solids: RPD • 60%	Sporadic Marginal Failures[1]: Water: RPD • 60% Solids: RPD • 60%
Surrogates (I.10.2.5 / I.11.4.5)	%Interference-Free Matrix[2]: Water: %Rec = 60% - 120% B/N cmpds %Rec = 45% - 135% A cmpds Solids: %Rec = 60% - 120% B/N cmpds %Rec = 45% - 135% A cmpds Project Sample Matrix[2] Water: %Rec = 45% - 135% B/N cmpds %Rec = 35% - 140% A cmpds Solids: %Rec = 45% - 135% B/N cmpds %Rec = 35% - 140% A cmpds	Sporadic Marginal Failures[1]: Water: %Rec = 15% - 150% Solids: %Rec = 20% - 150%

[1] The number of sporadic marginal failure (SMF) allowances depends upon the number of target analytes reported from the analysis. For instance, if the full list of target compounds as presented in Table 13 are reported, then 5 SMFs are allowed to the expanded criteria presented for the LCS. If the MS includes only a subset of compounds and for Surrogates, allow up to 1 SMF.
[2] B = base, N = neutral, and A = acid compounds (cmpds).

Number of Allowable QC Failures

N	X
5 - 15	1
16 - 30	2
31 - 45	3
46 - 60	4
61 - 75	5
76 - 90	6
91 - 105	7

Note: N = number of reported method target analytes
X = sporadic marginal failures allowed

SOP No.	Revision No.	Effective Date	Page
AMB-8270C-66	6	August 30, 2005	39 of 46

TITLE: ANALYTICAL METHODS FOR GC/MS SEMIVOLATILE SAMPLES BY SW846 3RD EDITION METHOD 8270C

SUPERCEDES: Revision 5

TABLE 1

Semivolatiles Target Compound List and Contract
Estimated Quantitation Limits

			Estimated Quantitation Limits	
	Semivolatiles	CAS Number	Water µg/L	Low Soil µg/Kg
34.	Phenol	108-95-2	10	330
35.	bis-(2-Chloroethyl)ether	111-44-4	10	330
36.	2-Chlorophenol	95-57-8	10	330
37.	1,3-Dichlorobenzene	541-73-1	10	330
38.	1,4-Dichlorobenzene	106-46-7	10	330
39.	1,2-Dichlorobenzene	95-50-1	10	330
40.	2-Methylphenol	95-48-7	10	330
41.	Bis(2-chloroisopropl)ether	108-60-1	10	330
42.	4-Methylphenol	106-44-5	10	330
43.	N-Nitroso-di-n-propylamine	621-64-7	10	330
44.	Hexachloroethane	67-72-1	10	330
45.	Nitrobenzene	98-95-3	10	330
46.	Isophorone	78-59-1	10	330
47.	2-Nitrophenol	88-75-5	10	330
48.	2,4-Dimethylphenol	105-67-9	10	330
49.	bis(2-Chloroethoxy) methane	111-91-1	10	330
50.	2,4-Dichlorophenol	120-83-2	10	330
51.	1,2,4-Trichlorobenzene	120-82-1	10	330
52.	Naphthalene	91-20-3	10	330
53.	4-Chloroaniline	106-47-8	20	1300
54.	Hexachlorobutadiene	87-68-3	10	330
55.	4-Chloro-3-methylphenol	59-50-7	20	1300
56.	2-Methylnaphthalene	91-57-6	10	330
57.	Hexachlorocyclopenta-diene	77-47-4	10	330
58.	2,4,6-Trichlorophenol	88-06-2	10	330
59.	2,4,5-Trichlorophenol	95-95-4	10	330
60.	2-Chloronaphthalene	91-58-7	10	330
61.	2-Nitroaniline	88-74-4	50	3300
62.	dimethylphthalate	131-11-3	10	330
63.	Acenaphthylene	208-96-8	10	330
64.	2,6-Dinitrotoluene	606-20-2	10	330
65.	3-Nitroanline	99-09-2	50	3300
66.	Acenaphthene	83-32-9	10	330
67.	2,4-Dinitrophenol	51-28-5	50	330
68.	4-Nitrophenol	100-02-7	50	3300
69.	Dibenzofuran	132-64-9	10	330
70.	2,4-Dinitrotoluene	121-14-2	10	330
71.	Diethlphthalate	84-66-22	10	330
72.	4-Chlorophenyl-phenyl ether	7005-72-3	10	330
73.	Fluorene	86-73-7	10	330

TITLE: ANALYTICAL METHODS FOR GC/MS SEMIVOLATILE SAMPLES BY SW846 3RD EDITION METHOD 8270C

SUPERCEDES: Revision 5

			Estimated Quantitation Limits	
	Semivolatiles	CAS Number	Water µg/L	Low Soil µg/Kg
74.	4-Nitroaniline	100-01-6	50	3300
75.	4,6-Dinitro-2-methylphenol	534-52-1	50	3300
76.	N-Nitroso-diphenylamine	86-30-6	10	330
77.	4-Bromophenyl-phenylether	101-55-3	10	330
78.	Hexachlorobenzene	118-74-1	10	330
79.	Pentachlorophenol	87-86-5	50	3300
80.	Phenanthrene	85-01-8	10	330
81.	Anthracene	120-12-7	10	330
82.	Benzyl Alcohol	100-51-6	20	1300
83.	Di-n-butylphthalate	84-74-2	10	330
84.	Fluoranthene	206-44-0	10	330
85.	Pyrene	129-00-0	10	330
86.	Butylbenzylphthalate	85-68-7	10	330
87.	3,3-Dichlorobenzidine	91-94-1	20	1300
88.	Benzo(a)anthracene	56-55-3	10	330
89.	Chrysene	218-01-9	10	330
90.	bis(2-Ethylhexyl)phthalate	117-81-7	10	330
91.	Di-n-octylphthalate	117-84-0	10	330
92.	Benzo(b)fluoranthene	205-99-2	10	330
93.	Benzo(k)fluoranthene	207-08-9	10	330
94.	Benzo(a)pyrene	50-32-8	10	330
95.	Indeno(1,2,3-cd)-pyrene	193-39-5	10	330
96.	Dibenzo(a,h)-anthracene	53-70-3	10	330
97.	Benzo(g,h,i)perylene	191-24-2	10	330
98.	Benzoic Acid	65-85-0	50	3300

TABLE 2

DFTPP Key Ions and Ion Abundance Criteria

Mass	Ion Abundance Criteria
51	30.0 - 60.0 percent of mass 198
68	Less than 2.0 percent of mass 69
70	Less than 2.0 percent of mass 69
127	40.0 - 60.0 percent of mass 198
197	Less than 1.0 percent of mass 198
198	Base peak, 100 percent relative abundance (see Note)
199	5.0-9.0 percent of mass 198
275	10.0-30.0 percent of mass 198
365	Greater than 1.0% of than mass 198
441	Present but less than mass 443
442	40.0 – 110.0 percent of mass 198
443	17.0 - 23.0 percent of mass 442

Note: All ion abundances MUST be normalized to m/z 198, the nominal base peak, even though the ion abundance of m/z 442 may be greater to 110 percent that of m/z 198.

TABLE 3

Characteristic Ions for Internal Standards
for Semivolatile Compounds

Internal Standards	Primary Quantitation Ion	Secondary Ions
1,4-Dichlorobenzene-d4	152	115, 150
Naphthalene-d8	136	68
Acenaphthene-d10	164	162,160
Phenanthrene-d10	188	94, 80
Chrysene-d12	240	120, 236
Perylene-d12	264	260, 265

SOP No.	Revision No.	Effective Date	Page
AMB-8270C-66	6	August 30, 2005	42 of 46

TITLE: ANALYTICAL METHODS FOR GC/MS SEMIVOLATILE SAMPLES BY SW846 3RD EDITION METHOD 8270C

SUPERCEDES: Revision 5

TABLE 4
Semivolatile Internal Standards with Corresponding Target Compounds and Surrogates Assigned for Quantitation

1,4-Dichlorobenzene-d_4	Naphthalene-d_8	Acenaphthene-d_{-10}	Phenanthrene-d	Chrysene-d_{12}	Perylene-d_{12}
Phenol	Nitrobenzene	Hexachlorocyclopentadiene	4,6-Dinitro-2-methylphenol	Pyrene	Benzo(b)fluoranthene
bis(2-Chloroethyl)ether	Isophorone	2,4,6-Trichlorophenol	N-nitroso-di-phenylamine	Butylbenzylphthalate	Benzo(k)fluoranthene
2-Chlorophenol	2-Nitrophenol	2,4,5-Trichlorophenol	4-Bromophenylphenolether	3,3'-Dichlorobenzidine	Benzo(a)phyrne
1,3-Dichlorobenzene	2,4-Dimethylphenol	2-Chloroaphthalene	Hexachlorobenzene	Benzo(a)-anthracene	Indeno(1,2,3-cd)-pyrene
1,4-Dichlorobenzene	bis(2-Chloroethoxy) methane	2-Nitroaniline	Pentachlorophenol	bis(2-ethyl-hexyl)phthalate	Benzo(g,h,i)-perylene
1,2-Dichlorobenzene	2,4-Dichlorophenol	Dimethylphthalate	Carbzole	Chrysene	Dibenzo(a,h)-anthracene
2-Methylphenol	1,2,4-Trichlorobenzene	Acenaphthylene	Phenanthrene	Terphenyl-d_{14} (surr)	
2,2'-oxybis-(1-Chloropropane)	Naphthalene	3-Nitroaniline	Anthracene	Di-n-octyl-phthalata	
4-Methylphenol	4-Chloroanaline	Acenaphthene	Di-n-butylphthalate		
N-Nitroso-Di-n-propylamine	Hexachlorobutadiene	2,4-Dinitorphenol	Fluoranthene		
Hexachloroethane	4-Chloro-3-methylphenol	4-Nitrophenol			
2-Fluorophenol(surr)	2-Methylnaphthalene	Dibenzofuran			
Phenol-d_5 (surr)	Nitrobenzene-d_5 (surr)	2,4-Dinitrotoluene			
4-methylphenol	Benzoic acid	2,6-Dinitrotoluene			
Aniline	4-chloroaniline	Diethylphthalate			
Benzyl Alcohol	N-Nitrosobutyl-amine	4-Chlorophenyl-phenylether			
		Fluorene			
		4-Nitroaniline			

1,4-Dichlorobenzene-d_4	Naphthalene-d_8	Acenaphthene-d_{10}	Phenanthrene-d	Chrysene-d_{12}	Perylene-d_{12}
		2-Fluorobiphenyl (surr)			
		2,4,6-Tribromophenol (surr)			

TABLE 5

Relative Response Factor Criteria for Initial and Continuing
Calibration of Semivolatile Target Compounds and Surrogates

Semivolatile Compounds	Minimum RRF	Maximum % RSD	Maximum % Diff
Acenaphthene (CCC)	none	30	±20
1,4-Dichlorobenzene (CCC)	none	30	±20
Hexachlorobutadiene (CCC)	none	30	±20
N-Nitrosodiphenylamine (CCC)	none	30	±20
Di-n-octylphthalate (CCC)	none	30	±20
Flouranthene (CCC)	none	30	±20
Benzo(a)pyrene (CCC)	none	30	±20
4-Chloro-3-methylphenol (CCC)	none	30	+20
2,4-Dichlorophenol (CCC)	none	30	±20
2-Nitrophenol (CCC)	none	30	±20
Phenol (CCC)	none	30	±20
Pentachlorophenol(CCC)	none	30	±20
2,4,6-Trichlorophenol (CCC)	none	30	±20
N-Nitroso-di-n-propylamine (SPCC)	0.050	None	none
Hexachlorocyclopentadiene (SPCC)	0.050	None	none
2,4-Dinitrophenol (SPCC)	0.050	None	none
4-Nitrophenol (SPCC)	0.050	None	none

TABLE 6

Characteristic Ions for Semivolatile
Target Compounds and Surrogates

Parameters	Primary Quantitation Ion	Secondary Ion(s)
Phenol	94	65, 66
bis(2-Chloroethyl)ether	93	63, 95
2-Chlorophenol	128	64, 130
1,3-Dichlorobenzene	146	148, 113
1,4-Dichlorobenzene	146	148, 113
1,2-Dichlorobenzene	146	148, 113
2-Methylphenol	108	107
Bis(2-chloroisopropyl)ether	45	77, 79
4-Methylphenol	108	107
N-Nitroso-di-n-propylamine	70	42, 101, 130
Hexachloroethane	117	201, 199
Nitrobenzene	77	123, 65
Isophorone	82	95, 138
2-Nitrophenol	139	65, 109
2,4-Dimethylphenol	107	121, 122
bis(2-Chloroethoxy)methane	93	95, 123
2,4-Dichlorophenol	162	164, 98
1,2,4-Trichlorobenzene	180	182, 145
Naphthalene	128	129, 127
4-Chloroaniline	127	129
Hexachlorobutadiene	225	223, 227
4-Chloro-3-methylphenol	107	144, 142
2-Methylnaphthalene	142	141
Hexachlorocyclopentadiene	237	235, 272

Parameters	Primary Quantitation Ion	Secondary Ion(s)
2,4,6-Trichlorophenol	196	198, 200
2,4,5-Trichlorophenol	196	198, 200
2-Chloronaphthalene	162	164, 127
2-Nitroaniline	65	92, 138
Dimethylphthalate	163	194, 164
Acenaphthylene	152	151, 153
3-Nitroaniline	138	108, 92
Acenaphthene	153	152, 154
2,4-Dinitrophenol	184	63, 154
4-Nitrophenol	109	139, 65
Dibenzofuran	168	139
2,4-Dinitrotoluene	165	63, 182
2,6-Dinitrotoluene	165	89, 121
Diethylphthalate	149	177, 150
4-Chlorophenyl-phenylether	204	206, 141
Fluorene	166	165, 167
4-Nitroaniline	138	92, 108
4,6-Dinitro-2-methylphenol	198	182, 77
N-Nitrosodiphenylamine	169	168, 167
4-Bromophenyl-phenylether	248	250, 141
Hexachlorobenzene	284	142, 249
Pentachlorophenol	266	264, 268
Phenanthrene	178	179, 176
Anthracene	178	179, 176
Benzyl Alcohol	108	79, 77
Di-n-butylphthalate	149	150, 104
Fluoranthene	202	101, 100

Parameters	Primary Quantitation Ion	Secondary Ion(s)
Pyrene	202	101, 100
Butylbenzylphthalate	149	91, 206
3,3'-Dichlorobenzidine	252	254, 126
Benzo(a)anthracene	228	229, 226
bis(2-Ethylhexyl)phthalate	149	167, 279
Chrysene	228	226, 229
Di-n-octylphthalate	149	---
Benzo(b)fluoranthene	252	253, 125
Benzo(k)fluoranthene	252	253, 125
Benzo(a)pyrene	252	253, 125
Indeno(1,2,3-cd)pyrene	276	138, 227
Dibenzo(a,h)anthracene	278	139, 279
Benzo(g,h,i)perylene	276	138,277
Benzoic Acid	122	105, 77
SURROGATES		
Phenol-d5	99	42, 71
2-Fluorophenol	112	64
2,4,6-Tribormophenol	330	332, 141
Nitrobenzene-d5	82	128, 54
2-Fluorobiphenyl	172	171
Terphenyl-d14	244	122, 212

SOP No.	Revision No.	Effective Date	Page
AMB-8270C-66	6	August 30, 2005	47 of 46

TITLE: ANALYTICAL METHODS FOR GC/MS SEMIVOLATILE SAMPLES BY SW846 3RD EDITION METHOD 8270C

SUPERCEDES: Revision 5

Attachment D: Job Summary Checklist

Job Number: _____ Instrument # _____
Method: _____

Yes/No	Primary Review
	Tunes passed?
	ICC's passed?
	CCV's passed?
	Quantitations have been performed correctly?
	Qualitative identifications are accurate?
	Client specific requirements have been followed?
	Method and process SOP's have been followed?
	Method and/or QUAPP specific QC criteria have been met?
	QC samples are within established limits?
	Dilution factors are correctly recorded and applied?
	Non-conformances and/or anomalous data have been properly documented and communicated?
	Job folder complete?

Check	Secondary Review
	Qualitative identification.
	Quantitative accuracy.
	Calibration.
	QC samples.
	Method and/or QUAPP specific QC criteria.
	Adherence to method and process SOP's.

Comments: _____
_____

Analyst: _____ Date: _____
AIMS Entry: _____ Date: _____
Review: _____ Date: _____

CHEVRON PRODUCTS COMPANY
CHEVRON - HASTINGS ON HUDSON
METHOD 8270 - HASTINGS SEMIVOLATILES
ANALYSIS DATA SHEET

Client No.

SAMPLE A

Lab Name: TestAmerica Laborato Contract: TBD

Lab Code: RECNY Case No.: _____ SAS No.: _____ SDG No.: _____

Matrix: (soil/water) WATER Lab Sample ID: A7C59001

Sample wt/vol: 1000.0 (g/mL) ML Lab File ID: _____

Level: (low/med) LOW Date Samp/Recv: 10/31/2007 10/31/2007

% Moisture: _____ decanted: (Y/N) N Date Extracted: 10/31/2007

Concentrated Extract Volume: 1000 (uL) Date Analyzed: 10/31/2007

Injection Volume: 1.00 (uL) Dilution Factor: 1.00

GPC Cleanup: (Y/N) N pH: ____

CAS NO.	COMPOUND	CONCENTRATION UNITS: (ug/L or ug/Kg) UG/L	Q
100-52-7	Benzaldehyde	5	U
108-95-2	Phenol	5	U
95-57-8	2-Chlorophenol	5	U
95-48-7	2-Methylphenol	5	U
108-60-1	2,2'-Oxybis(1-Chloropropane)	5	U
98-86-2	Acetophenone	5	U
106-44-5	4-Methylphenol	5	U
621-64-7	N-Nitroso-Di-n-propylamine	5	U
67-72-1	Hexachloroethane	5	U
98-95-3	Nitrobenzene	5	U
78-59-1	Isophorone	5	U
88-75-5	2-Nitrophenol	5	U
105-67-9	2,4-Dimethylphenol	5	U
111-91-1	Bis(2-chloroethoxy) methane	5	U
120-83-2	2,4-Dichlorophenol	5	U
91-20-3	Naphthalene	5	U
106-47-8	4-Chloroaniline	5	U
87-68-3	Hexachlorobutadiene	5	U
105-60-2	Caprolactam	5	U
59-50-7	4-Chloro-3-methylphenol	5	U
91-57-6	2-Methylnaphthalene	5	U
77-47-4	Hexachlorocyclopentadiene	5	U
88-06-2	2,4,6-Trichlorophenol	5	U
95-95-4	2,4,5-Trichlorophenol	5	U
92-52-4	Biphenyl	5	U
91-58-7	2-Chloronaphthalene	5	U
88-74-4	2-Nitroaniline	10	U
131-11-3	Dimethyl phthalate	5	U
208-96-8	Acenaphthylene	5	U
606-20-2	2,6-Dinitrotoluene	5	U
99-09-2	3-Nitroaniline	10	U
83-32-9	Acenaphthene	5	U

CHEVRON PRODUCTS COMPANY
CHEVRON - HASTINGS ON HUDSON
METHOD 8270 - HASTINGS SEMIVOLATILES
ANALYSIS DATA SHEET

Client No.

SAMPLE A

Lab Name: TestAmerica Laborato Contract: TBD

Lab Code: RECNY Case No.: _____ SAS No.: _____ SDG No.: _____

Matrix: (soil/water) WATER Lab Sample ID: A7C59001

Sample wt/vol: 1000.0 (g/mL) ML Lab File ID: _____

Level: (low/med) LOW Date Samp/Recv: 10/31/2007 10/31/2007

% Moisture: _____ decanted: (Y/N) N Date Extracted: 10/31/2007

Concentrated Extract Volume: 1000 (uL) Date Analyzed: 10/31/2007

Injection Volume: 1.00 (uL) Dilution Factor: 1.00

GPC Cleanup: (Y/N) N pH: ____

CAS NO.	COMPOUND	CONCENTRATION UNITS: (ug/L or ug/Kg) UG/L	Q
51-28-5	2,4-Dinitrophenol	10	U
100-02-7	4-Nitrophenol	10	U
132-64-9	Dibenzofuran	5	U
121-14-2	2,4-Dinitrotoluene	5	U
84-66-2	Diethyl phthalate	5	U
7005-72-3	4-Chlorophenyl phenyl ether	5	U
86-73-7	Fluorene	5	U
100-01-6	4-Nitroaniline	10	U
534-52-1	4,6-Dinitro-2-methylphenol	10	U
86-30-6	N-nitrosodiphenylamine	5	U
101-55-3	4-Bromophenyl phenyl ether	5	U
118-74-1	Hexachlorobenzene	5	U
1912-24-9	Atrazine	5	U
87-86-5	Pentachlorophenol	10	U
85-01-8	Phenanthrene	5	U
120-12-7	Anthracene	5	U
86-74-8	Carbazole	5	U
84-74-2	Di-n-butyl phthalate	5	U
206-44-0	Fluoranthene	5	U
129-00-0	Pyrene	5	U
85-68-7	Butyl benzyl phthalate	5	U
91-94-1	3,3'-Dichlorobenzidine	5	U
56-55-3	Benzo(a)anthracene	5	U
218-01-9	Chrysene	5	U
117-81-7	Bis(2-ethylhexyl) phthalate	5	U
117-84-0	Di-n-octyl phthalate	5	U
205-99-2	Benzo(b)fluoranthene	5	U
207-08-9	Benzo(k)fluoranthene	5	U
50-32-8	Benzo(a)pyrene	5	U
193-39-5	Indeno(1,2,3-cd)pyrene	5	U
53-70-3	Dibenzo(a,h)anthracene	5	U
191-24-2	Benzo(ghi)perylene	5	U

CHEVRON PRODUCTS COMPANY
CHEVRON - HASTINGS ON HUDSON
METHOD 8270 - HASTINGS SEMIVOLATILES
ANALYSIS DATA SHEET

Client No.

SAMPLE A

Lab Name: TestAmerica Laborato Contract: TBD

Lab Code: RECNY Case No.: _____ SAS No.: _____ SDG No.: _____

Matrix: (soil/water) WATER Lab Sample ID: A7C59001

Sample wt/vol: 1000.0 (g/mL) ML Lab File ID: _____

Level: (low/med) LOW Date Samp/Recv: 10/31/2007 10/31/2007

% Moisture: _____ decanted: (Y/N) N Date Extracted: 10/31/2007

Concentrated Extract Volume: 1000 (uL) Date Analyzed: 10/31/2007

Injection Volume: 1.00 (uL) Dilution Factor: 1.00

GPC Cleanup: (Y/N) N pH: ____

CAS NO.	COMPOUND	CONCENTRATION UNITS: (ug/L or ug/Kg) UG/L	Q
84-65-1	9,10-Anthracenedione	10	U
81-64-1	1,4-Dihydroxy-9,10-anthracendione	40	U
129-43-1	1-Hydroxy-9,10-anthracenedione	20	U
95-51-2	o-Chloroaniline	10	U
301-02-0	(z)-9-octadecenamide	100	U
95-53-4	2-Methyl-Benzenamine	10	U
106-49-0	p-Aminotoluene	10	U

Response Factor Report HP5973X

Method Path : C:\MSDCHEM\1\METHODS\
Method File : ADD 3.M
Title : ADD#3
Last Update : Tue Sep 04 15:56:43 2007
Response Via : Initial Calibration

Calibration Files
5 =X19302.D 20 =X19303.D 50 =X19304.D
80 =X19305.D 120 =X19306.D 160 =X19307.D

		Compound	5	20	50	80	120	160	Avg	%RSD
1)	I	CI30 1,4-Dichlorobenz	ISTD							
2)		E150 O-Toluidine	2.007	2.149	2.224	2.360	2.303	2.357	2.233	6.17
3)		C829 p-Aminotoluene	2.579	2.553	2.706	2.660	2.458	2.357	2.552	5.04
4)		C951 O-Chloroanilin	1.721	1.847	1.834	1.870	1.793	1.864	1.822	3.09
5)	I	CI40 Naphthalene-d8	ISTD							
6)	I	CI50 Acenaphthene-d8	ISTD							
7)	I	CI60 Phenanthrene-d10	ISTD							
8)		C831 9,10-Anthracen	0.160	0.242	0.295	0.302	0.301	0.316	-----	
							L	M= 0.319	R=0.999	
								B= -0.032		
9)		C826 1-Hydroxy-9,10	0.136	0.214	0.288	0.319	0.325	0.349	-----	
							L	M= 0.356	R=0.997	
								B= -0.063		
10)		C827 1,4-Dihydro-9,	0.082	0.181	0.269	0.315	0.322	0.357	-----	
							L	M= 0.366	R=0.994	
								B= -0.086		
11)		C828 (Z)-9-Octadeca	0.038	0.046	0.085	0.106	0.125	0.152	-----	
							Q	A= 0.023	R=1.000	
								B= 0.059		
								C= -0.006		
12)	I	CI70 Chrysene-d12	ISTD							
13)	I	CI75 Perylene-d12	ISTD							
								Total Average %RSD		4.77

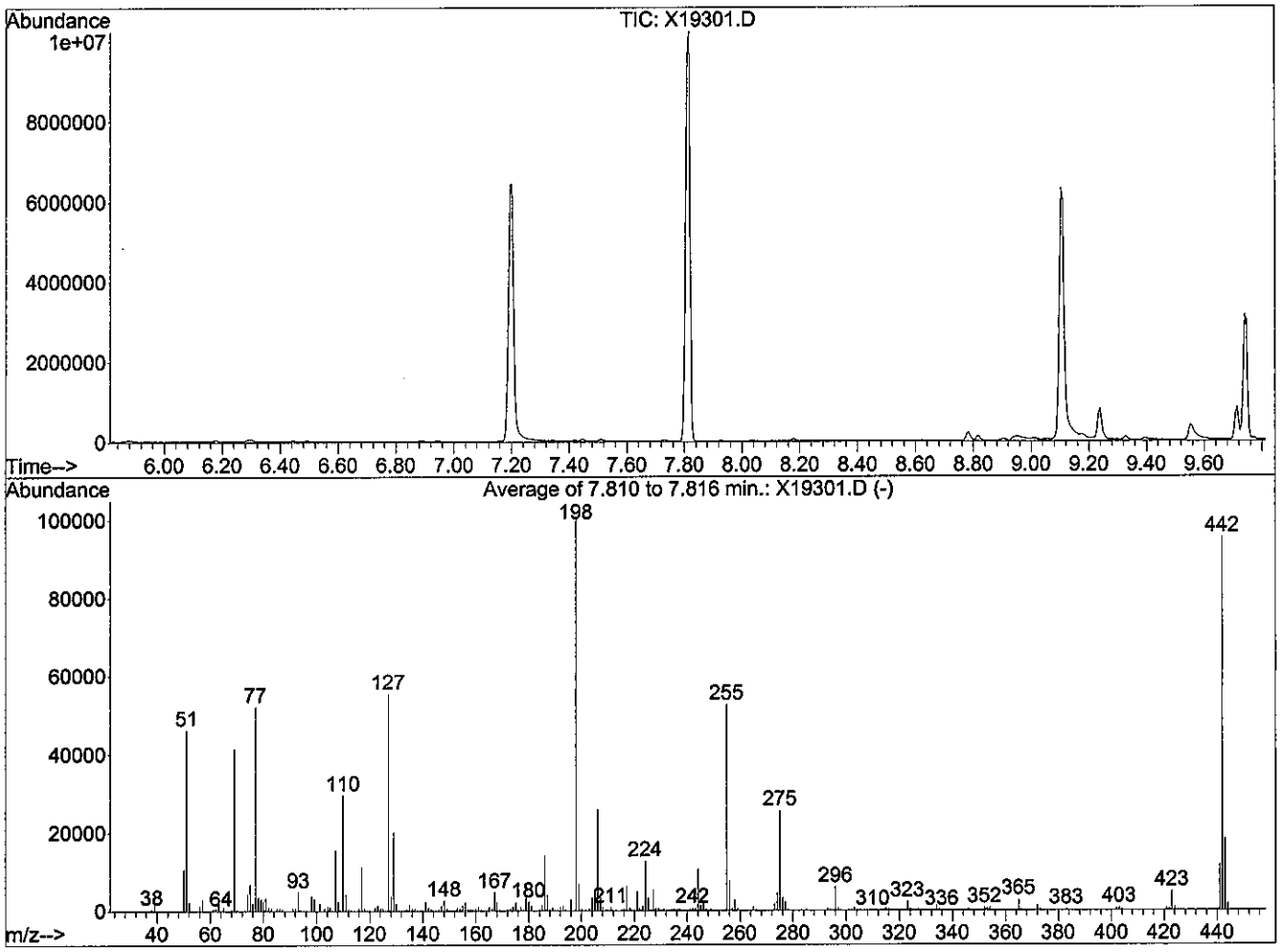
L = Linear LO = Linear+Origin Q = Quad QO = Quad+Origin R = Corr. Coef
(#) = Out of Range

ADD 3.M Tue Sep 04 15:57:28 2007

Data File : D:\DATA\082107\X19301.D
Acq On : 22 Aug 2007 1:26 am
Sample : DFTPP 50NG
Misc :
MS Integration Params: NA

Vial: 36
Operator: PM
Inst : HP5973X
Multiplr: 1.00

Method : C:\MSDCHEM\1\METHODS\ADD 3.M (RTE Integrator)
Title : ADD#3
Last Update : Wed Aug 22 10:08:03 2007
Response via : Initial Calibration



Spectrum Information: Average of 7.810 to 7.816 min.

Target Mass	Rel. to Mass	Lower Limit%	Upper Limit%	Rel. Abn%	Raw Abn	Result Pass/Fail
51	198	30	60	46.2	46180	PASS
68	69	0.00	2	0.9	383	PASS
69	198	0.00	100	41.5	41451	PASS
70	69	0.00	2	0.5	216	PASS
127	198	40	60	55.4	55435	PASS
197	198	0.00	1	0.1	105	PASS
198	198	100	100	100.0	100000	PASS
199	198	5	9	6.9	6941	PASS
275	198	10	30	25.6	25567	PASS
365	198	1	100	2.8	2774	PASS
441	198	0.01	100	11.7	11714	PASS
442	198	39	110	95.5	95470	PASS
443	442	17	23	19.3	18449	PASS

Average of 7.810 to 7.816 min.: X19301.D
DFTPP 50NG
Modified:subtracted

m/z	abund.	m/z	abund.	m/z	abund.	m/z	abund.
36.05	74	51.05	46180	63.10	1735	76.00	2060
37.10	182	52.10	2359	64.10	275	77.10	52171
38.05	486	53.00	46	65.10	975	78.10	3570
39.10	3278	55.05	259	66.20	50	79.10	3045
40.00	12	56.00	1349	67.95	383	80.00	2372
41.05	13	57.10	2873	69.00	41451	81.00	3443
43.10	11	58.05	177	70.05	216	82.05	906
45.00	61	59.05	36	70.80	26	83.10	894
47.00	6	60.10	30	73.05	309	85.10	774
48.10	26	61.10	577	74.10	4415	86.05	774
50.10	10634	62.10	676	75.10	6956	87.10	511

Average of 7.810 to 7.816 min.: X19301.D
DFTPP 50NG
Modified:subtracted

m/z	abund.	m/z	abund.	m/z	abund.	m/z	abund.
88.00	158	100.10	113	111.10	4226	121.10	34
89.05	98	101.10	1904	112.05	534	122.05	890
91.00	805	102.10	108	113.10	130	123.05	1476
92.05	774	102.95	612	114.15	64	124.05	703
93.10	4921	104.10	1256	116.00	653	125.05	693
94.00	390	105.00	1055	117.05	11323	127.10	55435
94.95	122	105.90	275	118.05	757	128.05	3694
96.10	238	107.10	15555	118.95	140	129.05	20203
97.00	67	108.10	2434	119.90	100	130.05	1867
98.05	3908	108.90	228	120.10	120	131.10	312
99.05	3171	110.00	29589	120.80	27	132.10	116

Average of 7.810 to 7.816 min.: X19301.D
DFTPP 50NG
Modified:subtracted

m/z	abund.	m/z	abund.	m/z	abund.	m/z	abund.
133.00	93	143.95	151	154.00	522	165.00	989
134.05	550	145.00	171	155.10	1381	166.05	566
135.05	1536	146.05	406	156.10	2232	167.10	4752
136.10	556	147.05	1218	157.10	439	168.05	2375
137.05	698	148.05	2655	158.05	442	169.10	501
138.05	257	149.10	642	159.05	334	169.95	135
139.00	110	150.05	206	160.00	730	171.00	186
140.00	76	151.00	134	161.05	1128	172.00	470
141.05	2447	151.20	187	162.05	325	173.00	522
142.05	812	152.15	122	163.00	96	174.10	1044
143.10	564	153.00	862	164.00	124	175.10	2099

Average of 7.810 to 7.816 min.: X19301.D
DFTPP 50NG
Modified:subtracted

m/z	abund.	m/z	abund.	m/z	abund.	m/z	abund.
176.00	307	187.10	4126	198.00	100000	209.05	207
177.10	989	188.05	452	199.00	6941	210.00	323
178.00	256	189.10	810	200.05	539	211.05	934
179.00	3385	190.05	153	201.60	208	211.70	245
180.05	2344	191.05	317	202.20	84	213.00	121
181.10	1225	192.10	1125	203.00	613	215.00	309
182.00	169	193.10	1321	204.10	3401	216.00	440
183.15	115	194.00	283	205.10	5588	217.00	6387
184.00	301	194.90	87	206.10	25870	218.10	744
185.10	1503	196.05	2945	207.10	3364	218.90	62
186.10	14239	196.70	105	208.00	830	219.85	89

Average of 7.810 to 7.816 min.: X19301.D
DFTPP 50NG
Modified:subtracted

m/z	abund.	m/z	abund.	m/z	abund.	m/z	abund.
221.10	5026	232.10	49	242.05	716	253.05	247
221.85	742	232.90	47	243.05	614	253.80	110
223.00	1199	233.10	27	244.10	10759	255.00	52650
224.05	12764	234.00	363	245.10	1347	256.05	7749
225.10	3349	235.00	385	246.10	1928	257.00	529
226.00	329	236.05	282	247.10	398	258.00	2833
227.05	5274	237.05	474	248.05	118	259.05	489
228.05	716	238.10	49	249.05	366	260.05	71

m/z	abund.	m/z	abund.	m/z	abund.	m/z	abund.
229.00	585	239.00	117	249.90	108	261.00	114
229.95	206	240.10	167	250.90	108	264.00	107
231.10	512	241.05	326	251.85	128	265.05	1112

Average of 7.810 to 7.816 min.: X19301.D
DFTPP 50NG
Modified:subtracted

m/z	abund.	m/z	abund.	m/z	abund.	m/z	abund.
266.00	262	278.95	79	291.90	49	304.00	226
267.90	34	281.95	71	293.00	491	305.00	24
269.90	50	282.90	110	294.00	155	308.00	121
271.05	128	283.10	158	294.90	76	309.05	93
272.00	189	284.10	170	296.00	5997	310.00	120
273.00	1616	285.00	382	297.05	782	312.90	33
274.00	4441	286.10	36	297.90	40	313.95	288
275.00	25567	288.00	48	298.20	24	315.00	690
276.05	3384	288.80	32	300.95	90	316.05	412
277.00	2174	289.00	55	301.95	122	316.95	96
278.05	361	289.95	83	303.05	763	321.10	219

Average of 7.810 to 7.816 min.: X19301.D
DFTPP 50NG
Modified:subtracted

m/z	abund.	m/z	abund.	m/z	abund.	m/z	abund.
322.00	124	334.00	1479	352.95	526	373.05	306
323.05	2270	335.05	405	354.05	679	373.90	25
324.05	446	336.00	68	355.05	153	376.90	28
324.90	24	339.00	25	359.05	71	383.00	346
325.90	32	340.00	35	362.90	24	383.95	108
327.00	410	340.90	121	364.95	2774	389.95	171
327.90	104	341.10	174	365.95	411	391.05	80
328.10	113	341.95	102	367.10	25	391.80	26
328.95	52	345.95	507	370.00	50	392.00	87
332.05	145	346.90	103	371.00	202	400.90	33
333.05	207	352.05	702	372.00	1345	401.95	577

Average of 7.810 to 7.816 min.: X19301.D
DFTPP 50NG
Modified:subtracted

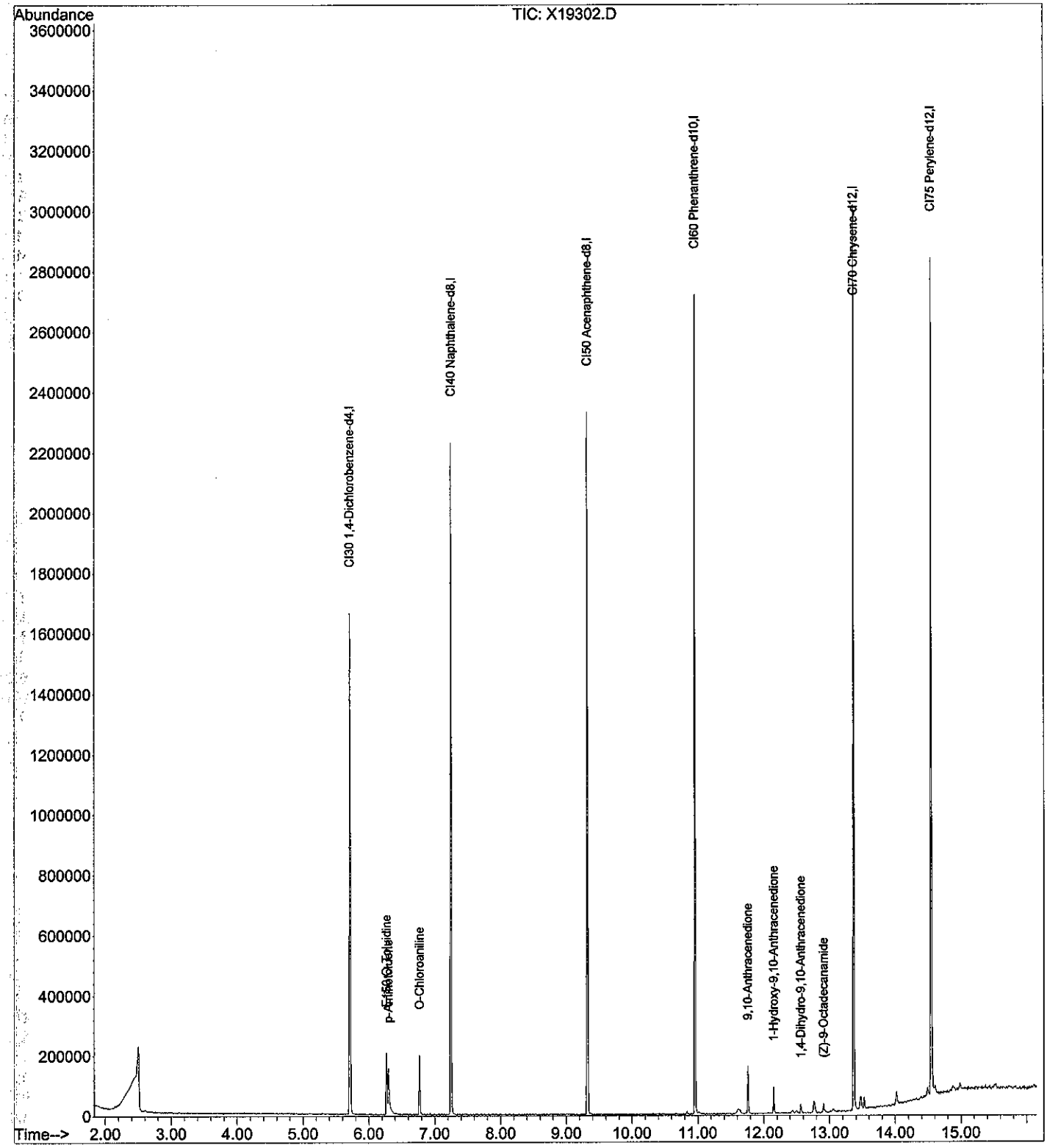
m/z	abund.	m/z	abund.	m/z	abund.	m/z	abund.
403.05	747	438.10	24				
404.00	298	438.90	36				
404.90	32	441.00	11714				
419.60	27	442.00	95470				
420.95	640	443.00	18449				
422.00	553	444.00	1714				
423.00	4869	444.95	107				
424.05	996						
425.00	111						
426.10	28						
437.30	31						

Quantitation Report (QT Reviewed)

Data File : D:\DATA\082107\X19302.D
Acq On : 22 Aug 2007 1:42 am
Sample : SSTD005
Misc : ADD#3
MS Integration Params: rteint.p

Vial: 37
Operator: PM
Inst : HP5973X
Multiplr: 1.00

Quant Time: Aug 22 10:13:00 2007 Results File: ADD 3.RES
Quant Method : C:\MSDCHEM\1\METHODS\ADD 3.M (RTE Integrator)
Title : ADD#3
Last Update : Wed Aug 22 10:08:03 2007
Response via : Initial Calibration
DataAcq Meth : 8270BP

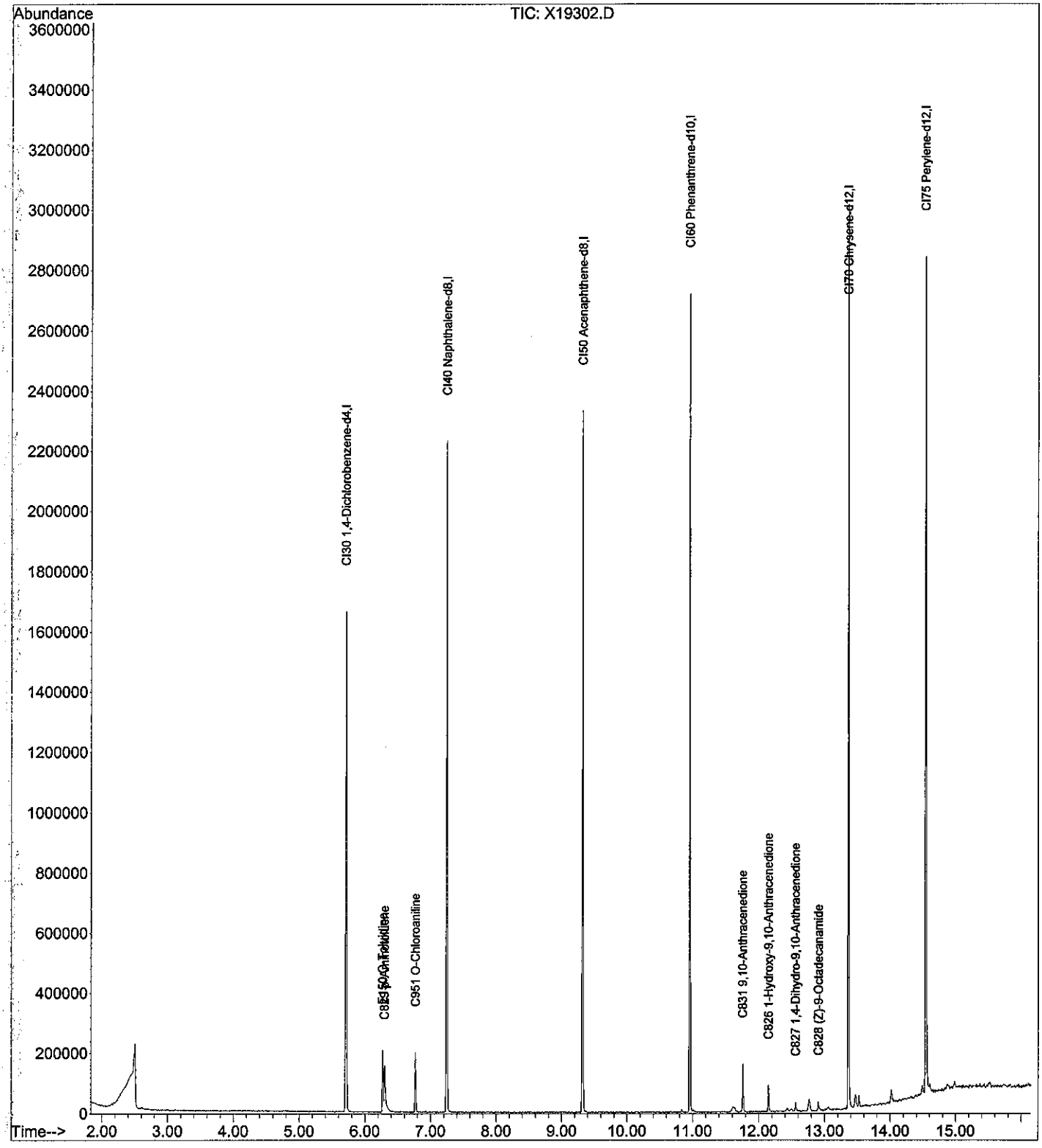


Quantitation Report (Not Reviewed)

Data File : D:\DATA\082107\X19302.D
Acq On : 22 Aug 2007 1:42 am
Sample : SSTD005
Misc : ADD#3
MS Integration Params: rteint.p

Vial: 37
Operator: PM
Inst : HP5973X
Multiplr: 1.00

Quant Time: Sep 04 15:54:57 2007 Results File: ADD 3.RES
Quant Method : C:\MSDCHEM\1\METHODS\ADD 3.M (RTE Integrator)
Title : ADD#3
Last Update : Tue Sep 04 15:54:53 2007
Response via : Initial Calibration
DataAcq Meth : 8270BP



Quantitation Report (Not Reviewed)

Data File : D:\DATA\082107\X19302.D Vial: 37
Acq On : 22 Aug 2007 1:42 am Operator: PM
Sample : SSTD005 Inst : HP5973X
Misc : ADD#3 Multiplr: 1.00
MS Integration Params: rteint.p
Quant Time: Sep 04 15:54:57 2007 Results File: ADD 3.RES

Quant Method : C:\MSDCHEM\1\METHODS\ADD 3.M (RTE Integrator)
Title : ADD#3
Last Update : Tue Sep 04 15:54:53 2007
Response via : Initial Calibration
DataAcq Meth : 8270BP
IS QA File : CC level for IS QA unknown. No recoveries calculated.

Internal Standards	R.T.	QIon	Response	Conc	Units		Dev(Min) Rcv(Ar)
1) CI30 1,4-Dichlorobenzene-d	5.71	152	267228	40.00	ng		0.00 NA%
5) CI40 Naphthalene-d8	7.25	136	992920	40.00	ng		0.00 NA%
6) CI50 Acenaphthene-d8	9.32	164	530976	40.00	ng		0.00 NA%
7) CI60 Phenanthrene-d10	10.96	188	919741	40.00	ng		0.00 NA%
12) CI70 Chrysene-d12	13.36	240	860138	40.00	ng		0.00 NA%
13) CI75 Perylene-d12	14.54	264	950588	40.00	ng		0.00 NA%
Target Compounds							Qvalue
2) E150 O-Toluidine	6.27	106	67033	4.49	ng		96
3) C829 p-Aminotoluene	6.30	106	86140	5.16	ng		94
4) C951 O-Chloroaniline	6.77	127	57496	4.72	ng		97
8) C831 9,10-Anthracenedione	11.76	180	18451	6.49	ng	#	88
9) C826 1-Hydroxy-9,10-Anthra	12.15	224	15588	9.00	ng		94
10) C827 1,4-Dihydro-9,10-Anth	12.56	240	9454	10.51	ng	#	91
11) C828 (Z)-9-Octadecanamide	12.91	72	4421	6.06	ng	#	17

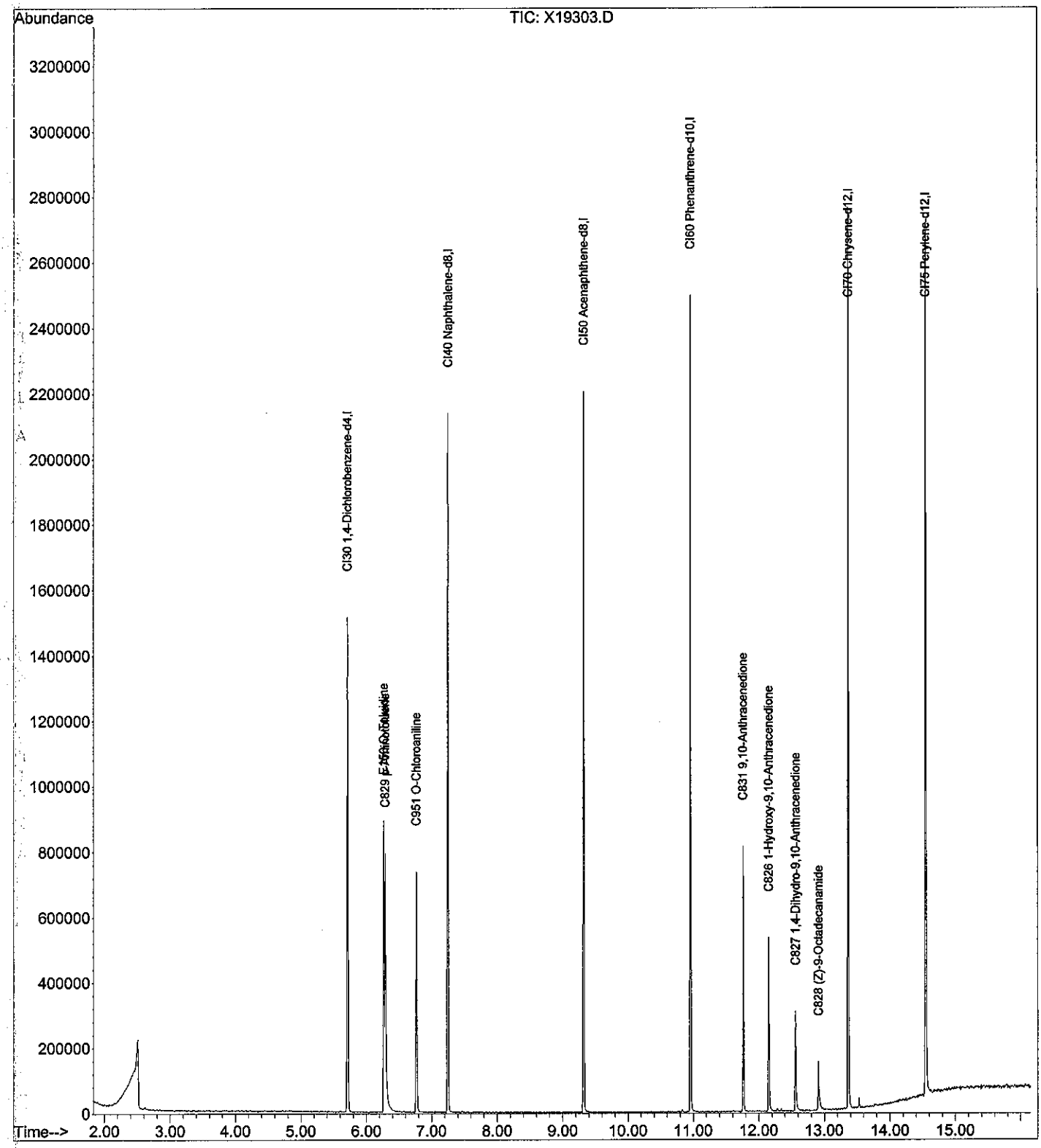
(#) = qualifier out of range (m) = manual integration (+) = signals summed

Quantitation Report (Not Reviewed)

Data File : D:\DATA\082107\X19303.D
Acq On : 22 Aug 2007 2:04 am
Sample : SSTD020
Misc : ADD#3
MS Integration Params: rteint.p

Vial: 38
Operator: PM
Inst : HP5973X
Multiplr: 1.00

Quant Time: Sep 04 15:55:18 2007 Results File: ADD 3.RES
Quant Method : C:\MSDCHEM\1\METHODS\ADD 3.M (RTE Integrator)
Title : ADD#3
Last Update : Tue Sep 04 15:55:12 2007
Response via : Initial Calibration
DataAcq Meth : 8270BP



Quantitation Report (Not Reviewed)

Data File : D:\DATA\082107\X19303.D Vial: 38
Acq On : 22 Aug 2007 2:04 am Operator: PM
Sample : SSTD020 Inst : HP5973X
Misc : ADD#3 Multiplr: 1.00
MS Integration Params: rteint.p
Quant Time: Sep 04 15:55:18 2007 Results File: ADD 3.RES

Quant Method : C:\MSDCHEM\1\METHODS\ADD 3.M (RTE Integrator)
Title : ADD#3
Last Update : Tue Sep 04 15:55:12 2007
Response via : Initial Calibration
DataAcq Meth : 8270BP
IS QA File : CC level for IS QA unknown. No recoveries calculated.

Internal Standards	R.T.	QIon	Response	Conc	Units	Dev(Min) Rcv(Ar)
1) CI30 1,4-Dichlorobenzene-d	5.71	152	248048	40.00	ng	0.00 NA%
5) CI40 Naphthalene-d8	7.25	136	938572	40.00	ng	0.00 NA%
6) CI50 Acenaphthene-d8	9.32	164	497458	40.00	ng	0.00 NA%
7) CI60 Phenanthrene-d10	10.96	188	836650	40.00	ng	0.00 NA%
12) CI70 Chrysene-d12	13.36	240	800509	40.00	ng	0.00 NA%
13) CI75 Perylene-d12	14.54	264	858881	40.00	ng	0.00 NA%
Target Compounds						Qvalue
2) E150 O-Toluidine	6.27	106	266502	19.24	ng	97
3) C829 p-Aminotoluene	6.29	106	316588	20.00	ng	95
4) C951 O-Chloroaniline	6.77	127	229099	20.28	ng	99
8) C831 9,10-Anthracenedione	11.76	180	101313	19.15	ng	91
9) C826 1-Hydroxy-9,10-Anthra	12.15	224	89501	19.11	ng	98
10) C827 1,4-Dihydro-9,10-Anth	12.56	240	75546	19.26	ng	98
11) C828 (Z)-9-Octadecanamide	12.91	72	19336	12.18	ng	# 51

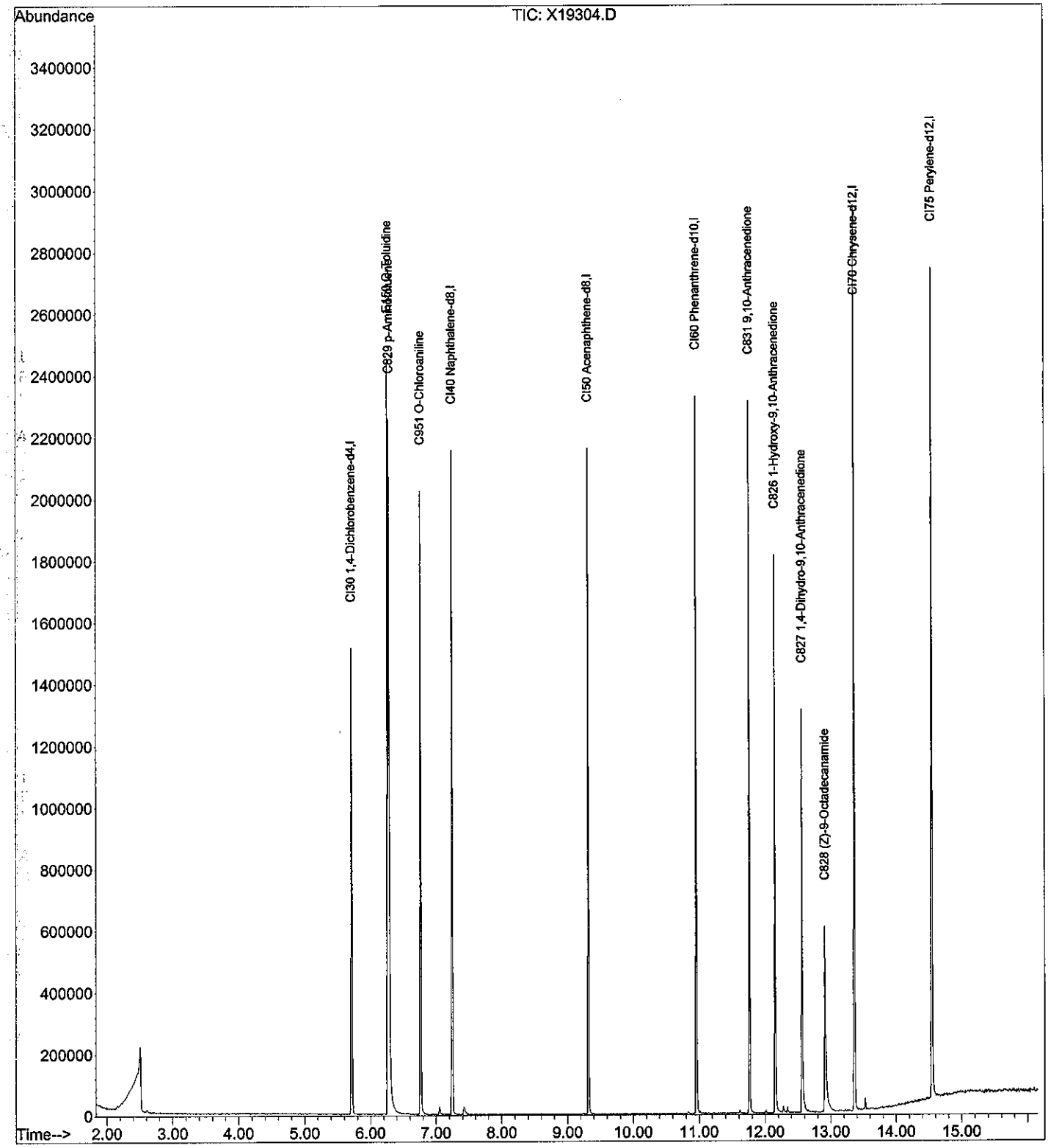
(#) = qualifier out of range (m) = manual integration (+) = signals summed

Quantitation Report (Not Reviewed)

Data File : D:\DATA\082107\X19304.D
Acq On : 22 Aug 2007 2:26 am
Sample : SSTD050
Misc : ADD#3
MS Integration Params: rteint.p

Vial: 39
Operator: PM
Inst : HP5973X
Multiplr: 1.00

Quant Time: Sep 04 15:55:38 2007 Results File: ADD 3.RES
Quant Method : C:\MSDCHEM\1\METHODS\ADD 3.M (RTE Integrator)
Title : ADD#3
Last Update : Tue Sep 04 15:55:25 2007
Response via : Initial Calibration
DataAcq Meth : 8270BP



Quantitation Report (Not Reviewed)

Data File : D:\DATA\082107\X19304.D Vial: 39
Acq On : 22 Aug 2007 2:26 am Operator: PM
Sample : SSTD050 Inst : HP5973X
Misc : ADD#3 Multiplr: 1.00
MS Integration Params: rteint.p
Quant Time: Sep 04 15:55:38 2007 Results File: ADD 3.RES

Quant Method : C:\MSDCHEM\1\METHODS\ADD 3.M (RTE Integrator)
Title : ADD#3
Last Update : Tue Sep 04 15:55:25 2007
Response via : Initial Calibration
DataAcq Meth : 8270BP
IS QA File : CC level for IS QA unknown. No recoveries calculated.

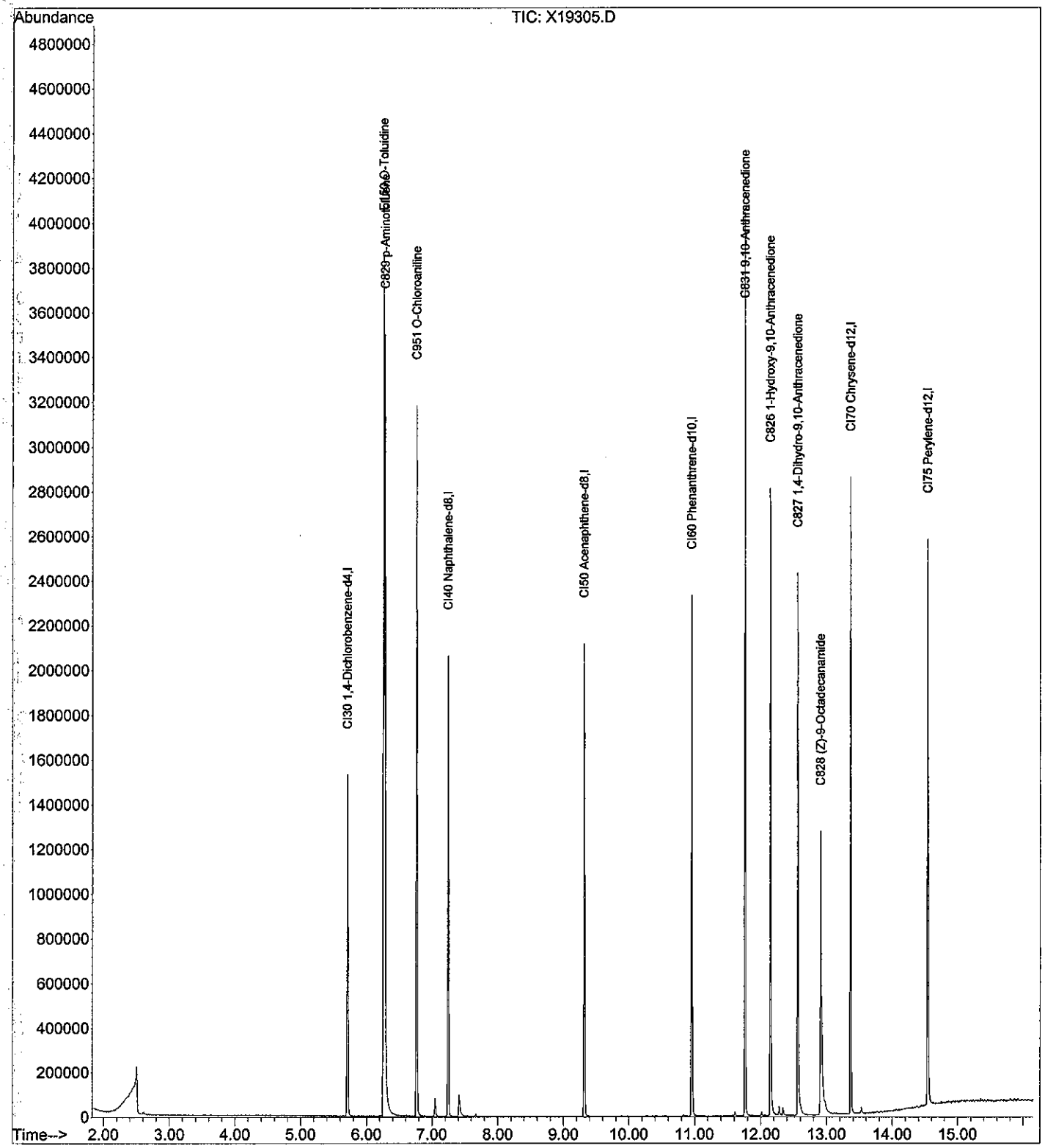
Internal Standards	R.T.	QIon	Response	Conc	Units	Dev(Min) Rcv(Ar)
1) CI30 1,4-Dichlorobenzene-d	5.71	152	253300	40.00	ng	0.00 NA%
5) CI40 Naphthalene-d8	7.24	136	939433	40.00	ng	0.00 NA%
6) CI50 Acenaphthene-d8	9.32	164	499501	40.00	ng	0.00 NA%
7) CI60 Phenanthrene-d10	10.96	188	854748	40.00	ng	0.00 NA%
12) CI70 Chrysene-d12	13.36	240	825082	40.00	ng	0.00 NA%
13) CI75 Perylene-d12	14.54	264	862071	40.00	ng	0.00 NA%
Target Compounds						Qvalue
2) E150 O-Toluidine	6.26	106	704122	49.79	ng	96
3) C829 p-Aminotoluene	6.28	106	856822	53.02	ng	97
4) C951 O-Chloroaniline	6.76	127	580731	50.34	ng	99
8) C831 9,10-Anthracenedione	11.76	180	315624	50.27	ng	95
9) C826 1-Hydroxy-9,10-Anthra	12.15	224	308109	47.60	ng	98
10) C827 1,4-Dihydro-9,10-Anth	12.56	240	287766	46.19	ng	96
11) C828 (Z)-9-Octadecanamide	12.91	72	90652	36.07	ng	# 68

(#) = qualifier out of range (m) = manual integration (+) = signals summed

Quantitation Report (Not Reviewed)

Data File : D:\DATA\082107\X19305.D Vial: 40
Acq On : 22 Aug 2007 2:48 am Operator: PM
Sample : SSTD080 Inst : HP5973X
Misc : ADD#3 Multiplr: 1.00
MS Integration Params: rteint.p

Quant Time: Sep 04 15:55:55 2007 Results File: ADD 3.RES
Quant Method : C:\MSDCHEM\1\METHODS\ADD 3.M (RTE Integrator)
Title : ADD#3
Last Update : Tue Sep 04 15:55:50 2007
Response via : Initial Calibration
DataAcq Meth : 8270BP



Quantitation Report (Not Reviewed)

Data File : D:\DATA\082107\X19305.D Vial: 40
Acq On : 22 Aug 2007 2:48 am Operator: PM
Sample : SSTD080 Inst : HP5973X
Misc : ADD#3 Multiplr: 1.00
MS Integration Params: rteint.p
Quant Time: Sep 04 15:55:55 2007 Results File: ADD 3.RES

Quant Method : C:\MSDCHEM\1\METHODS\ADD 3.M (RTE Integrator)
Title : ADD#3
Last Update : Tue Sep 04 15:55:50 2007
Response via : Initial Calibration
DataAcq Meth : 8270BP
IS QA File : CC level for IS QA unknown. No recoveries calculated.

Internal Standards	R.T.	QIon	Response	Conc	Units	Dev(Min) Rcv(Ar)
1) CI30 1,4-Dichlorobenzene-d	5.71	152	244533	40.00	ng	0.00 NA%
5) CI40 Naphthalene-d8	7.24	136	920485	40.00	ng	0.00 NA%
6) CI50 Acenaphthene-d8	9.32	164	483762	40.00	ng	0.00 NA%
7) CI60 Phenanthrene-d10	10.96	188	828837	40.00	ng	0.00 NA%
12) CI70 Chrysene-d12	13.36	240	805612	40.00	ng	0.00 NA%
13) CI75 Perylene-d12	14.54	264	852823	40.00	ng	0.00 NA%
Target Compounds						Qvalue
2) E150 O-Toluidine	6.26	106	1154428	84.55	ng	96
3) C829 p-Aminotoluene	6.28	106	1300679	83.37	ng	99
4) C951 O-Chloroaniline	6.76	127	914721	82.13	ng	100
8) C831 9,10-Anthracenedione	11.76	180	500188	79.63	ng	95
9) C826 1-Hydroxy-9,10-Anthra	12.15	224	528589	78.76	ng	97
10) C827 1,4-Dihydro-9,10-Anth	12.56	240	522779	78.33	ng	96
11) C828 (Z)-9-Octadecanamide	12.91	72	175444	61.49	ng	# 62

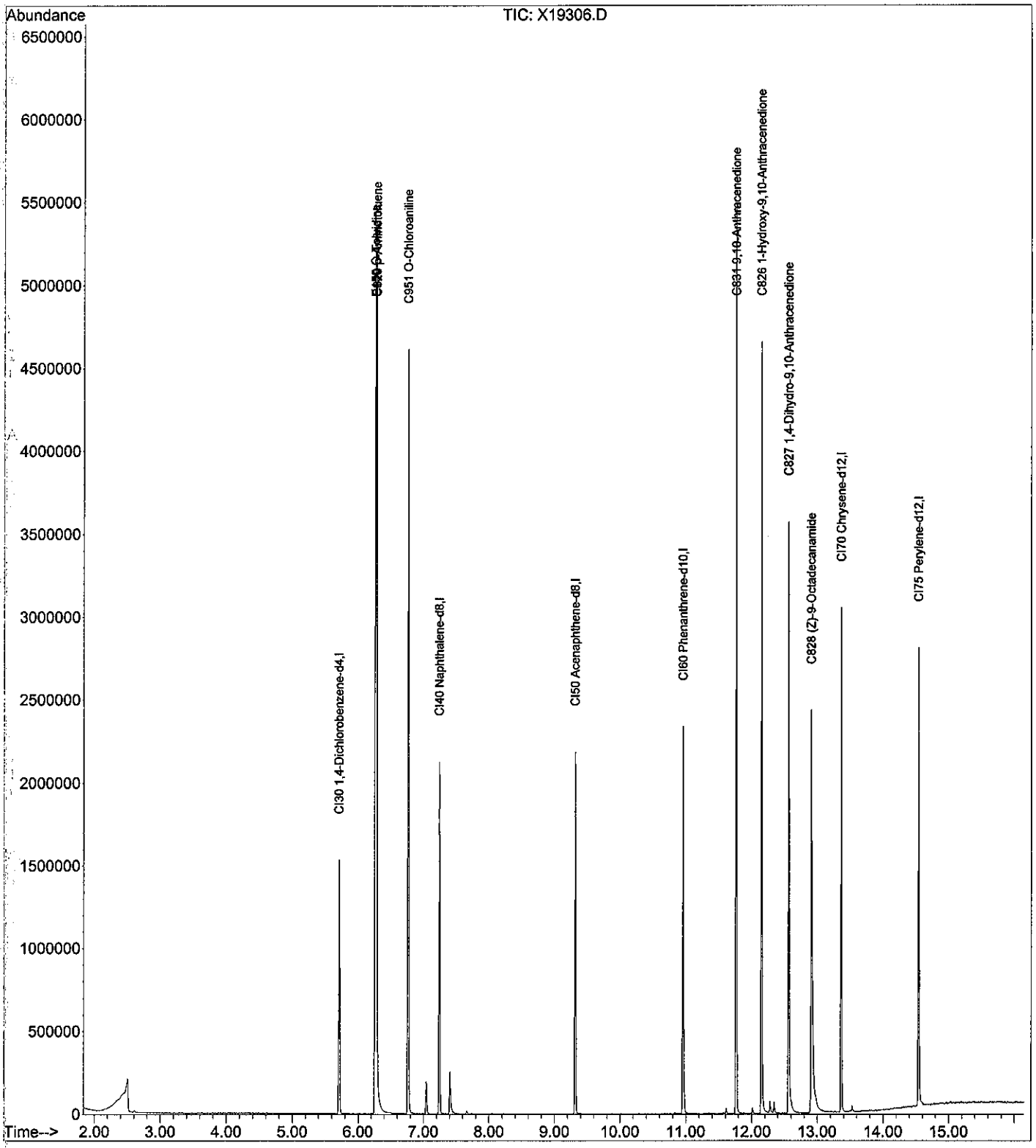
(#) = qualifier out of range (m) = manual integration (+) = signals summed

Quantitation Report (Not Reviewed)

Data File : D:\DATA\082107\X19306.D
Acq On : 22 Aug 2007 3:09 am
Sample : SSTD120
Misc : ADD#3
MS Integration Params: rteint.p

Vial: 41
Operator: PM
Inst : HP5973X
Multiplr: 1.00

Quant Time: Sep 04 15:56:16 2007 Results File: ADD 3.RES
Quant Method : C:\MSDCHEM\1\METHODS\ADD 3.M (RTE Integrator)
Title : ADD#3
Last Update : Tue Sep 04 15:56:11 2007
Response via : Initial Calibration
DataAcq Meth : 8270BP



Quantitation Report (Not Reviewed)

Data File : D:\DATA\082107\X19306.D Vial: 41
Acq On : 22 Aug 2007 3:09 am Operator: PM
Sample : SSTD120 Inst : HP5973X
Misc : ADD#3 Multiplr: 1.00
MS Integration Params: rteint.p
Quant Time: Sep 04 15:56:16 2007 Results File: ADD 3.RES

Quant Method : C:\MSDCHEM\1\METHODS\ADD 3.M (RTE Integrator)
Title : ADD#3
Last Update : Tue Sep 04 15:56:11 2007
Response via : Initial Calibration
DataAcq Meth : 8270BP
IS QA File : CC level for IS QA unknown. No recoveries calculated.

Internal Standards	R.T.	QIon	Response	Conc	Units		Dev(Min) Rcv(Ar)
1) CI30 1,4-Dichlorobenzene-d	5.71	152	252333	40.00	ng		0.00 NA%
5) CI40 Naphthalene-d8	7.24	136	935914	40.00	ng		0.00 NA%
6) CI50 Acenaphthene-d8	9.32	164	510130	40.00	ng		0.00 NA%
7) CI60 Phenanthrene-d10	10.96	188	871404	40.00	ng		0.00 NA%
12) CI70 Chrysene-d12	13.36	240	856687	40.00	ng		0.00 NA%
13) CI75 Perylene-d12	14.54	264	906200	40.00	ng		0.00 NA%
Target Compounds							Qvalue
2) E150 O-Toluidine	6.26	106	1743637	123.76	ng		98
3) C829 p-Aminotoluene	6.28	106	1860719	115.58	ng		100
4) C951 O-Chloroaniline	6.76	127	1357654	118.13	ng		99
8) C831 9,10-Anthracenedione	11.77	180	787036	117.20	ng		95
9) C826 1-Hydroxy-9,10-Anthra	12.16	224	849938	116.70	ng		98
10) C827 1,4-Dihydro-9,10-Anth	12.57	240	842251	115.04	ng		95
11) C828 (Z)-9-Octadecanamide	12.91	72	327726	96.15	ng	#	60

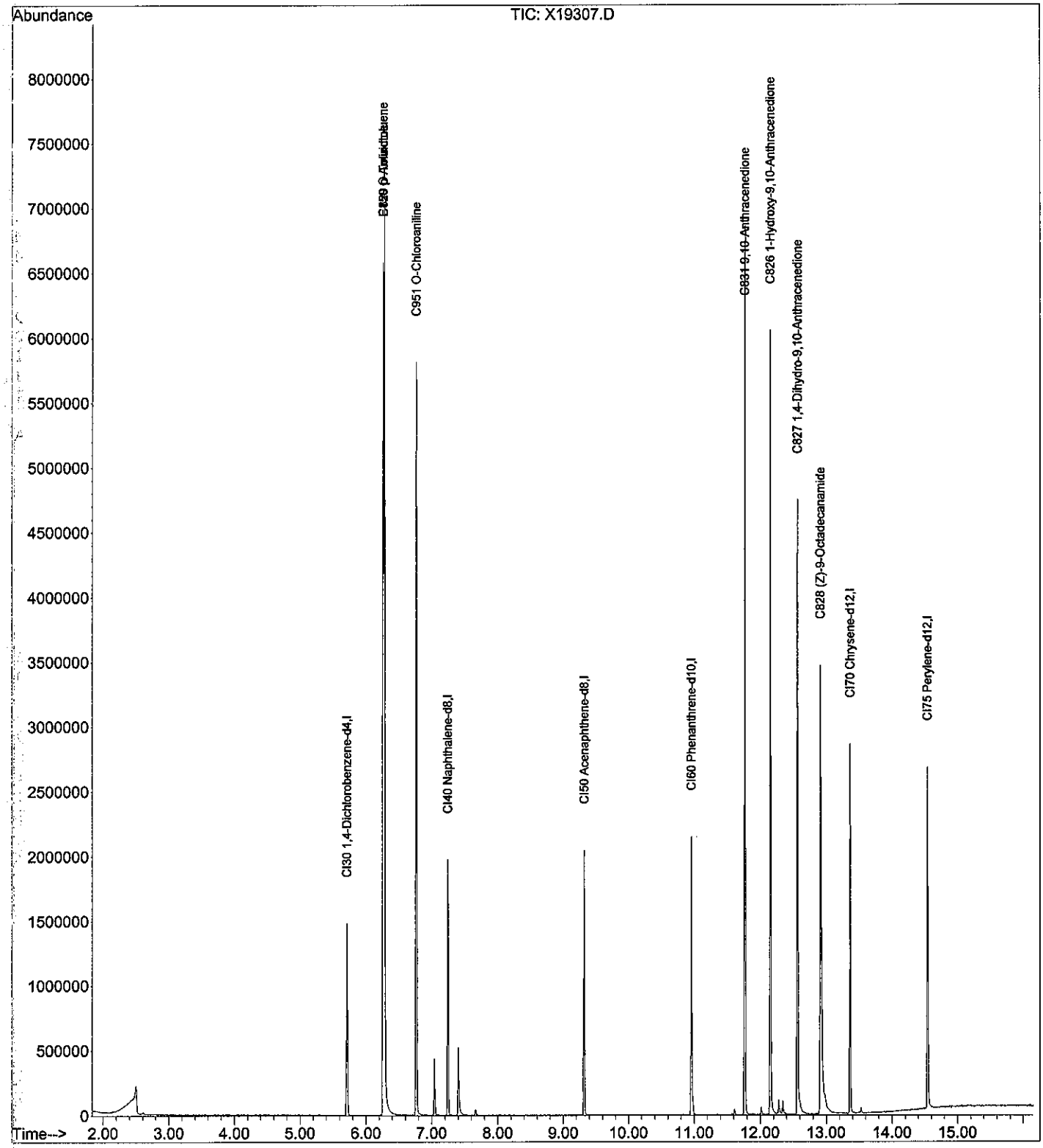
(#) = qualifier out of range (m) = manual integration (+) = signals summed

Quantitation Report (Not Reviewed)

Data File : D:\DATA\082107\X19307.D
Acq On : 22 Aug 2007 3:31 am
Sample : SSTD160
Misc : ADD#3
MS Integration Params: rteint.p

Vial: 42
Operator: PM
Inst : HP5973X
Multiplr: 1.00

Quant Time: Sep 04 15:56:37 2007 Results File: ADD 3.RES
Quant Method : C:\MSDCHEM\1\METHODS\ADD 3.M (RTE Integrator)
Title : ADD#3
Last Update : Tue Sep 04 15:56:32 2007
Response via : Initial Calibration
DataAcq Meth : 8270BP



Quantitation Report (Not Reviewed)

Data File : D:\DATA\082107\X19307.D Vial: 42
Acq On : 22 Aug 2007 3:31 am Operator: PM
Sample : SSTD160 Inst : HP5973X
Misc : ADD#3 Multiplr: 1.00
MS Integration Params: rteint.p
Quant Time: Sep 04 15:56:37 2007 Results File: ADD 3.RES

Quant Method : C:\MSDCHEM\1\METHODS\ADD 3.M (RTE Integrator)
Title : ADD#3
Last Update : Tue Sep 04 15:56:32 2007
Response via : Initial Calibration
DataAcq Meth : 8270BP
IS QA File : CC level for IS QA unknown. No recoveries calculated.

Internal Standards	R.T.	QIon	Response	Conc	Units	Dev(Min) Rcv(Ar)
1) CI30 1,4-Dichlorobenzene-d	5.71	152	236649	40.00	ng	0.00 NA%
5) CI40 Naphthalene-d8	7.24	136	890482	40.00	ng	0.00 NA%
6) CI50 Acenaphthene-d8	9.32	164	467880	40.00	ng	0.00 NA%
7) CI60 Phenanthrene-d10	10.96	188	809574	40.00	ng	0.00 NA%
12) CI70 Chrysene-d12	13.36	240	802933	40.00	ng	0.00 NA%
13) CI75 Perylene-d12	14.54	264	843338	40.00	ng	0.00 NA%
Target Compounds						Qvalue
2) E150 O-Toluidine	6.26	106	2231485	168.88	ng	97
3) C829 p-Aminotoluene	6.26	106	2231485	147.79	ng	90
4) C951 O-Chloroaniline	6.77	127	1764878	163.74	ng	100
8) C831 9,10-Anthracenedione	11.77	180	1022171	162.26	ng	94
9) C826 1-Hydroxy-9,10-Anthra	12.16	224	1129138	163.83	ng	98
10) C827 1,4-Dihydro-9,10-Anth	12.57	240	1157407	165.66	ng	96
11) C828 (Z)-9-Octadecanamide	12.91	72	491633	131.27	ng	# 62

(#) = qualifier out of range (m) = manual integration (+) = signals summed

The purity and supplier of reference standards for the added analytes

Absolute St ndards, Inc.
800-368-1131
www.absolutestandards.com

ISO 9 ISC | QS Registered 025 Accredited

CERTIFIED WEIGHT REPORT

Part Number: 94788 **Solvent(s):** Lot # E04467 Methylene chloride
Lot Number: 081307
Description: Multi-Component Standard
4 components
Expiration Date: 081310

Nominal Concentration (µg/mL): 1000

5E-05 Balance Uncertainty
Weight(s) shown below were combined and diluted to (mL): 25.0 0.011 Flask Uncertainty

Justin Dippold 081307
Formulated By: Justin Dippold DATE

Pedro Rentas 081307
Reviewed By: Pedro L. Rentas DATE

MSDS Information
(Solvent Safety Info. On Attached pg.)

	Compound	RM#	Lot Number	Nominal Conc (µg/mL)	Purity	Uncertainty ± Purity	Target Weight(g)	Actual Weight(g)	*Actual Conc (µg/mL)	Expanded Uncertainty	CAS#	OSHA PEL (TWA)	LD50
1.	Anthraquinone (9,10-Anthracenedione)	537	12416DX	1000	97	0.2	0.02580	0.02584	1001.4	0.0057	00084-65-1	N/A	orl-mus >5g/kg
2.	2-Chloroaniline (o-Chloroaniline)	1708	02303LQ	1000	98	0.2	0.02554	0.02564	1003.9	0.0057	00095-51-2	N/A	N/A
3.	o-Toluidine (2-Methyl-Benzenamine)	401	10614BY	1000	99	0.2	0.02528	0.02535	1002.6	0.0057	00095-53-4	5 ppm (22mg/m3/8H)(skin)	orl-rat 670mg/kg
4.	p-Toluidine (p-Aminotoluene)	1135	08812BZ	1000	99	0.2	0.02528	0.02540	1004.6	0.0057	00106-49-0	2 ppm (9mg/m3/8H)(skin)	orl-rat 656mg/kg
5.													
6.													
7.													
8.													
9.													
10.													
11.													
12.													
13.													
14.													
15.													
16.													
17.													
18.													
19.													
20.													

SC 34-2 Thru 6
recd 8/21/07

Certificate of Analysis

Printed on Oct 27, 2007 (JST)

TOKYO CHEMICAL INDUSTRY CO.,LTD.
4-10-1 Nihonbashi-Honcho, Chuo-ku, Tokyo 103-0023 Japan

Chemical Name: Oleamide		
Product Number: O0107	Lot: K8OSA	

Tests	Results	Specifications
Purity(GC)	71.6 %	min. 70.0 %
Melting point	73.2 deg-C	65.0 to 75.0 deg-C
Solubility in Methanol	transparency	within almost transparency

Customer service:

TCI AMERICA
Tel: +1-800-423-8616 / +1-503-283-1681
Fax: +1-888-520-1075 / +1-503-283-1987
E-mail: sales@tciamerica.com

Certificate of Analysis

TOKYO CHEMICAL INDUSTRY CO., LTD.
4-10-1 Nihonbashi-Honcho, Chuo-ku, Tokyo 103-0023 Japan

Chemical Name: Quinizarin		
Product Number: D0243	Lot: OGM01	

Tests	Results	Specifications
Purity (HPLC) Melting Point	96.2 area % 197.0 °C	min. 95.0 area % 196.0 to 202.0

Customer Service:
TCI America
Tel: 1-800-423-8616 / 1-503-283-1681
Fax: 1-888-520-1075 / 1-503-283-1987
Email: sales@tciamerica.com

Certificate of Analysis

Printed on Oct 26, 2007 (JST)

TOKYO CHEMICAL INDUSTRY CO.,LTD.
4-10-1 Nihonbashi-Honcho, Chuo-ku, Tokyo 103-0023 Japan

Chemical Name: 1-Hydroxyanthraquinone		
Product Number: H0354	Lot: FIH01	

Tests	Results	Specifications
Purity(Neutralization titration)	97.7 %	min. 95.0 %
Melting point	197.5 deg-C	195.0 to 199.0 deg-C

Customer service:

TCI AMERICA
Tel: +1-800-423-8616 / +1-503-283-1681
Fax: +1-888-520-1075 / +1-503-283-1987
E-mail: sales@tciamerica.com

Demonstration of Capability for added analytes (per National Environmental Laboratory Accreditation Conference requirements)

SEVERN TRENT STL

DOC Cert. Statement
Revision 6
October 12, 2005

SEVERN TRENT LABORATORIES - BUFFALO

TRAINING & DEMONSTRATION OF CAPABILITY CERTIFICATION STATEMENT

Employee: Paul McNamara Page ____ of ____

Method Number: 8270 Date: 10/20/2007

Parameters or Analytes: Arcadis Addn for waters

Initial Demonstration of Capability: ☑

SOP Number: AMS-8270C-GC Revision # 7 Date Read NA

Trained By: NA

Date training began: NA Date training completed: NA

Continued Demonstration of Capability: ☐

SOP Number: _____ Revision # _____ Date Read _____

I CERTIFY that I have read and understand the SOP identified above. I have also submitted data associated with the demonstration of capability.

Employee Signature — Date: 10/24/07

We, the undersigned, CERTIFY that:

1. The analyst identified above, using the cited test method(s), which is in use at this facility for the analyses of samples under the National Environmental Laboratory Accreditation Program, have met the Demonstration of Capability.

2. The test method(s) was performed by the analyst(s) identified on this certification.

3. A copy of the test method(s) and the laboratory-specific Sops are available for all personnel on-site.

4. The data associated with the demonstration capability are true, accurate, complete and self-explanatory.

5. All raw data (including a copy of this certification form) necessary to reconstruct and validate these analyses have been retained at this facility, and that the associated information is well organized and available for review by authorized assessors.

John Schove
Operations Manager — Signature — Date: 10/26/07

Verl Preston
Quality Assurance Manager — Signature — Date: 10/25/2007

8270 WATER IDOC FOR ARCADIS ADDS (ADD#3) Rept: AN1341
Study Type: DEMO - Percent Recoveries Page: 1

Instrument: HP5973X
File Extension: RR
Unit of Measure: UG/L

Analyst: PM
Analysis Date: 10/20/2007 - 10/20/2007
Analisys Time: 03:54

Prep Analyst: AL
Prep Method: 3510 WATER
Expiration Date: 10/20/2008

			% Recovery					
No		Compound Name	X20663	X20664	X20665	X20666	Avg	Spk Amt
2	E150	O-Toluidine	83.4	85.7	81.2	82.1	83.1	100.00
3	C829	p-Aminotoluene	77.7	71.8	70.4	74.2	73.5	100.00
4	C951	O-Chloroaniline	78.0	75.5	73.6	70.6	74.4	100.00
8	C831	9,10-Anthracenedione	105.5	106.2	97.9	85.6	98.8	100.00
9	C826	1-Hydroxy-9,10-Anthracenedione	93.3	92.7	87.0	77.2	87.5	100.00
10	C827	1,4-Dihydro-9,10-Anthracenedion	97.3	98.1	90.1	80.0	91.4	100.00
11	C828	(Z)-9-Octadecanamide	107.5	105.8	99.1	90.5	100.7	100.00

8270 WATER IDOC FOR ARCADIS ADDS (ADD#3)
Study Type: DEMO
Rept: AN0751
Page:

Instrument: HP5973X
File Extension: RR
Unit of Measure: UG/L

Analyst: PM
Analysis Dates: 10/20/2007 - 10/20/2007
Analysis Time: 03:54

Prep Analyst: AL
Prep Method: 3510 WATER
Expiration Date: 10/20/2008

No	Compound Name	X20663	X20664	X20665	X20666	Avg	Spike Amt
2	E150 O-Toluidine	83.44580	85.67710	81.24640	82.09590	83.11630	100.0000
3	C829 p-Aminotoluene	77.72520	71.78830	70.42710	74.16470	73.52633	100.0000
4	C951 O-Chloroaniline	78.03400	75.51030	73.62930	70.55630	74.43248	100.0000
8	C831 9,10-Anthracenedione	105.52050	106.21590	97.90750	85.57580	98.80493	100.0000
9	C826 1-Hydroxy-9,10-Anthracenedione	93.31440	92.67630	87.00220	77.18880	87.54543	100.0000
10	C827 1,4-Dihydro-9,10-Anthracenedion	97.27560	98.14470	90.10700	79.98410	91.37785	100.0000
11	C828 (Z)-9-Octadecanamide	107.47190	105.83020	99.07640	90.52900	100.72688	100.0000

SEVERN TRENT STL

DOC Cert. Statement
Revision 6
October 12, 2005

SEVERN TRENT LABORATORIES - BUFFALO

TRAINING & DEMONSTRATION OF CAPABILITY CERTIFICATION STATEMENT

Employee: Paul McNamara Page _____ of _____

Method Number: 8270 Date: 10/23/2007

Parameters or Analytes: Arcadis Abbs for Soils

Initial Demonstration of Capability: ☑

SOP Number: AMS-8270C-66 Revision # 7 Date Read NA

Trained By: NA

Date training began: NA Date training completed: NA

Continued Demonstration of Capability: ☐

SOP Number: _____ Revision # _____ Date Read _____

I CERTIFY that I have read and understand the SOP identified above. I have also submitted data associated with the demonstration of capability.

Employee Signature — Date: 10/24/07

We, the undersigned, CERTIFY that:

1. The analyst identified above, using the cited test method(s), which is in use at this facility for the analyses of samples under the National Environmental Laboratory Accreditation Program, have met the Demonstration of Capability.

2. The test method(s) was performed by the analyst(s) identified on this certification.

3. A copy of the test method(s) and the laboratory-specific Sops are available for all personnel on-site.

4. The data associated with the demonstration capability are true, accurate, complete and self-explanatory.

5. All raw data (including a copy of this certification form) necessary to reconstruct and validate these analyses have been retained at this facility, and that the associated information is well organized and available for review by authorized assessors.

John Schove
Operations Manager — Signature — Date: 10/26/07

Verl Preston
Quality Assurance Manager — Signature — Date: 10/25/2007

Standard calibration curves for added analytes

Instrument: HP5973X
Analyst: PM
Prep Analyst: AL
File Extension: RR
Analysis Date: 10/23/2007 - 10/23/2007
Prep Method: SOIL
Unit of Measure: UG/KG
Analisys Time: 17:16
Expiration Date: 10/23/2008

No	Compound Name	% Recovery X20715	X20716	X20717	X20718	Avg	Spk Amt
2	E150 O-Toluidine	84.1	76.6	70.3	83.5	78.6	3300.00
3	C829 p-Aminotoluene	72.3	81.9	53.1	102.1	77.3	3300.00
4	C951 O-Chloroaniline	84.0	80.1	60.7	91.0	78.9	3300.00
8	C831 9,10-Anthracenedione	115.2	109.5	87.2	122.3	108.6	3300.00
9	C826 1-Hydroxy-9,10-Anthracenedione	103.1	96.5	78.3	107.2	96.3	3300.00
10	C827 1,4-Dihydro-9,10-Anthracenedion	106.0	101.3	81.4	111.5	100.0	3300.00
11	C828 (Z)-9-Octadecanamide	128.0	118.0	98.7	118.3	115.8	3300.00

Instrument: HP5973X
File Extension: RR
Unit of Measure: UG/KG

Analyst: PM
Analysis Dates: 10/23/2007 - 10/23/2007
Analysis Time: 17:16

Prep Analyst: AL
Prep Method: SOIL
Expiration Date: 10/23/2008

No	Compound Name	X20715	X20716	X20717	X20718	Avg	Spike Amt
2	E150 O-Toluidine	2774.45667	2529.30000	2320.81667	2756.79333	2595.34167	3300.0000
3	C829 p-Aminotoluene	2385.12000	2701.64333	1751.05667	3370.33333	2552.03833	3300.0000
4	C951 O-Chloroaniline	2771.07000	2644.03667	2003.11000	3001.79667	2605.00334	3300.0000
8	C831 9,10-Anthracenedione	3801.58667	3614.90000	2878.61333	4035.18333	3582.57083	3300.0000
9	C826 1-Hydroxy-9,10-Anthracenedione	3402.05000	3185.36000	2582.94667	3537.23333	3176.89750	3300.0000
10	C827 1,4-Dihydro-9,10-Anthracenedion	3498.69667	3341.70667	2685.91667	3680.20000	3301.63000	3300.0000
11	C828 (Z)-9-Octadecanamide	4224.76000	3894.14333	3258.62000	3903.73333	3820.31417	3300.0000

Typical sample analysis results

CHEVRON PRODUCTS COMPANY
CHEVRON - HASTINGS ON HUDSON
METHOD 8270 - HASTINGS SEMIVOLATILES
ANALYSIS DATA SHEET

Client No.

SAMPLE A

Lab Name: TestAmerica Laborato Contract: TBD

Lab Code: RECNY Case No.: _____ SAS No.: _____ SDG No.: _____

Matrix: (soil/water) WATER Lab Sample ID: A7C59001

Sample wt/vol: 1000.0 (g/mL) ML Lab File ID: _____

Level: (low/med) LOW Date Samp/Recv: 10/31/2007 10/31/2007

% Moisture: _____ decanted: (Y/N) N Date Extracted: 10/31/2007

Concentrated Extract Volume: 1000 (uL) Date Analyzed: 10/31/2007

Injection Volume: 1.00 (uL) Dilution Factor: 1.00

GPC Cleanup: (Y/N) N pH: ____

CAS NO.	COMPOUND	CONCENTRATION UNITS: (ug/L or ug/Kg) UG/L	Q
100-52-7	Benzaldehyde	5	U
108-95-2	Phenol	5	U
95-57-8	2-Chlorophenol	5	U
95-48-7	2-Methylphenol	5	U
108-60-1	2,2'-Oxybis(1-Chloropropane)	5	U
98-86-2	Acetophenone	5	U
106-44-5	4-Methylphenol	5	U
621-64-7	N-Nitroso-Di-n-propylamine	5	U
67-72-1	Hexachloroethane	5	U
98-95-3	Nitrobenzene	5	U
78-59-1	Isophorone	5	U
88-75-5	2-Nitrophenol	5	U
105-67-9	2,4-Dimethylphenol	5	U
111-91-1	Bis(2-chloroethoxy) methane	5	U
120-83-2	2,4-Dichlorophenol	5	U
91-20-3	Naphthalene	5	U
106-47-8	4-Chloroaniline	5	U
87-68-3	Hexachlorobutadiene	5	U
105-60-2	Caprolactam	5	U
59-50-7	4-Chloro-3-methylphenol	5	U
91-57-6	2-Methylnaphthalene	5	U
77-47-4	Hexachlorocyclopentadiene	5	U
88-06-2	2,4,6-Trichlorophenol	5	U
95-95-4	2,4,5-Trichlorophenol	5	U
92-52-4	Biphenyl	5	U
91-58-7	2-Chloronaphthalene	5	U
88-74-4	2-Nitroaniline	10	U
131-11-3	Dimethyl phthalate	5	U
208-96-8	Acenaphthylene	5	U
606-20-2	2,6-Dinitrotoluene	5	U
99-09-2	3-Nitroaniline	10	U
83-32-9	Acenaphthene	5	U

CHEVRON PRODUCTS COMPANY
CHEVRON - HASTINGS ON HUDSON
METHOD 8270 - HASTINGS SEMIVOLATILES
ANALYSIS DATA SHEET

Client No.

SAMPLE A

Lab Name: TestAmerica Laborato Contract: TBD

Lab Code: RECNY Case No.: _____ SAS No.: _____ SDG No.: _____

Matrix: (soil/water) WATER Lab Sample ID: A7C59001

Sample wt/vol: 1000.0 (g/mL) ML Lab File ID: _____

Level: (low/med) LOW Date Samp/Recv: 10/31/2007 10/31/2007

% Moisture: _____ decanted: (Y/N) N Date Extracted: 10/31/2007

Concentrated Extract Volume: 1000 (uL) Date Analyzed: 10/31/2007

Injection Volume: 1.00 (uL) Dilution Factor: 1.00

GPC Cleanup: (Y/N) N pH: ____

CAS NO.	COMPOUND	CONCENTRATION UNITS: (ug/L or ug/Kg) UG/L	Q
51-28-5	2,4-Dinitrophenol	10	U
100-02-7	4-Nitrophenol	10	U
132-64-9	Dibenzofuran	5	U
121-14-2	2,4-Dinitrotoluene	5	U
84-66-2	Diethyl phthalate	5	U
7005-72-3	4-Chlorophenyl phenyl ether	5	U
86-73-7	Fluorene	5	U
100-01-6	4-Nitroaniline	10	U
534-52-1	4,6-Dinitro-2-methylphenol	10	U
86-30-6	N-nitrosodiphenylamine	5	U
101-55-3	4-Bromophenyl phenyl ether	5	U
118-74-1	Hexachlorobenzene	5	U
1912-24-9	Atrazine	5	U
87-86-5	Pentachlorophenol	10	U
85-01-8	Phenanthrene	5	U
120-12-7	Anthracene	5	U
86-74-8	Carbazole	5	U
84-74-2	Di-n-butyl phthalate	5	U
206-44-0	Fluoranthene	5	U
129-00-0	Pyrene	5	U
85-68-7	Butyl benzyl phthalate	5	U
91-94-1	3,3'-Dichlorobenzidine	5	U
56-55-3	Benzo(a)anthracene	5	U
218-01-9	Chrysene	5	U
117-81-7	Bis(2-ethylhexyl) phthalate	5	U
117-84-0	Di-n-octyl phthalate	5	U
205-99-2	Benzo(b)fluoranthene	5	U
207-08-9	Benzo(k)fluoranthene	5	U
50-32-8	Benzo(a)pyrene	5	U
193-39-5	Indeno(1,2,3-cd)pyrene	5	U
53-70-3	Dibenzo(a,h)anthracene	5	U
191-24-2	Benzo(ghi)perylene	5	U

CHEVRON PRODUCTS COMPANY
CHEVRON - HASTINGS ON HUDSON
METHOD 8270 - HASTINGS SEMIVOLATILES
ANALYSIS DATA SHEET

Client No.

SAMPLE A

Lab Name: TestAmerica Laborato Contract: TBD

Lab Code: RECNY Case No.: _____ SAS No.: _____ SDG No.: _____

Matrix: (soil/water) WATER Lab Sample ID: A7C59001

Sample wt/vol: 1000.0 (g/mL) ML Lab File ID: _____

Level: (low/med) LOW Date Samp/Recv: 10/31/2007 10/31/2007

% Moisture: _____ decanted: (Y/N) N Date Extracted: 10/31/2007

Concentrated Extract Volume: 1000 (uL) Date Analyzed: 10/31/2007

Injection Volume: 1.00 (uL) Dilution Factor: 1.00

GPC Cleanup: (Y/N) N pH: ____

CAS NO.	COMPOUND	CONCENTRATION UNITS: (ug/L or ug/Kg) UG/L	Q
84-65-1	9,10-Anthracenedione	10	U
81-64-1	1,4-Dihydroxy-9,10-anthracendione	40	U
129-43-1	1-Hydroxy-9,10-anthracenedione	20	U
95-51-2	o-Chloroaniline	10	U
301-02-0	(z)-9-octadecenamide	100	U
95-53-4	2-Methyl-Benzenamine	10	U
106-49-0	p-Aminotoluene	10	U

STL BUFFALO
LABORATORY STANDARD OPERATING PROCEDURES

SOP No.	Revision No.	Effective Date	Page
AME-3010-75	3	September 26, 2006	1 of 11

TITLE: METHOD 3010A ACID DIGESTION OF AQUEOUS SAMPLES AND EXTRACTS FOR TOTAL METALS FOR ANALYSIS BY ICP-AES

SUPERCEDES: Revision 2

REVIEWED AND APPROVED BY:	SIGNATURE	DATE
Verl D. Preston, Quality Manager		
Christopher A. Spencer, Laboratory Director		
Jennifer Pierce, Metals Supervisor		

1.0 IDENTIFICATION OF TEST METHODS

1.1. Method 3010A– Acid Digestion of Aqueous Samples for AFCEE and Extracts for Total Metals for Analysis by ICP-AES.

2.0 APPLICABLE MATRIX

2.1. Aqueous samples, TCLP and mobility-procedure extracts, and wastes that contain suspended solids for total metals analysis

3.0 REPORTING LIMIT N/A

4.0 SCOPE AND APPLICATION

4.1. This digestion procedure is used for the preparation of aqueous samples, TCLP and mobility-procedure extracts, and wastes that contain suspended solids for analysis, by inductively coupled plasma atomic emission spectroscopy (ICP-AES). This procedure is used to determine total metals.

TITLE: METHOD 3010A ACID DIGESTION OF AQUEOUS SAMPLES AND EXTRACTS FOR TOTAL METALS FOR ANALYSIS BY ICP-AES

SUPERCEDES: Revision 2

4.2. Samples prepared by Method 3010A may be analyzed by ICP-AES for the following elements.

Aluminum	Calcium	Magnesium	Silver
Antimony	Chromium	Manganese	Sodium
Arsenic	Cobalt	Molybdenum	Thallium
Barium	Copper	Nickel	Tin
Beryllium	Iron	Potassium	Titanium
Boron	Lead	Selenium	Vanadium
Cadmium			Zinc

5.0 SUMMARY OF TEST METHOD

5.1. A mixture of Nitric acid and the sample is refluxed in a digestion cup. This step is repeated with additional portions of Nitric acid until the digestate is light in color or until its color has stabilized. After the digestate has been brought to a low volume between 5 and 10mls, it is refluxed with Hydrochloric acid and finally brought up to the final volume of 50mls. If the sample should go to dryness, it must be discarded and the sample reprepared.

6.0 DEFINITIONS

6.1 Total Metals – The concentration determined on an unfiltered acidified sample following vigorous digestion.

6.2 Trace ICP – an ICP with the viewing angle along the long axis of the torch.

7.0 INTERFERENCES

7.1. Potential sources of trace metals contamination include: metallic or metal-containing labware (e.g., talc gloves which contain high levels of zinc), containers, impure reagents, dirty glassware, improper sample transfers, dirty work areas, atmospheric inputs such as dirt and dust, etc. Be aware of potential sources of contamination and take appropriate measures to minimize or avoid them.

7.2. Physical interference effects may contribute to inaccuracies in the determination of trace elements. Oils, solvents and other matrices may not be digested using this method if they are not soluble with acids. If physical interferences are present, they should be documented.

7.3. Visual interferences or anomalies (such as foaming, emulsions, precipitates, etc.) must be documented on the digestion log.

7.4. Allowing samples to boil or go dry during digestion may result in the loss of volatile metals. If this occurs the sample must be reprepared. Antimony is easily lost by volatilization from hydrochloric acid media.

7.5. Precipitation of silver chloride (AgCl) may occur when chloride ions and high concentrations of silver (i.e., greater than 1 mg/L) are present in the sample. Samples containing more than 1 mg/L of silver can be diluted, redigested and reanalyzed to produce more accurate results upon project manager/client request.

8.0 SAFETY

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

8.2. Blue Nitrile Gloves are to be used when handling all standards and samples. Safety glasses must be worn at all times. Extra care is taken when dispensing concentrated acids. Concentrated acids are to be dispensed only in the fume hood.

8.3. SPECIFIC SAFETY CONCERNS OR REQUIREMENTS

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

8.4. PRIMARY MATERIALS USED

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

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

TITLE: METHOD 3010A ACID DIGESTION OF AQUEOUS SAMPLES AND EXTRACTS FOR TOTAL METALS FOR ANALYSIS BY ICP-AES

SUPERCEDES: Revision 2

Material (1)	Hazards	Exposure Limit (2)	Signs and symptoms of exposure
Nitric Acid	Corrosive Oxidizer Poison	2 ppm-TWA 4 ppm-STEL	Nitric acid is extremely hazardous; it is corrosive, reactive, an oxidizer, and a poison. Inhalation of vapors can cause breathing difficulties and lead to pneumonia and pulmonary edema, which may be fatal. Other symptoms may include coughing, choking, and irritation of the nose, throat, and respiratory tract. Can cause redness, pain, and severe skin burns. Concentrated solutions cause deep ulcers and stain skin a yellow or yellow-brown color. Vapors are irritating and may cause damage to the eyes. Contact may cause severe burns and permanent eye damage.
1 – Always add acid to water to prevent violent reactions.			
2 – Exposure limit refers to the OSHA regulatory exposure limit.			

9.0 EQUIPMENT AND SUPPLIES

9.1 *Environmental Express* Hot Blocks

9.2 *Environmental Express* 50 mL Polypropylene digestion cups

9.3 Eppendorf pipettes and pipette tips

9.4 NIST Certified Thermometer

9.5 Filters

10.0 REAGENTS AND STANDARDS

10.1. Reagent grade chemicals shall be used in all tests. Unless otherwise indicated, it is intended that all reagents shall conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available. Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination.

10.2. Laboratory Reagent Water – produced by a Millipore de-ionized system. The maximum allowed conductivity is 1.0 ohms-cm at 25°C. The reagent water must be free of the analytes of interest as demonstrated through the analysis of method blanks.

10.3. Nitric Acid (HNO3), concentrated, trace metal grade or better.

10.4. Hydrochloric acid (HCl), concentrated, trace metal grade or better.

10.4.1. The certificates of analysis for the concentrated acids are listed on the bottle. Whenever the purity of the acid is suspect, the acid should be analyzed by ICP-MS to determine

levels of impurities. If impurity concentrations are at such levels that method blanks are <MDL, the acid may be used.

10.5. 1:2 HCl prepared by mixing equal volume of reagent water and concentrated Hydrochloric acid. Pour concentrated acid to water; never pour water to concentrated acid.

10.6. Spike standards:

10.6.1. Silver (Ag) - 10 µg/mL in 2% HNO3: Add 0.5 mL of 1000 µg/mL Ag stock standard to 30 ml reagent water in a 50 ml volumetric flask. Add 1 ml concentrated HNO3. Dilute to volume with reagent water.

10.6.2. Tin (Sn) - 40 µg/mL in 2% HNO3: Add 2.0 mL of 1000 µg/mL Sn stock standard to 30 ml reagent water in a 50 ml volumetric flask. Add 1 ml concentrated HNO3. Dilute to volume with reagent water.

10.6.3. ICP-AES spikes: ICUS-1370, ICUS-574, 10 µg/mL Ag, 40 µg/mL Sn and ICUS-1454 (this spike is used for all TCLP's). See Table 21.1 for detail.

10.6.4. The Certificates of Analysis for these standards are kept by analysts in the Digestion Lab.

11.0 SAMPLE COLLECTION, PREPARATION AND STORAGE

11.1. Aqueous wastewaters must be acidified to a pH of < 2 with concentrated HNO_3. Refrigeration is not required.

11.2. Sample holding time for metals is 180 days from the date of collection to the date of analysis.

11.3. If Boron is to be determined, collection into a plastic container is preferred.

11.4. If samples are received unpreserved, the Project Manager must be contacted immediately so that the client can be informed.

11.5. The matrix spike solution must be added to TCLP leachates before the samples are acidified.

12.0 QUALITY CONTROL

12.1. Method Blank/Prep Blank (MB, PB) is a volume of reagent water processed through the sample preparation and analysis procedure. For each batch of samples (not to exceed 20 samples), a Method Blank must be employed. This blank is useful in monitoring any contamination.

12.2. Laboratory Fortified Blank (LFB) is a volume of reagent water spiked with known concentrations of analytes and carried through the preparation and analysis procedure. For each batch of samples (not to exceed 20 samples), a LFB must be employed to determine analyte recovery.

12.3. A Matrix Spike and or Matrix Spike Duplicate is an aliquot of sample that has been fortified with known concentrations of analytes and carried through the preparation and analysis procedure. For each batch of samples (not to exceed 20 samples), a matrix spike (MS) should be processed on a routine basis to determine biases in the analytical results due to sample matrix. Matrix Spike Duplicates (SD) samples may also be used to determine matrix effects on digestion and detection.

12.4. For each batch of samples (not to exceed 20 samples), replicate samples should be processed on a routine basis. Replicate samples are either matrix duplicate (MD) or matrix *spike* duplicate (SD) depending on the clients' request, but are usually matrix spike duplicates. Replicate samples will be used to determine precision. MD is just another aliquot of the selected sample. SD is just another MS that is processed through the preparation and analysis procedure.

13.0 CALIBRATION AND STANDARDIZATION:

13.1. The Environmental Express digestion cups are Class-A-calibrated. The certificates are kept in digestion lab. The lot number of the cups used is recorded in the comment section on the digestion log. Each lot is verified at the 50ml final volume mark.

13.2. Analytical balances should be checked and calibrated using NIST Class "1" Certified weights (See SOP AGP-BAL-05) daily. These weights are recorded in a weight calibration logbook stored in the digestion laboratory.

13.3. Hot block temperatures are to be checked daily and documented in the digestion hot block temp logbook. The Hot block temperature is verified by measuring the temperature of a tube of reagent water placed in the apparatus.

13.4. Pipettes /Eppendorf's are verified weekly and calibrated quarterly by a delivery of reagent water on a Certified Balance (See SOP AGP-PIPET-01). These results are entered into a QA approved spreadsheet, copies of these spreadsheets are in the digestion laboratory.

14.0 PROCEDURES

14.1. One time procedural variations are allowed only if deemed necessary in the professional judgment of supervision to accommodate variation in sample matrix, chemistry, sample size or other parameters. Any variation in procedure shall be completely documented using a Job Exception Form. The Job Exception is routed to the Metals supervisor and then to the lab Project Manager and QA staff for possible client notification. The Job Exception should be placed in the project file. Any unauthorized deviations from this procedure must also be documented as a nonconformance, with cause and corrective addition described.

14.2. Method Blank/Prep Blank (MB, PB): For each digestion batch of 20 samples or less, transfer 50 mL of laboratory reagent water to a digestion cup and carry through the entire analytical process.

14.3. Laboratory Fortified Blank (LFB): For each digestion batch of 20 samples or less, transfer 50 mL of laboratory reagent water to a digestion cup, fortify with the following spike solutions and carry through the entire analytical process.

Spike Standards	Volume
ICUS-1370	0.25 ml
ICUS-574	0.25 ml
Ag (10 ug/ml)	0.25 ml
Sn (40ug/ml)	0.25 ml
ICUS-1454 *use for TCLP's	2.0 ml / 400ml

14.4. Matrix Spike (MS) and Matrix Spike Duplicate (SD): For each digestion batch of 20 samples or less, prepare one sample in triplicate. Analyze one aliquot and fortify two aliquots with the same spiking solutions as listed in 14.3 for the LFB. These three samples are Sample, MS and SD.

14.5. Matrix Duplicate: For each digestion batch of 20 samples or less, prepare one sample in triplicate and fortify one aliquot with the spiking solutions indicated above for the MS. Analyze the other two aliquots. These three samples are treated as sample, MD and MS. MD is not routinely prepared. It is only done on the basis of the clients' requests.

14.6. Analysis Procedure:

14.6.1. Transfer a 50 mL representative aliquot of the well mixed sample to a 50 mL digestion cup.

14.6.2. Add 3.0 ml of concentrated HNO_3.

14.6.3. Place the cup on a hot block (sample temperature 95° ±3°C) and cautiously evaporate to a low volume of approximately 5 mL, making certain that the sample does not boil and that no portion of the bottom of the digestion cup is allowed to go dry.

14.6.3.1. NOTE: If a sample is allowed to go to dryness, low recoveries will result. Should this occur, discard the sample and reprepare.

14.6.3.2. NOTE: If samples are evaporated unevenly, reagent water might be added to bring all samples to the same volume and continue the evaporation. This note is also applicable to other evaporation steps.

14.6.4. Continue heating, adding additional HNO_3 (3 mL as an example) as necessary, until the digestion is complete (generally indicated when the digestate is light in color or does not change in appearance with continued refluxing).

14.6.5. Evaporate to a low volume (approximately 5 ml), not allowing any portion of the bottom of the digestion cup to go dry. Cool the digestion cup.

14.6.6. Add 5.0 mL 1:2 HCl (equal parts of concentrated Hydrochloric acid and blank water).

14.6.7. Reflux for an additional 15 minutes to dissolve any precipitate or residue resulting from evaporation.

14.6.8. Wash down the cup walls. Bring to final volume of 50 mL with reagent water.

14.6.9. Turbid samples are filtered with 2 µm Teflon filters or .45um membrane disk filters prior to analysis.

15.0 CALCULATIONS NA

16.0 METHOD PERFORMANCE

16.1. On an annual basis, Method Detection Limit studies are performed in accordance with 40 CFR 136, Appendix B.

17.0 DATA ASSESSMENT AND ACCEPTANCE CRITERIA FOR QUALITY CONTROL MEASURES

17.1 A representative digestion batch and the quality control criteria are illustrated below:

Sample/QC	Acceptance Criteria
Method Blank	< LAB PQL , <2.2x MDL for 200 series
Lab Fortified Blank	80-120% SW846, 85-115% for 200 series
Matrix Spike	75-125% Recovery
Matrix Spike Duplicate or Matrix Duplicate	<20% RPD
≤ 20 Samples	N/A

TITLE: METHOD 3010A ACID DIGESTION OF AQUEOUS SAMPLES AND EXTRACTS FOR TOTAL METALS FOR ANALYSIS BY ICP-AES

SUPERCEDES: Revision 2

18.0 CONTINGENCIES FOR HANDLING OUT-OF-CONTROL OR UNACCEPTABLE DATA

18.1. Contingencies for unacceptable data will have to be evaluated on a client-by–client or even by a sample-by-sample basis by the supervisor, the lab director or the project manager. Corrective action will be prescribed accordingly.

18.2. A job exception form should be completed for the following issues:

- Insufficient Sample
- Unusual Matrix
- Loss of Digestate
- Holding Time exceedance

19.0 WASTE MANAGEMENT /POLLUTION PREVENTION

19.1. All samples, reagents, and laboratory wastes must be handled with caution. Appropriate safety measures should be employed as detailed in STL's Laboratory Safety Manual and Chemical Hygiene Plan. 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."

19.2. Waste Streams Produced by the Method:
The following waste streams are produced when this method is carried out.

19.2.1. All acidic waste generated should be disposed of as HNO_3 waste in a "AN" waste container. All laboratory wastes and used samples must be disposed in an "AN" waste container as detailed in STL's Laboratory Safety Manual, Chemical Hygiene Plan, and SOP AWM-HazMg-01.

20.0 REFERENCES

20.1. *Method 3010A*; Test Methods for Evaluating Solid Waste, Physical/Chemical Methods; SW846, Third Edition; 9/86 with all applicable updates (I-7/92; II-9/94; IIA-8/93; IIB-1/95; III-12/96; IIIA-4/98)

21.0 TABLES AND DIAGRAMS

21.1 Table 1: ICP-AES Spikes

21.2 Diagram 1: Digestion log

TITLE: METHOD 3010A ACID DIGESTION OF AQUEOUS SAMPLES AND EXTRACTS FOR TOTAL METALS FOR ANALYSIS BY ICP-AES

SUPERCEDES: Revision 2

22.0 CHANGES FROM PREVIOUS REVISION

22.1 Section 5.1: Defined low volume

22.2 Section 10.2: Corrected spelling of Ohms

22.3 Section 17.1: Method Blank (MB) added 200 series criteria

22.4 Section 22.1: Deleted 2 columns that were duplicates

21.1 Spike Solutions and Final Concentrations for ICP-AES

Analyte	**ICUS-1370** (µg/mL)	**ICUS-574** (µg/mL)	10 ug/mL Ag Stock (ug/mL)	10 µg/mL Sn Stock (µg/mL)	**ICUS-1454** *(ug/mL)*	Final Conc. In Digestate if using **ICUS-1454** (ug/mL)	Final Conc. In Digestate if using **ICUS-1370** and **ICUS-574** (ug/mL)
Aluminum		2000					10
Antimony	40				200	1.0	0.2
Arsenic	40				200	1.0	0.2
Barium		40			200	1.0	0.2
Beryllium	40				200	1.0	0.2
Boron		40					0.2
Cadmium	40				200	1.0	0.2
Calcium	2000						10
Chromium	40				200	1.0	0.2
Cobalt	40				200	1.0	0.2
Copper	40				200	1.0	0.2
Iron	2000						10
Lead	40				200	1.0	0.2
Magnesium	2000						10
Manganese	40				200	1.0	0.2
Molybdenum	40				200	1.0	0.2
Nickel	40				200	1.0	0.2
Potassium		2000					10
Selenium	40				200	1.0	0.2
Silver			10		200	1.0	0.05
Sodium		2000					10
Thallium	40				200	1.0	0.2
Tin				40			0.2
Vanadium	40				200	1.0	0.2
Zinc	40				200	1.0	0.2
Titanium	40						0.2

SOP No.	Revision No.	Effective Date	Page
AME-3010-75	3	September 26, 2006	11 of 11

TITLE: METHOD 3010A ACID DIGESTION OF AQUEOUS SAMPLES AND EXTRACTS FOR TOTAL METALS FOR ANALYSIS BY ICP-AES

SUPERCEDES: Revision 2

21.2 Digestion Log

STL Buffalo
Date: 09/24/2003
Time: 12:26:37

METALS DIGESTION LOG
A3B10706 - 09/22/2003 TCLP WATER 3010 (Closed)
AQUEOUS

Page: 1
Rept: AN0764

Date	Time	Dig Emp	Jobno	Sample ID	Bot ID	Sample Type	Digest ID	V1	Analysis Type	Initial V1 (ml)	Final (ml)	Color Before/After	Clarity Before/After	Textur
09/22/03	13:00	AH	A03-8995	A3899501	A	FS	AD346711	A	TCLP	50.00	50.00	COLORLES / COLORLES	CLEAR / CLEAR	NONE
09/22/03	13:00	AH	A03-8999	A3899901	A	FS	AD346712	A	TCLP	50.00	50.00	COLORLES / COLORLES	CLEAR / CLEAR	NONE
09/22/03	13:00	AH	A03-8999	A3899901MS	A	MS	AD346713	A	TCLP	50.00	50.00	COLORLES / COLORLES	CLEAR / CLEAR	NONE
09/22/03	13:00	AH	A03-8999	A3899901SD	A	SD	AD346714	A	TCLP	50.00	50.00	COLORLES / COLORLES	CLEAR / CLEAR	NONE
09/22/03	13:00	AH		A3B1070601	A	LCS	AD346715	A	TCLP	50.00	50.00	COLORLES / COLORLES	CLEAR / CLEAR	NONE
09/22/03	13:00	AH		A3B1070602	A	EBLK	AD346716	A	TCLP	50.00	50.00	COLORLES / COLORLES	CLEAR / CLEAR	NONE
09/22/03	13:00	AH		A3B1070603	A	MBLK	AD346717	A	TCLP	50.00	50.00	COLORLES / COLORLES	CLEAR / CLEAR	NONE

Comments: EPPENDORF'S USED IN PARENTHESIS:
8.) MD-03A-2702E 2.00ml SN 388671
QUALITY CONTROL ADDITIVES:
SPIKES ADDED / EPPENDORF (*) USED FOR SPIKING
A- 1 - W1 MDL1920 (8) 2.0ml/400ml INT. VOL .25ml/50ML FIN VOL
2 - W2 MDL2020 (8) 2.0ml/400ml INT. VOL .25ml/50ML FIN VOL
3 - Ag MSL4019 (8) 2.0ml/400ml INT. VOL .25ml/50ML FIN VOL
4 - Sn MSL4020 (8) 2.0ml/400ml INT. VOL .25ml/50ML FIN VOL
CONC. NITRIC ACID = 1-MDL-21
1:1 Hcl ACID = MSL4018
EBLANKS in NUM.ORDER = J-1033
HOT BLOCK TEMPERATURE = A/124
SAMPLE TEMPERATURE = 96
BATCH ENDED = 22:30
DIGESTIVE CUP LOT = A305LP029

Color: Black Gray Red Yellow
Blue Green Violet Colorless
Brown Orange White

Clarity: Clear
Cloudy
Opaque

Texture: Fine (powdery)
Medium (sand)
Coarse (large crystals or rocks)

* Redigestion

STL BUFFALO
LABORATORY STANDARD OPERATING PROCEDURES

SOP No.	Revision No.	Effective Date	Page
AME-6010-30	16	June 13, 2006	1 of 71

TITLE: METHOD 6010B/ 200.7/CLP USING THE THERMO JARRELL ASH 61E TRACE

SUPERCEDES: Revision 15

REVIEWED AND APPROVED BY:	SIGNATURE	DATE
Verl Preston, Quality Manager		
Christopher Spencer, Laboratory Director		
Jennifer Pierce, Supervisor		

1.0 IDENTIFICATION OF TEST METHODS

1.1 This SOP is used for the determination of Dissolved (Soluble) and Total elements by Thermo Jarrell Ash ICAP 61E Trace Analyzer (referred to also as the Trace). This SOP is specific for methods (SW-846) 6010B, 200.7, and CLP.

2.0 APPLICABLE MATRIX

2.1 Soluble water samples and digestates of waters, TCLPs, total recoverables, soils, sludges, sediments, and other wastes.

3.0 REPORTING LIMIT

3.1 Tables 22.2 and 22.3 list achievable Instrumental Detection Limits (IDLs) and Method Detection Limits. The laboratory IDLs are updated quarterly and the MDLs are updated annually. The current IDLs and MDLs are maintained in the laboratory LIMs system.

3.2 The laboratory standard Practical Quantitation Limits (PQLs) are also listed in Tables 22.2 and 22.3. The standard laboratory PQLS remain static and are only changed if there is a major update to the analytical system.

TITLE: METHOD 6010B/ 200.7/CLP USING THE THERMO JARRELL ASH 61E TRACE

SUPERCEDES: Revision 15

4.0 SCOPE AND APPLICATION

4.1 This SOP is specific for methods (SW-846) 6010B, 200.7, and CLP. This SOP discusses the procedures as they are performed at STL Buffalo. Table 22.15 summarizes the actual method criteria.

4.2 At STL Buffalo, there are two 61E Trace Analyzers. They are designated as Trace #1 and Trace #2.

4.3 Table 22.1 lists the elements that are analyzed on each Trace.

4.4 Tables 22.2 and 22.3 list the approximate instrument detection limits (IDL's) which can be achieved on each Trace. IDL's are recalculated quarterly or when a significant instrumentation change occurs.

4.5 Table 22.4 lists the wavelengths and typical background points used on each Trace.

4.6 The linear range is the concentration range over which the instrument response to an analyte is linear. Table 22.5 lists the approximate linear ranges of each Trace. Linear ranges are recalculated quarterly or when a significant instrumentation change occurs.

4.7 Interelement correction factors (IECs) are used to correct for interferences caused by spectral overlap of elemental lines. At STL, IECs are verified and calculated quarterly or when an instrumentation change occurs.

4.8 All samples, standards, and blanks are matrix matched to achieve an aqueous solution containing 6% HNO_3 and 5% HCl by volume.

5.0 SUMMARY OF TEST METHOD

5.1 Samples are nebulized and the aerosol that is produced is transported to the plasma torch where excitation occurs. Characteristic atomic-line emission spectra are produced by radio frequency inductively coupled plasma (ICP). The spectra are dispersed by a grating spectrometer and the intensities of the emission lines are monitored by photomultiplier tubes. The photocurrents from the photomultiplier tubes are processed and controlled by a computer system. A background correction technique is required to compensate for variable background contribution to the determination of trace elements. Background must be measured adjacent to analyte lines during analysis. The position selected for the background intensity measurement, on either or both sides of the analytical line, will be determined by the complexity of the spectrum adjacent to the analyte line. The position used must be free of spectral interferences and reflect the same change in background intensity as occurs at the analyte wavelength measured.

5.2 This SOP contains the procedures for the daily operation of the ICAP 61E Trace Analyzer. This SOP also contains procedures for calibration, standard and sample preparation, maintenance, data handling, and quality control. This SOP is based on methods 6010B (SW-846), 200.7 and CLP.

5.3 On a given day the normal steps in operating the Trace include:

- Perform any routine maintenance, if required.
- Instrument start-up and warm-up
- Preparation of standards. All standards and quality control standards are prepared from stock solutions, as needed (6-month expiration date.) The Calibration Standards are made every 3-7 days.
- Type-up a run to analyze. A run is simply a sequence of samples with all required quality control that is analyzed as a single unit.
- Set-up the autosampler.
- Prepare all the samples for analysis including the required spikes, serial dilutions, and other quality control samples.
- Analyze the samples.
- When the analysis is complete, check the data for compliance with 6010B, 200.7, AFCEE or CLP whichever is applicable.
- Log in compliant data.
- Dispose of samples and standards appropriately. Clean-up area.

5.4 If the instrument is not operating properly or requires any maintenance, refer to Section 14 for help with routine maintenance and troubleshooting.

6.0 DEFINITIONS

6.1 Trace - Abbreviation for Thermo Jarrell Ash ICAP 61E Trace Analyzer. The Trace ICP has a viewing angle along the long axis of the torch.

6.2 IECs - Interelement correction factors. Used to correct for interferences caused by spectral overlap of element lines. See Section 12.17.1 for procedures on determining IECs.

6.3 Linear Range - Also referred to as linear dynamic range. The linear range is the concentration range over which the instrument response to an analyte is linear. Refer to Section 12.17.3 for the determination of linear ranges.

6.4 IDL - Instrument detection limit. The IDL of an element is the lowest calculated concentration that the instrument can measure. See section 12.17.2 for procedures on determining IDLs.

6.5 MDL – Method Detection Limit. The minimum amount of a substance that can be measured with a specified degree of confidence that the amount is greater than zero.

6.6 PQL – Practical Quantitation Limit. The minimum amount of a substance that can be *quantitatively* measured with a specified degree of confidence and within *accuracy and precision guidelines*.

6.6 Calibration Standards - A series of solutions containing known amounts of each element with a matrix similar to samples. These solutions are used to calibrate the instrument.

6.7 ICL - The highest calibration standard re-run directly after calibrating the instrument.

6.8 ICV - Initial calibration verification, which must be from a source different from that of the calibration standard

6.9 ICB - Initial calibration blank.

6.10 ICSA - Interference check sample containing only high levels of Al, Fe, Ca, and Mg.

6.11 ICSAB - Interference check sample containing high levels of Al, Fe, Ca, and Mg, and low levels of all other elements that are analyzed by the Trace.

6.12 CCV - Continuing calibration verification.

6.13 CCB - Continuing calibration blank.

6.14 LCS - Laboratory control sample. A quality control sample containing known concentration of analytes that is taken through the entire digestion and analysis procedure.

6.15 Method Blank - A blank sample that is taken through each step of the analytical procedure, including the digestion procedure if it is used.

6.16 Method of Standard Addition - Involves the analysis of an unknown sample and the analysis of an unknown sample with a known amount of a standard added. This procedure may be used when matrix interference is suspected.

6.17 Calibration Blank - A blank solution containing 6% HNO_3 and 5% HCl for calibration.

6.18 Total Metals – The concentration determined on an unfiltered sample following vigorous digestion.

6.19 Soluble or Dissolved Metals – The concentration determined on a sample after passing through a 0.45 um membrane. Acidification and digestion are performed after filtration.

6.20 ELGA water – This is blank reagent water that is deionized, filtered, and has a resistivity of 18 $M\Omega cm^{-1}$.

6.21 Calibration Curvefit- This process allows individual element wavelengths to be standardized using an extended calibration with more standards to produce a more linear and precise response over a greater range. The calibration is then fitted to a curve, which can be resloped during the instrument standardization.

TITLE: METHOD 6010B/ 200.7/CLP USING THE THERMO JARRELL ASH 61E TRACE

SUPERCEDES: Revision 15

7.0 INTERFERENCES

7.1 There are four main types of interferences. They are - spectral, physical, chemical and memory interferences.

7.1.1 Spectral Interferences

These types of interferences are caused primarily from the overlap of elemental lines and background contributions. Interferences from spectral overlap are eliminated by the use of interelement correction factors. Interferences caused by background contributions are eliminated by the use of background correction. Table 22.3 lists the typical background points.

7.1.2 Physical Interferences

These types of interferences are caused by differences between the physical properties of standards and samples. The major source of these interferences is a high dissolved solids concentration in a sample. Physical interferences are minimized by using an internal standard, diluting the samples and/or performing the method of standard addition.

Additionally, high salt concentrations can cause a buildup of salt at the tip of the nebulizer. This effect is minimized on the Trace by use of an Argon Saturator and a Noordermeer V-Groove nebulizer designed for high dissolved solid use.

7.1.3 Chemical Interferences

These are generally caused by molecular compound formation, ionization effects, and solvent evaporization effects. These effects can be minimized by careful selection of the operating conditions, by buffering the sample, or by standard addition procedures. At STL Buffalo, buffer solution of $Li(NO_3)$ is added on-line to minimize the ionization effects of the high level of easily ionized elements such as K and Na.

7.1.4 Memory Interferences

Memory interferences result when analytes in a previous sample contribute to the signals measured in a new sample. To minimize memory effects, a rinse period of at least 60 seconds is used between samples and standards. If memory interference is suspected, the sample must be reanalyzed after a rinse period of sufficient length.

7.2 The following tests may be performed to check for physical and chemical interferences. A serial dilution is performed on a representative sample from each sample batch. A post digestion spike is performed based upon client requirements. The sample batch does not exceed twenty samples.

7.2.1 Serial Dilution

A serial dilution (1:5) is performed on a representative sample of each matrix of each sample group. See Table 22.15 for recovery criteria for each method. If the element concentration is high enough, such that the analyte in the diluted sample is at least a factor of 10 above the IDL, the serial dilution must agree within ±10% of the original sample. If the serial dilution is outside the 10% limit, a chemical or physical interference effect should be suspected.

7.2.2 Spike Addition

A post-spike is performed when required per method being analyzed. A representative sample within the sample group (client job) is spiked. Generally, the spike is performed on the same sample as the one on which the serial dilution is performed, unless there is limited volume. Spiking a sample consists of adding a specified amount of four separate spike solutions to the unknown sample. Each spike solution contains various elements of interest. See Table 22.15 for recovery criteria for each analyte.

7.2.2.1 The four spike solutions for Non-CLP samples are:

- **Spike 1 (Custom Inorganic Standard) Made by Ultra Scientific**
  This ULTRAgrade ™ standard was gravimetrically prepared and the true value listed is the concentration calculated from gravimetric and volumetric measurements performed during the preparation of the standard.

ANALYTE	TRUE VALUE
Antimony	40.0 µg/mL
Arsenic	40.0 µg/mL
Beryllium	40.0 µg/mL
Cadmium	40.0 µg/mL
Chromium	40.0 µg/mL
Cobalt	40.0 µg/mL
Copper	40.0 µg/mL
Lead	40.0 µg/mL
Manganese	40.0 µg/mL
Molybdenum	40.0 µg/mL
Nickel	40.0 µg/mL
Selenium	40.0 µg/mL
Thallium	40.0 µg/mL
Vanadium	40.0 µg/mL

Zinc	40.0 µg/mL
Titanium	40.0 µg/mL
Calcium	2000.0 µg/mL
Iron	2000.0 µg/mL
Magnesium	2000.0 µg/mL

Matrix: 5% HNO_3 in water

All weights are traceable to NIST traceable weights CAT#ICUS-1370.

NOTE: These concentrations might be slightly different between different lots. Current concentrations may be found in the binder of the Certificates of Analysis. This NOTE is also applicable to Spike 2, Spike 3 and Spike 4.

- **Spike 2 (Custom Inorganic Standard) Made by Ultra Scientific**

This ULTRAgrade ™ standard was gravimetrically prepared and the true value listed is the concentration calculated from gravimetric and volumetric measurements performed during the preparation of the standard.

ANALYTE	TRUE VALUE
Barium	40.0 µg/mL
Boron	40.0 µg/mL
Aluminum	2000.0 µg/mL
Potassium	2000.0 µg/mL
Sodium	2000.0 µg/mL

Matrix: 5% HNO_3 in water

All weights are traceable to NIST traceable weights CAT# ICUS-574

- **Spike 3**

ANALYTE	TRUE VALUE
Silver	10 µg/mL

See 10.5.1 for preparation.

- **Spike 4**

ANALYTE	TRUE VALUE
Tin	40 µg/mL

See 10.5.2 for preparation.

Table 22.5 lists the final concentration of each element spiked.

To prepare a spike, add 0.05 mL of Spike 1, Spike 2, Spike 3 and Spike 4 to 9.80 mL of sample. Mix thoroughly and analyze.

7.2.2.2 The three spike solutions for CLP samples are:

CLP-1 Made by ULTRA SCIENTIFIC

ANALYTE	TRUE VALUE (4.1)	TRUE VALUE (5.2)
Aluminum	2000 µg/mL	2000 µg/mL
Barium	2000 µg/mL	2000 µg/mL
Beryllium	50.0 µg/mL	50.0 µg/mL
Chromium	200.0 µg/mL	200.0 µg/mL
Cobalt	500.0 µg/mL	500.0 µg/mL
Copper	250.0 µg/mL	250.0 µg/mL
Iron	1000 µg/mL	1000 µg/mL
Manganese	500.0 µg/mL	500.0 µg/mL
Nickel	500.0 µg/mL	500.0 µg/mL
Silver	50.0 µg/mL	50.0 µg/mL
Vanadium	500.0 µg/mL	500.0 µg/mL
Zinc	500.0 µg/mL	500.0 µg/mL

CLP-2 Made by ULTRA SCIENTIFIC

ANALYTE	TRUE VALUE (4.0)	TRUE VALUE (5.0)
Antimony	500.0 µg/mL	100.0 µg/mL

CLP-3 Made by ULTRA SCIENTIFIC

ANALYTE	TRUE VALUE (4.0)	TRUE VALUE (5.0)
Arsenic	2000 µg/mL	40 µg/mL
Cadmium	50 µg/mL	50 µg/mL
Thallium	2000 µg/mL	50 µg/mL
Selenium	2000 µg/mL	10 µg/mL
Lead	500 µg/mL	20 µg/mL

Refer to sample preparation SOPs for the preparation of matrix spikes for CLP samples.

The spike recovery criteria may be found in table 22.15.

8.0 SAFETY

8.1 Many of the metallic elements analyzed for in this method are known to be hazardous to health. Care should be taken in the handling and disposing of all standards and samples. See section 20.0 for procedures on the disposal of standard and sample waste.

8.2 The matrix of all ICP standards and samples is 6% HNO_3, 5% HCl by volume. Gloves should be used when handling all standards and samples. Safety glasses must be worn at all times. Extra care

should be taken when dispensing concentrated acids. Concentrated acids should be dispensed only in the fume hood.

8.3 The plasma emits strong UV light and is harmful to vision. **AVOID LOOKING DIRECTLY AT THE PLASMA.**

8.4 The RF generator produces strong radio frequency waves, most of which are unshielded. People with pacemakers should not go near the instrument while in operation.

8.5 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.			

TITLE: METHOD 6010B/ 200.7/CLP USING THE THERMO JARRELL ASH 61E TRACE

SUPERCEDES: Revision 15

9.0 EQUIPMENT AND SUPPLIES

9.1 Thermo Jarrell Ash ICAP 61E Trace Analyzer is equipped with an autosampler, computer, printer, and source of argon. There are two Trace Analyzers at STL Buffalo. They are designated as Trace #1 and Trace #2.

9.2 Volumetric flasks in various sizes from 50 mL to 1000 mL. These are used for standard preparation and sample dilution.

9.3 Eppendorfs in various sizes. These are used for standard and sample preparation. The Eppendorfs are verified using an analytical balance on a weekly basis. They are calibrated on a quarterly basis along with the repipettors. A logbook of the calibration results is kept as a record. At least one Eppendorf in each of the following ranges are used:

10 µL → 100 µL
50 µL → 200 µL
50 µL → 250 µL
100 µL → 1000 µL
500 µL → 2500 µL
2000 µL → 10000 µL

9.4 Disposable polypropylene pipette tips for the Eppendorfs in various sizes.

9.5 Disposable 17x100 mm polypropylene culture tubes used in the autosampler as the sample containers.

9.6 28 mL Nalgene brand disposable sample vials used to hold standards and quality control samples in the autosampler.

9.7 Spare parts for the Trace:

- nebulizers
- torches
- spray chambers
- platens

9.8 Red/Red/Red pump tubing (TJA #13017701)

9.9 Orange/Green/Orange pump tubing

9.10 Orange/Orange/Orange pump tubing

9.11 Internal Standard tubing mixing kit

9.12 Autosampler sample probes (TJA #4097-30)

9.13 500 mL graduated cylinder

9.14 Para-ilm

9.15 Repipettors for acids and dilutions.

10.0 REAGENTS AND STANDARDS

10.1 All standards and samples are prepared such that the matrices are matched.

10.1.1 All standards and samples are prepared using 18 $M\Omega cm^{-1}$ ELGA water. The metals lab has an ELGA water system attached to a deionized water system. The ELGA water is monitored daily by the Wet Chemistry department and maintenance is performed as needed.

10.1.2 All standards are prepared with volumetric flasks, and calibrated Eppendorfs.

10.1.3 All standards and samples are prepared with Trace Metals Grade Nitric and Hydrochloric Acids.

10.1.4 All the standards and samples are prepared in the same matrix containing 6%HNO_3 and 5% HCl by volume.

10.1.5 Standards are prepared as needed, every 3 to 7 days for Calibration Standards.

10.2 Table 22.7 lists all the reagents and stock solutions that are purchased as starting materials. All stock solutions are certified and the certifications are kept for a record. All stock solutions are logged into an incoming logbook that is stored in the lab.

The multi-element calibration standards and other solutions required (except the quality control sample used for ICVs and CCVs) are prepared from stock solutions purchased from ULTRA SCIENTIFIC. The quality control sample used for ICVs and CCVs are prepared from stock solutions purchased from HIGH PURITY. The use of two vendors ensures a second source verification of standards. The ionization buffer $LiNO_3$ is purchased as a solid from MALLINCKRODT.

10.3 There are two types of solutions that are prepared from the purchased stock standards. They are prepared stock solutions and the working standards. Prepared stock solutions are used as intermediate standards for preparing the working standards. Prepared stock solutions are recorded in the standards logbook. They expire in six months or when the original starting stock standards expire, whichever is first. Prepared stock solutions are labeled with their name, the preparation date, the expiration date, and the initials of the analyst preparing the solution.

The working standards are prepared from the purchased stock standards and the prepared stock solutions. The working standards are also recorded in the standards logbook.

Solutions prepared by the analyst are recorded in the standards logbook. The following information is recorded:

- Name or concentration of the solution
- Date prepared
- Initials of analyst preparing the solution
- The manufacturer of the starting stock solution
- The lot number of the starting stock solution
- The name or concentration of the starting stock solution
- The volume of the starting stock solution used
- The final volume of the solution being prepared
- The source of the HNO_3
- The source of the HCl

10.4 Blank solutions contain 6% HNO_3 and 5% HCl in ELGA water. The blank solution is used for the following:

- Calibration blank
- ICB
- CCBs
- Sample dilutions

10.4.1 The Blank Solution is prepared by adding 1200 mL concentrated HNO_3 and 1000 mL concentrated HCl to a 20 liter plastic carboy half filled with ELGA water. Bring up to volume with ELGA water. This procedure may be scaled up or down. Use a 500 mL graduated cylinder to add the acids. Be extremely careful when handling conc. acids in these amounts (work in the fume hood wearing lab coat, gloves and safety glasses).

10.4.2 The instrument rinse is prepared in a 20-liter plastic carboy. The rinse blank is prepared by adding 1200 mL of concentrated HNO_3 to the carboy half filled with ELGA water. Fill the carboy to the 20-liter mark with ELGA water.

10.5 The following stock solutions are prepared:

- Spike 3, containing 10 µg/mL Ag and Spike 4, containing 40 µg/mL Sn
- 100 g/L $LiNO_3$ solution

Begin with ULTRA SCIENTIFIC stock standards.

10.5.1 Spike 3 is prepared by adding 1.0 mL of 1,000 µg/mL Ag to a 100 mL volumetric flask half filled with Blank Solution. Bring up the final volume with Blank Solution. This spike is used for the postspike.

10.5.2 Spike 4 is prepared by adding 4.0 ml of 1,000 ug/ml Sn to a 100 ml volumetric flask filled with blank solution. Bring up the final volume with blank solution. This spike is used for the post-spike.

10.5.3 The 200.0 g/L $LiNO_3$ (0.1%) solution is prepared by weighing out 200.0 g of $LiNO_3$ and dissolving in a 1000 mL volumetric flask half filled with Blank Solution. Dilute to one liter with Blank Solution. This is the buffer for the internal standard.

10.6 The following calibration standards and solutions are to be prepared in the laboratory:

- Std. 1
- Std. 2
- Std. 3
- ICSA
- ICSAB
- CCV
- Internal Standard
- CRI
- NAKCAMG100
- NAKCAMG400

These standards and solutions are prepared from ULTRA SCIENTIFIC stock standards and prepared stock solutions.

10.6.1 Std. 1 is prepared by adding 20 mL of Std. 3 (Section 10.6.3) to a 200 mL volumetric flask half filled with Blank Solution. Bring up to final volume with Blank Solutions. See Table 22.8 for concentrations of elements in Std. 1.

10.6.2 Std. 2 is prepared by adding 100 mL of Std. 3 (Section 10.6.3) a 200 mL volumetric flask half filled with Blank Solution. Bring up to final volume with Blank Solution. See Table 22.8 for concentrations of elements in Std. 2.

10.6.3 Std. 3 is prepared by adding 5.0 mL ICUS-575, 5.0 mL ICUS-576, 0.5 mL 1000 µg/mL Sn and 0.5 mL 1000 µg/mL Ag to a 500 mL volumetric flask half filled with Blank Solution. Bring up to final volume with Blank Solution. See Table 22.8 for concentrations of elements in Std. 3.

10.6.4 The ICSA is prepared by adding 50.0 mL of ICSA stock solution (ICM-441) to a 500 mL volumetric flask half filled with Blank Solution. Bring up to final volume with Blank Solution. See Table 22.9 for concentrations of elements in the ICSA.

10.6.5 The ICSAB is prepared by adding 50.0 mL of stock solution (ICUS-919) to a 500 mL volumetric flask half filled with Blank Solution. Bring up to final volume with Blank Solution. See Table 22.9 for concentrations of elements in the ICSAB.

10.6.6 The NAKCAMG100 is prepared by adding 1.0 mL of each 10,000ppm stock solutions Na (ICP-111-5), K (ICP-119-5), Ca (ICP-120-5), Mg (ICP-112) to a 100 mL volumetric flask half filled with Blank Solution. See Table 22.9 for concentrations of elements in the NAKCAMG100.

10.6.7 The NAKCAMG400 is prepared by adding 4.0 mL of each 10,000ppm stock solutions Na (ICP-111-5), K (ICP-119-5), Ca (ICP-120-5), Mg (ICP-112) to a 100 mL volumetric flask half filled with Blank Solution. See Table 22.9 for concentrations of elements in the NAKCAMG400.

10.7. The following standards are prepared from HIGH PURITY

10.7.1. A quality control sample (called the CCV) is prepared from HIGH PURITY stock standards. See Table 22.10 for true values for the CCV. The CCV is prepared by adding 5.0 mL CAL STD. #2 –R Solution A, 5.0 mL CAL STD.#2-R Solution B, 0.5 mL of 1000 µg/mL Ag and 0.5 mL of 1000 µg/mL Sn to a 1000 mL volumetric flask half filled with Blank Solution. Bring up to volume with Blank Solution. Final concentrations can be found in Table 22.10.

10.7.2. The Initial Calibration Verification (ICV) is prepared using the same stock as the CCV. It is prepared by adding 75.0ml of the CCV to a 100ml volumetric flask and bringing it up to volume with Blank Solution. Final concentrations can be found in Table 22.10.

10.8. Yttrium – 5 mg/l: The internal standard/profile is prepared by adding 5 mL of 1,000 µg/mL Y stock solution to a 1000 mL volumetric flask half filled with Blank Solution. Add 50.0 mL of the 200 g/L $LiNO_3$ solution. Bring up to final volume with Blank Solution.

10.9. Low Level Verification Standards:

10.9.1. The CRI is prepared by adding 50 ml ICUS-1241 to a 500 mL volumetric flask half filled with Blank Solution. Bring up to final volume with Blank Solution.

11.0 SAMPLE COLLECTION, PRESERVATION, SHIPMENT AND STORAGE

11.1 The maximum holding time for metals samples is 180 days from sample collection. Aqueous samples are preserved with nitric acid to a pH<2. Soil samples do not require additional preservation.

11.2 Soil and total water samples are prepared by a digestion procedure in the digestion lab. The digestates are brought to the instrumental lab by the Digestion analyst. The digestates are stored on a shelf in the instrumental lab. When analysis on the digestates are complete, the digestates are placed in a main sample storage area. The main storage area is located near the digestion lab. The main storage area is used to store the original total samples, digestates, and soluble samples. The main storage area is kept locked when unattended. The digestates are kept for 6 months before they are finally disposed of. For CLP work the digestates are stored for 365 days after delivery of the data

package. For all CLP, AFCEE and USACE samples, samples must be refrigerated at 4 degrees C from the time of collection until digestion. CLP samples may be disposed of after 60 days in a manner that complies with all applicable regulations.

11.3 Soluble samples are stored in the main sample storage area with the digestates and the original total samples. All samples taken from the storage area must be logged out in the sample custody logbook that is kept in the digestion lab. Samples are logged back in when complete. The main storage area is kept locked when unattended. The key to the storage area can be obtained from the sample control personnel and returned to them when finished.

11.3.1 **Controlled Access Storage**: CLP, AFCEE and USACE samples require controlled access storage with strict Chain-of –Custody procedures. Digestates for these samples are obtained from and returned to the cooler custodian. The custodian maintains both the original samples and the digestates in the locked controlled access storage cooler.

11.3.1.1 The original samples are kept for 60 days following delivery of the final report package.

11.3.1.2 Digestates are maintained for 365 days for CLP samples; 60 days for AFCEE and USACE samples.

11.4 Most total and soluble samples have already been preserved by sample control when they were received or in the field when the samples were taken. Preservation is required by the laboratory analyst in cases that samples have not been filtered and preserved. A comment, listing lot numbers of the acid and filter used, is placed in the particular job affected

12.0 QUALITY CONTROL:

Overview: This section provides the guidelines of the quality control that are used to determine if data are useable or not. Depending on the clients' requests and each specific protocol, some QC samples may not be prepared and/or analyzed to each job. Any observed deviations must be documented for future references. If the analyst cannot make a decision about the usability of data, the supervisor must be consulted and the resolution must be documented. If data are unusable, the samples must be redigested and/or reanalyzed depending on the situation. For details on how an actual analytical run is laid out see Section 14.0 - Procedures. For details on any of the calculations which are required in this section see Section 15.0 - Calculations.

12.1 Standards - To insure quality data, all working standards are prepared from high quality certified stock standards. All prepared standards are logged into the standards logbook to insure traceability. Stock solutions are purchased as often as necessary to insure a fresh source.

12.2 Instrument Calibration - The instrument is calibrated daily at the beginning of each analytical run. All relevant information is printed for reference. A blank and 3 levels of standards are used to calibrate each element. A linear plot of each element is produced. The correlation coefficient (which is printed right after the calibration standards have been analyzed) for each element must be 0.995 or greater. If

the correlation is less than 0.995 for a particular element, then the data for that element may not be used from that particular analytical run.

12.3 ICL - The ICL is the highest calibration standard that is analyzed after the instrument is calibrated. Results for the ICL must agree within ±5% of the true value of each element. If the ICL is outside the control limits for an element, then the instrument must be re-calibrated or that element cannot be used from that analytical run. See Table 22.8 for the true values of the highest standard (STD. 3). The ICL is called STD 3 Ver on the analytical run.

12.4 ICV - The ICV is prepared from a separate source other than the calibration standards. It is analyzed after the ICL. See Table 22.15 for ICV control limit criteria. If the ICV is outside the control limits for an element, then the instrument must be recalibrated or that element cannot be used from that analytical run. The measured values must be within +/- 10% of the true value for CLP and method 6010B. The measured values must be within +/- 5% of the true value for method 200.7. See Table 22.10 for the true values of the ICV.

12.5 CCV's - The CCV is prepared from a separate source other than the calibration standards. It is analyzed after every ten samples and at the end of the analytical run. See Table 22.15 for CCV control limit criteria. If the CCV is outside the control limits for an element, the ten samples before and after that CCV should be reanalyzed for that element. See Table 22.10 for the true values of the CCV.

12.6 ICB and CCB's - After analyzing the ICV, analyze an ICB. After analyzing each CCV, analyze a CCB. See Table 22.15 for blank control criteria.

12.7 ICSA – See Table 22.15 for recovery criteria for the ICSA standard. If the ICSA is outside the control limit for an element, that element cannot be used from that analytical run. See Table 22.9 for the true values of the ICSA.

12.8 ICSAB- After analyzing the ICSA, analyze an ICSAB. See Table 22.15 for recovery criteria. If the ICSAB is outside the control limits for an element, that element cannot be used from that analytical run. See Table 22.9 for the true values of the ICSAB.

12.9 Method Blank – For each batch of digestions, one method blank is digested for every 20 samples. Table 22.15 summarizes method blank compliance criteria.

12.10. LFB - For each batch of digestions of water samples, an LFB (Lab Fortified Blank) is prepared. Refer to Table 22.15 for compliance criteria. If the LFB for an element is outside the control limits, then all the samples for that element must be re-digested. See Table 22.6 for the concentrations of each analyte.

12.11. LCS - For each batch of digestions of soil samples, a LCS (Laboratory Control Sample) is prepared. The certified values are different from lot to lot, and the certified values can be found in the binder of

the Certificate of Analysis. The acceptance limits are provided by the supplier. If the LCS for an element is outside the control limits, all the samples for that element must be re-digested.

12.11.1. Instead of this LCS, AFCEE and USACE require a water aliquot to be spiked in a similar fashion to the LFB in 12.10. See Table 22.6 for the concentration of each analyte.

12.12 Matrix Duplicate (MD) - For CLP + per client request, one matrix duplicate is performed per digestion batch. See table 22.15 for duplicate compliance criteria. If the RPD is outside the control limits for an element, the data should be reviewed to determine cause. If lab error suspected, reanalyze or redigest. Generally MD is performed only for CLP digestions.

12.13 Matrix Spike (MS) and Matrix Spike Duplicate (SD) - For each batch of samples, two matrix spikes are performed (one is MS and the other is SD). See Table 22.6 for the concentrations of the matrix spikes for each element. See table 22.15 for criteria for spike recovery and precision. If the RPD is outside the control limits for an element, the data should be reviewed to determine cause. If lab error suspected, reanalyze or redigest. If the recovery for an element is outside the control limits, matrix effect is suspected for digestion and/or the determination. Generally SD is performed for SW-8463 and CFR protocols.

12.14 Post Spike - A post digestion spike is performed based on client requirements. It is performed on the base sample that has an MS associated with it. The spike recovery must agree within the limits specified in Table 22.15. If the post spike for an element is outside the control limits, the matrix effect is suspected in the ICP determination.

12.15 Serial Dilution - A serial dilution is performed on the base sample in the batch of 20 that has a matrix spike. If the serial dilution is outside the control limit, the matrix effect is suspected in the ICP determination. The dilution is a 1:5 (one part of the sample to four parts of the blank solution).

12.16 IEC, IDL, AND LINEAR RANGE DETERMINATION

12.16.1 IEC's - The IEC's (interelement correction factors) are determined by first analyzing the following solutions:

- 100 µg/mL Cr
- 100 µg/mL Mn
- 100 µg/mL V
- 1000 µg/mL Fe
- 1000 µg/mL Al
- 1000 µg/mL Ca
- 1000 µg/mL Mg
- 100 µg/mL Tl
- 100 µg/mL Ti

- 100 µg/mL Mo
- 100 µg/mL Co
- 100 µg/mL Cu

Each of the above solutions is prepared from individual 10,000 ug/ml (Ultra Scientific) stock standards at the above concentrations.

Next check to see whether there are any false positive or negative readings for the other elements in each of the solutions. If there are any, then an IEC calculation is necessary.

To calculate the IEC factor, divide the false reading for an element by the actual reading of the interfering element.

Sample Calculation:
The following results are obtained after running a 200.0 µg/mL Fe solution:

Fe = 208.0 µg/mL
Cd = 1.21 µg/mL

The IEC factor for Cadmium would be:

$$\frac{1.21}{208.0} = 0.00582$$

12.16.2 IDLs – For non-CLP protocols, the IDL is determined by analyzing a blank solution 7 times. Calculate the standard deviation of the 7 readings for each element. The IDL is 3 times the standard deviation. For CLP protocol, the IDL is determined by multiplying 3, the average of the standard deviations obtained on three nonconsecutive days from the analysis of a standard solution at a concentration 3x-5x the estimated IDLs, with seven consecutive measurements per day.

12.16.3 Linear Ranges - The linear range is the highest standard the instrument can read which is ±5% of the known value. Analyze a series of standards for each element. The highest that is within ±5% establishes the linear range.

12.17 Additional AFCEE and USACE specific quality control requirements are detailed in Tables 22.18 and 22.19.

12.18 The internal standard counts are monitored for every analysis. The internal standard counts must fall between 50 and 150 percent of the counts of the internal standard in the initial calibration blank. If the internal standard fails to fall between 50 and 150 percent of the initial blank the data from that particular sample may not be used from that analytical run. Recalibrate and reanalyze the sample.

TITLE: METHOD 6010B/ 200.7/CLP USING THE THERMO JARRELL ASH 61E TRACE

SUPERCEDES: Revision 15

13.0 CALIBRATION AND STANDARDIZATION

13.1 The daily stardization of the 61E Trace analyzer is done automatically at the beginning of each run and approximately every 60 samples. The calibration standards are programmed into the autosampler table. The programming procedure and the standards used are outlined in section 14.

13.2 A Calibration Curvefit is used to calibrate the Na, K, Ca, and Mg wavelengths. This procedure uses an extended calibration with more standards to produce a more linear and precise response over a greater range. The calibration is then fitted to a curve, which can be resloped during a instrument standardization using the low and high standards of the daily calibration. A Calibration Curvefit is preformed quarterly or if significant instrument changes occur. The procedures below explain the steps to run a calibration curvefit

13.2.1 Calibration Curvefit Sample Table Setup and Modification

The following procedure is used to select the elements to be standardized and the concentration of the standards used during a curvefit, or to modify an existing standards table.

- Starting from the main menu, go to “Development”.
- Go to “Calibration-Standards”.
- Type table name to be create or modified.
- To add a standard press <F1>.
- Add elements to the standard by pressing <F1>.
- To add an element on the periodic table highlight the element to be added and press <F1>. To remove an existing element press <F2>.
- Press <F9> when done.
- Type in the concentration of the each element in the standard.
- Press <F9> when done.
- To modify an existing standard in the table, highlight it, and press <F1>. This will allow you to change the elements and concentrations in the standard by following the instructions above.
- To delete a standard press <F5>.

NOTE: The blank and highest standard of the daily curve must be represented exactly in curvfit table including the same concentrations. If this is not done the fitted curve will not automatically be resloped during daily calibration.

13.2.2 Analysis of the Calibration Curvefit Standards

This procedure is used when analyzing a curve to be fitted.

- Starting from the main menu go to “Development”.
- Go to “Calibration –Analysis”

- Enter the standards table name and designate a calibration data file using a one digit run number, three letter month abbreviation, two number day date, and the last two digits of the year (example: 1MAR0705).
- Press <F9> Done to continue.
- To analyze a standard, begin by manually aspirating the standard, then press<F1>Run Standard, and skip comments section by pressing <F1>Run.
- When analysis is complete press <F9>Done.
- Repeat above two steps for each standard in the table.
- When all standards have been analyzed press <F9>Done.

13.2.3 Fitting the Curve

These steps are used to fit the analyzed standards or refit an existing data set.

- Starting at the main menu, go to “Development”.
- Go to “Calibration-Curvefit”.
- Select or enter the standards table and data file used during analysis.
- Press <F9>Done to continue.
- Select the element/line to be fitted.
- Press <F1>Fit Element.
- Press <F1>Fit Element.
- Under fit type select ”Full Fit”.
- Under Weight select “Concentration Zero Factor :100.000”.
- Press <F9>Done Calc.
- Check that the Correlation Coefficient > 0.995.
- Press <F9>Done.
- Repeat for the rest of the elements to be fitted.

13.2.4 Integrating the Calibration Curvefit into the Instrument Method

The following procedure is used to integrate the Calibration Curvefit into the chosen method. This will allow the Calibration Curvfit to be resloped with the daily curve using the blank and high standard.

- Starting on the main menu, go to “Development”.
- Go to “Methods”.
- Type the method name.
- Press <F5>Element Info
- Highlight the fitted element. The drop down menu below the line marked “Stdzn Method” should read “2-pt Calib” and the high and low standards names should match both the daily curves high and low standards as well as the two matching standards in the curve fit table. If these are correct check the remaining curvefit elements.
- If any of the criteria are incorrect press <PgDwn> twice.
- Highlight the incorrect item and type correction or press spacebar for drop down.

- Press <F9>Done to save.
- Repeat for the remaining curvefit elements.
- Press <F9>Done/Keep to save changes.
- Next from "Modifying Existing Method" page press <F3>Method Info.
- At the bottom left of the page enter the "Calibration Data File" and the "Calibration Stds" table name.
- Press <F9>Done/Keep.

These steps should have completed Calibration Curvefit standardization and integration into the method. Run a daily curve to confirm.

14.0 PROCEDURES

14.1 The following is a daily checklist for the operation of 61E Trace analyzer. This is only a summary of the basic steps. Each step is followed by a reference for further information.

1. Empty the main drain waste and the autosampler drain waste, if necessary. See Section 14.2.2.1
2. Fill the autosampler rinse, if necessary. See Section 14.2.2.2
3. Refill the internal standard, if necessary. See Section 14.2.2.3.
4. Inspect the pump tubing daily. Either change tubing or change side to side. See section 14.2.2.7.
5. Change the torch, if necessary. The torch should be changed when necessary. See Section 14.2.2.4. The procedure for cleaning the torch is described in Section 14.2.2.5.
6. Check the argon pressure, if necessary. See Section 14.2.2.6.
7. Fill the Argon Saturator if necessary. See Section 14.2.2.12
8. Ignite the plasma. See Section 14.3.
9. Start the peristaltic pump. See Section 14.3.2.
10. Prepare the standards and QC samples. These must be re-poured daily. See Section 10.0.
11. Type the run into an autosampler table and print the table. See section 14.5.
12. Change the data file name for each run. See Section 14.6.
13. Place the standards and QC samples in the autosampler. See Section 14.7.
14. Run a profile before each run. See Section 14.8.
15. Prepare the samples and place them into the autosampler. See Section 14.9.
16. Start the analysis. See Section 14.10.
17. When the analysis is complete, print the data in a condensed format. See Section 14.11.
18. Turn off the plasma unless you want to perform another analysis. See Section 14.12.
19. Empty the samples and standards into a waste receptacle.
20. Perform a validation of the data. See Section 14.13.

14.2 Instrument Layout and Maintenance

This section contains information on the layout of the instrument and any maintenance that the analyst might perform. Any maintenance beyond what is covered here, will be performed by Thermo Jarrell Ash service personnel.

14.2.1 There are two 61E Trace Analyzers at STL Buffalo. They are both set up in a similar manner.

14.2.1.1 See Section 22.11 for a diagram of the main instrument layout.

14.2.1.2 See Section 22.12 for a diagram of the tubing layout.

14.2.1.3 See Section 22.13 for a diagram of the torch/spray chamber/nebulizer assembly.

14.2.1.4 See Section 22.14 for a diagram showing the layout of the autosampler. Rack #1 holds the 28 mL disposable polypropylene sample vials and are used for the calibration standards and quality control standards.

14.2.1.5 Racks #2, #3, #4, and #5 hold the disposable polypropylene culture tubes and are used for the samples.

14.2.2 The following is a list of maintenance topics and problems that need troubleshooting:

- Emptying the waste (See Section 14.2.2.1).
- Filling the autosampler rinse (See Section 14.2.2.2).
- Filling the Internal Standard (See Section 14.2.2.3).
- Filling Argon Saturator (See Section 14.2.2.12).
- Changing the torch (See Section 14.2.2.4).
- Cleaning the torch (See Section 14.2.2.5).
- Check the argon pressure (See Section 14.2.2.6).
- Changing the pump tubing (See Section 14.2.2.7).
- The cooling water level is low (See Section 14.2.2.8).
- The red warning light on the power supply comes on (See Section 14.2.2.9).

- Droplets form on the wall on the inside of the spray chamber (See Section 14.2.2.10).

- The "By Pass" light comes on (See Section 14.2.2.11).

14.2.2.1 Emptying the Waste

14.2.2.1.1 There is one waste container for the 61E Trace Analyzer. One waste line comes from the excess sample draining from the spray chamber (this is called main instrument drain). The other waste line comes from the autosampler rinse station. Both lines run into a central container and are acid wastes and must be disposed of properly. The analyst is responsible for disposal of the acid wastes. Make sure that the 5 gal. waste container is labeled for nitric acid (AN) waste.

14.2.2.1.2 The following steps are used when emptying the instrument waste container:

- Unscrew the instrument waste cap.
- Replace full carboy container with an empty one.
- Replace the cap being sure not to tangle the tubing.

CAUTION: Always wear gloves and safety glasses when handling wastes.

14.2.2.2 Filling the Autosampler Rinse

- Pull the tubing from the 20 liter rinse container.
- Refill the rinse container with reagent blank prepared according to Section 10.4.2.
- Replace the tubing into the rinse container.
- Place a piece of parafilm on the top of the rinse container to keep dust out.

14.2.2.3 Filling the Internal Standard

- Pull the tubing out of the internal standard flask.
- Fill the flask with the internal standard prepared according to Section 10.8.
- Place the tubing back into the internal standard flask.
- Place a piece of parafilm on the top of the flask to keep dust out.

14.2.2.4 Changing the Torch - Refer to the diagram in Section 22.13 to aid in changing the torch. Use the following steps:

- Turn the plasma off.
- Pull the spray chamber out.
- Remove the two screws holding the white cover in place.
- Remove the white cover.
- Pull out the gray collar. Be careful not to bump the quartz end.
- Remove the two argon lines from the gray collar by unscrewing the connectors.
- Remove the black O ring from the end of the gray collar.
- Tip the gray collar until the torch slides out.
- Place a clean torch in the instrument and follow steps 1 through 8 in reverse order.
- Ignite the plasma

14.2.2.5 Cleaning the Torch – Soak the torch overnight in a 1:1 HNO_3 solution (equal parts of HNO_3 acid and ELGA water). Rinse the torch with ELGA water and allow to dry.

Prepare the 1:1 HNO_3 solution (equal parts of HNO_3 acid and ELGA water) by adding 100 mL of ELGA water using a graduated cylinder to a 400 mL beaker. Carefully add 100 mL of conc. HNO_3 acid. Be very careful when working with concentrated acids in this quantity. Work in the fume hood and wear gloves, lab coat, and safety glasses.

14.2.2.6 Checking the Argon Pressure

- The argon regulator is installed on the wall next to the door. Check the pressure to make sure it is 80 psi.

14.2.2.7 Changing the Pump Tubing

There are three types of pump tubing used (use the diagram in Section 22.12 for reference):

- orange/orange/orange - used for the samples.
- orange/green/orange - used for the internal Standard
- red/red/red - used for the autosampler rinse and main instrument waste.

To replace the pump tubing, first pull the tubing from each end of the pump tubing. Insert the tubing into the ends of the new pump tubing.

14.2.2.8 The Cooling Water Level is Low

If the cooling system light comes on, the water level is low. Use the following steps to add water to the cooling system:

- Remove the cover from the top of the cooling system.
- Remove the cap inside.
- Add DI water.
- Replace the inside cap.
- Replace the top cover.

14.2.2.9 The Red Warning light on the Power Supply Comes On

This indicates that the filter on the back of the power supply is dirty. Use the following steps to clean the filter.

- Remove the filter from the power supply.
- Rinse the filter with water.
- Pat the filter dry with a paper towel.
- Allow the filter to dry.
- Replace the filter on the power supply.

14.2.2.10 Droplets Form on the Wall on the Inside of the Spray Chamber

Aspirate a solution of 1:1 Nitric acid (equal parts of HNO_3 acid and ELGA water). Do not aspirate for more than 10 seconds at a time. Prepare the 50% Nitric acid solution by adding 50 mL of conc. Nitric acid to ~50 mL of ELGA water.

WARNING!: Conc. Nitric acid is extremely hazardous! Work in the fume hood and wear gloves, lab coat, and safety glasses. Pull the hood sash down to protect your face.

14.2.2.11 The "By Pass" Light Comes On

The "By Pass" light comes on if the instrument loses the vacuum due to a power outage or pump failure. If the pump fails, it needs to be fixed first. Use the following steps to regain the vacuum:

- Go to the back of the instrument and turn the switch labeled HV (high voltage) to the off position.
- Dial the Red needle on the vacuum gauge all the way to the right.
- Push the start button.

- When the black needle reads less than 20, dial the Red needle to 20.
- Turn the HV (high voltage) to on.
- Ignite the plasma.

14.2.2.12 Filling the Argon Saturator
If the water level is below "min" line, refilling is necessary with the following steps:

- Lower the neb. pressure to 15 PSI on the plasma screen.
- Turn off neb. pressure.
- Unscrew Argon Saturator cap.
- Fill with Elga Water to the red "Max" line.
- Replace cap
- Turn neb. pressure on.

14.3 Instrument Start-Up

14.3.1 The instrument should always be left on. This is required to maintain the vacuum. If it ever becomes necessary to turn off the instrument, make sure that the high voltage (HV) switch on the back of the instrument is turned to off. If the instrument is not being used on a given day, it is only necessary to turn the computer, monitor, and printer off.

If the computer has been turned off, and if only the C:\ prompt comes up when it is turned on, then type STNRUN and press ⟨Enter⟩ to get into the software. Once in the software the plasma is ready to ignite.

14.3.2 The following steps are used to ignite the plasma and start the peristaltic pump.

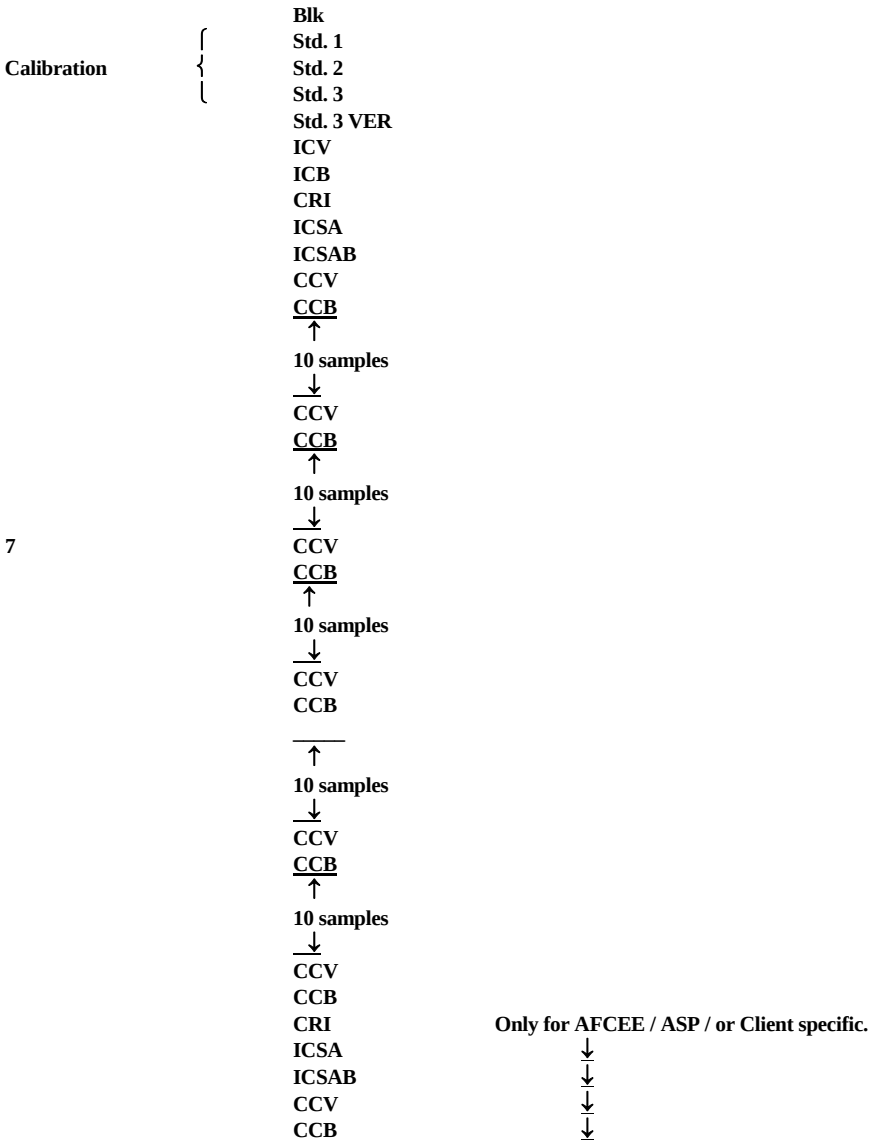
- Starting from the main menu. Go to "Setup".
- Go to "Plasma Control Panel".
- Press ⟨Enter⟩.
- Press ⟨F1⟩ for plasma on.
- Press ⟨F9⟩ to continue.
- If the plasma does not ignite, then press ⟨F1⟩ and try again. Make sure there is sufficient argon first and that the valve on the argon tank is open.
- When the plasma ignites, press ⟨Enter⟩ for O.K.
- Press ⟨ESC⟩ to go back to the main menu.
- Go to "Operation".
- Press ⟨Enter⟩.
- Type in the name of the method you want to run.
- Press ⟨Enter⟩. This will start the peristaltic pump. The instrument is now warming up. Allow the instrument to warm up for 30 to 60 minutes.

TITLE: METHOD 6010B/ 200.7/CLP USING THE THERMO JARRELL ASH 61E TRACE

SUPERCEDES: Revision 15

14.4 Typing an Analytical Run

14.4.1 Each Non-CLP analytical run is typed in the following format:



Run a CCV and CCB after every 10 samples and at the end of the analytical run. Run the CRI, ICSA, and ICSAB at the beginning and end of the analytical run.

NOTE: To be compliant with all protocols and clients' particular requests, extensive QC samples are routinely prepared and run. However, not all these QC samples are required for a particular protocol. For example, the ending CRI, ICSA and ICSAB are not required by SW-864 and 40 CFR protocol.

Therefore, a particular run may not include ending CRI, ICSA and ICSAB if that procedure only involves standard SW-864 and 40 CFR protocols. This note is also applicable to CLP procedure.

14.4.2 Each CLP analytical batch is typed in the following format:

Calibration
- **Blk**
- **Std. 1**
- **Std. 2**
- **Std. 3**

Std. 3 VER
ICV
ICB
CRI
ICSA
ICSAB
CCV
CCB
↑
10 samples
↓
CCV
CCB
↑
7 samples
CRI
ICSA
ICSAB
↓
CCV
CCB
↑
10 samples
↓
CCV
CCB
↑
7samples
CRI
ICSA
ICSAB
↓
CCV
CCB
CRI
ICSA
ICSAB
CCV
CCB

14.5 Typing the Autosampler Table

Type the analytical run into the autosampler table according to the following steps:

- Starting from the main menu, go to "Exit".
- Go to "ASEDIT.EXE".
- Press ⟨Enter⟩.
- Type in the name of the autosampler table. Use one of the saved templates.
- Press ⟨Enter⟩.
- Press ⟨F1⟩ to edit set.
- Press ⟨F1⟩ to edit samples.
- Use the arrow keys to toggle down to the CCB.
- Holding the ⟨ALT⟩ key down, press:
  - ⟨F1⟩ - to add a sample,
  - ⟨F3⟩ - to add a QC,
  - ⟨F5⟩ - to add calibration standards, or
  - ⟨F7⟩ - to add a blank.
- Type in the ID's of the samples and QC as written in the analytical run log.
- Under the check table column type:
  - ICV - for each ICV,
  - CCV – for each CCV
  - FUCRI – for each CRI,
  - B - for each ICB and CCB,
  - ICSA - for each ICSA
  - ICSAB - for each ICSAB
- After all the samples have been typed in, note the position of the last sample.
- Press ⟨F9⟩ for Done/Keep.
- Press ⟨F5⟩ for modify set.
- Type in the last sample position (from step 12).
- Press ⟨F9⟩ for Done/Keep.
- Press ⟨F9⟩, again, for Done/Keep.
- Press ⟨F2⟩ to print the autosampler table.
- Press ⟨F9⟩ for Done/Keep.

14.6 Entering the Data File Name

Each analytical run requires its own specific data file name. Use the following procedure to enter the data file name prior to the start of the analytical run:

- Starting from the main instrument menu, go to "Development".
- Go to "Methods".
- Press ⟨Enter⟩.
- Type in the method name of the method you are going to run.
- Press ⟨Enter⟩.

- Press ⟨Enter⟩ for method info.
- Use the arrow keys to toggle down to "Analysis Data File".
- Enter the new data file name.

Examples:

For Trace #1

1100500

Key: 1 = run# (1, 2, 3, etc.)
10 = month
05 = day
00= the last two digits of the year

For Trace #2

A100500

Key: A = run # (A,B,C, etc.)
10 = month
05 = day
00 = the last two digits of the year

- Press ⟨F9⟩ for Done/Keep.
- Press ⟨F9⟩, again, for Done/Keep.

14.7 Using the autosampler printout and the autosampler layout diagram (See Section 22.14), set-up the calibration standards and the quality control samples. Use the 28 mL disposable polypropylene sample vials to hold the calibration standards and the quality control samples.

14.8 Instrument Profile

Before each analytical run, a profile of the instrument must be performed. The profile is performed using the Internal Standard, which is a 5 PPM Yitrium solution according to the following steps:

- Starting from the main menu, go to "Operation".
- Go to "analysis".
- Press ⟨Enter⟩.
- Type in the name of the method you are going to run.
- Press ⟨Enter⟩.
- Press ⟨F5⟩ for profile.
- Press ⟨F3⟩ for automatic..
- Press ⟨F1⟩ to run scan.
- When the scan comes up, check the "Peak Position". The "Peak Position" should be between negative 0.05 and positive 0.05.
  If yes, then press ⟨F9⟩ for done/keep.

If no, then press ⟨F1⟩ to calc. SS. Check the vernier position on the computer. This should be the same as the vernier position on the instrument. Press ⟨Enter⟩. Dial in the new vernier position.

- Press ⟨F9⟩ for Done/Keep.

14.9 Preparing Samples for the Autosampler

14.9.1 Using the autosampler table printout and the autosampler layout diagram (See Section 22.14), set-up the samples in the autosampler. Use the disposable polypropylene culture tubes. Pour the samples into the culture tubes and place in the autosampler.

14.9.1.1 For 'Total Metals' and 'Soluble Metals', the samples consist of the digestates received from the metals preparation department.

14.9.2 To prepare post spikes, add the following amounts of each spike solution to 9.80 mL of sample:

50 µL - Spike 1 (Section 7.2.2.1)
50 µL - Spike 2 (Section 7.2.2.1)
50 µL – Spike 3 (Section 7.2.2.1)
50 µL – Spike 4 (Section 7.2.2.1)

Mix each post spike thoroughly and place in autosampler.

14.9.3 To prepare the 1:5 serial dilution, add 2.0 mL of sample to 8.0 mL of calibration blank.

14.10 Starting an Analysis

The determination parameters, such as integration time (13 seconds), rinsing time (60 seconds), number of replicates (2), standard concentrations are already entered, and in very rare cases these parameters may be changed. Refer to the instrument operation manual for modifying these values when necessary.

Once the autosampler table has been prepared, the samples, standard and quality control samples have been placed in the autosampler, and the instrument has been profiled, you are ready to begin the analysis. Use the following steps to begin the analysis.

- From the main menu, go to "Operation".
- Go to "Analysis".
- Press ⟨Enter⟩.
- Enter the method name of the method you wish to run.
- Press ⟨Enter⟩.
- Press ⟨F9⟩ for autosampler.
- Type in the autosampler table name under "Sample Name:".

- Press ⟨Enter⟩.
- If you want to have the instrument shut off automatically then press ⟨F7⟩ until the "Terminating Action:" reads "shutdown".
- Press ⟨F1⟩ to start the analysis.

14.11 Printing Data in Condensed Format

The trace instrument will print data during an analysis. A simplified, condensed version of the data may be prepared after the analysis is completed according to the following procedure:

- Starting from the main instrument menu, go to "IMS".
- Go to "Report Writer".
- Press ⟨Enter⟩.
- Enter the file name of the file you wish to print according to the following steps:
  - Press ⟨F6⟩ for a list of files.
  - Press ⟨F2⟩ to deselect all files.
  - Enter the number preceding the file you wish to print.
  - Press ⟨Enter⟩.
  - Press ⟨F9⟩ for Done/Keep.
- Use the arrow keys to go to "Method name".
- Enter the name of the method used to generate the data file.
- Press ⟨Enter⟩.
- Using the arrow keys to toggle back and forth, enter the start date and the end date of the data file you wish to print. You don't need to enter anything for the times.
- Use the arrow keys to go to "Sample type".
- Press the space bar to bring up a list of options.
- Use the arrow keys to toggle up to "All Types".
- Press ⟨Enter⟩.
- Press ⟨F9⟩ to continue.
- Use the arrow keys to toggle down to "Show Internal Standards".
- Change the No to Yes by pressing the "y" key.
- Use the arrow keys to toggle down to "Report format".
- Press the space bar to bring up a list of options.
- Use the arrow keys to toggle down to "vertical".
- Press ⟨Enter⟩.
- Press ⟨F9⟩ for Done/Go to start printing the data file.
- After printing the data file, press ⟨ESC⟩ to return to the main menu.

14.12 Plasma Shutoff

To turn the plasma off, use the following steps:

- From the main menu, go to "Setup".
- Go to "Plasma Control Panel".

- Press ⟨Enter⟩.
- Press ⟨F5⟩.
- Press ⟨Enter⟩.
- After about one minute, press ⟨ESC⟩.

14.13 Validation of the Data

When the analytical run is complete, the data must be checked for compliance with the method. Using Section 12.0 - Quality Control - check all the quality control samples (ICV, ICB, CCVs, CCBs, ICSA, ICSAB, Cal. Standards, ICL, and digested blank and LCS) for compliance. If a quality control sample falls outside the required limits for an element, then that element must be rerun on another analytical run.

Also check the spikes and serial dilution for any matrix effects that might require a diluted sample run.

15.0 CALCULATIONS

15.1 Refer to sections 12.1 through 12.15 and section 14.13 to determine if data are valid for each element. Any sample reading over the linear range must be diluted. Diluted samples must be run on required samples. Analyzing the sample and a series of spiked aliquots of the sample at different known concentrations performs an MSA.

15.2 The following calculations are illustrated:
- Relative Percent Difference (RPD) (See Section 15.2.1).
- Post spike calculation (See Section 15.2.2).
- Method of Standard Addition (MSA) calculation (See Section 15.2.3).

15.2.1 The formula for calculating the relative percent difference is:

$$\%\,RPD = \frac{D_1 - D_2}{(D_1 + D_2)/2}\ X\ 100$$

Where,

RPD = relative percent difference
D_1 = first sample value
D_2 = second sample value (replicate)

Sample calculation: A sample gave a reading of 2.51 µg/mL and the replicate reading was 2.39 µg/mL.

$$\%RPD = \frac{2(2.51-2.39)}{2.51+2.39} X100$$

$$RPD = 4.90\%.$$

15.2.2 The formula for calculating the post spike recovery is:

$$\% \ Recovery = \frac{S_2 - S_1}{SA} X \ 100$$

Where, S_2 = the post spiked sample reading
S_1 = the sample reading
SA = the spike added

Sample Calculations:
A sample gave a reading of 0.250 µg/mL. The sample was post spiked with 2.000 µg/mL and gave a reading of 2.289 µg/mL.

$$\%Recovery = \frac{2.289 - .250}{2.000} X \ 100$$

$$\% \ Recovery = \frac{2.039}{2.000} X \ 100$$

$$\% \ Recovery = 102.0\%$$

15.2.3 The formula for calculating the simplest version of MSA (single-addition method) is:

Where,

$$C_x = \frac{S_A V_S C_S}{(S_B - S_A) V_x}$$

S_B = the concentration of the spiked sample
S_A = the concentration of the unspiked sample
V_S = volume of spike solution added.
C_S = concentration of spike solution
V_x = volume of sample before adding spike
C_x = the unknown sample concentration

Sample calculation:

A sample gave a reading of 0.792 µg/mL. 50 µL of a 200 µg/mL spike solution was added to 10.0 mL of the sample. The spiked sample reading was 1.512 µg/mL.

$$C_x = \frac{(0.792)(0.05)(200.0)}{1.512 - 0.792)(10.0)}$$

$$C_x = \frac{7.92}{7.20}$$

$$C_x = 1.10ppm$$

16.0 METHOD PERFORMANCE

16.1 This SOP is applicable to digested sample matrices and soluble water samples.

16.2 Extensive quality control is used to insure compliance with method 6010B, 200.7 and CLP protocol.

16.3 Thorough documentation is employed to insure traceability of reagents and standards.

16.4. Approximate detection and reporting limits for Trace #1 and Trace #2 are found in Section 22.2 and 22.3.

16.5. Samples that read above the instrument's linear range must be diluted.

16.5.1. For USACE all samples above the high level calibration standard shall be diluted to within the calibration curve.

17.0 DATA ASSESSMENT AND ACCEPTANCE CRITERIA FOR QUALITY CONTROL MEASURES

17.1 Refer to Table 22.15 for acceptance criteria for QC measurements

18.0 CORRECTIVE ACTIONS FOR OUT-OF-CONTROL DATA

18.1. If calibration fails, i.e., correlation coefficient is lower than 0.995, ICV and/or ICB are out of control limit, ICSA and/or ICSAB are not recovered quantitatively, the analysis procedure must be terminated, the problems must be solved, and re-calibration must be started over.

18.2. If CCV and/or CCB fail, affected analytes in the 10 samples before and after that CCV/CCB pair must be reanalyzed.

18.3. If LFB or LCS fail, the whole digestion batch must be reanalyzed. If the reanalysis still fails, that whole digestion batch must be reprepared.

18.4. If the Method Blank fails for a analyte, but samples do not contain that analyte higher than the reporting limit or samples contain that analyte higher than 10x the Method Blank, the data is usable and reportable. Otherwise, the digestion batch must be reprepared for that analyte.

18.4.1. For USACE projects, target analytes in the Method Blank must be less than ½ the reporting limit.

18.5. If RPD for SD or MD is out of control limits, the data should be reviewed to determine cause. If redigestion and reanalysis are still out of limits, the sample might be inhomogeneous and the data should be reported with qualification. Refer to table 22.15 for RPD criteria.

18.6 If Post Spike or Serial Dilution fail, matrix effects in determination are suspected.

18.7 If LCS, Post Spike and Serial Dilution are within QC limits but MS fail, matrix interference can be assumed and corrective action is not required.

18.8 For CLP if the percent recovery of the CRI falls outside the control limits of 70-130% (50-150%for Sb, Pb, Tl), the CRI must be re-analyzed for the outlying analytes.

18.8.1 For USACE the control limits are +/- 20% of the true values. If the analysis is not compliant, recalibrate and re-analyze the CRI and all samples associated with it.

18.9 Additional AFCEE and USACE corrective action requirements are detailed in Tables 22.18 and 22.19.

18.10 If the internal standard counts for any analysis fail to fall between 50 and 150 percent of the counts of the internal standard in the initial calibration blank, recalibrate and reanalyze the affected sample/samples.

19.0 CONTINGENCIES FOR HANDLING OUT-OF-CONTROL OR UNACCEPTABLE DATA

19.1. Contingencies for unacceptable data will have to be evaluated on a client-by -client or even by a sample-by-sample basis by the supervisor, the lab manager or the QA manager. Corrective action will be prescribed accordingly.

19.2. In the event acceptable data can not be obtained, a Job Exception Form must be filed with the Project Manager and the client notified.

20.0 WASTE MANAGEMENT /POLLUTION PREVENTION

20.1. 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."

20.2. Waste Streams Produced by the Method: The following waste streams are produced when this method is carried out.

20.2.1. All acidic waste consisting of sample and rinse solution: Dispose of as HNO_3 waste in a "AN" waste container.

21.0 REFERENCES

21.1 Method 6010B, Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW-846, 3rd Edition, Final Update III, Revision 2, December 1996.

21.2 ICAP 61E Trace Analyzer Operator's Manual.

21.3 ILM04.1, USEPA Contract Laboratory Program. Statement of Work for Inorganic Analysis and Classical Chemistry Parameters.

21.4 ILM05.2, USEPA Contract Laboratory Program, Statement of Work for Inorganic Analysis and Classical Chemistry Parameters.

21.5 Method 200.7, "Determination of Metals and Trace Elements in Water and Wastes by Inductively Coupled Plasma-Atomic Emission Spectrometry", Revision 3.3, 40CFR Part 136, Appendix C, April 1991. (Approved for CWA compliance testing)

21.6 Method 200.7, "Determination of Metals and Trace Elements in Water and Wastes by Inductively Couple Plasma-Atomic Emission Spectrometry", Revision 4.4, US EPA / EMSL, May 1994. (Approved for SDWA compliance testing)

22.0 TABLES, DIAGRAMS, FLOWCHARTS AND VALIDATION DATA

22.1. Elements which are analyzed on ICAP 61E Trace analyzer

22.2. Approximate Water Detection Limits for the ICAP 61E Trace Analyzers

22.3. Approximate Soil Detection Limits for the ICAP 61E Trace Analyzers

22.4. Wavelengths and Background Points Used for Each Element on the ICAP 61E Trace Analyzer

22.5. Approximate Linear Dynamic Range of Each Element on the ICAP 61E Trace Analyzer

22.6. Concentration of each analyte for LFB, LCS, Post-digestion Spike, Non-CLP matrix spike and CLP matrix spike.

22.7. Reagents and Stock Solution which are Purchased as Starting Materials for Preparation of Trace Standards

22.8. Concentration of Calibration Standards

22.9. Values for ICSA and ICSAB

22.10. Values for CCV and ICV

22.11. Main Instrument Layout

22.12. Tubing Layout

22.13. Torch/Spray Chamber/Nebulizer Assembly

22.14. Autosampler Layout

22.15. Blank Page form Analytical Run Log

22.16. Method Summary

22.17. CLP Contract Required Detection Limits (CRDLs)

22.18. Concentration of Each Element in the CRI Standard Solutions

22.19. USACE Requirements

22.20. AFCEE 3.1 Requirements

22.21. Data Review Summary for Metals

22.22. Certificates of Analysis for Custom Blend Standards

23.0 CHANGES FROM PREVIOUS REVISION

23.1 Section 10.5.3 modified from 100g/L of LiNo3 to 200 g/L and 100g LiNo3 to read 200g LiNo3

23.2 Section 10.8 modified 100g/L to 200g/L

23.3 22.20 Replaced Review Summary form with current form.

23.4 Table 22.15: Revised frequency of CRI and ICSA/ICSAB 200.7 and SW-846 to beginning of analytical run only

22.1 Elements Which are Analyzed on the ICAP 61E Trace Analyzer:

Aluminum	Al	Magnesium	Mg
Arsenic	As	Manganese	Mn
Antimony	Sb	Molybdenum	Mo (not on Trace 1)
Barium	Ba	Nickel	Ni
Beryllium	Be	Sodium	Na
Boron	B	Potassium	K
Cadmium	Cd	Selenium	Se
Calcium	Ca	Silver	Ag
Chromium	Cr	Thallium	Tl
Cobalt	Co	Vanadium	V
Copper	Cu	Zinc	Zn
Iron	Fe	Tin	Sn
Lead	Pb	Titanium	Ti

TITLE: METHOD 6010B/ 200.7/CLP USING THE THERMO JARRELL ASH 61E TRACE

SUPERCEDES: Revision 15

22.2 Approximate Water Detection Limits for the ICAP 61E Trace Analyzers.

Element	Estimated IDL (mg/L)	Estimated MDL (mg/L)	Lab PQL (mg/L)
Al	0.020	0.025	0.2
Sb	0.004	0.005	0.02
As	0.003	0.005	0.01
Ba	0.0002	0.0002	0.002
Be	0.0001	0.0003	0.002
B	0.001	0.005	0.05
Cd	0.00032	0.0006	0.001
Ca	0.014	0.02	0.5
Cr	0.0008	0.001	0.004
Co	0.0008	0.001	0.004
Cu	0.001	0.002	0.01
Fe	0.02	0.030	0.05
Pb	0.002	0.004	0.006
Mg	0.015	0.02	0.2
Mo	0.001	0.002	0.01
Ni	0.00090	0.002	0.01
K	0.052	0.075	0.5
Se	0.003	0.005	0.015
Na	0.250	0.3	1.0
Ag	0.00075	0.0008	0.003
Tl	0.003	0.005	0.02
V	0.001	0.002	0.005
Zn	0.004	0.007	0.02
Sn	0.003	0.004	0.01
Ti	0.00035	0.0008	0.005
Mn	0.0005	0.0003	0.003

TITLE: METHOD 6010B/ 200.7/CLP USING THE THERMO JARRELL ASH 61E TRACE

SUPERCEDES: Revision 15

22.3 Approximate Soil Detection Limits for the ICAP 61E Trace Analyzers.

Element	Estimated IDL (mg/kg)	Estimated MDL (mg/kg)	Lab PQL (mg/kg)
Al	2.0	2.0	10.0
Sb	0.4	5	15.0
As	0.3	0.5	2.0
Ba	0.02	0.1	0.5
Be	0.01	0.03	0.2
B	0.1	0.5	2.0
Cd	0.03	0.06	0.2
Ca	1.0	2.0	10.0
Cr	0.08	0.1	0.5
Co	0.1	0.1	0.5
Cu	0.1	0.2	1.0
Fe	2.0	3.0	10.0
Pb	0.2	0.4	1.0
Mg	2.0	2.0	20.0
Mo	0.1	0.2	1.0
Ni	0.1	0.2	0.5
K	5.0	8.0	30.0
Se	0.3	0.5	4.0
Na	25	30	140.0
Ag	0.1	0.1	0.5
Tl	0.3	1.0	6.0
V	0.1	0.2	0.5
Zn	0.4	0.7	2.0
Sn	0.3	0.4	2.0
Ti	0.05	0.1	0.5
Mn	0.05	0.05	0.2

TITLE: METHOD 6010B/ 200.7/CLP USING THE THERMO JARRELL ASH 61E TRACE

SUPERCEDES: Revision 15

22.4 Wavelengths and Background Points Used for Each Element on the ICAP 61E Trace Analyzer.

		Background Points	
Element	Wavelength	Trace #1	Trace #2
Al	3082.15	+10	+10
Sb	2068.38	+10	+10
As	1890.42	-10	-10
Ba	4934.09	+10	+10
Be	3130.42	-10	+10
B	2946.78	+10	+10
Cd	2265.02	-10	-10
Ca	3179.33	-10	-10
Cr	2677.16	-10	+10
Co	2286.16	+10	-10
Cu	3247.53	+10	+10
Fe	2714.41	+10	+10
Pb	2203.53	+10/-10	+10/-10
Mg	2790.78	-10	+10
Mo	2020.30	NA	-10
Ni	2316.04	+10	+10
K	4047.35/7664.35	-28	-28
Se	1960.26	+10/-10	+10/-10
Na	3302.32	+10	-10
Ag	3280.68	-10	-10
Tl	1908.64	+10	-10
V	2924.02	+10	+10
Zn	3062.00	+10	+10
Sn	2899.89	-10	-10
Ti	3372.80	+10	-10
Mn	2576.10	-10	+10

TITLE: METHOD 6010B/ 200.7/CLP USING THE THERMO JARRELL ASH 61E TRACE

SUPERCEDES: Revision 15

22.5 Approximate Linear Dynamic Range of Each Element on the ICAP 61E Trace Analyzer.

Element	Trace #1 (mg/L)	Trace #2 (mg/L)
Al	500	500
Sb	25	25
As	50	50
Ba	10	10
Be	10	10
B	25	25
Cd	5	5
Ca	500	500
Cr	25	25
Co	5	5
Cu	20	20
Fe	200	200
Pb	10	25
Mg	500	500
Mo	NA	25
Ni	10	10
K	500	500
Se	10	50
Na	500	500
Ag	10	10
Tl	25	50
V	200	200
Zn	200	200
Sn	10	10
Ti	5	5
Mn	50	50

TITLE: METHOD 6010B/ 200.7/CLP USING THE THERMO JARRELL ASH 61E TRACE

SUPERCEDES: Revision 15

22.6 Concentration of each analyte for LFB, LCS, Post-digestion Spike, Non-CLP matrix spike and CLP matrix spike:

Element	LFB, Post-digestion Spike and Non-CLP Matrix Spike (mg/L)	CLP Matrix Spike (4.1) (mg/L)	CLP Matrix Spike (5.2) (mg/L)	Soil Post-digestion Spike and Non-CLP Matrix Spikes (mg/kg)	Estimated Soil LCS (mg/kg) Changes with new lot	CLP Matrix Spike Soil (4.1) (mg/kg)	CLP Matrix Spike Soil (5.2) (mg/kg)
Al	10.0	2.00	2.00	100	6340		
Sb	0.20	0.500	0.100	20	34	20	20
As	0.20	2.00	0.040	20	192	8	8
Ba	0.20	2.00	2.00	20	417	400	400
Be	0.20	0.050	0.050	20	99.9	10	10
B	0.20			20	131		
Cd	0.20	0.050	0.050	20	125	10	10
Ca	10.0			100	3370		
Cr	0.20	0.200	0.200	20	133	40	40
Co	0.20	0.500	0.500	20	56.8	100	100
Cu	0.20	0.250	0.250	20	93.9	50	50
Fe	10	1.00	1.00	100	11600		
Pb	0.20	0.500	0.020	20	160	4	4
Mg	10.0			100	2000		
Mo	0.20			20	62.9		
Ni	0.20	0.500	0.500	20	174	100	100
K	10.0			100	1890		
Se	0.20	2.00	0.010	20	97	2	10
Na	10.0			100	241		
Ag	0.20	0.050	0.050	20	115	10	10
Tl	0.20	2.00	0.050	20	79.1	10	10
V	0.20	0.500	0.500	20	92.7	100	100
Zn	0.20	0.500	0.500	20	246	100	100
Sn	0.20			20	117		
Ti	0.20			20	327		
Mn	0.20	0.500	0.500	20	320	100	100

TITLE: METHOD 6010B/ 200.7/CLP USING THE THERMO JARRELL ASH 61E TRACE

SUPERCEDES: Revision 15

22.7 Reagents and Stock Solutions which are Purchased as Starting Materials for Preparation of Trace Standards.

From ULTRA SCIENTIFIC:

ICUS-575	10,000 µg/mL	Al
ICUS-576	10,000 µg/mL	Sb
ICM-441	10,000 µg/mL	As
ICUS-573	10,000 µg/mL	Ba
ICUS-574	10,000 µg/mL	Be
ICUS-919	10,000 µg/mL	Cd
ICUS-1241	10,000 µg/mL	Ca
1,000 µg/mL Ag	10,000 µg/mL	Cr
1000 ug/ml Y	10,000 µg/mL	Co
1,000 µg/mL Sn	10,000 µg/mL	Cu
CLP-1	10,000 µg/mL	Fe
CLP-2	10,000 µg/mL	Pb
CLP-3	10,000 µg/mL	Mg
	10,000 µg/mL	Mn
	10,000 µg/mL	Mo
	10,000 µg/mL	Ni
	10,000 µg/mL	K
	10,000 µg/mL	Se
	10,000 µg/mL	Na
	10,000 µg/mL	Ag
	10,000 µg/mL	Tl
	10,000 µg/mL	Zn
	10,000 µg/mL	Sn
	10,000 µg/mL	Ti

Certificates of Analysis are attached for the custom blend standards listed as ICUS-(…) above.

From JT-BAKER

Concentration HCl (Trace Metals Grade)
Concentration HNO_3 (Trace Metals Grade)

From HIGH PURITY:

Solid $LiNO_3$	CAL STD #2-R Solution A
10,000 µg/mL Y	CAL STD #2-R Solution B
1,000 µg/mL Ag	
1,000 µg/mL Sn	

TITLE: METHOD 6010B/ 200.7/CLP USING THE THERMO JARRELL ASH 61E TRACE

SUPERCEDES: Revision 15

Table 22.8 Concentrations of Calibration Standards: (in mg/L)

Element	Std. 1	Std. 2	Std. 3	NAKCAMG100	NAKCAMG400
Al	5.0	25.0	50.0		
Sb	0. 1	0.5	1.0		
As	0. 1	0.5	1.0		
Ba	0. 1	0.5	1.0		
Be	0. 1	0.5	1.0		
Cd	0. 1	0.5	1.0		
Ca	5	25	50	100	400
Cr	0.1	0.5	1.0		
Co	0.1	0.5	1.0		
Cu	0.1	0.5	1.0		
Fe	5	25	50		
Mg	5	25	50	100	400
Mn	0.1	0.5	1.0		
Ni	0.1	0.5	1.0		
Ag	0.1	0.5	1.0		
Tl	0.1	0.5	1.0		
Zn	0.1	0.5	1.0		
V	0.1	0.5	1.0		
B	0.1	0.5	1.0		
Mo	0.1	0.5	1.0		
Ti	0.1	0.5	1.0		
Sn	0.1	0.5	1.0		
Se	0.1	0.5	1.0		
Na	5	25	50.0	100	400
K	5	25	50.0	100	400
Pb	0.1	0.5	1.0		

TITLE: METHOD 6010B/ 200.7/CLP USING THE THERMO JARRELL ASH 61E TRACE

SUPERCEDES: Revision 15

Table 22.9 Values for ICSA and ICSAB (in mg/L)

Element	ICSAB	ICSA
Al	500.0	500
Ca	500.0	500
Fe	100.0	200
Mg	500.0	500
Ba	0.5	-
Be	0.5	-
Cd	1.0	-
Co	0.5	-
Cr	0.5	-
Cu	0.5	-
Mn	0.5	-
Ni	1.0	-
Pb	0.05	-
V	0.5	-
Zn	1.0	-
Sb	0.6	-
As	0.1	-
Tl	0.1	-
Se	0.05	-
Ag	0.2	-

TITLE: METHOD 6010B/ 200.7/CLP USING THE THERMO JARRELL ASH 61E TRACE

SUPERCEDES: Revision 15

Table 22.10 Values for CCV and ICV (in mg/L):

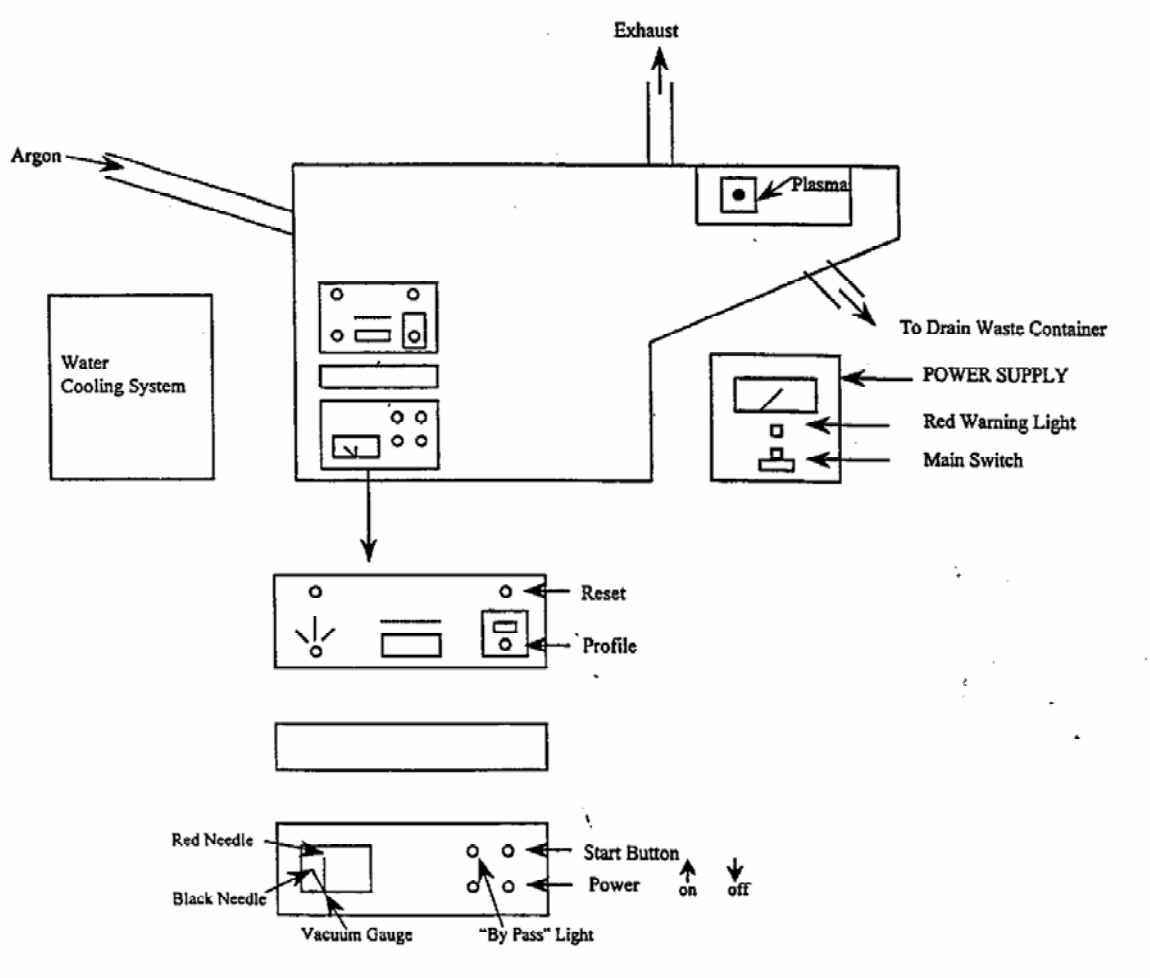
Element	CCV	ICV
Al	25.0	18.75
Sb	0.5	0.375
As	0.5	0.375
Ba	0.5	0.375
Be	0.5	0.375
B	0.5	0.375
Cd	0.5	0.375
Ca	25.0	18.75
Cr	0.5	0.375
Co	0.5	0.375
Cu	0.5	0.375
Fe	25.0	18.75
Pb	0.5	0.375
Mg	25.0	18.75
Mn	0.5	0.375
Mo	0.5	0.375
Ni	0.5	0.375
K	25.0	18.75
Se	0.5	0.375
Na	25.0	18.75
Ag	0.5	0.375
Tl	0.5	0.375
V	0.5	0.375
Zn	0.5	0.375
Sn	0.5	0.375
Ti	0.5	0.375
Mn	0.5	0.375

TITLE: METHOD 6010B/ 200.7/CLP USING THE THERMO JARRELL ASH 61E TRACE

SUPERCEDES: Revision 15

22.11 Main Instrument Layout:

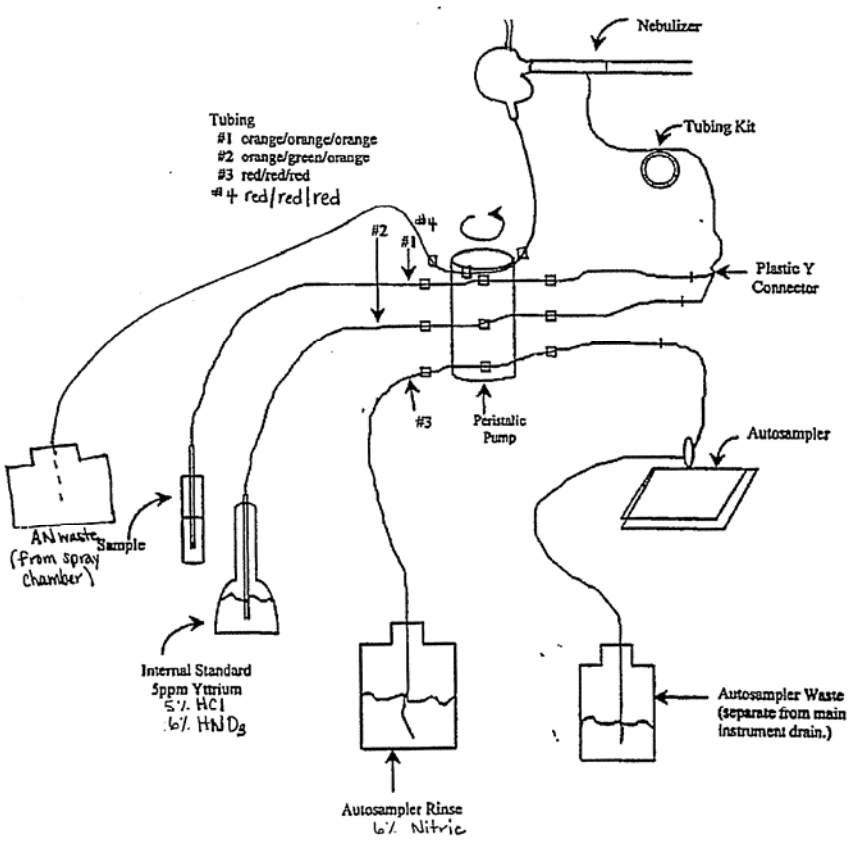
The vacuum pump –not shown- is located behind the instrument. The auto sampler – not shown- is placed in front of the instrument.



TITLE: METHOD 6010B/ 200.7/CLP USING THE THERMO JARRELL ASH 61E TRACE

SUPERCEDES: Revision 15

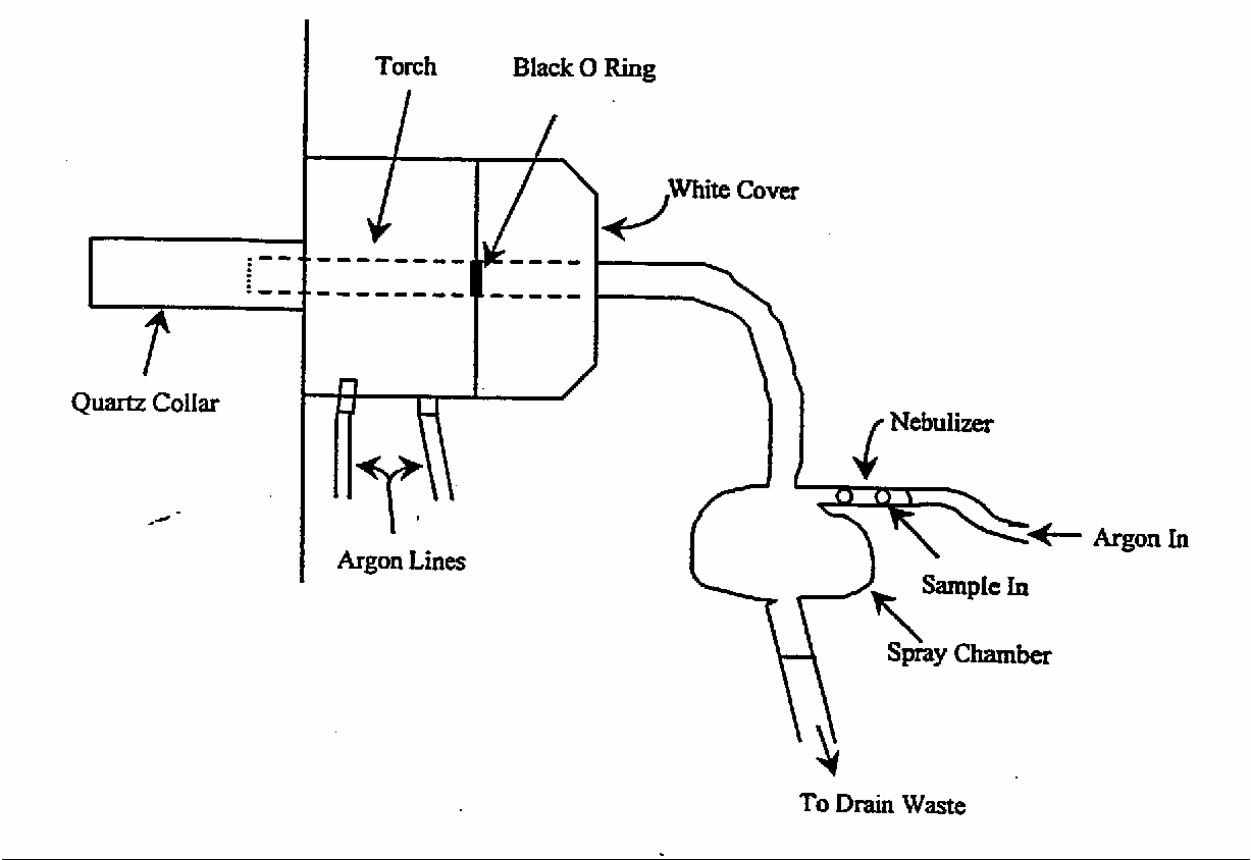
22.12 Tubing Layout:



TITLE: METHOD 6010B/ 200.7/CLP USING THE THERMO JARRELL ASH 61E TRACE

SUPERCEDES: Revision 15

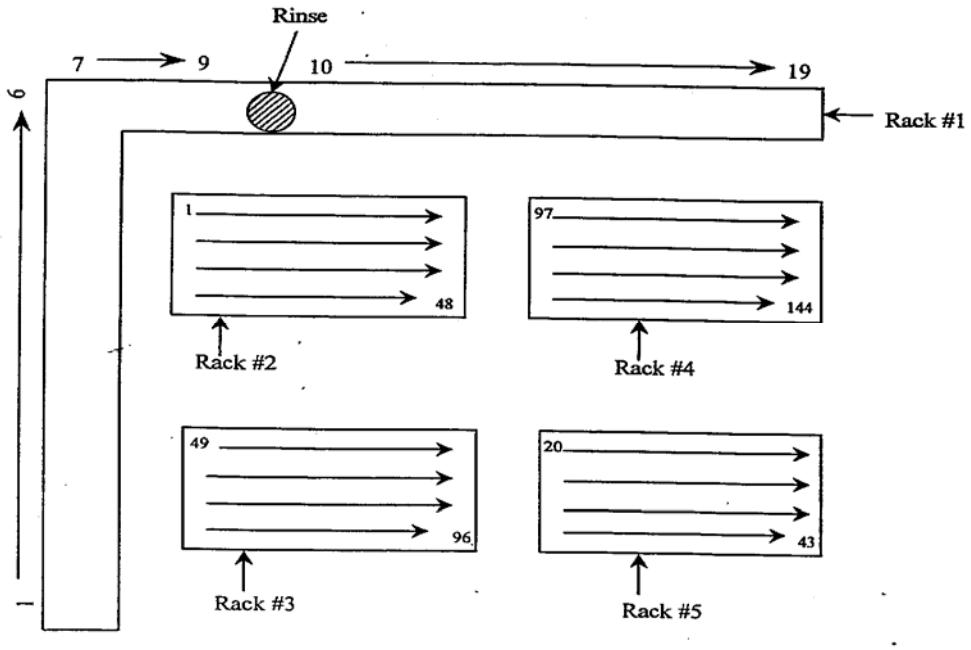
22.13 Torch/Spray Chamber/Nebulizer Assembly:



TITLE: METHOD 6010B/ 200.7/CLP USING THE THERMO JARRELL ASH 61E TRACE

SUPERCEDES: Revision 15

22.14 Autosampler Layout:



TITLE: METHOD 6010B/ 200.7/CLP USING THE THERMO JARRELL ASH 61E TRACE

SUPERCEDES: Revision 15

Table 22.15: Method Summary

Method ⇒ / Parameter ⇓	EPA Series Method 200.7	SW-846B Method 6010B	CLP
Method Validation (2)	Initial demonstration of performance: 1. Determination of the linear dynamic range. Verify linear calibration range limit by analyzing a high concentration standard. Results must be within 5% of the true value. 2. Analyze LCS within ±10% of stated value. 3. Establish MDLs using Laboratory fortified blanks. MDLs must meet regulatory levels.	Same	Same
QC Check Standards/Samples (ICV)	Verify each element calibration with a Control Sample (ICV) prepared from a source different than the calibration standards at the following concentrations: Silver at a maximum of 0.5mg/L, other elements ≥1.0 mg/L. The ICV must be within 3% RSD. %R = 95-105	Verify each element calibration with an Instrument Check Standard prepared from a source different from the calibration standards at a concentration equivalent to the midpoint of the calibration curves. The ICV must be within 5% RSD. %R=90-110	Initial calibration verified with independent standard. %R=90-110
Method Detection Limit	IDL's are determined quarterly. MDLs are determined annually	IDL's are determined quarterly. MDLs are determined annually	IDL's are determined quarterly. MDLs are determined annually.
Standard Solution Expiration(3)	Stocks: yearly or specified by supplier Intermediate: 6 mos. Working: 3 days to 7 days	(same)	(same)
Initial Calibration	Per instrument manufacturer's specification. Minimum of a blank and one standard.	Per instrument manufacturer's specifications (should consist of 3 levels and a blank). The criteria is that the RSD <3%.	Per instrument manufacturer's specifications. Minimum of a blank and one standard.
Continuing Calibration	Analyze instrument performance check (ICV) solution immediately following calibration, after every 10 samples and at the end of the run. For initial analysis, %R=95-105, for subsequent analyses of standard. %R=90-110.	Analyze instrument check standard after every 10 samples and at the end of the run. %R=90-110.	Calibration checked after every 10 samples or 2 hours whichever is more frequent with a mid-range calibration standard. %R=90-110.
Accuracy/Precision	-One MS/MD per 10 samples or each batch %R=75-125%.	-One MS/MD per 10 samples or each batch %R=75-125%.	One MS and one duplicate per sample delivery group or per similar matrix type.%R=75-125. %RPD<20.

TITLE: METHOD 6010B/ 200.7/CLP USING THE THERMO JARRELL ASH 61E TRACE

SUPERCEDES: Revision 15

Method ⇒ / Parameter ⇓	EPA Series Method 200.7	SW-846B Method 6010B	CLP
	%RPD < 20% - A laboratory fortified blank (LFB) prepared with each batch of samples digested. %R=85-115.	%RPD < 20% - A laboratory fortified blank (LFB) prepared with each batch of samples digested. %R=80-120	Analyze a post-digestion spike if the pre-digestion spike recovery is outside control limits and the sample result does not exceed 4 times the spike added.
Blanks	One method blank with each batch of samples. When values constitute 10% or more of the analyte level or are 2.2 times the analyte MDL, whichever is greater, the entire preparation and analysis is repeated. Analyze calibration blank after each instrument check standard (CCV). Blank < 3x IDL.	One method blank per batch of samples processed at the same time. Analyze calibration blank after each Instrument Check Standard (CCV). Blank < 3x IDL.	One method blank per sample delivery group or per sample process batch, whichever is more frequent. Analyze calibration blanks after initial and continuing calibration verification or every 10 samples. If absolute value of blank for any analyte >CRDL, terminate analysis, correct problem, and reanalyze all samples since last compliant blank.
Interference Check Standard	Interference Check Solutions containing known concentrations of interfering elements and the elements of interest are analyzed at the beginning of each analytical run. %R=80-120. Reanalyze highest standard after calibration for checking (not required by the method). %R=95-105.	Interference Check Solutions containing known concentrations of interfering elements and the elements of interest are analyzed at the beginning of each analytical run. %R=80-120. Reanalyze highest standard after calibration for checking (not required by the method). %R=95-105.	Analyze ICSA and ICSAB solutions at a frequency of not greater than 20 analytical samples. Must be followed immediately by CCV/CCB pair. ICSA: For target analytes with CRDL $\leq$10 µg/L, results should fall within $\pm$ CRDL of the analyte's true value, otherwise use alternate method to quantitate results for affected analytes. ICSAB: %R=80-120
IDL Standard (CRI)	Run CRI Standard at the beginning of each analytical run directly following ICB. %R=50-150. Not required per method..	Run CRI Standard at the beginning of each analytical run directly following ICB.. %R=50-150. Not required per method.	Run CRI Standard every 20 analytical samples and at the beginning and end of each analytical run. %R=50-150 For Antimony, Lead, and Thallium %R=70-130 All other elements
Serial Dilution	1:5 Dilution on each new matrix per job. % Difference=10%	1:5 Dilution on each new matrix per job. % Difference=10%	1:5 Dilution on each new matrix per SDG. % Difference=10%
Matrix Spike	%R= 75-125	%R=75-125	%R=75-125
Post Digestion Spike	%R=85-115	%R=75-125	%R=75-125
Holding Time (4)	180 days from collection	180 days from collection	180 days from receipt (VTSR)

TITLE: METHOD 6010B/ 200.7/CLP USING THE THERMO JARRELL ASH 61E TRACE

SUPERCEDES: Revision 15

Table 22.16 Contract Required Detection Limits (CRDL)

Analyte	CRDL (4.0) (ng/mL)	CRDL (5.0) (ng/mL)
Aluminum	200	200
Antimony	60	5
Arsenic	10	5
Barium	200	20
Beryllium	5	1
Cadmium	5	2
Calcium	5000	5000
Chromium	10	5
Cobalt	50	5
Copper	25	5
Iron	100	100
Lead	3	3
Magnesium	5000	5000
Manganese	15	10
Mercury	0.2	0.1
Nickel	40	20
Potassium	5000	5000
Selenium	5	5
Silver	10	5
Sodium	5000	5000
Thallium	10	5
Vanadium	50	10
Zinc	20	10

TITLE: METHOD 6010B/ 200.7/CLP USING THE THERMO JARRELL ASH 61E TRACE

SUPERCEDES: Revision 15

22.17 Concentration of each element in the CRI standard solution and reading on the instrument.

Analyte	CRI Stock Std	CRI
Aluminum	2.0	0.2
Antimony	0.2	0.02
Arsenic	0.1	0.01
Barium	0.02	0.002
Beryllium	0.02	0.002
Boron	0.5	0.005
Cadmium	0.01	0.001
Calcium	5.0	0.05
Chromium	0.04	0.004
Cobalt	0.04	0.004
Copper	0.1	0.001
Iron	0.5	0.05
Lead	0.05	0.005
Magnesium	2.0	0.02
Manganese	0.03	0.003
Nickel	0.1	0.01
Potassium	5.0	0.5
Selenium	0.15	0.0015
Silver	0.03	0.003
Sodium	10.0	1.0
Thallium	0.2	0.02
Tin	0.1	0.01
Vanadium	0.05	0.005
Zinc	0.2	0.02

TITLE: METHOD 6010B/ 200.7/CLP USING THE THERMO JARRELL ASH 61E TRACE

SUPERCEDES: Revision 15

22.18 USACE Requirements

EM 200-1-3
1 Feb 01

Table I-1
Summary of Measurement quality objectives for Method 6010 Inductively Coupled Plasma (ICP) Metals

Quality Control Element	Description of Element	Frequency of Implementation	Acceptance Criteria
Initial Calibration (I.9.2.1.1)	Option 1- 1 std and blank, and a low-level check standard at MQL Option 2- 3 stds and blank	Daily	Option 1- Low-level check standard ± 20% Option 2- r • 0.995
Instrumental Precision (I.9.2.1.1)	%RSD 3 integrations (exposures)	Each calibration and calibration verification standards (ICV/CCV)	%RSD < 5%
Initial Calibration Verification (ICV) (I.9.3)	Midlevel (2nd source) verification	After initial calibration	%Recovery ± 10%
Initial Calibration Blank (ICB) (I.9.4)	Interference-free matrix to assess analysis contamination	After initial calibration	Analytes < MDL
Interelement Check Standards (ICS) (I.8.1)	ICS-A - interferents only ICS-B - interferents and target analytes	Beginning of analytical sequence	%Recovery ± 20% for target analytes
Continuing Calibration Blank (CCB) (I.9.4)	Interference-free matrix to assess analysis contamination	Every 10 samples and at end of analytical sequence	Analytes < MDL
Continuing Calibration Verification (CCV) (I.9.5 / I.9.5.1)	Midlevel verification	Every 10 samples and at end of analytical sequence	%Recovery ± 10%
Method Blank (MB) (I.10.2.1 / I.11.4.1)	Interference-free matrix to assess overall method contamination	1 per sample batch	Analytes < one-half MRL
Laboratory Control Sample (LCS) (I.10.2.2 / I.11.4.2)	Interference-free matrix containing all target analytes	1 per sample batch	%Rec = 80% - 120% Sporadic marginal failures[1]: %Rec = 60% - 140%
Matrix Spike (MS) (I.10.2.3 / I.11.4.3 / I.11.4.3.1)	Sample matrix spiked with all/subset of target analytes prior to digestion	1 per sample batch	%Rec = 75% - 125%
Matrix Duplicate (MD) or Matrix Spike Duplicate (MSD) (I.10.2.4 / I.11.4.4)	Refer to text for MD or MS.	1 per sample batch	RPD • 25%
Post Digestion Spike (PDS) (I.10.3.1 / I.11.4.6)	Sample digestate spiked with all/subset of target analytes	1 per sample batch on MS sample	%Rec = 75% - 125%
Serial Dilution (SD) (I.10.3.2)	1:4 dilution analyzed to assess matrix effects	As needed to assess new and unusual matrices	Agreement between undiluted and diluted results ± 10%
Method of Standard Additions (MSA) (I.12.2.1)	Method of quantitation	As needed for samples with suspected or confirmed matrix effects	r • 0.995

[1] The number of Sporadic Marginal Failure (SMF) allowances depends upon the number of target analytes reported from the analysis. For instance, if between 7 to 15 metals are reported from the ICP analysis, one (1) SMF is allowed to the expanded criteria presented. If greater than 15 metals are reported from the ICP analysis, two (2) SMFs are allowed.

I-54

TITLE: METHOD 6010B/ 200.7/CLP USING THE THERMO JARRELL ASH 61E TRACE

SUPERCEDES: Revision 15

22.19 AFCEE 3.1 Requirements

AFCEE QAPP Version 3.1 August 2001 Page 7-106

Table 7.2.15-2. QC Acceptance Criteria for Method SW6010B

Method	Analyte	Accuracy Water (% R)	Precision Water (% RPD)	Accuracy Soil (% R)	Precision Soil (% RPD)
SW6010B	Aluminum	80-120	≤20	79-120	≤30
	Antimony	80-120	≤20	80-120	≤30
	Arsenic	80-120	≤20	80-120	≤30
	Barium	80-120	≤20	80-120	≤30
	Beryllium	80-120	≤20	80-120	≤30
	Cadmium	80-120	≤20	80-120	≤30
	Calcium	80-120	≤20	80-120	≤30
	Chromium	80-120	≤20	80-120	≤30
	Cobalt	80-120	≤20	80-120	≤30
	Copper	80-120	≤20	80-120	≤30
	Iron	80-120	≤20	80-120	≤30
	Lead	80-120	≤20	80-120	≤30
	Magnesium	80-120	≤20	80-120	≤30
	Manganese	80-120	≤20	80-120	≤30
	Molybdenum	79-120	≤20	80-120	≤30
	Nickel	80-120	≤20	80-120	≤30
	Potassium	80-120	≤20	80-120	≤30
	Selenium	80-120	≤20	80-120	≤30
	Silver	80-120	≤20	75-120	≤30
	Sodium	80-120	≤20	80-120	≤30
	Thallium	80-120	≤20	80-120	≤30
	Vanadium	80-120	≤20	80-120	≤30
	Zinc	80-120	≤20	80-120	≤30

AFCEE QAPP Version 3.1 August 2001 Page 7-105

Table 7.2.15-1. RLs for Method SW6010B

Parameter/Method	Analyte	Water RL	Water Unit	Soil RL	Soil Unit
ICP Screen for Metals SW6010B	Aluminum	0.2	mg/L	20.0	mg/kg
	Antimony	0.05	mg/L	10.0	mg/kg
	Arsenic	0.03	mg/L	5.0	mg/kg
	Barium	0.05	mg/L	1.0	mg/kg
	Beryllium	0.004	mg/L	1.0	mg/kg
	Cadmium	0.005	mg/L	0.50	mg/kg
	Calcium	1.1	mg/L	100	mg/kg
	Chromium	0.01	mg/L	1.0	mg/kg
	Cobalt	0.06	mg/L	1.0	mg/kg
	Copper	0.01	mg/L	2.0	mg/kg
	Iron	0.20	mg/L	3.0	mg/kg
	Lead	0.025	mg/L	3.0	mg/kg
	Magnesium	1.0	mg/L	100	mg/kg
	Manganese	0.01	mg/L	1.0	mg/kg
	Molybdenum	0.015	mg/L	3.0	mg/kg
	Nickel	0.02	mg/L	2.0	mg/kg
	Potassium	1.0	mg/L	200	mg/kg
	Selenium	0.03	mg/L	3.0	mg/kg
	Silver	0.01	mg/L	1.0	mg/kg
	Sodium	1.0	mg/L	100	mg/kg
	Thallium	0.08	mg/L	6.0	mg/kg
	Vanadium	0.01	mg/L	1.0	mg/kg
	Zinc	0.02	mg/L	2.0	mg/kg

SOP No.	Revision No.	Effective Date	Page
AME-6010-30	16	June 13, 2006	59 of 71

TITLE: METHOD 6010B/ 200.7/CLP USING THE THERMO JARRELL ASH 61E TRACE

SUPERCEDES: Revision 15

AFCEE QAPP Version 3.1 August 2001 Page 7-106

Table 7.2.15-2. QC Acceptance Criteria for Method SW6010B

Method	Analyte	Accuracy Water (% R)	Precision Water (% RPD)	Accuracy Soil (% R)	Precision Soil (% RPD)
SW6010B	Aluminum	80-120	≤20	79-120	≤30
	Antimony	80-120	≤20	80-120	≤30
	Arsenic	80-120	≤20	80-120	≤30
	Barium	80-120	≤20	80-120	≤30
	Beryllium	80-120	≤20	80-120	≤30
	Cadmium	80-120	≤20	80-120	≤30
	Calcium	80-120	≤20	80-120	≤30
	Chromium	80-120	≤20	80-120	≤30
	Cobalt	80-120	≤20	80-120	≤30
	Copper	80-120	≤20	80-120	≤30
	Iron	80-120	≤20	80-120	≤30
	Lead	80-120	≤20	80-120	≤30
	Magnesium	80-120	≤20	80-120	≤30
	Manganese	80-120	≤20	80-120	≤30
	Molybdenum	79-120	≤20	80-120	≤30
	Nickel	80-120	≤20	80-120	≤30
	Potassium	80-120	≤20	80-120	≤30
	Selenium	80-120	≤20	80-120	≤30
	Silver	80-120	≤20	75-120	≤30
	Sodium	80-120	≤20	80-120	≤30
	Thallium	80-120	≤20	80-120	≤30
	Vanadium	80-120	≤20	80-120	≤30
	Zinc	80-120	≤20	80-120	≤30

AFCEE QAPP Version 3.1 August 2001 Page 7-105

Table 7.2.15-1. RLs for Method SW6010B

Parameter/Method	Analyte	Water RL	Water Unit	Soil RL	Soil Unit
ICP Screen for Metals SW6010B	Aluminum	0.2	mg/L	20.0	mg/kg
	Antimony	0.05	mg/L	10.0	mg/kg
	Arsenic	0.03	mg/L	5.0	mg/kg
	Barium	0.05	mg/L	1.0	mg/kg
	Beryllium	0.004	mg/L	1.0	mg/kg
	Cadmium	0.005	mg/L	0.50	mg/kg
	Calcium	1.1	mg/L	100	mg/kg
	Chromium	0.01	mg/L	1.0	mg/kg
	Cobalt	0.06	mg/L	1.0	mg/kg
	Copper	0.01	mg/L	2.0	mg/kg
	Iron	0.20	mg/L	3.0	mg/kg
	Lead	0.025	mg/L	3.0	mg/kg
	Magnesium	1.0	mg/L	100	mg/kg
	Manganese	0.01	mg/L	1.0	mg/kg
	Molybdenum	0.015	mg/L	3.0	mg/kg
	Nickel	0.02	mg/L	2.0	mg/kg
	Potassium	1.0	mg/L	200	mg/kg
	Selenium	0.03	mg/L	3.0	mg/kg
	Silver	0.01	mg/L	1.0	mg/kg
	Sodium	1.0	mg/L	100	mg/kg
	Thallium	0.08	mg/L	6.0	mg/kg
	Vanadium	0.01	mg/L	1.0	mg/kg
	Zinc	0.02	mg/L	2.0	mg/kg

SOP No.	Revision No.	Effective Date	Page
AME-6010-30	16	June 13, 2006	60 of 71

TITLE: METHOD 6010B/ 200.7/CLP USING THE THERMO JARRELL ASH 61E TRACE

SUPERCEDES: Revision 15

Table 7.2.15-2. QC Acceptance Criteria for Method SW6010B

Method	Analyte	Accuracy Water (% R)	Precision Water (% RPD)	Accuracy Soil (% R)	Precision Soil (% RPD)
SW6010B	Aluminum	80-120	≤20	79-120	≤30
	Antimony	80-120	≤20	80-120	≤30
	Arsenic	80-120	≤20	80-120	≤30
	Barium	80-120	≤20	80-120	≤30
	Beryllium	80-120	≤20	80-120	≤30
	Cadmium	80-120	≤20	80-120	≤30
	Calcium	80-120	≤20	80-120	≤30
	Chromium	80-120	≤20	80-120	≤30
	Cobalt	80-120	≤20	80-120	≤30
	Copper	80-120	≤20	80-120	≤30
	Iron	80-120	≤20	80-120	≤30
	Lead	80-120	≤20	80-120	≤30
	Magnesium	80-120	≤20	80-120	≤30
	Manganese	80-120	≤20	80-120	≤30
	Molybdenum	80-120	≤20	80-120	≤30
	Nickel	79-120	≤20	80-120	≤30
	Potassium	80-120	≤20	80-120	≤30
	Selenium	80-120	≤20	80-120	≤30
	Silver	80-120	≤20	80-120	≤30
	Sodium	80-120	≤20	75-120	≤30
	Thallium	80-120	≤20	80-120	≤30
	Vanadium	80-120	≤20	80-120	≤30
	Zinc	80-120	≤20	80-120	≤30

Table 7.2.15-1. RLs for Method SW6010B

Parameter/Method	Analyte	Water RL	Water Unit	Soil RL	Soil Unit
ICP Screen for Metals SW6010B	Aluminum	0.2	mg/L	20.0	mg/kg
	Antimony	0.05	mg/L	10.0	mg/kg
	Arsenic	0.03	mg/L	5.0	mg/kg
	Barium	0.05	mg/L	1.0	mg/kg
	Beryllium	0.004	mg/L	1.0	mg/kg
	Cadmium	0.005	mg/L	0.50	mg/kg
	Calcium	1.1	mg/L	100	mg/kg
	Chromium	0.01	mg/L	1.0	mg/kg
	Cobalt	0.06	mg/L	1.0	mg/kg
	Copper	0.01	mg/L	2.0	mg/kg
	Iron	0.20	mg/L	3.0	mg/kg
	Lead	0.025	mg/L	3.0	mg/kg
	Magnesium	1.0	mg/L	100	mg/kg
	Manganese	0.01	mg/L	1.0	mg/kg
	Molybdenum	0.015	mg/L	3.0	mg/kg
	Nickel	0.02	mg/L	2.0	mg/kg
	Potassium	1.0	mg/L	200	mg/kg
	Selenium	0.03	mg/L	3.0	mg/kg
	Silver	0.01	mg/L	1.0	mg/kg
	Sodium	1.0	mg/L	100	mg/kg
	Thallium	0.08	mg/L	6.0	mg/kg
	Vanadium	0.01	mg/L	1.0	mg/kg
	Zinc	0.02	mg/L	2.0	mg/kg

SOP No.	Revision No.	Effective Date	Page
AME-6010-30	16	June 13, 2006	61 of 71

TITLE: METHOD 6010B/ 200.7/CLP USING THE THERMO JARRELL ASH 61E TRACE

SUPERCEDES: Revision 15

Table 7.2.15-2. QC Acceptance Criteria for Method SW6010B

Method	Analyte	Accuracy Water (% R)	Precision Water (% RPD)	Accuracy Soil (% R)	Precision Soil (% RPD)
SW6010B	Aluminum	80-120	≤20	79-120	≤30
	Antimony	80-120	≤20	80-120	≤30
	Arsenic	80-120	≤20	80-120	≤30
	Barium	80-120	≤20	80-120	≤30
	Beryllium	80-120	≤20	80-120	≤30
	Cadmium	80-120	≤20	80-120	≤30
	Calcium	80-120	≤20	80-120	≤30
	Chromium	80-120	≤20	80-120	≤30
	Cobalt	80-120	≤20	80-120	≤30
	Copper	80-120	≤20	80-120	≤30
	Iron	80-120	≤20	80-120	≤30
	Lead	80-120	≤20	80-120	≤30
	Magnesium	80-120	≤20	80-120	≤30
	Manganese	80-120	≤20	80-120	≤30
	Molybdenum	79-120	≤20	80-120	≤30
	Nickel	80-120	≤20	80-120	≤30
	Potassium	80-120	≤20	80-120	≤30
	Selenium	80-120	≤20	80-120	≤30
	Silver	80-120	≤20	75-120	≤30
	Sodium	80-120	≤20	80-120	≤30
	Thallium	80-120	≤20	80-120	≤30
	Vanadium	80-120	≤20	80-120	≤30
	Zinc	80-120	≤20	80-120	≤30

Table 7.2.15-1. RLs for Method SW6010B

Parameter/Method	Analyte	Water RL	Water Unit	Soil RL	Soil Unit
ICP Screen for Metals SW6010B	Aluminum	0.2	mg/L	20.0	mg/kg
	Antimony	0.05	mg/L	10.0	mg/kg
	Arsenic	0.03	mg/L	5.0	mg/kg
	Barium	0.05	mg/L	1.0	mg/kg
	Beryllium	0.004	mg/L	1.0	mg/kg
	Cadmium	0.005	mg/L	0.50	mg/kg
	Calcium	1.1	mg/L	100	mg/kg
	Chromium	0.01	mg/L	1.0	mg/kg
	Cobalt	0.06	mg/L	1.0	mg/kg
	Copper	0.01	mg/L	2.0	mg/kg
	Iron	0.20	mg/L	3.0	mg/kg
	Lead	0.025	mg/L	3.0	mg/kg
	Magnesium	1.0	mg/L	100	mg/kg
	Manganese	0.01	mg/L	1.0	mg/kg
	Molybdenum	0.015	mg/L	3.0	mg/kg
	Nickel	0.02	mg/L	2.0	mg/kg
	Potassium	1.0	mg/L	200	mg/kg
	Selenium	0.03	mg/L	3.0	mg/kg
	Silver	0.01	mg/L	1.0	mg/kg
	Sodium	1.0	mg/L	100	mg/kg
	Thallium	0.08	mg/L	6.0	mg/kg
	Vanadium	0.01	mg/L	1.0	mg/kg
	Zinc	0.02	mg/L	2.0	mg/kg

TITLE: METHOD 6010B/ 200.7/CLP USING THE THERMO JARRELL ASH 61E TRACE

SUPERCEDES: Revision 15

22.20 Example of a Data Review Summary Form for Metals

Date: 05/31/2006
Time: 15:35:36

STL Buffalo
Metals Job Summary
Analysis Type: TOTAL METALS (WATER)

Page: 2
Rept: AN1500

Job No: A06-5562
Project/Task: NYSA586010 7

Lab Due Date: 05/30/2006
Client Due Date: 06/08/2006

Test No	Description	QAPP	Protcl	Method	Mtx	TCLP	Holding Tp	Holding Tclp	Holding Extr	Holding Anal	Prep Type	Unit Meas	Detect Limit Type	Detect Limit Value	Spikes Code	Spikes Amount	Spikes Conc	QC Limits	RPD
CGA01566	TOTAL METALS-G/W-(15) M.PENINSULA																		
CTA06201	AG - WMI - SILVER BY ICP(0.010)-TOT		SW8463	6010	Water	N	S	0	180	180	D	MG/L	CDL	0.01000	A00067	0.25 ML	10.00 UG/ML	(75-125)	20.0
CTA05737	AS - WMI - ARSENIC-ICP TJA - TOTAL		SW8463	6010	Water	N	S	0	180	180	D	MG/L	CDL	0.01000	A00067	1.00 ML	10.00 UG/ML	(75-125)	20.0
CTA06195	BA - WMI - BARIUM BY ICP(0.020)-TOT		SW8463	6010	Water	N	S	0	180	180	D	MG/L	CDL	0.02000	A00067	1.00 ML	10.00 UG/ML	(75-125)	20.0
CTA06203	BE - WMI - BERYLLIUM BY ICP-TOTAL(0		SW8463	6010	Water	N	S	0	180	180	D	MG/L	CDL	0.00300	A00067	1.00 ML	10.00 UG/ML	(80-120)	20.0
CTA05813	CD - WMI - CADMIUM BY ICP(0.0050)-T		SW8463	6010	Water	N	S	0	180	180	D	MG/L	CDL	0.00500	A00067	1.00 ML	10.00 UG/ML	(75-125)	20.0
CTA05444	CO - COBALT - TOTAL - W WITH RL= 0.		SW8463	6010	Water	N	S	0	180	180	D	MG/L	CDL	0.02000	A00067	1.00 ML	10.00 UG/ML	(80-120)	20.0
CTA05725	CR - WMI - CHROMIUM BY ICP(0.010)-T		SW8463	6010	Water	N	S	0	180	180	D	MG/L	CDL	0.01000	A00067	1.00 ML	10.00 UG/ML	(75-125)	20.0
CTA05446	CU - COPPER - TOTAL - W - WITH RL =		SW8463	6010	Water	N	S	0	180	180	D	MG/L	CDL	0.01000	A00067	1.00 ML	10.00 UG/ML	(80-120)	20.0
CTA05816	NI - WMI - NICKEL BY ICP(0.040)-TOT		SW8463	6010	Water	N	S	0	180	180	D	MG/L	CDL	0.04000	A00067	1.00 ML	10.00 UG/ML	(75-125)	20.0
CTA05734	PB - WMI - LEAD-ICP TJA - TOTAL		SW8463	6010	Water	N	S	0	180	180	D	MG/L	CDL	0.00500	A00067	1.00 ML	10.00 UG/ML	(75-125)	20.0
CTA05872	SB - WMI - ANTIMONY-ICP TJA - TOTAL		SW8463	6010	Water	N	S	0	180	180	D	MG/L	CDL	0.00600	A00067	1.00 ML	10.00 UG/ML	(75-125)	20.0
CTA05882	V - WMI - VANADIUM BY ICP-TOTAL(0.		SW8463	6010	Water	N	S	0	180	180	D	MG/L	CDL	0.05000	A00067	1.00 ML	10.00 UG/ML	(80-120)	20.0
CTA05453	ZN - ZINC - TOTAL - W - WITH RL= 0.		SW8463	6010	Water	N	S	0	180	180	D	MG/L	CDL	0.00500	A00067	1.00 ML	10.00 UG/ML	(80-120)	20.0

Comments: _____

Criteria:

Initial Calibration/Second Source Criteria Met? Y / N
CCV/CCB Criteria Met? Y / N
Method Blank Criteria Met? Y / N
LCS Criteria Met? Y / N
MS/MD Criteria Met? Y / N

Analyst Approval: _____ Date: _____

Data Processor: _____ Date: _____

Data Reviewer: _____ Date: _____

TITLE: METHOD 6010B/ 200.7/CLP USING THE THERMO JARRELL ASH 61E TRACE

SUPERCEDES: Revision 15

22.21 Certificates of Analysis for Custom Blend Standards

MDL-1-27

Certificate of Analysis

Custom Inorganic Standard — **Catalog Number: ICUS-575**

Lot Number: D00396

Expiration Date: 08/2004

This ULTRAgrade(TM) standard was gravimetrically prepared, and the true value listed is the concentration calculated from gravimetric and volumetric measurements performed during the preparation of the standard. The standard uncertainty is ± 0.5% relative, unless otherwise specified.

Analyte	True Value		Analytical Method
antimony	100.0	µg/mL	gravimetric
arsenic	100.0	µg/mL	gravimetric
beryllium	100.0	µg/mL	gravimetric
cadmium	100.0	µg/mL	gravimetric
chromium	100.0	µg/mL	gravimetric
cobalt	100.0	µg/mL	gravimetric
copper	100.0	µg/mL	gravimetric
lead	100.0	µg/mL	gravimetric
manganese	100.0	µg/mL	gravimetric
molybdenum	100.0	µg/mL	gravimetric
nickel	100.0	µg/mL	gravimetric
selenium	100.0	µg/mL	gravimetric
thallium	100.0	µg/mL	gravimetric
titanium	100.0	µg/mL	gravimetric
vanadium	100.0	µg/mL	gravimetric
zinc	100.0	µg/mL	gravimetric
calcium	4999.7	µg/mL	gravimetric
iron (99.999%)	5000.0	µg/mL	gravimetric
magnesium	5000.0	µg/mL	gravimetric

Matrix: 5% nitric acid in water

All weights are traceable to NIST traceable weights

ISO-9001:2000 Registered by
Reg. No. 00-R1192rev.1

ISO 17025 Certified by
Cert. No. 0851-01

John E. Russo, Chem. Eng.
Quality Control Manager

TITLE: METHOD 6010B/ 200.7/CLP USING THE THERMO JARRELL ASH 61E TRACE

SUPERCEDES: Revision 15

Certificate of Analysis MDL-2-27

Custom Inorganic Standard

Catalog Number: ICUS-576

Lot Number: D00385

Expiration Date: 08/2004

This ULTRAgrade(TM) standard was gravimetrically prepared, and the true value listed is the concentration calculated from gravimetric and volumetric measurements performed during the preparation of the standard. The standard uncertainty is ± 0.5% relative, unless otherwise specified.

Analyte	True Value		Analytical Method
barium	100.0	µg/mL	gravimetric
boron	100.0	µg/mL	gravimetric
aluminum	5000.0	µg/mL	gravimetric
potassium	5000.0	µg/mL	gravimetric
sodium	5000.0	µg/mL	gravimetric

Matrix: 5% nitric acid in water

All weights are traceable to NIST traceable weights

John E. Russo, Chem. Eng.
Quality Control Manager

TITLE: METHOD 6010B/ 200.7/CLP USING THE THERMO JARRELL ASH 61E TRACE

SUPERCEDES: Revision 15

Certificate of Analysis

27-NOL-15

CLP ICP Interference Check Standard I

Catalog Number: ICM-441

Lot Number: D00226

Expiration Date: 05/2006

This ULTRAgrade(TM) standard was gravimetrically prepared, and the true value listed is the concentration calculated from gravimetric and volumetric measurements performed during the preparation of the standard. The standard uncertainty is ± 0.5% relative, unless otherwise specified.

Analyte	True Value		Analytical Method	NIST SRM#
aluminum	5000.0	µg/mL	ICP	3101a
calcium	5000.0	µg/mL	ICP	3109a
iron	2000.0	µg/mL	ICP	3126a
magnesium	4999.8	µg/mL	ICP	3131a

Matrix: 5% nitric acid in water

All weights are traceable to NIST traceable weights

John E. Russo, Chem. Eng.
Quality Control Manager

TITLE: METHOD 6010B/ 200.7/CLP USING THE THERMO JARRELL ASH 61E TRACE

SUPERCEDES: Revision 15

Certificate of Analysis

MDL-7-26

Custom Inorganic Standard

Catalog Number: ICUS-574

Lot Number: D00347

Expiration Date: 08/2005

This ULTRAgrade(TM) standard was gravimetrically prepared, and the true value listed is the concentration calculated from gravimetric and volumetric measurements performed during the preparation of the standard. The standard uncertainty is ± 0.5% relative, unless otherwise specified.

Analyte	True Value		Analytical Method
barium	40.0	µg/mL	gravimetric
boron	40.0	µg/mL	gravimetric
aluminum	2000.0	µg/mL	gravimetric
potassium	2000.0	µg/mL	gravimetric
sodium	2000.0	µg/mL	gravimetric

Matrix: 5% nitric acid and water

All weights are traceable to NIST traceable weights

John E. Russo, Chem. Eng.
Quality Control Manager

ULTRA Scientific

TITLE: METHOD 6010B/ 200.7/CLP USING THE THERMO JARRELL ASH 61E TRACE

SUPERCEDES: Revision 15

Certificate of Analysis

Custom Inorganic Standard

Catalog Number: ICUS-919
Lot Number: E00155
Job Number: J00003691
Expiration Date: 06/2005

This ULTRAgrade(TM) standard was gravimetrically prepared, and the true value listed is the concentration calculated from gravimetric and volumetric measurements performed during the preparation of the standard The standard uncertainty is ± 0 5% relative, unless otherwise specified

Analyte	True Value		Analytical Method
silver	2.0	mg/L	gravimetric
arsenic	1 0	mg/L	gravimetric
barium	5 0	mg/L	gravimetric
beryllium	5 0	mg/L	gravimetric
cadmium	10 0	mg/L	gravimetric
cobalt	5 0	mg/L	gravimetric
chromium	5 0	mg/L	gravimetric
copper	5 0	mg/L	gravimetric
manganese	5 0	mg/L	gravimetric
nickel	10 0	mg/L	gravimetric
lead	0 5	mg/L	gravimetric
antimony	6 0	mg/L	gravimetric
selenium	0 5	mg/L	gravimetric
thallium	1 0	mg/L	gravimetric
vanadium	5 0	mg/L	gravimetric
zinc	10 0	mg/L	gravimetric
aluminum	5000 0	mg/L	gravimetric
calcium	5000 0	mg/L	gravimetric
iron	1000 0	mg/L	gravimetric
magnesium	5000 0	mg/L	gravimetric

Matrix. 5% nitric acid in water

All weights are traceable to NIST traceable weights

ISO-9001:2000
Registered by
Reg No 00-R1192rev 1

ISO 17025
Certified by
Cert. No 0851-01

John E Russo, Chem. Eng
Quality Control Manager

ULTRA SCIENTIFIC 250 Smith Street, North Kingstown, RI 02852 • 401-294-9400 • fax 401-295-2330 • www.ultrasci.com

TITLE: METHOD 6010B/ 200.7/CLP USING THE THERMO JARRELL ASH 61E TRACE

SUPERCEDES: Revision 15

Certificate of Analysis

Inorganic Custom Standard

Catalog Number: ICUS-1241
Lot Number: E00337
Job Number: J00003929
Expiration Date: 08/2005

This ULTRAgrade(TM) standard was gravimetrically prepared, and the true value listed is the concentration calculated from gravimetric and volumetric measurements performed during the preparation of the standard The standard uncertainty is ± 0 5% relative, unless otherwise specified

Analyte	True Value		Analytical Method
aluminum	2 0	µg/mL	gravimetric
antimony	0 2	µg/mL	gravimetric
arsenic	0 1	µg/mL	gravimetric
barium	0 02	µg/mL	gravimetric
beryllium	0 02	µg/mL	gravimetric
boron	0 5	µg/mL	gravimetric
cadmium	0 01	µg/mL	gravimetric
calcium	5 0	µg/mL	gravimetric
chromium	0 04	µg/mL	gravimetric
cobalt	0 04	µg/mL	gravimetric
copper	0 1	µg/mL	gravimetric
iron	0 5	µg/mL	gravimetric
lead	0 05	µg/mL	gravimetric
magnesium	2 0	µg/mL	gravimetric
manganese	0 03	µg/mL	gravimetric
molybdenum	0 1	µg/mL	gravimetric
nickel	0 1	µg/mL	gravimetric
potassium	5 0	µg/mL	gravimetric
selenium	0 15	µg/mL	gravimetric
silver	0 03	µg/mL	gravimetric
sodium	10 0	µg/mL	gravimetric
thallium	0 2	µg/mL	gravimetric
tin	0 1	µg/mL	gravimetric
titanium	0 05	µg/mL	gravimetric
vanadium	0 05	µg/mL	gravimetric
zinc	0 2	µg/mL	gravimetric

Matrix: 5% nitric acid in water

ISO-9001:2000 Registered by
Reg No 00-R1192rev 1

ISO 17025 Certified by
Cert No 0851-01

All weights are traceable to NIST traceable weights

John E Russo, Chem. Eng
Quality Control Manager

ULTRA SCIENTIFIC 250 Smith Street, North Kingstown, RI 02852 • 401-294-9400 • fax 401-295-2330 • www.ultrasci.com

P.O. Box 41727
Charleston, SC 29423
TEL: (843) 767-7900
FAX: (843) 767-7906

HIGH-PURITY STANDARDS

Certificate of Analysis

SM-606-044 (CAL STD. #2-RR)

Solution A

Lot # 417517

Source	Source Purity	Matrix	Standard Concentration
High Purity Metals Salts or Oxides	99.99+%	HNO_3, 5%	µg/mL ± 0.5% See elements listed on back

This spectrometric standard solution has been prepared from high-purity reference materials. Subboiled high-purity acid has been used to place the materials in solution and to stabilize the standard. The matrix is as noted above in 18 megaohm deionized water. The reference materials have been assayed by optical emission spectrometry and atomic absorption spectrometry.

The standard has been prepared gravimetrically by weighing the reference material to 5 significant figures. Volumetric glassware has been calibrated gravimetrically to 5 significant figures.

The Standard Concentration has been certified by spectrometric analysis against an independent source which is directly traceable to National Institute of Standards and Technology, Standard Reference Material No. 3100 series, and checked by ICP prior to shipping.

This solution is valid for a period of one year from the shipping date provided the solution is kept tightly capped and stored under normal laboratory conditions.

Theodore C. Rains

Theodore C. Rains, Ph.D.
President

Exp. Date JUL 05
MSDS ATTACHED

P.O. Box 41727
Charleston, SC 29423
TEL: (843) 767-7900
FAX: (843) 767-7906

HIGH-PURITY STANDARDS

Certificate of Analysis

SM-606-044 (CAL STD. #2-RR)

Solution B

Lot # 417517

Source	Source Purity	Matrix	Standard Concentration
High Purity Metals Salts or Oxides	99.99+%	HNO_3, 5% + Tr HF	100 µg/mL ± 0.5% Antimony Molybdenum Titanium

This spectrometric standard solution has been prepared from high-purity reference materials. Subboiled high-purity acid has been used to place the materials in solution and to stabilize the standard. The matrix is as noted above in 18 megaohm deionized water. The reference materials have been assayed by optical emission spectrometry and atomic absorption spectrometry.

The standard has been prepared gravimetrically by weighing the reference material to 5 significant figures. Volumetric glassware has been calibrated gravimetrically to 5 significant figures.

The Standard Concentration has been certified by spectrometric analysis against an independent source which is directly traceable to National Institute of Standards and Technology, Standard Reference Material No. 3100 series, and checked by ICP prior to shipping.

This solution is valid for a period of one year from the shipping date provided the solution is kept tightly capped and stored under normal laboratory conditions.

Theodore C. Rains, Ph.D.
President

JUL 0 8
Exp. Date
MSDS ATTACHED

TITLE: METHOD 6010B/ 200.7/CLP USING THE THERMO JARRELL ASH 61E TRACE

SUPERCEDES: Revision 15

ULTRA SCIENTIFIC
Analytical Solutions

Certificate of Analysis

Inorganic Custom Standard

Catalog Number: ICUS-1370

Lot Number: F00019

Job Number: J00004351

Expiration Date: 02/2006

This ULTRAgrade(TM) standard was gravimetrically prepared, and the true value listed is the concentration calculated from gravimetric and volumetric measurements performed during the preparation of the standard. The standard uncertainty is ± 0.5% relative, unless otherwise specified.

Analyte	True Value				Analytical Method
antimony	40.00	±	0.20	µg/mL	gravimetric
arsenic	40.00	±	0.20	µg/mL	gravimetric
beryllium	40.00	±	0.20	µg/mL	gravimetric
cadmium	40.00	±	0.20	µg/mL	gravimetric
chromium	40.00	±	0.20	µg/mL	gravimetric
cobalt	40.00	±	0.20	µg/mL	gravimetric
copper	40.00	±	0.20	µg/mL	gravimetric
lead	40.00	±	0.20	µg/mL	gravimetric
manganese	40.00	±	0.20	µg/mL	gravimetric
molybdenum	40.00	±	0.20	µg/mL	gravimetric
nickel	40.00	±	0.20	µg/mL	gravimetric
selenium	40.00	±	0.20	µg/mL	gravimetric
thallium	40.00	±	0.20	µg/mL	gravimetric
vanadium	40.00	±	0.20	µg/mL	gravimetric
zinc	40.00	±	0.20	µg/mL	gravimetric
titanium	40.00	±	0.20	µg/mL	gravimetric
calcium	2000	±	10	µg/mL	gravimetric
iron	2000	±	10	µg/mL	gravimetric
magnesium	2000	±	10	µg/mL	gravimetric

Matrix: 5% nitric acid in water

All weights are traceable to NIST traceable weights

Quality System
Quality Endorsed Company ISO 9001 SAI Global Registered

ACCREDITED
ISO 17025 Cert. No. 0851- 01

250 Smith Street, North Kingstown, RI 02852 USA
401-294-9400 Fax: 401-295-2330
www.ultrasci.com

Dr. Edward Fitzgerald,
Senior Scientist

STL BUFFALO
LABORATORY STANDARD OPERATING PROCEDURES

SOP No.	Revision No.	Effective Date	Page
AMV-5030-42	6	February 28, 2007	1 of 7

TITLE: METHOD 5030: PURGE AND TRAP FOR VOLATILE ORGANICS

SUPERCEDES: Revision 5

REVIEWED & APPROVED BY:	Signature	Date
Verl Preston, Quality Manager		
Christopher A. Spencer, Laboratory Director		
James J. Lis, Supervisor		

1.0 IDENTIFICATION OF TEST METHODS

1.1. Method 5030B: Purge and Trap

2.0 APPLICABLE MATRIX

2.1. This method is applicable to all aqueous samples.

2.2. Soil and solid samples should be prepared using Method 5035, however if bulk soil samples are received, this procedure may be followed if approved through the client and the project plan.

3.0 REPORTING LIMIT

3.1. Reporting Limits are specific to the determinative method.

4.0 SCOPE AND APPLICATION

4.1. This method describes sample preparation and extraction for the analysis of volatile organics by a purge and trap procedure. The gas chromatographic determinative steps are found in Methods 8260B, 624, 524.2, NYSDEC Analytical Services Protocols, and USEPA OLMO4.3.

TITLE: METHOD 5030: PURGE AND TRAP FOR VOLATILE ORGANICS

SUPERCEDES: Revision 5

5.0 SUMMARY OF TEST METHOD

5.1. An inert gas, helium is bubbled through a sample (solution) at ambient temperature and the volatile components are efficiently transferred from the aqueous phase to the vapor phase. The vapor is swept through a sorbent column where the volatile components are adsorbed. After sample purging is completed, the sorbent column is heated and backflushed with inert gas to desorb the components onto a gas chromatographic column.

6.0 DEFINITIONS

6.1. Standard definitions are found in STL Buffalo's Laboratory Quality Manual.

7.0 INTERFERENCES

7.1. Purchasing high-quality helium minimizes impurities from the purge gas (helium). The purge and trap system is highly susceptible to carryover from high level samples. Sample lines are flushed twice with volatile free water after each sampling. The trap is baked at 260 degrees C for a minimum of eight minutes.

7.2. The laboratory analyzes weekly volatile holding blanks to ensure an environment free of volatile organic solvent vapors.

7.3. Methylene chloride can permeate through a septum seal, a trip blank is carried through the sampling and handling protocols to serve as a check on such contamination.

7.4. The purge and trap system will also be demonstrated to be clean by the use of VBLKs and IBLKs.

7.5. Contamination by carryover can occur whenever a high-concentration and low-concentration samples are analyzed sequentially. Unusually high-concentration samples should be followed by an analysis of organic-free reagent water to check for cross-contamination.

8.0 SAFETY

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

8.2. *SPECIFIC SAFETY CONCERNS OR REQUIREMENTS*

8.2.1. Special precautions are taken when working with a purge and trap system. Due to the amount of gas utilized by the system, all employees are required to wear approved safety glasses. Parts of the system are under pressure, always allowing for the possibility of shattered glass.

8.3. *PRIMARY MATERIALS USED*

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

Material (1)	Hazards	Exposure Limit (2)	Signs and symptoms of exposure
Hydrochloric Acid	Corrosive Poison	5 ppm-Ceiling	Inhalation of vapors can cause coughing, choking, inflammation of the nose, throat, and upper respiratory tract, and in severe cases, pulmonary edema, circulatory failure, and death. Can cause redness, pain, and severe skin burns. Vapors are irritating and may cause damage to the eyes. Contact may cause severe burns and permanent eye damage.
Methanol	Flammable Poison Irritant	200 ppm-TWA	A slight irritant to the mucous membranes. Toxic effects exerted upon nervous system, particularly the optic nerve. Symptoms of overexposure may include headache, drowsiness and dizziness. Methyl alcohol is a defatting agent and may cause skin to become dry and cracked. Skin absorption can occur; symptoms may parallel inhalation exposure. Irritant to the eyes.
1 – Always add acid to water to prevent violent reactions.			
2 – Exposure limit refers to the OSHA regulatory exposure limit.			

8.4 The following analytes have been tentatively classified as known or suspected, human or mammalian carcinogens:

8.4.1 Benzene

8.4.2 Carbon tetrachloride

8.4.3 Chloroform

8.4.4 Vinyl chloride

TITLE: METHOD 5030: PURGE AND TRAP FOR VOLATILE ORGANICS

SUPERCEDES: Revision 5

9.0 EQUIPMENT AND SUPPLIES

9.1. Purge and trap device that consists of three parts.

9.2. Sample purger designed to accept 5ml samples and have a total volume of less than 15 mls. In low level drinking water methods 25ml sample purge vessels are utilized. A heater pocket capable of reaching 40 degrees C for soils is fitted onto the purge vessel.

9.3. A VOCARB 3000 trap ~30cm long containing the following materials is utilized for all methods:

9.3.1. 10cm Carbopack B

9.3.2. 6cm Carboxen 1000

9.3.3. 1cm Carboxen 1001

9.4. The desorber rapidly pre-heats the trap to 245 degrees C and then desorbs at 250 degrees C. The trap is then baked at 260 degrees C.

10.0 REAGENTS AND STANDARDS

10.1. Volatile free water for making sample dilutions and method blanks

10.2. Purge and trap grade methanol

11.0 SAMPLE COLLECTION, PRESERVATION, SHIPMENT AND STORAGE

11.1. Samples should be collected in 40 ml capped vials with zero headspace and stored at 4 +/-2°C until time of analysis

11.2. Aqueous samples preserved with HCl must be analyzed within 14 days of collection.

11.3. Aqueous samples not preserved with HCl must be analyzed within 7 days of collection.

11.4. Soil samples must be analyzed within 14 days of collection.

12.0 QUALITY CONTROL

12.1. A standard, MSB, and VBLK is analyzed in each run as well as a MS/SD every 20 samples.

13.0 CALIBRATION AND STANDARDIZATION

13.1. See appropriate determinative method(s).

TITLE: METHOD 5030: PURGE AND TRAP FOR VOLATILE ORGANICS

SUPERCEDES: Revision 5

14.0 PROCEDURE

14.1. Instrument Operating Conditions (Suggested)

14.1.1.	Purge temperature	<35-40ºC
14.1.2.	Desorb Temperature	250ºC
14.1.3.	Line Temperature	110ºC
14.1.4.	Purge Gas (Helium)	40ml/min.
14.1.5.	Purge Total Time	11min.
14.1.6.	Desorb Time	2min.

14.2. Instrument Maintenance

14.2.1. Upon verification of established operating conditions, the following is performed on a sequence basis:

14.2.1.1. check purge flow;

14.2.1.2. analyze blank to insure system is free of contamination (daily).

14.2.1.3. vessel and lines are flushed three times after each analysis.

14.3. Note: System must be leak free. System can be checked by purging 5mls water in sample vessel and capping off vent on purge device. If purge flow stops system is leak free, if purge flow continues (within 2-3 minutes) this means there is a leak within the system. Leak must be located and corrected.

15.0 CALCULATIONS NA

16.0 METHOD PERFORMANCE

16.1. MDLs are performed yearly, per analytical method, and kept on file with the Quality Department.

17.0 DATA ASSESSMENT AND ACCEPTANCE CRITERIA FOR QUALITY CONTROL MEASURES

17.1. See appropriate determinative method(s).

18.0 CORRECTIVE ACTIONS FOR OUT-OF-CONTROL DATA

18.1. If the standard fails, rerun calibration curve.

18.2. If MSB or VBLK fails, re-analyze samples.

19.0 CONTINGENCIES FOR HANDELING OUT-OF-CONTROL OR UNACCEPTABLE DATA

19.1. Inform project manager for client input and fill out job exception report.

19.2. Rerun samples to confirm results.

19.3. Resample if client or project manager requests.

20.0 WASTE MANAGEMENT/ POLLUTION PREVENTION

20.1. 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."

20.2. *Waste Streams Produced by the Method*

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

20.3.1. Acidic material from the auto-sampler: Waste stream must be collected in "A" waste receptacles and neutralized before discharge to a sewer system.

20.3.2. Methanol waste from rinses and standards: Collect in "C" waste receptacles. In the case of medium level soil extractions, the methanol is decanted off the soil and collected in the "C" receptacle. Waste receptacles are then taken to sample control where they are properly disposed of.

20.3.3. Excess samples (acidic and non-acidic). Collect in "A" waste receptacles and neutralize samples before disposal into drain/sewer.

20.3.4. Excess soil sample from medium level extraction: Wrap in tin foil and place in solid waste receptacle. Soils for dry weight measurements are also disposed in this manner.

TITLE: METHOD 5030: PURGE AND TRAP FOR VOLATILE ORGANICS

SUPERCEDES: Revision 5

21.0 REFERENCE

21.1. U.S. EPA 40 CFR Part 136, "Guidelines Establishing Test Procedures for the Analysis of Pollutants Under the Clean Water Act; Final Rule and Interim Final Rule and Proposed Rule," October 26, 1984

21.2. U.S. EPA "Method 5030B, Purge and Trap for Aqueous Samples", Test Methods for Evaluating Solid Waste, Volume 1B, Revision 2, December 1996.

21.3. U.S. EPA "Method 5030A, Purge and Trap for Aqueous and Soil Samples", Test Methods for Evaluating Solid Waste, Update 11B, January 1995.

22.0 TABLES, DIAGRAMS, FLOWCHARTS AND VALIDATION DATA NA

23.0 CHANGES FROM PREVIOUS REVISION

23.1. Section 11.3: Clarified hold time for non-preserved aqueous samples.

STL BUFFALO
LABORATORY STANDARD OPERATING PROCEDURES

SOP No.	Revision No.	Effective Date	Page
AMV-8260B-56	8	May 31, 2006	1 of 62

TITLE: ANALYTICAL METHODS FOR THE ANALYSIS OF GC\MS VOLATILE SAMPLES 8260B

SUPERCEDES: Revision 7

REVIEWED & APPROVED BY:	Signature	Date
Verl Preston, Quality Manager		
Christopher Spencer, Laboratory Director		
James Lis, Supervisor		

1.0 IDENTIFICATION OF TEST METHODS

1.1. Methods 8260B -5 mL aqueous purge, 8260B - 25mL aqueous purge, 8260B – 5gr soil and 8260B – medium level soil.

2.0 APPLICABLE MATRIX

2.1. Applicable matrices include all aqueous samples, sediment, and soil.

3.0 REPORTING LIMIT

3.1. The standard reporting limit (RL) is established at or above the lo-level standard in the calibration curve. For a 5-ml purge volume, the RL for the majority of compounds is 1 ug/l.

4.0 SCOPE AND APPLICATION

4.1. The analytical method is utilized for the analysis of water, sediment and soil from hazardous waste sites for the organic compounds listed in table 1.

4.2. The method includes sample preparation and analyses by purge and trap gas chromatograph/mass spectrometer (GC/MS). Method can be used for 5mL purge or 25mL purge (concentrations adjusted accordingly).

TITLE: ANALYTICAL METHODS FOR THE ANALYSIS OF GC\MS VOLATILE SAMPLES 8260B

SUPERCEDES: Revision 7

5.0 SUMMARY OF TEST METHOD

5.1. Volatile compounds are extracted from sample matrix by the purge and trap method. Analytes are desorbed onto a capillary column. An appropriate ramping temperature program is applied to maximize separation and achieve the correct resolution between the analytes. A mass spectrometer detector (MSD) interfaced to the gas chromatograph (GC) is utilized to detect analytes of interest.

5.2. Analytes eluted from the capillary column are introduced into the mass spectrometer via a jet separator or a direct connection. Identification of target analytes is accomplished by comparing their mass spectra with the electron impact spectra of authentic standards. Quantitation is accomplished by comparing the response of a major (quantitation) ion relative to an internal standard using a five-point calibration curve.

6.0 DEFINITIONS

6.1. VBLK - Volatile blank: VBLK's are made from laboratory produced volatile free water. They are analyzed before samples to ensure a clean laboratory environment and analytical system.

6.2. IBLK – Instrument Blank: IBLK's are made from laboratory produced volatile free water. They are analyzed after high level samples to verify that the system is clean and demonstrate the absence of carryover.

7.0 INTERFERENCES

7.1. Airborne contamination may result from solvent vapors. VBLKs and IBLKs will be utilized to demonstrate a clean system and laboratory environment.

7.2. Some volatile compounds can permeate through a sample septum seal during storage or shipment. A weekly volatile holding blank is stored will all samples in the sample incubator to monitor contamination.

7.3. Contamination by carryover can occur whenever a sample with high concentrations of target compounds precedes a sample with low levels. The purging device, syringe and lines are flushed between every analysis to reduce carry over contamination. The trap is baked at 260^0 C between each analysis.

8.0 SAFETY

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

TITLE: ANALYTICAL METHODS FOR THE ANALYSIS OF GC\MS VOLATILE SAMPLES 8260B

SUPERCEDES: Revision 7

8.2. *SPECIFIC SAFETY CONCERNS OR REQUIREMENTS*

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

8.2.2. The mass spectrometer is under deep vacuum. The mass spectrometer must be brought to atmospheric pressure prior to working on the source.

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

8.3. PRIMARY MATERIALS USED

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

Material (1)	Hazards	Exposure Limit (2)	Signs and symptoms of exposure
Methanol	Flammable Poison Irritant	200 ppm-TWA	A slight irritant to the mucous membranes. Toxic effects exerted upon nervous system, particularly the optic nerve. Symptoms of overexposure may include headache, drowsiness and dizziness. Methyl alcohol is a defatting agent and may cause skin to become dry and cracked. Skin absorption can occur; symptoms may parallel inhalation exposure. Irritant to the eyes.
1 – Always add acid to water to prevent violent reactions.			
2 – Exposure limit refers to the OSHA regulatory exposure limit.			

9.0 EQUIPMENT AND SUPPLIES

9.1. Glassware

9.1.1. Syringes - Hamilton Syringes size, 10ul, 25ul, 50ul, 100ul, 500ul, 1ml, 5ml, 10ml, 25ml

9.1.2. Pasteur Pipets – disposable

9.1.3. Vials and caps - 2ml disposable

9.1.4. Vials and caps - 40ml disposable

9.1.5. Volumetric flasks - pyrex 2ml, pyrex 10ml, pyrex 50ml, pyrex 100ml

9.2. pH paper - wide range -.EM Science

9.3. Analytical Balance Mettler - Toledo Inc. Mettler AE160

9.4. Purge and trap devices

9.4.1. PTA-30 W/S by Dynatech Autosampler

9.4.2. Tekmar 2000 Concentrator

9.4.3. Varian Archon Autosampler

9.4.4. Tekmar 3000 Concentrator

9.4.5. Encon Concentrator

9.5. Trap Packing - Supelco Vocarb 3000

9.5.1. Packing Material:

9.5.1.1. 10cm Carbpack B

9.5.1.2. 6cm Carboxen 1000

9.5.1.3. 1cm Carboxen 1001

9.6. Gas Chromatograph/Mass Spectrometer (GC/MS) - GC: HP5890, MS: Finnigan INCOS 50 and Finnigan INCOS 50XL

9.7. Gas chromatograph Column - J&W Scientific DB-624 or Phenomenex ZB-624

9.7.1. Internal diameter: 0.53mm

9.7.2. Length: 75m

9.7.3. Coating: Cyanopropylphenyl Methyl Silicone

9.7.4. Film thickness: 3.0µm

9.8. Data System -

9.8.1. Finnigan INCOS software/Data General DG-10 computer

9.8.2. Dell computer with Teknivent Vector/Warp enviroquant software

9.8.3. Gas Chromatograph/Mass Spectrometer (GC/MS) - GC: HP6890, MS: Hewlett-Packard/ Agilent 5973N

9.9. Gas chromatograph Column - J&W Scientific DB-624 or Phenomenex ZB-624

9.9.1. Internal diameter: 0.25mm or .18mm

9.9.2. Length: 60m or20m

9.9.3. Coating: Cyanopropylphenyl Methyl Silicone

9.9.4. Film thickness: 1.4µm or 1µm

9.10. Data System - Hewlett-Packard Kayak XM600 computer with Chemstation software.

10.0 REAGENTS AND STANDARDS

10.1. Reagent Water - For volatile analysis, the reagent water is volatile free and is prepared by passing water through a carbon trap.

10.2. Methanol - Burdick & Jackson, purge and trap grade

10.3. Stock Standards- Are purchased as certified standard mixtures. Traceability is documented following the procedures in the "Standards Traceability and Preparation Logbooks" SOP# AGP-STD-14. Individual compounds are prepared using reagent grade chemicals following the "Primary Standards Preparation" SOP# AMV-STD-25.

10.3.1. Stock Target Compound Mix – Is composed of three different mixtures.

10.3.1.1. The Gas Mix (See Table 6 for component list) is purchased from Supelco (or equivalent vendor) at a concentration of 2000ug/ml.

10.3.1.2. The 54 Component Mix (See Table 7 for component list) is purchased from Supelco (or equivalent vendor) at a concentration of 2000ug/ml.

10.3.1.3. The 8260+ Mix (See Table 8 for component list) is purchased from Restek (or equivalent vendor) and is composed of four separate mixtures.

10.3.1.3.1. 8260+ Mix #1 is purchased at a concentration of 1000ug/ml.

10.3.1.3.2. 8260+ Mix #2 is purchased at a concentration of 5000ug/ml.

10.3.1.3.3. 8260+ Mix #3 is purchased at a concentration of 20000ug/ml.

10.3.1.3.4. 8260+ Mix #4 is purchased at a concentration of 5000ug/ml.

10.3.2. Stock Calibration Verification Mix – Is composed of two different mixtures.

10.3.2.1. The Second Source Mix (See Table 9 for component list) is purchased from Ultra (or equivalent vendor) at a concentration of 2000ug/ml.

10.3.2.2. The 8260+ Second Source Mix (See Table 10 for component list) is purchased from Supelco (or equivalent vendor) and is composed of two separate mixtures.

10.3.2.2.1. 8260+ Second Source Mix #1 is purchased at a concentration of 1000ug/ml.

10.3.2.2.2. 8260+ Second Source Mix #2 is purchased at a concentration of 5000ug/ml.

10.3.3. Stock Internal Standard Solution – A mixture of 1,4-Dichlorobenzene-d4, Chlorobenzene-d5 and 1,4-Difluorobenzene in Methanol is purchased from Restek (or equivalent vendor) at a concentration of 2500ug/ml.

10.3.4. Stock System Monitoring Solution – A mixture of Dibromofluoromethane, Toluene-D8, 4-Bromofluorobenzene and 1,2-Dichloroethane-d4 in Methanol is purchased from Ultra (or equivalent vendor) at a concentration of 2500ug/ml.

10.3.5. Stock Matrix Spike Solution – A 5 component mixture of 1,1-dichloroethene, trichloroethene, chlorobenzene, toluene, and benzene in Methanol is purchased from Restek (or equivalent vendor) at a concentration of 2500ug/ml.

10.3.6. Stock BFB Solution – A solution of 4-Bromofluorobenzene in Methanol is purchased from Supelco (or equivalent vendor) at a concentration of 25000ug/ml.

10.4. Secondary IS and System Monitoring Calibration Dilution Standards – these solutions are used for the manual injections required to prepare the initial calibration.

10.4.1. Internal Standard Solution: 80ul of stock standard IS solution (2500ug/ml) is added to approximately 1 ml of purge and trap grade methanol in a 2 ml Class A volumetric, and then brought up to final volume of 2 ml with additional purge and trap grade methanol for a final concentration of 100ng/ul.

10.4.2. System Monitoring Compound Solution: 80ul of stock standard Surrogate solution (2500ug/ml) is added to approximately 1 ml of purge and trap methanol in a 2 ml Class A volumetric, and then brought up a final volume of 2ml with additional purge and trap grade methanol for a final concentration of 100ng/ml.

10.4.3. To calculate appropriate expiration dates, refer to "Standards Traceability and Preparation Logbooks", SOP No. AGP-STD-14.

10.5. Working Standards

10.5.1. Intermediate Calibration Solution: (Three individual mixtures)

10.5.1.1. 250ul of stock standard Gas Mix solution (2000ug/ml) is added to approximately 4 ml of purge and trap methanol in a 5ml Class A volumetric, and then brought up a final volume of 5ml with additional purge and trap grade methanol for a final concentration of 100ng/ul.

10.5.1.2. 500ul of stock standard 54 Component Mix solution (2000ug/ml) is added to approximately 9ml of purge and trap methanol in a 10ml Class A volumetric, and then brought up a final volume of 10ml with additional purge and trap grade methanol for a final concentration of 100ng/ul.

10.5.1.3. 1000ul of each of the four stock standard 8260+ Mixes are added to approximately 9ml of purge and trap methanol in a 10ml Class A volumetric, and then brought up a final volume of 10ml with additional purge and trap grade methanol.

10.5.2. Matrix Spike Solution: 100ul of stock standard 5 component solution (2500ug/ml) is added to approximately 4 ml of purge and trap methanol in a 5 ml Class A volumetric, and then brought up a final volume of 5ml with additional purge and trap grade methanol for a final concentration of 50ng/ul.

10.5.3. A Full List Matrix Spike standard is made from stock Calibration Verification Standards and is composed of two mixes.

10.5.3.1. 250ul of stock standard Gas Mix solution (2000ug/ml) is added to approximately 4 ml of purge and trap methanol in a 5ml Class A volumetric, and then brought up a

final volume of 5ml with additional purge and trap grade methanol for a final concentration of 100ng/ul.

10.5.3.2. 200ul of each of the two stock standard 8260+ Second Source Mixes are added to approximately 1ml of purge and trap methanol in a 2ml Class A volumetric, and then brought up a final volume of 2ml with additional purge and trap grade methanol.

10.5.4. Working Internal Standard and System Monitoring Compound Solutions – for autoinjection by instrument.

10.5.4.1. Working Internal Standard Solution: An Internal Standard Mixture is made from IS stock standard (2500ug/ml) at 140ng/ul to 175ng/ul, depending on sample loop size, for the Dynatek autosampler and is made at 220ng/ul to 280ng/ul, depending on sample loop size, for the Varian Archon autosampler.

10.5.4.2. Working System Monitoring Calibration Solution: A System Monitoring Compounds Mixture is made from Surrogate stock standard (2500ug/ml) at 140 ng/ul to 175 ng/ul depending on sample loop size, for the Dynatek autosampler and is made at 220ng/ul to 280ng/ul, , depending on sample loop size, for the Varian Archon autosampler.

10.5.5. Tuning Mixture: 4ul of stock solution 4-Bomofluorobenzene (BFB) tuning mixture is added to approximately 1 ml of purge and trap grade methanol in a 2 ml Class A volumetric, and then brought up to final volume of 2 ml with additional purge and trap grade methanol for a final concentration of 50ng/ul.

10.5.6. Working Initial Calibration Standards

10.5.6.1. Water: 25 ml

10.5.6.1.1. 20ul, 10ul and 5ul each of Intermediate Calibration Solution (10.5.1) is added to reagent water in each of three 50ml volumetric flasks. 20ul, 10ul and 5ul each of the Secondary IS Calibration Dilution Solution and also the Secondary System Monitoring Calibration Dilution Solution are added to the respective 50 ml volumetric flasks. The flasks are brought to volume with reagent water to prepare the 40, 20 and 10 ug/L standards respectively.

10.5.6.1.2. 4ul and 1ul each of Intermediate Calibration Solution ((10.5.1) plus 4ul and 1ul each of the Secondary IS Calibration Dilution Solution and also the Secondary System Monitoring Calibration Dilution Solution are added to reagent water in 100 ml volumetric flasks. The flasks are brought to volume with reagent water to prepare the 4f and 1 ug/L standards respectively.

10.5.6.1.3. Each standard is then transferred into a 40ml vial and loaded onto the Dynatech or Archon autosampler.

10.5.6.2. Water: 5 ml

10.5.6.2.1. 100ul, 50ul, 25ul, 10ul and 2.5ul each of Intermediate Calibration Solution (10.5.1) plus the same amounts of Secondary IS Calibration Soluti9on and Secondary System Monitoring Calibration Solution are (100ng/ml) are added to five individual 50ml volumetric flasks continuing reagent water to prepare 200, 100, 50, 20 and 5 ug/L standards respectively.

10.5.6.2.2. Each standard is then transferred into a 40ml vial and loaded onto the Dynatech or Archon autosampler.

10.5.6.3. Soil:

10.5.6.3.1. 100ul, 50ul, 25ul, 10ul and 2.5ul of Intermediate Calibration Solution (10.5.1) plus the same amounts of Secondary IS Calibration Solution and Secondary System Monitoring Calibration Solution (at 100ng/ml) are added to five individual 50ml volumetrics containing reagent water. The final concentration of each standard is 200, 100, 50, 20 and 5 ug/kg, respectively.

10.5.6.3.2. 5 ml of each standard is then transferred into five individual 40ml vials and loaded onto the Archon autosampler.

10.5.7. Continuing Calibration Standard

10.5.7.1. Water: 25 ml

10.5.7.1.1. 5ul of stock target compound mix is added to 50ml of DI water to make a final concentration of 10ppb. Pour standard into 40ml vial; working standard internal standard and system monitoring compounds are added by Dynatech or Archon autosampler.

10.5.7.2. Water: 5 ml

10.5.7.2.1. 25ul of stock target compound mix is added to 50ml of DI water to make a final concentration of 50ppb. Pour standard into 40ml vial; working standard internal standard and system monitoring compounds are added by Dynatech or Archon autosampler.

10.5.7.3. Soil:

10.5.7.3.1. 25ul of stock target compound is added to 50ml of DI water to make a final concentration of 50ppb.. Take 5ml and transfer it into a 40ml; working standard internal standard and system monitoring compounds are added by Archon autosampler.

10.6. Storage of standards

10.6.1. Stock and secondary dilution standards are stored in teflon-sealed crimp cap vials at -10^0 C to -20^0 C.

10.6.2. Aqueous standards are stored in teflon-sealed crimp cap bottles at 4^0 C plus or minus 2^o C.

11.0 SAMPLE COLLECTION, PRESERVATION, SHIPMENT AND STORAGE

11.1. Samples are collected in 40 mL vials with caps and septa, preserved to a pH < 2 with Hydrochloric Acid and stored at 4 ± 2 degrees C until time of analysis.

11.2. Holding time for unpreserved samples is 7 days from sample date. For preserved samples the holding time is 14 days from sample date.

11.3. For some clients, regulatory agencies or QAPPS, the specified holding times may be different than those described in 11.2. In those cases, consult the specific Protocol/Method/QAPP or Project Manager for holding time details.

11.4. Sample Storage

11.4.1. Volatile samples are stored at 4 ± 2^0C from the time of collection until analysis.

11.4.2. Volatile samples are stored together in refrigerators specifically designated for volatiles only.

11.4.3. Storage blanks are stored with samples until analysis.

11.4.4. Samples and extracts are stored separately.

11.4.5. Volatile samples and standards are stored separately.

TITLE: ANALYTICAL METHODS FOR THE ANALYSIS OF GC\MS VOLATILE SAMPLES 8260B

SUPERCEDES: Revision 7

11.5. Preparation Of MS/MSD Samples

11.5.1. Water Samples: 40ml vial is spiked with 8ul of 50ng/ul matrix spike standard for 25ml purge and 40ul for the 5ml purge. This corresponds to a final concentration in the samples of 10 ug/L and 50 ug/L respectively. Analysis proceeds according to procedures described for water analysis.

11.5.2. Low Level Soil/Sediment Samples: 5ul of matrix spiking solution is added to a 5g aliquot of sample. This corresponds to a final concentration in the samples of 50 ug/kg. Analysis proceeds according to procedures described for low-level soil/sediment samples.

11.5.3. Medium Level Soil/Sediment Samples: 1ml of methanol containing the soil extraction is combined with 50 mls of water and 50 ul of spiking solution is added to the water methanol extraction solution. Sample analysis proceeds according to procedures described for medium level soil/sediment samples.

12.0 QUALITY CONTROL

12.1. Blank Analysis:

12.1.1. Method Blank: A method blank consisting of a clean reference matrix (reagent water or purified quartz sand) must be analyzed prior to the analysis of samples but following any standard analysis.

12.1.1.1. Target compounds detected in a method blank must fall below the reporting limit, unless specified in client QAPP.

12.1.1.2. If internal standard or systems monitoring compound recoveries are not met, the method blank must be reanalyzed before the analysis of samples.

12.1.2. Storage (Holding) Blank: A weekly holding blank is analyzed to determine if cross contamination occurs within the volatile holding area. The results are reviewed by the quality assurance department and deemed acceptable or not acceptable. Corrective action, if necessary, will be taken.

12.1.3. Instrument Blank: An instrument blank consisting of a clean reference matrix analyzed after the analysis of samples containing target compounds which exceed the calibration range. Multiple instrument blanks are shot until the instrument blank meets the criteria for method blanks.

12.2. Matrix Spike Blank (MSB/LCS): An aliquot of clean reference material spiked with the matrix spiking solution is analyzed with each analytical batch. The standard from which the MSB/LCS is prepared is purchased from an alternate vender from the continuing (CCV) standard. The

solution is spiked at a concentration of 10ug/L for 25ml analysis and 50ug/L for 5ml analysis. A matrix spike blank duplicate is performed when insufficient volume is available for sample specific MS/MSD quality control.

12.2.1. The MSB/LCS must fall within internally derived statistical control limits or where applicable the limits specified by a project QAPP.

12.2.2. Routine compounds included in the MSB/LCS are:

1,1-Dichloroethene
Chlorobenzene
Toluene
Benzene

12.2.3. When required, the MSB/LCS a 'full-compound' spike will be prepared and the MSB/LCS will be spiked with all compounds of interest. Due to the potentially large number of target compounds for method 8260B, it is possible that a couple of spiking compound could fall outside limits in the MSB/LCS. If a compound falls outside limits biased high and that compound is not found in the samples, a comment will be made in the case narrative and the data will be found to be acceptable.

12.2.4. If the results of sample matrix spikes fall outside of the quality control range due to matrix, the MSB is used to verify that the laboratory can perform a spike on a clean matrix.

12.3. Matrix Spike And Matrix Spike Duplicate Analysis: A matrix spike and matrix spike duplicate consisting of an actual field sample which has been spiked with the matrix spiking solution.

12.3.1. Matrix spike and matrix spike duplicate analysis will not be performed on rinsates or field/trip blanks.

12.3.2. If a sample has not been designated for MS/MSD analysis by the client, a sample will be selected at the analyst's discretion. MS/MSD analysis will be performed at a minimum of every 20 samples.

12.3.3. If insufficient sample was received for a designated MS/MSD the client will be contacted with the laboratory's in-house designated sample for MS/MSD analysis. If no MS/MSD is required, the instance will be documented in the SDG narrative.

12.3.4. If medium level analysis is required on the client designated sample, the laboratory analyst will choose a low level sample on which to perform the quality control analysis. Medium level QC will also be performed.

TITLE: ANALYTICAL METHODS FOR THE ANALYSIS OF GC\MS VOLATILE SAMPLES 8260B

SUPERCEDES: Revision 7

13.0 CALIBRATION AND STANDARDIZATION

13.1. Instrument Tuning and Performance Check:

13.1.1. The GC/MS system is calibrated using Perflurotributylamine (PFTBA) according to the recommended tuning conditions suggested by the vendor.

13.1.2. An instrument performance check of Bromofluorobenzene (BFB) is analyzed at the beginning of each 12-hour analysis period.

13.1.2.1. The analysis of the instrument performance check is performed using the following procedure:

13.1.2.1.1. 1ul of a 50ng/ul solution is directly injected, resulting in a 50ng injection of BFB into the GC/MS.

13.1.2.1.2. A blank containing 50 ng BFB is purged.

13.1.2.2. The mass spectrum of BFB is acquired using the following procedure:

13.1.2.2.1. A single scan on the peak.

13.1.2.2.2. An average of the peak.

13.1.2.2.3. The apex scan, one scan immediately preceding the apex and one scan immediately following the apex are averaged. The spectrum is background subtracted using a single scan no more than 20 scans prior to the elution of BFB.

13.1.2.3. The mass spectrum of BFB must pass the technical acceptance criteria given in Table 2.

13.2. Initial Calibration (ICAL):

13.2.1. The instrument performance check must meet the technical acceptance criteria prior to the analysis of an initial curve or samples. The GC/MS system is calibrated using five levels of concentrations. All compounds of interest are included. (See section 17.0 for initial calibration acceptance criteria.)

13.2.2. Solutions containing target compounds and system monitoring compounds are analyzed at concentrations of 5, 20, 50, 100 and 200 ug/L. (1, 4, 10, 20 and 40 ug/L for 25 mL)

5 ml (aqueous) or 5 gram (soil) Purge Analysis

Standard	Solvent	Working Standard Conc.	Amount Added (ul)	Final Vol. (ml)	Final Conc. Water (ug/L)	Final Conc. Soil (ug/kg)
VSTD005	MeOH	100ng/ul	5	100	5	5
VSTD020	MeOH	100ng/ul	10	50	20	20
VSTD050	MeOH	100ng/ul	25	50	50	50
VSTD100	MeOH	100ng/ul	50	50	100	100
VSTD200	MeOH	100ng/ul	100	50	200	200

25 ml Purge Analysis

Standard	Solvent	Working Standard Conc.	Amount Added (ul)	Final Vol. (ml)	Final Conc Water (ug/L)
VSTD001	MeOH	100ng/ul	1	100	1
VSTD004	MeOH	100ng/ul	4	100	4
VSTD010	MeOH	100ng/ul	5	50	10
VSTD020	MeOH	100ng/ul	10	50	20
VSTD040	MeOH	100ng/ul	20	50	40

13.3. Continuing Calibration Verification (CCV):

13.3.1. Every 12 hours of sample analysis the laboratory must demonstrate that the instrument has drifted or changed minimally by performing an instrument performance check and continuing calibration verification. (See section 17.0 for continuing calibration acceptance criteria.)

14.0 PROCEDURE

14.1. Once initial calibration criteria has been met, and prior to analyzing samples and required blanks, Each GC/MS system must be routinely checked by analyzing a Continuing Calibration Verification (CCV) standard containing all compounds (including internal standards and system monitoring compounds) at a concentration of 50ug/L for 5ml or 10ug/L for 25ml analysis.

14.1.1. If time remains after initial calibration criteria have been met, it may not be necessary to perform a CCV. The 50 ug/L (10ug/L for 25ml) standard may be evaluated against the new initial curve and used as the CCV.

14.1.2. If there is no time remaining in the 12-hour period, the instrument performance check (BFB) must be analyzed along with a new CCV.

14.1.3. Procedure for Continuing Calibration: 25uls of internal standards, system monitoring compounds, and target compound mixture is added to a 50ml volumetric flask. A 5ml

aliquot is analyzed. 25ml purge analysis requires 5ul into a 50 ml volumetric flask filled with volatile free water.

14.2. Sample Analysis

14.2.1. BFB tuning criteria and GC/MS calibration verification must be met before sample analysis begins.

14.2.2. The acquisition time of the BFB tune establishes a 12hr. batch. The CCV, MSB, and VBLK must be analyzed within 12hrs, unless specified by the client request. The remaining time in the 12hr batch is utilized to run samples of similar matrix. All aqueous samples are considered a water matrix. All solid samples, with the exception of sludges, are considered soil matrix. Sludges are run medium level.

14.2.3. Samples and standard solutions are brought to ambient temperature before analysis.

14.2.4. Prior to the analysis of samples, a method blank must be analyzed in accordance with the associated procedures for a given matrix. Technical criteria for method blanks must be met prior to sample analysis.

14.3. Water Samples (See also SOP No. AMV-5030-42)

14.3.1. A 5ml sample aliquot is spiked with internal and system monitoring compounds to a final concentration of 50 ug/L each. 25ml analysis requires a final concentration of 10ug/L. The spike may be performed manually with a Hamilton gas tight syringe or the Dynatech/Archon auto sampler may be used. The sample is then loaded onto the auto sampler where it is in turn transferred to the purge chamber.

14.3.2. The sample is purged for 11.0 $\pm$ 1 minute at ambient temperature.

14.3.3. At the end of the purge time, the sample is desorbed onto the gas chromatograph column by rapidly heating the trap to 250^0C while the trap is back flushed with Helium between 20 - 60 ml/minute for two minutes. The sample is desorbed onto the column and the gas chromatograph temperature ramping program is commenced.

14.3.4. While the trap is in the bake mode, the purge chamber is flushed with two 5ml aliquots of reagent water in order to avoid possible contamination from carryover of target compounds.

14.3.5. After the sample has desorbed, the trap is conditioned at 260^0C for 8 minutes. After baking, the trap is ready for the next sample.

14.3.6. Dilutions may be necessary if the concentration of any target compound exceeds the working range of the calibration.

14.3.7. In the event that a dilution is required, a measured volume of sample is added to a volumetric flask then to volume with reagent water and inverted 3 times. The sample in the neck portion is discarded and a 5ml volume is taken for analysis. Analysis may then proceed as described.

14.4. Low Level Soil/Sediment Samples (See also SOP No. AMV-5035-43)

14.4.1. The low level soil method is based on a heated purge of a 5g sample mixed with reagent water containing a final concentration of 50 ug/L of internal and system monitoring compounds.

14.4.2. If a dilution of the soil/sediment is required, a smaller portion of soil may be used. The smallest amount of soil that may be used is 1g. If a higher dilution is required, the sample must be analyzed as a medium level soil/sediment.

14.4.3. Initial and continuing calibrations that are used for the quantitation of low soils/sediments are analyzed using the same purge and trap conditions as samples.

14.4.4. The sample consists of the entire contents of the sample container. The contents are mixed thoroughly with a narrow metal spatula. A 5g portion is taken for analysis. The weight is recorded to the nearest 0.01g.

14.4.5. A 5ml aliquot of reagent water containing internal standards and system monitoring compounds is added to the sample immediately prior to heating and purging.

14.4.6. After reagent water is added, the soil/sediment sample is heated to $40^0C \pm 1^0C$ then purged for 11 ± 1 minutes.

14.4.7. After purging, the sample is subjected to desorbing as described for water analysis.

14.5. Medium Level Soil/Sediment Samples

14.5.1. The medium level soil/sediment methods are based on an extraction of a portion of the sample with methanol. A portion of the extract is then added to a 5 ml aliquot of reagent water containing internal and system monitoring compounds at a final concentration of 50ug/L.

14.5.2. The sample consists of the entire contents of the sample container. The contents are thoroughly mixed with a thin metal spatula. A 4 g portion of the sample is weighed into a 20ml vial. The weight of the sample is recorded to the nearest 0.01g.

14.5.3. A 10ml aliquot of methanol is quickly added to the sample. The vial is capped and the sample is shaken for 2 minutes.

14.5.4. A determined amount of methanol extract is added to a 5ml aliquot of reagent water containing internal and system monitoring compounds at a final concentration of 50ug/L. Analysis may proceed according to procedures described for water samples.

14.5.5. Table 3 may be used to determine the volume of methanol extract required for a given dilution factor.

14.6. pH Determinations For Water Samples

14.6.1. After the sample aliquots are taken from the VOA vials, the pH of the sample is determined by placing several drops of sample, using a disposable pipet, onto pH paper. A checkmark will be entered in the injection logbook if the sample pH is <2, however if the sample demonstrates a pH>2, the actual pH will be noted in the injection logbook..

14.7. Percent Moisture Determinations

14.7.1. Immediately after weighing the sample for analysis, a 5-10g portion is weighed into a tared aluminum weigh pan. The sample is then dried overnight at 105^0C. The sample is allowed to cool. The final weight is recorded. Using equation 4, the percentage moisture, which is used for reporting concentrations relative to the dry weight of the soil/sediment samples, may be determined. The following calculation is used to determine percent moisture:

$$\%\text{moisture} = \frac{\text{Wt. of sample (g)} - \text{wt. of dried sample (g)}}{\text{wt. of sample (g)}} \times 100$$

15.0 CALCULATIONS

15.1. Calculations For MS/MSD Samples

15.1.1. The calculations to determine concentrations are the same equations described for sample analysis of a given matrix.

15.1.2. The percent recovery of the matrix spiking compounds is determined using equation:

$$\text{Matrix Spike Recovery} = \frac{SSR - SR}{SA} \times 100$$

Where,
SSR = Spiked sample result
SR = Sample results
SA = Spike added

15.1.3. The relative percent difference (RPD) of the recoveries of each compound between the matrix spike and matrix spike duplicate is determined using equation:

$$RPD = \frac{|MSR - MSDR|}{\frac{1}{2}(MSR + MSDR)} \times 100$$

Where,
MSR = Matrix spike recovery
MSDR = Matrix spike duplicate recovery

15.2. Calculations For Initial Calibration

15.2.1. The relative response factor (RRF) for each target compound and each system monitoring compound is calculated using equation.

$$RRF = \frac{Ax}{Ais} \times \frac{Cis}{Cx}$$

Where,
Ax = Area of the characteristic ion (EICP) for the compound to be measured (see Table 6)
Ais = Area of the characteristic ion (EICP for the specific internal standard (see Tables 5 and 6A)
Cis = Concentration of the internal standard
Cx = Concentration of the compound to be measured

15.2.2. The relative response factor of the Xylenes requires the use of the area response and the concentration of the peak that represents the single isomer.

15.2.3. The relative response factor of 1,2-dichloroethene is calculated using the sum of the areas of both isomers and the sum of the concentrations.

15.2.4. The average response factor (RRF) is calculated for all compounds of interest.

15.2.5. The relative standard deviation (% RSD) is calculated over the working range of the curve for all compounds using equation:

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

$$\text{Standard Deviation} = \sqrt{\frac{\sum_{i=1}^{n}(\chi i - \bar{\chi})2}{n-1}}$$

Where,
Xi = each individual value used to calculate the mean
X = the mean of n values
n = the total number of values

15.3. Calculations For Continuing Calibration

15.3.1. The relative response factor (RRF) for all target compounds and system monitoring compounds is calculated using equation 1.

15.3.2. The percent difference between the initial calibration and the continuing calibration is determined for all target compounds and system monitoring compound using equation:

$$\%\text{Difference} = \frac{\text{RRFc} - \overline{\text{RRFi}}}{\overline{\text{RRFi}}} \times 100$$

Where,
RRFc = Relative response factor from continuing calibration standard

$\overline{\text{RRFi}}$ = Mean relative response factor from the most recent initial calibration meeting technical acceptance criteria

15.4. Percent Moisture Determinations

15.4.1. Immediately after weighing the sample for analysis, a 5-10g portion is weighed into a tared aluminum weigh pan. The sample is then dried overnight at 105^0C. The sample is allowed to cool. The final weight is recorded. Using the equation for % moisture, concentrations relative to the dry weight of the soil/sediment samples, may be determined.

$$\%\text{moisture} = \frac{\text{g of wet sample - g of dry sample}}{\text{g of wet sample}} \times 100$$

15.5. Quantitation of volatile target compounds is done using the internal standard method. The internal standards used for each compound are assigned those indicated in table 5. The Internal Standard RRF of the continuing calibration is used in the quantitation calculation.

TITLE: ANALYTICAL METHODS FOR THE ANALYSIS OF GC\MS VOLATILE SAMPLES 8260B

SUPERCEDES: Revision 7

15.5.1. Water Samples: The following equation is used to calculate water samples:

$$\text{Concentration ug/L} = \frac{(Ax)\,(Is)\,(DF)}{(Ais)\,(RRF)\,(Vo)}$$

Where,

Ax = Area of the characteristic ion (EICP) for the compound to be measured (see Table 2)

Ais = Area of the characteristic ion (EICP) for the specific internal standard (see Tables 5 and 6A)

Is = Amount of internal standard added in nanograms (ng)

RRF= Relative response factor from the ambient temperature purge of the calibration standard.

Vo = Volume of water purged in milliliters (mL)

Df = Dilution factor. The dilution factor for analysis of water samples for volatiles by this method is defined as the ratio of the number of milliliters (mL) of water purged (i.e., Vo above) to the number of mL of the original water sample used for purging. For example, if 2.0 mL of sample is diluted to 5 mL with reagent water and purged, Df = 5 mL/2.0 mL = 2.5. If no dilution is performed, Df = 1.

15.5.1 Low Level Soil/Sediment Samples

15.5.1.1 The following equation is used for low level soil/sediment samples:

$$\text{Concentration ug/Kg (dry weight basis)} = \frac{(Ax)\,(Is)}{(Ais)\,(RRF)\,(Ws)\,(D)}$$

Where,

Ax, Is, Ais are as given for water.

RRF = Relative response factor form the heated purge of the calibration standard.

$$D = \frac{100 - \%\ \text{moisture}}{100}$$

Ws = Weight of sample added to the purge tube, in grams (g).

15.5.2 Medium Level Soil/Sediment Samples

15.5.2.1 The following equation is used for quantitation of medium level soil/sediment samples:

$$\text{Concentration ug/Kg (Dry weight basis)} = \frac{(Ax)\,(Is)\,(Vt)\,(1000)\,(Df)}{(Ais)\,(RRF)\,(Va)\,(Ws)\,(D)}$$

Where,
Ax, Is, Ais are as given for water.
RRF = Relative response factor from the ambient temperature purge of the calibration standard.
Vt = Total volume of the methanol extract in milliliters (mL).
NOTE: This volume is typically 10 mL, even though only 1 mL is transferred to the vial.
Va = Volume of the aliquot of the sample methanol extract (i.e., sample extract not including the methanol added to equal 100 uL) in microliters (uL) added to reagent water for purging.
Ws = Weight of soil/sediment extracted, in grams (g).
D = $\frac{100 - \%\ \text{moisture}}{100}$
Df = Dilution factor. The dilution factor for analysis of soil/sediment samples for volatiles by the medium level method is defined as:

$$\frac{\text{uL most conc. extract used to make dilution + uL clean solvent}}{\text{uL most conc. extract used to make dilution}}$$

(The dilution factor is equal to 1.0 in all cases other than those requiring dilution of the sample methanol extract (Vt). The factor of 1,000 in the numerator converts the value of Vt from mL to uL.)

15.6 When quantitating the sample concentration of Xylenes (total), the areas of both the M&P Xylene peak and the O-Xylene peak are summed and the RRF determined using equation 1 are used. The concentration of each peak may be determined separately and then summed to determine the concentration of Xylene (total).

15.7 When quantitating the concentration of 1,2-Dichloroethene (total), the concentrations of the two isomers (cis and trans) are summed.

15.8 Secondary ion quantitation may be used if interferences (such as matrix effects) may cause a bias in quantitation. If ions other then those listed in table 6 are used, the analyst will document the reason, and it will be noted in the job narrative.

15.9 If manual integration of any compound (including internal standards, system monitoring compounds, target or tentatively identified compounds) is required, the EICP of that compound will be provided. All manual integrations will be identified with an "m" and initialed and dated by the GC/MS analyst.

15.10 Tentatively Identified Compounds

15.10.1 An estimated concentration for tentatively identified compounds will be determined using the equations described above for a given matrix using the total area counts of both the tentatively identified compound and the nearest internal standard which is free of interferences.

15.10.2 The RRF used to determine all concentrations of tentatively identified compounds will be an assumed RRF of one (1).

15.10.3 All tentatively identified compounds will be qualified as "J" (estimated) and "N" (presumptive evidence).

15.11 System Monitoring Compounds

15.11.1 The recovery of all system monitoring compounds in samples, blanks matrix spikes and matrix spike duplicates, is calculated using equation:

$$\% \text{ Recovery} = \frac{\text{Concentration (amount) found}}{\text{Concentration (amount) spiked}} \times 100$$

15.11.2 The recovery limits for each system monitoring compound are laboratory established on an annual basis. The recoveries must be within the criteria limits. If they fall outside criteria limits, the results must be evaluated and the sample reanalyzed, if necessary.

15.11.3 The relative retention time (RRT) of each system monitoring compound must be within the acceptance windows of ±0.06 RRT.

15.12 Internal Standards

15.12.1 The internal standards of all samples, blanks, matrix spikes and matrix spike duplicates must be monitored. The EICP area of each internal standard must be within the range of -50.0 percent to 200.0 percent of those in the continuing calibration.

15.12.2 The relative retention time (RRT) of each internal standard must be within 0.5 minutes (30 seconds) of those in the continuing calibration.

15.13 Verification of Calculated Result

15.13.1 The laboratory analyst/data entry analyst will print out and review sample worksheets and hand calculate the result for positive hits, internal standards and surrogates for comparison to the AIMS calculated result. Corrective action will result, if needed.

TITLE: ANALYTICAL METHODS FOR THE ANALYSIS OF GC\MS VOLATILE SAMPLES 8260B

SUPERCEDES: Revision 7

16.0 METHOD PERFORMANCE

16.1. Each analyst prior to sample analysis will perform 4 replicate second source QC check standards, at 20ug/L, as an Initial Demonstration of Capability. The average recovery and standard deviation are keep in AIMS and kept with each analyst's training file.

17.0 DATA ASSESSMENT AND ACCEPTANCE CRITERIA FOR QUALITY CONTROL MEASURES

17.1. Technical Acceptance Criteria For Initial Calibration

17.1.1. SPCCs - System performance check compounds are compounds used to check compound instability degradation. The following average minimum average response factors must be met before the cuarbve can be used:

17.1.1.1.	Chloromethane	0.10
17.1.1.2.	1,1-Dichloroethane	0.10
17.1.1.3.	Bromoform	0.10
17.1.1.4.	Chlorobenzene	0.30
17.1.1.5.	1,1,2,2-Tetrachloroethane	0.30

17.1.2. CCCs - Calibration Check Compounds - Evaluates the calibration based on the integrity of the system. The % RSD for the CCCs MUST be equal or less than 30%. The CCCs are:

17.1.2.1. Vinyl chloride

17.1.2.2. 1,1-Dichloroethene

17.1.2.3. Chloroform

17.1.2.4. 1,2-Dichloropropane

17.1.2.5. Toluene

17.1.2.6. Ethylbenzene

17.1.3. If the % RSD of any of the target analytes is 15% or less, the average response factor is assumed constant and the average response factor may be used for quantitation.

OR

17.1.4. If the mean of the RSD values for all analytes is less than or equal to 15%, then the average response factor may be used for quantitation.

OR

17.1.5. If the % RSD of a target analyte is greater than 15%, linear regression may be used providing the coefficient of determination is greater than or equal to 0.99.

17.2. Technical Acceptance Criteria For Continuing Calibration

17.2.1. SPCCs - A system performance check is made daily or during every 12 hour analytical shift. Each compound must meet its minimum response factor (see Initial Calibration Criteria).

17.2.2. CCCs - Used to check the validity of the initial calibration. The % Difference for each CCC shall be less than or equal to 20% from the initial calibration for the continuing calibration to be valid. All non-CCC target compounds must be less than 100% difference.

17.2.3. Internal Standard Retention Time – The retention times for all internal standards must be evaluated to make sure that they are no more than 30 seconds from that of the midpoint of the initial calibration. If the retention time shift is greater than 30 seconds, the system must be inspected for malfunctions and maintenance must be performed, as required.

17.2.4. Internal Standard Response – The EICP area for all internal standards must be evaluated to make sure that they have not change by a factor greater than two (-50% to +100%) from that of the midpoint of the initial calibration. If the response exceeds these limits, the system must be inspected for malfunctions and maintenance must be performed, as required.

17.3. Technical Acceptance Criteria of Quality Control Samples

17.3.1. Samples, blanks, matrix spikes, and matrix spike duplicates must meet internal standard and system monitoring compound recovery limits. Where the Internal Standard recovery limit equals sample internal standard characteristic ion area (EICP) divided by the CCV internal standard characteristic ion area (EICP) , multiplied by 100.

TITLE: ANALYTICAL METHODS FOR THE ANALYSIS OF GC\MS VOLATILE SAMPLES 8260B

SUPERCEDES: Revision 7

18.0 CORRECTIVE ACTIONS FOR OUT-OF-CONTROL DATA.

18.1. Corrective Actions For MS/MSD

18.1.1 If the recoveries of the internal standards and system monitoring compounds do not agree with the unspiked sample (i.e. the sample recoveries were within control limits and MS/MSD recoveries were outside of control limits) the MS/MSD must be reanalyzed.

18.1.2 If the recoveries of the internal standards and system monitoring compounds agree with the unspiked sample (i.e. both the sample and MS/MSD recoveries were outside of control limits) re-analysis is not required. The instance will be documented in the SDG narrative.

18.1.3 Limits for the matrix spiking compounds are established by the laboratory on an annual basis. If the concentrations determined in the MS/MSD do not meet the control limits, no corrective action is necessary as long as the MSB/LCS was within control limits. The instance will be documented in the job narrative.

18.2 Corrective Actions For Initial Calibration

18.2.1 If technical acceptance criteria cannot be met, it may be necessary to re-analyze the initial calibration. If after re-analysis, the criteria has not been met, it may be necessary to inspect the GC/MS system for possible problems.

18.2.2 Corrective actions may require one or several of the following procedures:

18.2.2.1 Open new/remake standard mixes

18.2.2.2 The ion source may be cleaned

18.2.2.3 The column may be cut at the injection port end

18.2.2.4 Change the purge trap on the purge and trap unit

18.2.2.5 Correct purge gas flow to optimize response

18.2.2.6 The column may be baked out

18.2.2.7 The purge trap may be baked out

18.2.2.8 The column may be replaced

18.3 Corrective Actions for Failure to Meet the Continuing Calibration Acceptance Criteria

18.3.1 If the technical acceptance criteria given above are not met, it may be necessary to re-analyze the continuing calibration check. If, after re-analysis, the given criterion has not been met, it may be necessary to re-analyze the initial calibration.

18.3.2 Other Corrective actions may be taken. The following details possible corrective actions:

18.3.2.1 Open new/remake standard mixes

18.3.2.2 The ion source may be cleaned

18.3.2.3 The column may be cut at the injection port end

18.3.2.4 The trap on the purge and trap unit may be replaced

18.3.2.5 The purge gas flow may be adjusted

18.3.2.6 The column may be baked out

18.3.2.7 The purge trap may be baked out

18.3.2.8 The column may be replaced

18.4 Corrective Actions For Samples

18.4.1 If the internal standard or system monitoring criteria are not met, the sample must be re-analyzed to insure that it was not an internal problem that affected recoveries. If, after re-analysis, recoveries are outside of control limits, a matrix effect can be assumed.

18.4.2 When dilutions are performed, target compound concentration must fall within the upper range of the initial calibration. If any target compound exceeds the calibration range, the sample would require dilution. The sample immediately following a sample with target compounds above the calibration range must be monitored to insure that there is no carryover present. If there is a possibility of carryover, that sample must be re-analyzed.

18.4.3 If matrix effects exist, and both analyses exhibit recoveries outside of control limits, both analyses will be reported and documented in the job narrative.

18.4.4 If, after re-analysis, recovery criteria are met, only the second analyses will be reported. If the second analyses occurs outside of the contract required holding time, both analyses will be reported in that instance.

18.4.5 In the case of a matrix spike or matrix spike duplicate, these samples should only be reanalyzed if the recoveries do not agree with the unspiked sample. If recoveries agree, the unspiked sample will not require re-analysis. The instance will be documented in the SDG narrative.

18.5 Corrective Actions for Failure to Meet the Matrix Spike Blank Acceptance Criteria

18.5.1 Limits for the matrix spiking compounds are established by the laboratory on an annual basis. The MSB/LCS must fall within these control limits. When required, the MSB/LCS will be spiked with all compounds of interest, otherwise spiked to include a minimum of 1,1-dichloroethene, trichloroethene, chlorobenzene, toluene, and benzene. Due to the potentially large number of target compounds for method 8260B, it is possible that a couple of spiking compounds could fall outside limits in the MSB/LCS. If a compound falls outside limits biased high and that compound is not found in the samples, a comment will be made in the case narrative and the data will be found to be acceptable

18.5.2 If the technical acceptance criteria are not met, it may be necessary to re-analyze the matrix spike blank. If, after re-analysis, the given criterion has not been met, it may be necessary to re-analyze the initial calibration.

18.5.3 Other Corrective actions may be taken. The following details possible corrective actions:

18.5.3.1 Open new/remake standard mixes

18.5.3.2 The ion source may be cleaned

18.5.3.3 The column may be cut at the injection port end

18.5.3.4 The trap on the purge and trap unit may be replaced

18.5.3.5 The purge gas flow may be adjusted

18.5.3.6 The column may be baked out

18.5.3.7 The purge trap may be baked out

18.5.3.8 The column may be replaced

TITLE: ANALYTICAL METHODS FOR THE ANALYSIS OF GC\MS VOLATILE SAMPLES 8260B

SUPERCEDES: Revision 7

19.0 CONTINGENCIES FOR HANDELING OUT-OF-CONTROL OR UNACCEPTABLE DATA

19.1. Inform project manager for client input and fill out job exception report.

19.2. Rerun samples to confirm results.

19.3. Resample if client or project manager requests.

20.0 WASTE MANAGEMENT/ POLLUTION PREVENTION

20.1 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.”

20.2 Waste Streams Produced by the Method

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

20.2.2 Spill Response: Any spills must be cleaned up immediately and handled correctly. Any wastes that have a pH < 7 must be disposed of in an "A" waste container. Any wastes having a pH > 7 must be disposed of in a "D" waste container.

20.2.3 Aqueous waste generated from analysis: Any wastes that have a pH < 7 must be disposed of in an "A" waste container. Any wastes having a pH > 7 must be disposed of in a "D" waste container.

20.2.4 Solvent waste generated from analysis: Solvent waste is stored in laboratory approved metal waste receptacle and labeled "C" waste. Waste receptacles are then taken to sample control where they are then properly disposed of.

20.2.5 Solid waste generated from analysis: Solid volatile analysis waste consists of soils and glass. The soil is wrapped in tin foil and placed in the solid waste receptacle. Soils used for dry weight measurements are also disposed of in this manner. Glass waste such as pipets and vials are rinsed and disposed of in approved glass receptacles

20.2.6 Expired Standards. Expired and used standards are stored in a laboratory approved metal waste receptacle labeled “BV”. Waste receptacles are then taken to sample control where they are then properly disposed of.

TITLE: ANALYTICAL METHODS FOR THE ANALYSIS OF GC\MS VOLATILE SAMPLES 8260B

SUPERCEDES: Revision 7

21.0 REFERENCE

21.1. Method 8260B, "Test Methods for Evaluating Solid Waste"; SW846, Third Edition, December 1996.

22.0 TABLES, DIAGRAMS, FLOWCHARTS AND VALIDATION DATA

22.1. Compounds Determined by Method 8260B

22.2. BFB Key Ions and Ion Abundance Criteria

22.3. Volume of Medium Level Extracts for Dilution

22.4. Characteristic Masses (m/z) for Purgeable Organic Compounds

22.5. GCMS Volatile Job Summary and Data Review Checklist

22.6. Tables 6-12: Composition of Stock Standards

23.0 CHANGES FROM THE PREVIOUS REVISION

23.1. Laboratory Director change, signature added

23.2. Department Supervisor change, signature added.

23.3. Section 3.0: Changed section to Reporting Limit instead of Method Detection Limit and added appropriate information.

23.4. Section 14.7.1: Incorporated Interim Change IC-A

23.5. Updated Tables 6 through 10

23.6. Added Tables 11 & 12

23.7. Minor grammatical changes

23.8. Changed 'should' to 'shall' or 'must' as needed.

SOP No.	Revision No.	Effective Date	Page
AMV-8260B-56	8	May 31, 2006	30 of 62

TITLE: ANALYTICAL METHODS FOR THE ANALYSIS OF GC\MS VOLATILE SAMPLES 8260B

SUPERCEDES: Revision 7

TABLE 1: COMPOUNDS DETERMINED BY METHOD 8260B

		Appropriate Technique					
Compound	CAS No.[b]	5030/5035	5031	5032	5021	5041	Direct Injection
Acetone	67-64-1	pp	c	c	nd	c	c
Acetonitrile	75-05-8	pp	c	nd	nd	nd	c
Acrolein	107-02-8	pp	c	c	nd	nd	c
Acrylonitrile	107-13-1	pp	c	c	nd	c	c
Allyl alcohol	107-18-6	ht	c	nd	nd	nd	c
Allyl chloride	107-05-1	c	nd	nd	nd	nd	c
Benzene	71-43-2	c	nd	c	c	c	c
Benzyl chloride	100-44-7	c	nd	nd	nd	nd	c
Bis(2-chloroethyl)sulfide	505-60-2	pp	nd	nd	nd	nd	c
Bromoacetone	598-31-2	pp	nd	nd	nd	nd	c
Bromochloromethane	74-97-5	c	nd	c	c	c	c
Bromodichloromethane	75-27-4	c	nd	c	c	c	c
4-Bromofluorobenzene (surr)	460-00-4	c	nd	c	c	c	c
Bromoform	75-25-2	c	nd	c	c	c	c
Bromomethane	74-83-9	c	nd	c	c	c	c
n-Butanol	71-36-3	ht	c	nd	nd	nd	c
2-Butanone (MEK)	78-93-3	pp	c	c	nd	nd	c
t-Butyl alcohol	75-65-0	pp	c	nd	nd	nd	c
Carbon disulfide	75-15-0	pp	nd	c	nd	c	c
Carbon tetrachloride	56-23-5	c	nd	c	c	c	c
Chloral hydrate	302-17-0	pp	nd	nd	nd	nd	c
Chlorobenzene	108-90-7	c	nd	c	c	c	c
Chlorobenzene-d5 (IS)		c	nd	c	c	c	c
Chlorodibromomethane	124-48-1	c	nd	c	nd	c	c
Chloroethane	75-00-3	c	nd	c	c	c	c

TITLE: ANALYTICAL METHODS FOR THE ANALYSIS OF GC\MS VOLATILE SAMPLES 8260B

SUPERCEDES: Revision 7

		Appropriate Technique					
Compound	CAS No.[b]	5030/5035	5031	5032	5021	5041	Direct Injection
2-Chloroethanol	107-03-3	pp	nd	nd	nd	nd	c
2-Chloroethyl vinyl ether	110-75-8	c	nd	c	nd	nd	c
Chloroform	67-66-3	c	nd	c	c	c	c
Chloromethane	74-87-3	c	nd	c	c	c	c
Chloroprene	126-99-8	c	nd	nd	nd	nd	c
3-Chloropropionitrile	542-76-7	l	nd	nd	nd	nd	pc
Crotonaldehyde	4170-30-3	pp	c	nd	nd	nd	c
1,2-Dibromo-3-chloropropane	96-12-8	pp	nd	nd	c	nd	c
1,2-Dibromoethane	106-93-4	c	nd	nd	c	nd	c
Dibromomethane	74-95-3	c	nd	c	c	c	c
1,2-Dichlorobenzene	95-50-1	c	nd	nd	c	nd	c
1,3-Dichlorobenzene	541-73-1	c	nd	nd	c	nd	c
1,4-Dichlorobenzene	106-46-7	c	nd	nd	c	nd	c
1,4-Dichlorobenzene-d4 (IS)		c	nd	nd	c	nd	c
cis-1,4-Dichloro-2-butene	1476-11-5	c	nd	c	nd	nd	c
trans-1,4-Dichloro-2-butene	110-57-6	pp	nd	c	nd	nd	c
Dichlorodifluoromethane	75-71-8	c	nd	c	c	nd	c
1,1-Dichloroethane	75-34-3	c	nd	c	c	c	c
1,2-Dichloroethane	107-06-2	c	nd	c	c	c	c
1,2-Dichloroethane-d4 (surr)		c	nd	c	c	c	c
1,1-Dichloroethene	75-35-4	c	nd	c	c	c	c
trans-1,2-Dichloroethene	156-60-5	c	nd	c	c	c	c
1,2-Dichloropropane	78-87-5	c	nd	c	c	c	c
1,3-Dichloro-2-propanol	96-23-1	pp	nd	nd	nd	nd	c
cis-1,3-Dichloropropene	10061-01-5	c	nd	c	nd	c	c
trans-1,3-Dichloropropene	10061-02-6	c	nd	c	nd	c	c
1,2,3,4-Diepoxybutane	1464-53-5	c	nd	nd	nd	nd	c

TITLE: ANALYTICAL METHODS FOR THE ANALYSIS OF GC\MS VOLATILE SAMPLES 8260B

SUPERCEDES: Revision 7

		Appropriate Technique					
Compound	CAS No.[b]	5030/5035	5031	5032	5021	5041	Direct Injection
Diethyl ether	60-29-7	c	nd	nd	nd	nd	c
1,4-Difluorobenzene (I.S.)	540-36-3	nd	nd	nd	nd	c	c
1,4-Dioxane	123-91-1	pp	c	c	nd	nd	c
Epichlorohydrin	106-89-8	l	nd	nd	nd	nd	c
Ethanol	64-17-5	l	c	c	nd	nd	c
Ethyl acetate	141-78-6	l	c	nd	nd	nd	c
Ethylbenzene	100-41-4	c	nd	c	c	c	c
Ethylene oxide	75-21-8	pp	c	nd	nd	nd	c
Ethyl methacrylate	97-63-2	c	nd	c	nd	nd	c
Fluorobenzene (IS)	462-06-6	c	nd	nd	nd	nd	nd
Hexachlorobutadiene	87-68-3	c	nd	nd	c	nd	c
Hexachloroethane	67-72-1	l	nd	nd	nd	nd	c
2-Hexanone	591-78-6	pp	nd	c	nd	nd	c
2-Hydroxypropionitrile	78-97-7	l	nd	nd	nd	nd	pc
Iodomethane	74-88-4	c	nd	c	nd	c	c
Isobutyl alcohol	78-83-1	pp	c	nd	nd	nd	c
Isopropylbenzene	98-82-8	c	nd	nd	c	nd	c
Malononitrile	109-77-3	pp	nd	nd	nd	nd	c
Methacrylonitrile	126-98-7	pp	l	nd	nd	nd	c
Methanol	67-56-1	l	c	nd	nd	nd	c
Methylene chloride	75-09-2	c	nd	c	c	c	c
Methyl methacrylate	80-62-6	c	nd	nd	nd	nd	c
4-Methyl-2-pentanone (MIBK)	108-10-1	pp	c	c	nd	nd	c
Naphthalene	91-20-3	c	nd	nd	c	nd	c
Nitrobenzene	98-95-3	c	nd	nd	nd	nd	c
2-Nitropropane	79-46-9	c	nd	nd	nd	nd	c
N-Nitroso-di-n-butylamine	924-16-3	pp	c	nd	nd	nd	c

TITLE: ANALYTICAL METHODS FOR THE ANALYSIS OF GC\MS VOLATILE SAMPLES 8260B

SUPERCEDES: Revision 7

		Appropriate Technique					
Compound	CAS No.[b]	5030/5035	5031	5032	5021	5041	Direct Injection
Paraldehyde	123-63-7	pp	c	nd	nd	nd	c
Pentachloroethane	76-01-7	l	nd	nd	nd	nd	c
2-Pentanone	107-87-9	pp	c	nd	nd	nd	c
2-Picoline	109-06-8	pp	c	nd	nd	nd	c
1-Propanol	71-23-8	pp	c	nd	nd	nd	c
2-Propanol	67-63-0	pp	c	nd	nd	nd	c
Propargyl alcohol	107-19-7	pp	l	nd	nd	nd	c
B-Propiolactone	57-57-8	pp	nd	nd	nd	nd	c
Propionitrile (ethyl cyanide)	107-12-0	ht	c	nd	nd	nd	c
n-Propylamine	107-10-8	c	nd	nd	nd	nd	c
Pyridine	110-86-1	l	c	nd	nd	nd	c
Styrene	100-42-5	c	nd	c	c	c	c
1,1,1,2-Tetrachloroethane	630-20-6	c	nd	nd	c	c	c
1,1,2,2-Tetrachloroethane	79-34-5	c	nd	c	c	c	c
Tetrachloroethene	127-18-4	c	nd	c	c	c	c
Toluene	108-88-33	c	nd	c	c	c	c
Toluene-d8 (surr)	2037-26-5	c	nd	c	c	c	c
o-Toluene	95-53-4	pp	c	nd	nd	nd	c
1,2,4-Trichlorobenzene	120-82-1	c	nd	nd	c	nd	c
1,1,1-Trichloroethane	71-55-6	c	nd	c	c	c	c
1,1,2-Trichloroethane	79-00-5	c	nd	c	c	c	c
Trichloroethane	79-01-6	c	nd	c	c	c	c
Trichlorofluoromethane	75-69-4	c	nd	c	c	c	c
1,2,3-Trichloropropane	96-18-4	c	nd	c	c	c	c

SOP No.	Revision No.	Effective Date	Page
AMV-8260B-56	8	May 31, 2006	34 of 62

TITLE: ANALYTICAL METHODS FOR THE ANALYSIS OF GC\MS VOLATILE SAMPLES 8260B

SUPERCEDES: Revision 7

		Appropriate Technique					
Compound	CAS No.[b]	5030/5035	5031	5032	5021	5041	Direct Injection
Vinyl acetate	108-05-4	c	nd	c	nd	nd	c
Vinyl chloride	75-01-4	c	nd	c	c	c	c
Xylene (Total)	1330-20-7	c	nd	c	c	c	c

c= Adequate response by this technique
b= Chemical Abstract Services Registry Number
pp= Poor purging efficiency resulting in high EQLs
l= Inappropriate technique for this analyte
pc= Poor chromatographic behavior
nd= Not determined
surr= Surrogate
IS= Internal Standard
ht= Method analyte only when purged at 80 C

The following compounds are also amenable to analysis by Method 8260:

Bromombenzene
n-Butylbenzene
sec-Butlybenzene
tert-Butylbenzene
Chloroacetonitrile
1-Chlorobutane
1-Chlorohexane
2-Chlorotoluene
4-Chlorotoluene
Dibromofluoromethane
Cis-1,2-Dichloroethene
1,3-Dichloropropane
2,2-Dichloropropane
1,1-Dichloropropene
p-Isopropyltoluene
Methyl acrylate
Methyl-t-butyl ether
Pentafluorobenzene
n-Propylbenzene
1,2,3-Trichlorobenzene
1,2,4-Trimethylbenzene
1,3,5-Trimethylbenzene

TABLE 2
BFB Key Ions and Ion Abundance Criteria

mz	Required Intensity (relative abundance)
50	15 to 40% of m/z 95
75	30 to 60% of m/z 95
95	Base peak, 100% relative abundance
96	5 to 9% of m/z 95
173	less than 2% of m/z 174
174	Greater than 50% of m/z 95
175	5 to 9% of m/z 174
176	Greater than 95% but less than 101% of m/z 174
177	5 to9% of m/z 176

*Alternate tuning criteria may be used, (e.g. CLP, Method 524.2, or manufacturers' instructions), provided that method performance is not adversely affected.

TITLE: ANALYTICAL METHODS FOR THE ANALYSIS OF GC\MS VOLATILE SAMPLES 8260B

SUPERCEDES: Revision 7

TABLE 3

Volume of Medium Level Extracts for Dilution

Dilution Factor	Volume of Extract
1	100ul
2	50ul
5	20ul
10	10ul
20	5ul
25	4ul
40	2.5ul
50	2ul
100	1ul
200	50ul of a 1/10 Dilution

TITLE: ANALYTICAL METHODS FOR THE ANALYSIS OF GC\MS VOLATILE SAMPLES 8260B

SUPERCEDES: Revision 7

TABLE 4

Characteristic Masses (m/z) for Purgeable Organic Compounds

Analyte	Primary Characteristic Ion	Secondary Characteristic Ion(s)
Acetone	58	43
Acetonitrile	41	40,39
Acrolein	56	55,58
Acrylonitrile	53	52,51
Allyl alcohol	57	58,39
Allyl chloride	76	41,39,78
Benzene	78	-
Benzyl chloride	91	126,65,128
Bromoacetone	136	43,138,93,95
Bromobenzene	156	77,158
Bromochloromethane	128	49,130
Bromodichloromethane	83	85,127
Bromoform	173	175,254
Bromomethane	94	96
iso-Butanol	74	43
n-Butanol	56	41
2-Butanone	72	43
n-Butylbenzene	91	92,134
sec-Butylbenzene	105	134
tert-Butylbenzene	119	91,134
Carbon disulfide	76	78
Carbon tetrachloride	117	119
Chloral hydrate	82	44,84,86,111
Chloroacetonitrile	48	75

TITLE: ANALYTICAL METHODS FOR THE ANALYSIS OF GC\MS VOLATILE SAMPLES 8260B

SUPERCEDES: Revision 7

Analyte	Primary Characteristic Ion	Secondary Characteristic Ion(s)
Chlorobenzene	112	77,114
1-Chlorobutane	56	49
Chlorodibromomethane	129	208,206
Chloroethane	64 (49*)	66 (51*)
2-Chloroethanol	49	44,43,51,80
bis-(2-Chloroethyl) sulfide	109	111,158,160
2-Chloroethyl vinyl ether	63	65,106
Chloroform	83	85
Chloromethane	50 (49*)	52 (51*)
Chloroprene	53	88,90,51
3-Chloropropionitrile	54	49,89,91
3-Chlorotoluene	91	126
4-Chlorotoluene	91	126
1,2-Dibromo-3-chloropropane	75	155,157
Dibromochloromethane	129	127
1,2-Dibromoethane	107	109,188
Dibromomethane	93	95,174
1,2-Dichlorobenzene	146	111,148
1,2-Dichlorobenzene-d_4	152	115,150
1,3-Dichlorobenzene	146	111,148
1,4-Dichlorobenzene	146	111,148
cis-1,4-Dichloro-2-butene	75	53,77,124,89
trans-1,4-Dichloro-2-butene	53	88,75
Dichlorodifluoromethane	85	87
1,1-Dichlorothane	63	65,83
1,2-Dichloroethane	62	98
1,1-Dichlorothene	96	61,63

TITLE: ANALYTICAL METHODS FOR THE ANALYSIS OF GC\MS VOLATILE SAMPLES 8260B

SUPERCEDES: Revision 7

Analyte	Primary Characteristic Ion	Secondary Characteristic Ion(s)
cis-1,2-Dichloroethene	96	61,98
trans-1,2-Dichloroethene	96	61,98
1,2-Dichloropropane	63	112
1,3-Dichloropropane	76	78
2,2-Dichloropropane	77	97
1,3-Dichloro-2-propanol	79	43,81,49
1,1-Dichloropropene	75	110,77
cis-1,3-Dichloropropene	75	77,39
trans-1,3-Dichloropropene	75	77,39
1,2,3,4-Diepoxybutane	55	57,56
Diethyl ether	74	45,59
1,4-Dioxane	88	58,43,57
Epichlorohydrin	57	49,62,51
Ehtanol	31	45,27,46
Ethyl acetate	88	43,45,61
Ethylbenzene	91	106
Ethylene oxide	44	43,42
Ehtyl methacrylate	69	41,99,86,114
Hexachlorobutadiene	225	223,227
Hexachloroethane	201	166,199,203
2-Hexanone	43	58,57,100
2-Hydroxypropionitrile	44	43,42,53
Iodomethane	142	127,141
Isobutyl alcohol	43	41,42,74
Isopropylbenzene	105	120
p-Isopropyltoluene	119	134,91
Malonitrile	66	39,65,38

TITLE: ANALYTICAL METHODS FOR THE ANALYSIS OF GC\MS VOLATILE SAMPLES 8260B

SUPERCEDES: Revision 7

Analyte	Primary Characteristic Ion	Secondary Characteristic Ion(s)
Methacrylonitrile	41	67,39,52,66
Methyl acrylate	55	85
Methyl-t-butyl ether	73	57
Methylene chloride	84	86,49
Methyl ethyl ketone	72	43
Methyl iodide	142	127,141
Methyl methacrylate	69	41,100,39
4-Methyl-2-pentanone	100	43,58,85
Naphthalene	128	-
Nitrobenzene	123	51,77
2-Nitropropane	46	-
2-Picoline	93	66,92,78
Pentachloroethane	167	130,132,165,169
Propargyl alcohol	55	39,38,53
B-Propiolactone	42	43,44
Propionitrile (ethyl cyanide)	54	52,55,40
n-Propylamine	59	41,39
n-Propylbenzene	91	120
Pyridine	79	52
Styrene	104	78
1,2,3-Trichlorobenzene	180	182,145
1,2,4-Trichlorobenzene	180	182,145
1,1,1,2-Tetrachloroethane	131	133,119
1,1,2,2-Tetrachloroethane	83	131,85
Tetrachloroethene	164	129,131,166
Toluene	92	91
1,1,1-Trichloroethane	97	99,61

TITLE: ANALYTICAL METHODS FOR THE ANALYSIS OF GC\MS VOLATILE SAMPLES 8260B

SUPERCEDES: Revision 7

Analyte	Primary Characteristic Ion	Secondary Characteristic Ion(s)
1,1,2-Trichloroethane	83	97,85
Trichloroethene	95	97,130,132
Trichlorofluoromethane	151	101,153
1,2,3-Trichloropropane	75	77
1,2,4-Trimethylbenzene	105	120
1,3,5-Trimethylbenzene	105	120
Vinyl acetate	43	86
Vinyl chloride	62	64
o-Xylene	106	91
m-Xylene	106	91
p-Xylene	106	91
INTERNAL STANDARDS/SURROGATES		
Benzene-d6	84	83
Bromobenzene-d5	82	162
Bromochloromethane-d2	51	131
1,4-Difluorobenzene	114	
Chlorobenzene-d5	117	
1,4-Dichlorobenzene-d4	152	115,150
1,1,2-Trichloroethane-d3	100	
4-Bromofluorobenzene	95	174,176
Chloroform-d1	84	
Dibromofluoromethane	113	

SOP No.	Revision No.	Effective Date	Page
AMV-8260B-56	8	May 31, 2006	42 of 62

TITLE: ANALYTICAL METHODS FOR THE ANALYSIS OF GC\MS VOLATILE SAMPLES 8260B

SUPERCEDES: Revision 7

TABLE 5

Job Summary Check List (Page 1 & 2)

GCMS VOLATILE JOB SUMMARY

JOB No._____ METHOD_____
SOIL LEVEL_____
LAB DUE DATE_____ INSTRUMENT_____
PURGE VOLUME_____

TUNE
FILE#_____ FILE#_____ FILE#_____
PASSED? Y N PASSED? Y N PASSED? Y N

CONTINUING CALIBRATION VALUES CALCULATED FROM CCV ____ INIT ____
FILE#_____ FILE#_____ FILE#_____
PTS OUT?_____ PTS OUT_____ PTS OUT_____
ACCEPTABLE Y N ACCEPTABLE Y N ACCEPTABLE Y N

INITIAL CALIBRATION
REFERENCED CURVE ID_____ REFERENCED CURVE ID_____
PASSED? Y N PASSED? Y N

ADD STD REFERENCED CURVE ID_____
FILE#_____ FILE#_____ FILE#_____

MSB FILE#_____ FILE#_____ FILE#_____
PASSED? Y N PASSED? Y N PASSED? Y N
CMPDS OUT_____ CMPDS OUT_____ CMPDS OUT_____

MSBD FILE#_____ FILE#_____ FILE#_____
PASSED? Y N PASSED? Y N PASSED? Y N
CMPDS OUT_____ CMPDS OUT_____ CMPDS OUT_____

VBLK FILE#_____ FILE#_____ FILE#_____
VBLK____ VBLK____ VBLK____
ACCEPTABLE? Y N ACCEPTABLE Y N ACCEPTABLE Y N

COMMENTS AND CORRECTIVE MEASURES (see reverse side for comment # explanations)
#_____ Sample(s)_____
#_____ Sample(s)_____
#_____ Sample(s)_____
#_____ Sample(s)_____
#_____ Sample(s)_____

Other Comments_____
_____
_____
_____

ANALYST_____ DATE_____
2ND REVIEW_____ DATE_____
AIMS ENTRY_____ DATE_____
VALIDATOR _____ DATE_____

SOP No.	Revision No.	Effective Date	Page
AMV-8260B-56	8	May 31, 2006	43 of 62

TITLE: **ANALYTICAL METHODS FOR THE ANALYSIS OF GC\MS VOLATILE SAMPLES 8260B**

SUPERCEDES: Revision 7

Comment #	Comment
1	NA
2	NA
3	Sample(s) was diluted for excessive foaming.
4	Sample(s) was diluted for non-target compounds (TICS) exceeding 5X the total response of one of the Internal Standards.
5	Sample(s) was diluted for sample matrix which resulted in method non-compliance for an Internal standard.
6	Sample(s) was diluted for sample matrix which resulted in method non-compliance for a surrogate
7	Sample(s) was diluted for TCLP matrix
8	Sample(s) was diluted for high levels of target compound(s).
9	NA
10	NA
11	Sample(s) was diluted due to insufficient volume for a lower dilution.
12	Sample(s) was diluted for viscosity.
13	Sample(s) was diluted for other reason.
14	As a result of low volume, the sample was analyzed from a vial with headspace.
15	Sample(s) was re-analyzed for surrogate recoveries outside of limits.
16	Sample(s) was re-analyzed for Internal Standard recoveries outside of limits
17	Matrix effect on Surrogate was confirmed by the analysis of ms & sd
18	Sample contains compounds which saturated the detector.This will result in non-linear results between the sample and the "DL"
19	Samples were analyzed by method 8260B.
20	Sample pH was greater than 2.
21	There was insufficient volume for re-anaylsis of the sample(s).
22	There was insufficient volume for dilution of the sample(s).
23	The VBLK was contaminated with compounds below the reporting limit.
24	The VBLK was contaminated with compounds above the reporting limit.
25	The MSB had a compound(s) outside of the method limits.
26	Sample was re-run and confimed results not consistent with historical.
27	See accompaning Job Exception Report
28	
29	
30	

SOP No.	Revision No.	Effective Date	Page
AMV-8260B-56	8	May 31, 2006	44 of 62

TITLE: ANALYTICAL METHODS FOR THE ANALYSIS OF GC\MS VOLATILE SAMPLES 8260B

SUPERCEDES: Revision 7

Table 6

Certificate of Composition

DESCRIPTION: Volatile Organic Compounds Mix 6

MVSC 72 8-20

CATALOG NO.: 48799-U MFG DATE: Nov-2005

MVSC 73 1-7

LOT NO.: LB34727 EXPIRATION DATE: Feb-2007

SOLVENT: METHANOL

ANALYTE (1)	CAS NUMBER	PERCENT PURITY (2)	WEIGHT CONCENTRATION (3)	SUPELCO LOT NO
BROMOMETHANE	74-83-9	99.9 (a)	2000	LB22203
CHLOROETHANE	75-00-3	98.7 (a)	2000	LB29285
CHLOROMETHANE	74-87-3	99.9 (a)	2000	LA66620
DICHLORODIFLUOROMETHANE	75-71-8	99.9 (a)	2000	LB24923
TRICHLOROFLUOROMETHANE	75-69-4	99.9 (a)	2000	LA79530
VINYL CHLORIDE	75-01-4	99.9	2000	LB18727

(1) Listed in alphabetical order.
(2) Determined by capillary GC-FID, unless otherwise noted.
a) GC; detector HALL
(3) NIST traceable weights are used to verify balance calibration with the preparation of each lot. Concentration of analyte in solution is ug/ml +/- 0.5%, uncertainty based upon balance and Class A volumetric glassware. Weights are corrected for analytes less than 98% pure.

Elwood Doughty
QA Manager

Supelco warrants that its products conform to the information contained in this publication. Purchaser must determine the suitability of the product for its particular use. Please see the latest catalog or order invoice and packing slip for additional terms and conditions of sale.

SUPELCO
595 North Harrison Road • Bellefonte, PA 16823-0048 USA • Phone (814) 359-3441

TITLE: ANALYTICAL METHODS FOR THE ANALYSIS OF GC\MS VOLATILE SAMPLES 8260B

SUPERCEDES: Revision 7

Table 7
54 Component

Certificate of Analysis

PAGE 1 OF 2

DESCRIPTION: 502/524 Volatile Organics Calibration Mix

CATALOG NO.: 502111 MFG DATE: Nov-2003

LOT NO.: LB16275 EXPIRATION DATE: Mar-2006

SOLVENT: METHANOL

ANALYTE (1)	CAS NUMBER	PERCENT PURITY(2)	WEIGHT(3) CONCENTRATION	ANALYTICAL(4) CONCENTRATION		STD DEV	SUPELCO LOT NO
BENZENE	71-43-2	99.9	2000	2000	+/-	15.1	LB03979
BROMOBENZENE	108-86-1	99.9	2000	2009	+/-	17.4	LA97903
BROMOCHLOROMETHANE	74-97-5	99.7	2000	1967	+/-	33.3	LA67395
BROMODICHLOROMETHANE	75-27-4	99.9	2000	2103	+/-	0.1	LB15447
BROMOFORM	75-25-2	99.9	2000	1974	+/-	38.7	LB15898
CARBON TETRACHLORIDE	56-23-5	99.9	2000	1960	+/-	32.4	LA55581
CHLOROBENZENE	108-90-7	99.9	2001	2029	+/-	14.3	LB09884
CHLOROFORM	67-66-3	99.9	2000	2000	+/-	18.8	LA55585
CIS 1,3-DICHLOROPROPENE (Z)	10061-01-5	96.1	2000	2036	+/-	12.1	LA60646
CIS-1,2-DICHLOROETHYLENE	156-59-2	97.6	2000	1947	+/-	26.7	LA97197
DIBROMOCHLOROMETHANE	124-48-1	99.9	2001	2022	+/-	11.2	LA87237
DIBROMOMETHANE	74-95-3	99.8	2000	2000	+/-	33.6	LA39031
ETHYLBENZENE	100-41-4	99.5	2000	2040	+/-	8.0	LA40866
HEXACHLOROBUTADIENE	87-68-3	98.2	2001	1946	+/-	45.0	LA95300
ISOPROPYLBENZENE (CUMENE)	98-82-8	99.0	2000	2012	+/-	17.3	LB01119
M-XYLENE (5)	108-38-3	99.8	2001	*****			LB15074
METHYLENE CHLORIDE	75-09-2	99.9	2000	1957	+/-	28.9	LA88418
N-BUTYLBENZENE	104-51-8	98.7	2000	1996	+/-	25.3	LB09309
N-PROPYLBENZENE	103-65-1	99.9	2001	2028	+/-	15.6	LA92696
NAPHTHALENE	91-20-3	99.9	2000	1950	+/-	39.5	LA97766
O-XYLENE	95-47-6	99.5	2000	2022	+/-	9.8	LB08117
P-ISOPROPYLTOLUENE	99-87-6	99.9	2000	1986	+/-	20.7	LA41611
P-XYLENE (5)	106-42-3	99.9	2000	*****			LB04801
SEC-BUTYLBENZENE	135-98-8	99.4	2000	1993	+/-	31.6	LA51283
STYRENE	100-42-5	99.9	2001	2012	+/-	11.8	LB09037
TERT-BUTYLBENZENE	98-06-6	99.9	2000	1981	+/-	21.8	LB09550
TETRACHLOROETHENE	127-18-4	99.9	2001	2029	+/-	29.4	LB05248

(1) Listed in alphabetical order.
(2) Determined by capillary GC-FID, unless otherwise noted.
(3) NIST traceable weights are used to verify balance calibration with the preparation of each lot. Concentration of analyte in solution is ug/ml +/- 0.5%, based upon balance and Class A volumetric glassware. Weights are corrected for analytes less than 98% pure.
(4) Determined by chromatographic analysis against an independently prepared reference lot. Mean of replicate injections.
(5) These products coelute and are not quantified in the final mix.

Elwood Doughty
Quality Control Supervisor

Supelco warrants that its products conform to the information contained in this publication. Purchaser must determine the suitability of the product for its particular use. Please see the latest catalog or order invoice and packing slip for additional terms and conditions of sale.

SUPELCO
595 North Harrison Road
Bellefonte, PA 16823-0048 USA
Phone (814) 359-3441

SOP No.	Revision No.	Effective Date	Page
AMV-8260B-56	8	May 31, 2006	46 of 62

TITLE: ANALYTICAL METHODS FOR THE ANALYSIS OF GC\MS VOLATILE SAMPLES 8260B

SUPERCEDES: Revision 7

Certificate of Analysis

MUSC19 15-20
20 1-4

PAGE 2 of 2

DESCRIPTION: 502/524 Volatile Organics Calibration Mix

CATALOG NO.: 502111 MFG DATE: Nov-2003

LOT NO.: LB16275 EXPIRATION DATE: Mar-2006

SOLVENT: METHANOL

ANALYTE (1)	CAS NUMBER	PERCENT PURITY(2)	WEIGHT(3) CONCENTRATION	ANALYTICAL(4) CONCENTRATION		STD DEV	SUPELCO LOT NO
TOLUENE	108-88-3	99.7	2001	2020	+/-	15.8	LA90411
TRANS 1,3-DICHLOROPROPENE (E)	10061-02-6	98.5	2000	2052	+/-	12.9	LB06449
TRANS-1,2-DICHLOROETHYLENE	156-60-5	99.9	2000	1910	+/-	36.2	LB02428
TRICHLOROETHYLENE	79-01-6	98.5	2001	1980	+/-	20.2	LB04303
1,1-DICHLOROETHANE	75-34-3	97.0	2000	1968	+/-	32.1	LA54711
1,1-DICHLOROETHYLENE	75-35-4	99.9	2000	1980	+/-	46.1	LB04593
1,1-DICHLOROPROPENE	563-58-6	98.0	2000	1958	+/-	20.8	LB12558
1,1,1-TRICHLOROETHANE	71-55-6	99.9	2000	1973	+/-	26.8	LB14220
1,1,1,2-TETRACHLOROETHANE	630-20-6	99.1	2001	2000	+/-	16.1	LB01555
1,1,2-TRICHLOROETHANE	79-00-5	99.3	2000	2038	+/-	12.6	LB03464
1,1,2,2-TETRACHLOROETHANE	79-34-5	97.5	2000	1974	+/-	31.7	LA86969
1,2-DIBROMO-3-CHLOROPROPANE	96-12-8	97.9	2000	1978	+/-	43.5	LB06608
1,2-DIBROMOETHANE	106-93-4	99.6	2001	2029	+/-	0.1	LA87068
1,2-DICHLOROBENZENE	95-50-1	99.9	2000	2008	+/-	29.2	LA96474
1,2-DICHLOROETHANE	107-06-2	99.9	2000	1974	+/-	25.7	LA88777
1,2-DICHLOROPROPANE	78-87-5	99.9	2000	2019	+/-	9.6	LB08115
1,2,3-TRICHLOROBENZENE	87-61-6	99.75	2000	1962	+/-	18.9	LA50762
1,2,3-TRICHLOROPROPANE	96-18-4	99.1	2000	2006	+/-	17.8	LA39379
1,2,4-TRICHLOROBENZENE	120-82-1	98.6	2000	1957	+/-	52.1	LB12944
1,2,4-TRIMETHYLBENZENE	95-63-6	98.2	2000	2000	+/-	22.0	LA39081
1,3-DICHLOROBENZENE,	541-73-1	99.9	2001	2013	+/-	16.7	LA72024
1,3-DICHLOROPROPANE	142-28-9	99.9	2000	2024	+/-	11.8	LB00875
1,3,5-TRIMETHYLBENZENE	108-67-8	99.0	2000	2011	+/-	13.6	LA94493
1,4-DICHLOROBENZENE	106-46-7	99.9	2000	1992	+/-	16.2	LA50188
2-CHLOROTOLUENE	95-49-8	99.9	2000	2005	+/-	23.6	LA95842
2,2-DICHLOROPROPANE	594-20-7	98.3	2000	1968	+/-	19.4	LB01750
4-CHLOROTOLUENE	106-43-4	99.9	2001	1990	+/-	15.0	LB05252

(1) Listed in alphabetical order.
(2) Determined by capillary GC-FID, unless otherwise noted.
(3) NIST traceable weights are used to verify balance calibration with the preparation of each lot. Concentration of analyte in solution is ug/ml +/- 0.5%, based upon balance and Class A volumetric glassware. Weights are corrected for analytes less than 98% pure.
(4) Determined by chromatographic analysis against an independently prepared reference lot. Mean of replicate injections.
(5) These products coelute and are not quantified in the final mix.

Elwood Doughty
Quality Control Supervisor

Supelco warrants that its products conform to the information contained in this publication. Purchaser must determine the suitability of the product for its particular use. Please see the latest catalog or order invoice and packing slip for additional terms and conditions of sale.

SUPELCO
595 North Harrison Road
Bellefonte, PA 16823-0048 USA
Phone (814)359-3441

TITLE: ANALYTICAL METHODS FOR THE ANALYSIS OF GC\MS VOLATILE SAMPLES 8260B

SUPERCEDES: Revision 7

Table 8
8260 + Mix

8260+ #1A MXSL5 1/-720

Chemical Standard Batch Sheet
Lot #: A042263

Catalog #: 552504A	**Target:** 1000 - 40000 ug/ml	
Description: Custom Volatiles Standard Mix A		
Solvent: P&T Methanol	**Solvent Lot:** 44337	**Final Volume:** 100 ml

Made by: Joe Tallon	**Date:** 1/4/2006 8:09:50A	
Tested by:	**Date:**	
	By:	**Date:**
Packaged by: Jackie Glasgow / Staci Bodle	**Date:** 1/4/2006 10:49:12/	**No. Units:** 12
Balance Used: AT261	**Serial #:** 1119141429	

Compound	CAS	Storage Location	Lot #	Purity	Target Conc(ug/ml)	Target Weight	Actual Weight	Calc Conc(ug/ml)
Carbon disulfide	75-15-0	FA1A5D	J11J02	0.99	1,000.00	100.00	100.00	1,000.00
Methyl-tert-butyl ether (	1634-04-4	FA1B6C	10660BD	0.97	1,000.00	100.00	100.00	1,000.00
Iodomethane (methyl	74-88-4	FA1C2A	13906AB	0.99	1,000.00	100.00	100.00	1,000.00
Ethyl methacrylate	97-63-2	FA1C1D	09316HC	0.99	1,000.00	100.00	100.00	1,000.00
Tetrahydrofuran	109-99-9	FA1B8B	01057MC	0.99	5,000.00	500.00	500.00	5,000.00
trans-1,4-dichloro-2-butene	110-57-6	FA1C1C	160-22DD	0.99	5,000.00	500.00	500.00	5,000.00
Acetonitrile	75-05-8	FA1B13A	12067KC	0.99	40,000.00	4,000.00	4,000.00	40,000.00
1,1,2-Trichlorotrifluoroetha	76-13-1	FA1A11A	01404PV	0.99	1,000.00	100.00	100.00	1,000.00
Methyl acetate	79-20-9	FA1C11C	47640/1	0.99	1,000.00	100.00	100.00	1,000.00
Methylcyclohexane	108-87-2	FA1E4A	02759BC	0.99	1,000.00	100.00	100.00	1,000.00
Cyclohexane	110-82-7	FA1C7A	03145KB	0.99	1,000.00	100.00	100.00	1,000.00

SOP No.	Revision No.	Effective Date	Page
AMV-8260B-56	8	May 31, 2006	48 of 62

TITLE: **ANALYTICAL METHODS FOR THE ANALYSIS OF GC\MS VOLATILE SAMPLES 8260B**

SUPERCEDES: Revision 7

8260+ #1B MVSC5 1-720

Chemical Standard Batch Sheet
Lot #: A042264

Catalog #: 552504B	Target: 5000 ug/ml	
Description: Custom Volatiles Standard Mix B		
Solvent: P&T Methanol	Solvent Lot: A041266	Final Volume: 50 ml

Made by: Joe Tallon	Date: 1/4/2006 8:30:59A	
Tested by:	Date:	
	By:	Date:
Packaged by: Jackie Glasgow / Staci Bodle	Date: 1/4/2006 10:54:16/	No. Units: 12
Balance Used: AT261	Serial #: 1119141429	

Compound	CAS	Storage Location	Lot #	Purity	Target Conc(ug/ml)	Target Weight	Actual Weight	Calc Conc(ug/ml)
2-Chloroethyl vinyl ether	110-75-8	FA1A11D	03206CI	0.99	5,000.00	250.00	250.00	5,000.00

TITLE: ANALYTICAL METHODS FOR THE ANALYSIS OF GC\MS VOLATILE SAMPLES 8260B

SUPERCEDES: Revision 7

Certificate of Composition

8260 + #3

MVSC 42 4 → 13

DESCRIPTION: SEVERN TRENT LABS

QUOTE 20460869 LOT NO.: LB25705 MFG DATE: Dec-2004

SOLVENT: DEIONIZED WATER

ANALYTE (1)	CAS NUMBER	PERCENT PURITY (2)	WEIGHT CONCENTRATION (3)	SUPELCO LOT NO
ACROLEIN	107-02-8	98.4	20008 +/- 100.0	LB21530
ACRYLONITRILE	107-13-1	99.9	20000 +/- 100.0	LB25800

(1) Listed in alphabetical order.
(2) Determined by capillary GC-FID, unless otherwise noted.
(3) NIST traceable weights are used to verify balance calibration with the preparation of each lot. Concentration of analyte in solution is ug/ml +/- 0.5%, uncertainty based upon balance and Class A volumetric glassware. Weights are corrected for analytes less than 98% pure.

Elwood Doughty
QA Manager

Supelco warrants that its products conform to the information contained in this publication. Purchaser must determine the suitability of the product for its particular use. Please see the latest catalog or order invoice and packing slip for additional terms and conditions of sale.

SUPELCO
595 North Harrison Road • Bellefonte, PA 16823-0048 USA • Phone (814) 359-3441

STL BUFFALO
LABORATORY STANDARD OPERATING PROCEDURES

SOP No.	Revision No.	Effective Date	Page
AMV-8260B-56	8	May 31, 2006	50 of 62

TITLE: ANALYTICAL METHODS FOR THE ANALYSIS OF GC\MS VOLATILE SAMPLES 8260B

SUPERCEDES: Revision 7

8260+#4 mvsc 5 1→10

Chemical Standard Batch Sheet
Lot #: A042268

Catalog #: 556843	Target: 5000 ug/ml	
Description: Custom Vinyl Acetate Standard		
Solvent: P&T Methanol	Solvent Lot: A038421	Final Volume: 25 ml

Made by: Joe Tallon	Date: 1/4/2006 9:40:21A	
Tested by:	Date:	
	By:	Date:
Packaged by: Jackie Glasgow / Staci Bodle	Date: 1/4/2006 10:58:29A	No. Units: 12
Balance Used: AT261	Serial #: 1119141429	

Compound	CAS	Storage Location	Lot #	Purity	Target Conc(ug/ml)	Target Weight	Actual Weight	Calc Conc(ug/ml)
Vinyl acetate	108-05-4	FA1A9A	08831CW	0.99	5,000.00	125.00	125.00	5,000.00

SOP No.	Revision No.	Effective Date	Page
AMV-8260B-56	8	May 31, 2006	51 of 62

TITLE: ANALYTICAL METHODS FOR THE ANALYSIS OF GC\MS VOLATILE SAMPLES 8260B

SUPERCEDES: Revision 7

MVSC 23 6→20
24 1→75

RESTEK

Gravimetric Certificate

110 Benner Circle
Bellefonte, PA 16823-8812
Tel: (800)356-1688
Fax: (814)353-1309

FOR LABORATORY USE ONLY-READ MSDS PRIOR TO USE.

Catalog No.: 552501 Lot No.: A044128
Description: Custom Ketones Standard
Expiration Date[1]: March 2008 Storage: Freezer

Component #	Compound	CAS#	Percent Purity[2]	Concentration (weight/volume)[3]	Percent Uncertainty[4]
1	2-Butanone (MEK)	78-93-3	99%	5,000.00 ug/ml	+/-0.08 %
2	2-Hexanone	591-78-6	99%	5,000.00 ug/ml	+/-0.08 %
3	4-Methyl-2-pentanone (MIBK)	108-10-1	99%	5,000.00 ug/ml	+/-0.08 %
4	Acetone	67-64-1	99%	5,000.00 ug/ml	+/-0.08 %

Solvent: P/T Methanol/Water (90:10)

F. Joseph Fallon - Mix Technician Balance: 1119141429

[1] ...ation date of the unopened ampul stored at recommended temperature.
[2] ... was determined by one or more of the following techniques: GC/FID, HPLC, GC/ECD, GC/MS. Value rounded to the ...nearest LOWER whole percentage. In addition to detectors listed above, chemical identity and purity are confirmed using 1 or more of the following: MS, DSC, solid probe MS, GC/FPD, GC/NPD, GC/TC, FTIR, melting point, refractive index, and Karl Fisher. See data pack or contact Restek for further details.
[3] Based upon gravimetric preperation with balance calibration verified using NIST traceable weights (seven mass levels).
[4] Percent Uncertainty based upon balance AND ASTM Class A volumetric glassware accuracy.

ISO 9001 REGISTERED

Manufactured under Restek's ISO 9001 Registered Quality System Certificate #FM60387

TITLE: ANALYTICAL METHODS FOR THE ANALYSIS OF GC\MS VOLATILE SAMPLES 8260B

SUPERCEDES: Revision 7

Table 9
Second Source

ULTRA SCIENTIFIC
Analytical Solutions

Certificate of Analysis

VOC Mixture

Product DWM-588 **Expiration Date:** Dec-2008
Lot Number: CB-2659 **Page:** 2 of 3

Analyte	CAS#	Analyte Lot	True Value
1,2-dibromo-3-chloropropane	000096-12-8	OGF-01	2005 ± 10 µg/mL
1,2-dichloropropane	000078-87-5	DC-120777	2005 ± 10 µg/mL
1,3-dichloropropane	000142-28-9	PR-17916MR	2006 ± 10 µg/mL
2,2-dichloropropane	000594-20-7	CI-05304BI	2005 ± 10 µg/mL
1,1-dichloropropene	000563-58-6	34768-21	2006 ± 10 µg/mL
cis-1,3-dichloropropene	010061-01-5	35072-03	2006 ± 10 µg/mL
trans-1,3-dichloropropene	010061-02-6	34251-41	2005 ± 10 µg/mL
hexachlorobutadiene	000087-68-3	339923/1	2005 ± 10 µg/mL
1,2,3-trichloropropane	000096-18-4	12020TF	2006 ± 10 µg/mL
naphthalene	000091-20-3	14205KB	2005 ± 10 µg/mL
benzene	000071-43-2	31072	2006 ± 10 µg/mL
n-butylbenzene	000104-51-8	AA-28519CO	2005 ± 10 µg/mL
sec-butylbenzene	000135-98-8	MR-11305DN	2006 ± 10 µg/mL
tert-butylbenzene	000098-06-6	MQ-04010MQ	2006 ± 10 µg/mL
ethylbenzene	000100-41-4	033067	2005 ± 10 µg/mL
isopropylbenzene	000098-82-8	EN-00621TG	2006 ± 10 µg/mL
4-isopropyltoluene	000099-87-6	PP-05104CP	2006 ± 10 µg/mL
n-propylbenzene	000103-65-1	LO-14503MR	2006 ± 10 µg/mL
styrene	000100-42-5	MQ-11229MQ	2005 ± 10 µg/mL
toluene	000108-88-3	43045	2006 ± 10 µg/mL
1,2,4-trimethylbenzene	000095-63-6	BO-13528BI	2006 ± 10 µg/mL
1,3,5-trimethylbenzene	000108-67-8	KM-02011HM	2007 ± 10 µg/mL
o-xylene	000095-47-6	DO-06834CO	2006 ± 10 µg/mL
m-xylene	000108-38-3	DI-00459CJ	2006 ± 10 µg/mL

Balances used in the manufacture of this standard are calibrated with weights traceable to NIST in compliance with ANSI/NCSL Z-540-1 and ISO 9001.

Quality Endorsed Company ISO 9001 SAI Global Registered

ACCREDITED ISO 17025 Cert. No. 0851-01

250 Smith Street, North Kingstown, RI 02852 USA
401-294-9400 Fax: 401-295-2330
www.ultrasci.com

Dr. Edward Fitzgerald,
Senior Scientist

SOP No.	Revision No.	Effective Date	Page
AMV-8260B-56	8	May 31, 2006	53 of 62

TITLE: ANALYTICAL METHODS FOR THE ANALYSIS OF GC\MS VOLATILE SAMPLES 8260B

SUPERCEDES: Revision 7

ULTRA SCIENTIFIC
Analytical Solutions

Certificate of Analysis

VOC Mixture

Product DWM-588 **Expiration Date:** Dec-2008
Lot Number: CB-2659 **Page:** 3 of 3

Analyte	CAS#	Analyte Lot	True Value
p-xylene	000106-42-3	03747LN	2005 ± 10 µg/mL
1,4-dichlorobenzene	000106-46-7	06205KA	2005 ± 10 µg/mL
bromobenzene	000108-86-1	CG-02513MF	2006 ± 10 µg/mL
chlorobenzene	000108-90-7	63148HZ	2006 ± 10 µg/mL
2-chlorotoluene	000095-49-8	KS-06506BN	2005 ± 10 µg/mL
4-chlorotoluene	000106-43-4	CR-14512LQ	2005 ± 10 µg/mL
1,2-dichlorobenzene	000095-50-1	08946KY	2005 ± 10 µg/mL
1,3-dichlorobenzene	000541-73-1	JN-05902LZ	2006 ± 10 µg/mL
1,2,3-trichlorobenzene	000087-61-6	LI-12912PF	2006 ± 10 µg/mL
1,2,4-trichlorobenzene	000120-82-1	00334TQ	2006 ± 10 µg/mL
bromomethane	000074-83-9	06623AQ	2008 ± 10 µg/mL
chloroethane	000075-00-3	00223KG	2009 ± 10 µg/mL
chloromethane	000074-87-3	07-44048	2009 ± 10 µg/mL
dichlorodifluoromethane	000075-71-8	N960053	2008 ± 10 µg/mL
vinyl chloride	000075-01-4	UN-1086	2009 ± 10 µg/mL

Matrix: methanol (methyl alcohol)

Balances used in the manufacture of this standard are calibrated with weights traceable to NIST in compliance with ANSI/NCSL Z-540-1 and ISO 9001.

Quality System
Quality Endorsed Company
ISO 9001
SAI Global Registered

ACCREDITED
ISO 17025
Cert. No. 0851- 01

250 Smith Street, North Kingstown, RI 02852 USA
401-294-9400 Fax: 401-295-2330
www.ultrasci.com

Dr. Edward Fitzgerald,
Senior Scientist

SOP No.	Revision No.	Effective Date	Page
AMV-8260B-56	8	May 31, 2006	54 of 62

TITLE: ANALYTICAL METHODS FOR THE ANALYSIS OF GC\MS VOLATILE SAMPLES 8260B

SUPERCEDES: Revision 7

Table10
8260+ Second Source

8260+#1 Sec Source MUSC61→10

Certificate of Composition

DESCRIPTION: SEVERN TRENT LABS

QUOTE 20687608 LOT NO.: LB35787 EXPIRATION DATE: Jan-2007

SOLVENT: METHANOL

ANALYTE (1)	CAS NUMBER	PERCENT PURITY (2)	WEIGHT CONCENTRATION (3)	SUPELCO LOT NO
ACETONITRILE	75-05-8	99.9	40001 +/- 200.0	LB34175
CARBON DISULFIDE	75-15-0	99.9 (a)	999 +/- 5.0	LB09107
CYCLOHEXANE	110-82-7	99.9	1000 +/- 5.0	LB18076
ETHYL METHACRYLATE	97-63-2	99.3	1002 +/- 5.0	LA29651
FREON 113	76-13-1	99.9 (b)	1001 +/- 5.0	LA33286
METHYL ACETATE	79-20-9	98.1	1001 +/- 5.0	LB32233
METHYL CYCLOHEXANE	108-87-2	99.8	1001 +/- 5.0	LB06982
METHYL TERT-BUTYL ETHER	1634-04-4	99.9	1002 +/- 5.0	LB34302
TETRAHYDROFURAN	109-99-9	97.4	4999 +/- 25.0	LA58136
TRANS-1,4-DICHLORO-2-BUTENE	110-57-6	98.2	5002 +/- 25.0	LB10202
1-CHLOROHEXANE	544-10-5	99.9	1000 +/- 5.0	LB18907

(1) Listed in alphabetical order.
(2) Determined by capillary GC-FID, unless otherwise noted.
a) GC; detector FPD
b) GC; detector HALL
(3) NIST traceable weights are used to verify balance calibration with the preparation of each lot. Concentration of analyte in solution is ug/ml +/- 0.5%, uncertainty based upon balance and Class A volumetric glassware. Weights are corrected for analytes less than 98% pure.

Elwood Doughty
QA Manager

Supelco warrants that its products conform to the information contained in this publication. Purchaser must determine the suitability of the product for its particular use. Please see the latest catalog or order invoice and packing slip for additional terms and conditions of sale.

SUPELCO
595 North Harrison Road • Bellefonte, PA 16823-0048 USA • Phone (814) 359-3441

SOP No.	Revision No.	Effective Date	Page
AMV-8260B-56	8	May 31, 2006	55 of 62

TITLE: **ANALYTICAL METHODS FOR THE ANALYSIS OF GC\MS VOLATILE SAMPLES 8260B**

SUPERCEDES: Revision 7

8260+HL
See Source
MVSC6
11→20

Certificate of Composition

DESCRIPTION: SEVERN TRENT LABS

QUOTE 20687609 LOT NO.: LB35788 EXPIRATION DATE: Jan-2007

SOLVENT: DEIONIZED WATER 50 %
METHANOL 50 %

ANALYTE (1)	CAS NUMBER	PERCENT PURITY (2)	WEIGHT CONCENTRATION (3)	SUPELCO LOT NO
ACETONE	67-64-1	99.9	5004 +/- 25.0	LB31953
IODOMETHANE	74-88-4	99.9	1004 +/- 5.0	LA73149
VINYL ACETATE	108-05-4	99.9	5002 +/- 25.0	LB31606
2-BUTANONE	78-93-3	99.9	5004 +/- 25.0	LB19842
2-HEXANONE	591-78-6	99.9	5004 +/- 25.0	LB08447
4-METHYL-2-PENTANONE	108-10-1	99.9	5004 +/- 25.0	LA99226

(1) Listed in alphabetical order.
(2) Determined by capillary GC-FID, unless otherwise noted.
(3) NIST traceable weights are used to verify balance calibration with the preparation of each lot. Concentration of analyte in solution is ug/ml +/- 0.5%, uncertainty based upon balance and Class A volumetric glassware. Weights are corrected for analytes less than 98% pure.

Elwood Doughty
QA Manager

SUPELCO
595 North Harrison Road • Bellefonte, PA
16823-0048 USA • Phone (814) 359-3441

SOP No.	Revision No.	Effective Date	Page
AMV-8260B-56	8	May 31, 2006	56 of 62

TITLE: ANALYTICAL METHODS FOR THE ANALYSIS OF GC\MS VOLATILE SAMPLES 8260B

SUPERCEDES: Revision 7

Certificate of Analysis

MUSC 66 3-7

DESCRIPTION: 2-Chloroethyl vinyl ether

CATALOG NO.: 40017 MFG DATE: Feb-2005

LOT NO.: LB27794 EXPIRATION DATE: Feb-2008

SOLVENT: METHANOL

ANALYTE	CAS NUMBER	PERCENT PURITY(1)	WEIGHT(2) CONCENTRATION	ANALYTICAL(3) CONCENTRATION		STD DEV	SUPELCO LOT NO
2-CHLOROETHYL VINYL ETHER	110-75-8	99.9	5000	5000	+/-	55.9	LB01239

(1) Determined by capillary GC-FID, unless otherwise noted.
(2) NIST traceable weights are used to verify balance calibration with the preparation of each lot. Concentration of analyte in solution is ug/ml +/- 0.5%, uncertainty based upon balance and Class A volumetric glassware. Weights are corrected for analytes less than 98% pure.
(3) Determined by chromatographic analysis against an independently prepared reference lot. Mean of replicate injections.

Elwood Doughty
Quality Control Supervisor

Supelco warrants that its products conform to the information contained in this publication. Purchaser must determine the suitability of the product for its particular use. Please see the latest catalog or order invoice and packing slip for additional terms and conditions of sale.

SUPELCO
595 North Harrison Road
Bellefonte, PA 16823-0048 USA
Phone (814) 359-3441

SOP No.	Revision No.	Effective Date	Page
AMV-8260B-56	8	May 31, 2006	57 of 62

TITLE: ANALYTICAL METHODS FOR THE ANALYSIS OF GC\MS VOLATILE SAMPLES 8260B

SUPERCEDES: Revision 7

8260 #3 Sec. Source MVSC 7 1-7/10

Certificate of Composition

DESCRIPTION: SEVERN TRENT LABS

QUOTE 20687606 LOT NO.: LB35789 EXPIRATION DATE: Jul-2006

SOLVENT: DEIONIZED WATER

ANALYTE (1)	CAS NUMBER	PERCENT PURITY (2)	WEIGHT CONCENTRATION (3)	SUPELCO LOT NO
ACROLEIN	107-02-8	98.4	20012 +/- 100.1	LB21530
ACRYLONITRILE	107-13-1	99.9	20008 +/- 100.0	LB25800

(1) Listed in alphabetical order.
(2) Determined by capillary GC-FID, unless otherwise noted.
(3) NIST traceable weights are used to verify balance calibration with the preparation of each lot. Concentration of analyte in solution is ug/ml +/- 0.5%, uncertainty based upon balance and Class A volumetric glassware. Weights are corrected for analytes less than 98% pure.

Elwood Doughty
QA Manager

Supelco warrants that its products conform to the information contained in this publication. Purchaser must determine the suitability of the product for its particular use. Please see the latest catalog or order invoice and packing slip for additional terms and conditions of sale.

SUPELCO
595 North Harrison Road • Bellefonte, PA 16823-0048 USA • Phone (814) 359-3441

SOP No.	Revision No.	Effective Date	Page
AMV-8260B-56	8	May 31, 2006	58 of 62

TITLE: ANALYTICAL METHODS FOR THE ANALYSIS OF GC\MS VOLATILE SAMPLES 8260B

SUPERCEDES: Revision 7

Table 11
8260 Add

MVSC 71-18-20
72-01-07

Chemical Standard Batch Sheet
Lot #: A042005

Catalog #: 552546	Target: 2000-80000 ug/ml	
Description: Custom Volatiles Standard		
Solvent: P&T Methanol	Solvent Lot: 44337	Final Volume: 100 ml

Made by: Ryan Miller	Date: 12/19/2005 10:12:
Tested by:	Date:
	By: Date:
Packaged by: / SLB / JC	Date: 12-20-05 No. Units: 12
Balance Used: AT400	Serial #: 1113372841

Compound	CAS	Storage Location	Lot #	Purity	Target Conc(ug/ml)	Target Weight	Actual Weight	Calc Conc(ug/ml)
Allyl chloride (	107-05-1	FA1B13D	00305HO	0.99	2,000.00	200.00	200.00	2,000.00
Chloroprene	126-99-8	FA1D8B	051215JLM	0.99	2,000.00	200.00		0.00
Pentachloroethane	76-01-7	FA1C3B	OGL01	0.98	2,000.00	200.00	200.00	2,000.00
1,1,2-Trichlorotrifluoroetha	76-13-1	FA1A11A	01404PV	0.99	2,000.00	200.00	200.00	2,000.00
Dichlorodifluoromethane	75-71-8	HOOD	A042007	0.99	2,000.00		4.20 (ml)	1,978.41
Dichlorofluoromethane	75-43-4	HOOD	A042008	0.99	2,000.00		3.10 (ml)	1,974.39
Chlorodifluoromethane	75-45-6	VOA Lab	A042009	0.99	2,000.00		2.40 (ml)	2,016.62
Ethyl acetate	141-78-6	FA1C5B	11073ED	0.99	2,000.00	200.00	200.00	2,000.00
Diisopropyl ether (DIPE)	108-20-3	FA1C2B	13450CB	0.99	2,000.00	200.00	200.00	2,000.00
Hexachloroethane	67-72-1	RA1B6D	12719A0	0.99	2,000.00	200.00	200.00	2,000.00
Methyl methacrylate	80-62-6	FA1C2D	09505TO	0.99	2,000.00	200.00	200.00	2,000.00
Methacrylonitrile	126-98-7	FA1C2C	04406MI	0.99	2,000.00	200.00	200.00	2,000.00
Diethyl ether (ethyl ether)	60-29-7	FA1C1A	17676TQ	0.99	2,000.00	200.00	200.00	2,000.00
2-Nitropropane	79-46-9	RA1C11C	04609PN	0.98	10,000.00	1,000.00	1,000.00	10,000.00
Pr itrile	107-12-0	FA1C3D	10101EB	0.98	20,000.00	2,000.00	2,000.00	20,000.00
Cyclohexanone	108-94-1	RA1D2B	10513PA	0.99	20,000.00	2,000.00	2,000.00	20,000.00
ert-Butanol (TBA)	75-65-0	RA1H2D	06648PC	0.99	40,000.00	4,000.00	4,000.00	40,000.00
l-Butanol	71-36-3	FA1G1B	8238	0.99	80,000.00	8,000.00	8,000.00	80,000.00
sobutanol	78-83-1	FA1C3A	00439HD	0.99	80,000.00	8,000.00	8,000.00	80,000.00
,4-Dioxane	123-91-1	RA1H3B	03053BD	0.99	80,000.00	8,000.00	8,000.00	80,000.00

SOP No.	Revision No.	Effective Date	Page
AMV-8260B-56	8	May 31, 2006	59 of 62

TITLE: ANALYTICAL METHODS FOR THE ANALYSIS OF GC\MS VOLATILE SAMPLES 8260B

SUPERCEDES: Revision 7

Add + MVSC 74 18→20
L 75 1→7

RESTEK

CERTIFICATE OF COMPOSITION

FOR LABORATORY USE ONLY - READ MSDS PRIOR TO USE

110 Benner Circle
Bellefonte, PA 16823-8812
Tel: (800) 356-1688
Fax: (814) 353-1309

Catalog No.: 558661 Lot No.: A042271
Description: Custom Volatiles Standard
Expiration Date[1]: July 2007 Storage: Freezer

Elution Order	Compound	CAS#	Percent Purity[2]	Concentration[3]	Percent Uncertainty[4]
1	2-Propanol (isopropanol)	67-63-0	99%	20000 ug/mL	+/- 0.1
2	1-Propanol	71-23-8	99%	20000 ug/mL	+/- 0.1
3	n-Hexane (C6)	110-54-3	99%	1000 ug/mL	+/- 0.1
4	Acetaldehyde dimethyl acetal	534-15-6	99%	5000 ug/mL	+/- 0.1
5	Ethyl-tert-butyl ether (ETBE)	637-92-3	99%	1000 ug/mL	+/- 0.1
6	tert-Amyl methyl ether (TAME)	994-05-8	99%	1000 ug/mL	+/- 0.1
7	n-Heptane (C7)	142-82-5	99%	1000 ug/mL	+/- 0.1
8	2-Chlorobenzotrifluoride	88-16-4	99%	1000 ug/mL	+/- 0.1
9	3-Chlorobenzotrifluoride	98-15-7	99%	1000 ug/mL	+/- 0.1
10	4-Chlorobenzotrifluoride	98-56-6	98%	1000 ug/mL	+/- 0.1
11	3-Chlorotoluene	108-41-8	99%	1000 ug/mL	+/- 0.1
12	1,2,3-Trimethylbenzene	526-73-8	99%	1000 ug/mL	+/- 0.1
13	Dicyclopentadiene	77-73-6	98%	1000 ug/mL	+/- 0.1
14	1,3,5-Trichlorobenzene	108-70-3	99%	1000 ug/mL	+/- 0.1
	Solvent: P&T Methanol	67-56-1	99%		

Column:
105m x .32mm x 1.8um
Rtx-502.2 (cat #10921)

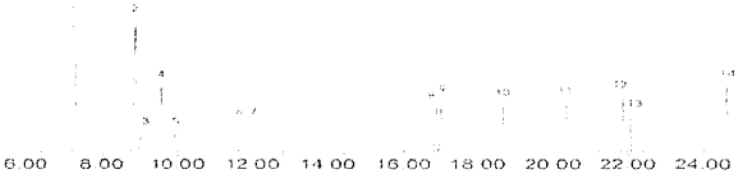
Carrier Gas:
helium @ 2.2 ml/min

Temp. Program:
40°C (hold 2 min.) to 240°C
@ 8°C/min. (hold 10 min.)

Inj. Temp:
200°C

Det. Temp:
250°C

Det. Type:
MSD



Manufactured By: FJT
John Liggett - QA Analyst

1 Expiration date of the unopened ampul stored at recommended temperature.
2 Purity was determined by one or more of the following techniques: GC/FID, HPLC, GC/ECD, GC/MS. Value rounded to the nearest LOWER whole percentage. In addition to detectors listed above, chemical identity and purity are confirmed using 1 or more of the following: MS, DSC, solid probe MS, GC/FPD, GC/NPD, GC/TC, FTIR, melting point, refractive index, and Karl Fisher. See data pack or contact Restek for further details.
3 Based upon gravimetric preperation with balance calibration verified using NISTtraceable weights (7 mass levels).
4 Percent Uncertainty based upon balance AND ASTM Class A volumetric glassware accuracy.

ISO 9001 REGISTERED
Manufactured Under Restek's ISO 9001 Registered Quality System Certificate #FM90397

TITLE: ANALYTICAL METHODS FOR THE ANALYSIS OF GC\MS VOLATILE SAMPLES 8260B

SUPERCEDES: Revision 7

Table 12
BFB
IS & SS

MVSC 3 1-710

RESTEK

CERTIFICATE OF ANALYSIS

FOR LABORATORY USE ONLY - READ MSDS PRIOR TO USE

110 Benner Circle
Bellefonte, PA 16823-8812
Tel: (800) 356-1688
Fax: (814) 353-1309

Catalog No.: 30067 Lot No.: A038850
Description: 4-Bromofluorobenzene Standard
Expiration Date[1]: January 2010 Storage: Freezer

Elution Order	Compound	CAS#	Percent Purity[2]	Concentration[3]	Percent Uncertainty[4]
1	1-Bromo-4-fluorobenzene (BFB)	460-00-4	99%	2500 ug/mL	+/- 0.1
	Solvent: P&T Methanol	67-56-1	99%		

Column:
105m x .53mm x 3.0um
Rtx-502.2 (cat.#10910)

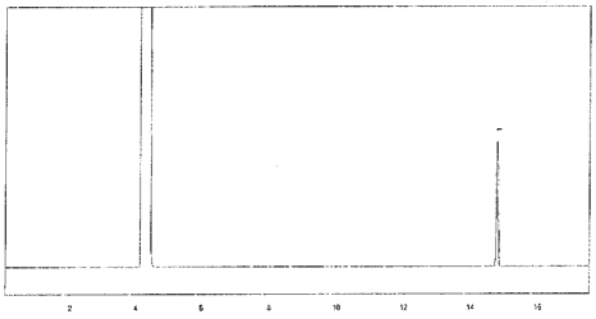
Carrier Gas:
hydrogen @ 40 cm/sec

Temp. Program:
50°C to 240°C @ 10°C/min.

Inj. Temp:
200°C

Det. Temp:
250°C

Det. Type:
FID



Manufactured By: MEW
John Lidgett - QA Analyst

1 Expiration date of the unopened ampul stored at recommended temperature.
2 Purity was determined by one or more of the following techniques: GC/FID, HPLC, GC/ECD, GC/MS. Value rounded to the nearest LOWER whole percentage. In addition to detectors listed above, chemical identity and purity are confirmed using 1 or more of the following: MS, DSC, solid probe MS, GC/FPD, GC/NPD, GC/TC, FTIR, melting point, refractive index, and Karl Fisher. See data pack or contact Restek for further details.
3 Based upon gravimetric preperation with balance calibration verified using NISTtraceable weights (7 mass levels).
4 Percent Uncertainty based upon balance AND ASTM Class A volumetric glassware accuracy.

ISO 9001 REGISTERED
Manufactured Under Restek's ISO 9001 Registered Quality System
Certificate #FM60397

Page 1 1

SOP No.	Revision No.	Effective Date	Page
AMV-8260B-56	8	May 31, 2006	61 of 62

TITLE: ANALYTICAL METHODS FOR THE ANALYSIS OF GC\MS VOLATILE SAMPLES 8260B

SUPERCEDES: Revision 7

MWSC08 (11-20)

RESTEK

CERTIFICATE OF ANALYSIS

FOR LABORATORY USE ONLY - READ MSDS PRIOR TO USE

110 Benner Circle
Bellefonte, PA 16823-8812
Tel: (800) 356-1688
Fax: (814) 353-1309

Catalog No.: 30091 Lot No.: A036981

Description: L/C VOA Internal Standard Mix

Expiration Date[1]: April 2010 Storage: Freezer

Elution Order	Compound	CAS#	Percent Purity[2]	Concentration[3]	Percent Uncertainty[4]
1	1,4-Difluorobenzene	540-36-3	99%	2500 ug/mL	+/- 0.1
2	Chlorobenzene-d5	3114-55-4	99%	2500 ug/mL	+/- 0.1
3	1,4-Dichlorobenzene-d4	3855-82-1	99%	2500 ug/mL	+/- 0.1
	Solvent: P&T Methanol	67-56-1	99%		

Column:
105m x .53mm x 3.0um
Rtx-502.2 (cat #10910)

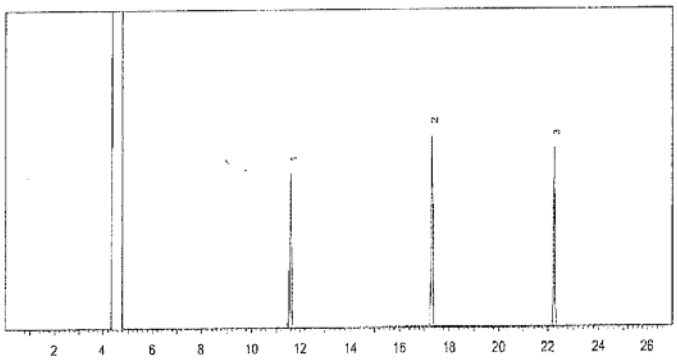
Carrier Gas:
hydrogen @ 40 cm/sec.

Temp. Program:
40°C (hold 2 min.) to 240°C
@ 8°C/min.

Inj. Temp:
200°C

Det. Temp:
250°C

Det. Type:
FID



Manufactured By: n/a
John Lidgett - QA Analyst

1 Expiration date of the unopened ampul stored at recommended temperature.
2 Purity was determined by one or more of the following techniques: GC/FID, HPLC, GC/ECD, GC/MS. Value rounded to the nearest LOWER whole percentage. In addtion to detectors listed above, chemical identity and purity are confirmed using 1 or more of the following: MS, DSC, solid probe MS, GC/FPD, GC/NPD, GC/TC, FTIR, melting point, reftactive index, and Karl Fisher. See data pack or contact Restek for further details.
3 Based upon gravimetric preperation with balance calibration verified using NISTtraceable weights (7 mass levels).
4 Percent Uncertainty based upon balance AND ASTM Class A volumetric glassware accuracy.

ISO 9001 REGISTERED
Manufactured Under Restek's ISO 9001 Registered Quality System Certificate #FM60397

SOP No.	Revision No.	Effective Date	Page
AMV-8260B-56	8	May 31, 2006	62 of 62

TITLE: **ANALYTICAL METHODS FOR THE ANALYSIS OF GC\MS VOLATILE SAMPLES 8260B**

SUPERCEDES: Revision 7

ULTRA SCIENTIFIC
Analytical Solutions

Certificate of Analysis

MVSC { 73 18-20
74 1-17

Method 8260 Surrogate Standard Mixture

Product STM-530 (4) Comp 5.0r **Expiration Date:** Sep-2008
Lot Number: CB-1899 **Page:** 1 of 1

This Certified Reference Material (CRM) was manufactured and verified in accordance with ULTRA's ISO 9001:2000 registered quality system, and the analyte concentrations were verified by our ISO 17025 accredited laboratory. The true value and uncertainty value at the 95% confidence level for each analyte, determined gravimetrically, is listed below.

Analyte	CAS#	Analyte Lot	True Value
4-bromofluorobenzene	000460-00-4	12515BO	2512 ± 13 µg/mL
dibromofluoromethane	001868-53-7	90004843	2512 ± 13 µg/mL
1,2-dichloroethane-d4	017060-07-0	PSO 5A-048	2512 ± 13 µg/mL
toluene-d8	002037-26-5	PSO AG-433	2510 ± 13 µg/mL

Matrix: methanol (methyl alcohol)

Balances used in the manufacture of this standard are calibrated with weights traceable to NIST in compliance with ANSI/NCSL Z-540-1 and ISO 9001.

Quality System
Quality Endorsed Company ISO 9001 SAI Global Registered

ACCREDITED
ISO 17025 Cert. No. 0851-01

250 Smith Street, North Kingstown, RI 02852 USA
401-294-9400 Fax: 401-295-2330
www.ultrasci.com

Dr. Edward Fitzgerald,
Senior Scientist

STL Buffalo
LABORATORY STANDARD OPERATING PROCEDURES

SOP No. ASP-1311-21	Revision No. 8	Effective Date October 26, 2005	Page 1 of 33

TITLE: TOXICITY CHARACTERISTIC LEACHING PROCEDURE (TCLP)

SUPERCEDES: Revision 7 – AWC-1311-21

REVIEWED AND APPROVED BY:	SIGNATURE	DATE
Verl Preston, Quality Manager		
Christopher Spencer, Laboratory Director		
Kathleen Aldrich, OP Department Manager		

1.0 IDENTIFICATION OF TEST METHODS

EPA Method 1311, Toxicity Characteristic Leachate Procedure (TCLP)

2.0 APPLICABLE MATRIX

This method is applicable to liquid, solid and multiphasic wastes.

3.0 METHOD DETECTION LIMIT

N/A

4.0 SCOPE AND APPLICATIONS

4.1 If a total analysis of any of the waste demonstrates that individual analytes are not present in the waste, or that they are present but at such low concentrations that the appropriate regulatory levels could not possibly be exceeded, the TCLP need not be run.

4.2 If an analysis of any one of the liquid fractions of the TCLP extract indicates that a regulated compound is present at such high concentrations that, even after accounting for dilution from the other fractions of the extract, the concentration would be above the regulatory level for that compound, then the waste is hazardous and it is not necessary to analyze the remaining fractions of the extract.

TITLE: TOXICITY CHARACTERISTIC LEACHING PROCEDURE (TCLP)

SUPERCEDES: Revision 7 – AWC-1311-21

5.0 SUMMARY OF TEST METHOD

5.1 The wastes are initially characterized and defined by matrix (liquid, solid or mixed phase) and by pH. This preliminary characterization determines the type of TCLP extraction procedure to be applied. Wastes containing less than 0.5 percent dry solid material are classified as liquid wastes and after filtration, are defined as the final TCLP extract. If the wastes contain greater than or equal to 0.5 percent solids, the liquid, if any, is separated from the solid phase and stored for later analysis. The solid phase is extracted with an amount of extraction fluid equal to 20 times the weight of the solid phase. Samples for volatiles analysis are extracted in a special pressurized extraction vessel. Extractions are conducted for a period of 18 $\pm$ 2 hours, followed by analysis of the extracts by approved EPA methodologies.

6.0 DEFINITIONS

6.1 TCLP = Toxicity Characteristic Leaching Procedure

7.0 INTERFERENCES

7.1 Potential interference that may be encountered during analysis are discussed in individual analytical methods.

7.2 Glassware and equipment contamination may result in analyte degradation. Soap residue on glassware and equipment may contribute to this. All glassware and equipment should be rinsed very carefully to avoid this problem.

7.3 Phthalates may be eliminated by proper glassware cleanup and by avoiding plastics. Only glass, Teflon or stainless steel tumblers may be used for leachates to be analyzed for organics. Plastic tumblers may be used for leachates to be analyzed for the metals.

8.0 SAFETY

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

8.2. The use of safety glasses and protective clothing are required throughout the entire procedure.

8.3. The solvents and reagents used in this extraction procedure are hazardous if improperly handled. Care must be taken during preparation and use of acetic acid, hydrochloric acid, nitric acid and sodium hydroxide solutions. Additional health and safety information is available and must be read from the Material Safety Data Sheets (MSDS) maintained in the laboratory.

8.4. The acetic acid extraction fluid in the nonvolatile extraction vessels may react with carbamates in the sample to form CO2 gas. Pressure buildup could potentially cause the vessels to explode. The vessels should be periodically vented during extraction, and once again prior to removal from the rotation apparatus to prevent this occurrence.

8.5. Proper precautions must be taken when using pressurized nitrogen during the filtration and pressurized procedures.

8.6. All steps of procedure should be done under a fume hood.

8.7. 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
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.
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.

TITLE: TOXICITY CHARACTERISTIC LEACHING PROCEDURE (TCLP)

SUPERCEDES: Revision 7 – AWC-1311-21

Material (1)	Hazards	Exposure Limit (2)	Signs and symptoms of exposure
Sodium Hydroxide	Corrosive	2 Mg/M3-Ceiling	Severe irritant. Effects from inhalation of dust or mist vary from mild irritation to serious damage of the upper respiratory tract, depending on severity of exposure. Symptoms may include sneezing, sore throat or runny nose. Contact with skin can cause irritation or severe burns and scarring with greater exposures. Causes irritation of eyes, and with greater exposures it can cause burns that may result in permanent impairment of vision, even blindness.
1 – Always add acid to water to prevent violent reactions.			
2 – Exposure limit refers to the OSHA regulatory exposure limit.			

9.0 EQUIPMENT AND SUPPLIES

9.1 Agitation apparatus: The rotation apparatus must be capable of rotating the extraction vessel in an end-over-end fashion at 30 $\pm$ 2 rpm.

9.2 Extraction Vessels

9.2.1 Zero-Headspace Extraction Vessel (ZHE)-This device is for use only when the waste is being tested for the mobility of volatile analytes. The ZHE allows for liquid/solid separation within the device and effectively precludes headspace. The vessels shall have an internal volume of 500-600 mL and be equipped to accommodate a 90-110mm filter. The device contains O-rings, which should be replaced frequently.

9.2.2 Bottle Extraction Vessel- Borosilicate medium walled glass, Teflon screw cap, for semi-volatile and pesticides.

9.2.3 2 Liter plastic extraction bottles with lids, for metal only extractions.

9.3 TCLP-ZHE Filtration Apparatus.

9.4 ZHE Extraction Fluid Transfer Device

9.5 A pH Meter and pH probe-accurate to $\pm$ 0.05 units at 25° C. Prior to TCLP pH measurements, calibrate the pH meter and electrode in accordance with the manufacturer's recommendations. Calibrate the pH meter using buffers, which bracket the pH of the samples and extraction fluid.

9.6 Laboratory Balance-balance must be accurate to within $\pm$ 0.01 grams. All weight measurements are to be within $\pm$ 0.1 grams.

9.7 Magnetic stirrer

9.8 Glassware:

9.8.1 Beakers, glass, 250-, 500-, 1000- and 2000-mL.

9.8.2 Graduated cylinders, glass 100- and 2000mL.

9.8.3 Erlenmeyer flasks, glass, 1000-mL.

9.8.4 Whatman Glass Microfiber Filters – grade GF/F.

9.8.5 Pre-washed Nitric filters purchased by ESS.

9.9 Mortar and Pestle.

9.10 Standard Sieve 9.5mm.

9.11 Compressed Nitrogen

10.0 REAGENTS AND STANDARDS

10.1 Reagent Water

10.1.1 Water for nonvolatile extractions is ASTM Type II from the deionized water system in the Wet Chemistry Lab..

10.1.2 Reagent Water for Volatile extractions is generated from the Volatile-Free purification system located in the GC/MS Lab.

10.2 Glacial Acetic Acid, ACS reagent grade.

10.3 Hydrochloric Acid (1N), ACS reagent grade.

10.4 Sodium hydroxide (1N), ACS reagent grade.

10.5 Nitric acid (1N), ACS reagent grade.

10.6 Extraction Fluid #1, pH=4.93 (Section 14.2).

10.7 Extraction Fluid #2, pH=2.88 (Section 14.2).

TITLE: TOXICITY CHARACTERISTIC LEACHING PROCEDURE (TCLP)

SUPERCEDES: Revision 7 – AWC-1311-21

11.0 SAMPLE COLLECTION, PRESERVATION, SHIPMENT AND STORAGE

11.1 The physical state of the waste sample may place requirements on the minimal size of the field sample for the TCLP procedure. Quality control requirements such as matrix spikes (Section 12.0) may require additional Aliquots be collected.

11.2 Preservatives shall not be added to samples prior to extraction.

11.3 Samples may be refrigerated unless refrigeration results in irreversible physical changes to the waste. If precipitation occurs, the entire sample (including precipitate) should be extracted.

11.4 When the waste is to be evaluated for volatiles analytes, care shall be taken to minimize the loss of volatiles. Samples shall be collected and stored in order to prevent the loss of volatiles analytes.

11.5 TCLP extractions and the analysis of the extracts must be conducted within the time period specified in Section 12.4. Extracts to be analyzed for metals must be preserved with 1N ACS reagent grade nitric acid to a pH of less than 2, unless precipitation occurs. If precipitation is observed, follow the steps outlined in Section 14.3.3.2. Extracts should be preserved for other analytes according to the guidance given in the individual analysis methods. Extracts for volatiles shall not be allowed to come into contact with the atmosphere (no headspace) to prevent losses.

12.0 QUALITY CONTROL

12.1 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. this applies to both volatile and nonvolatile extractor vessels.

12.2 A Matrix Spike must be analyzed with every 20 TCLP extractions.

12.2.1 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.

12.2.2 Matrix spike analyte concentrations should approximate the regulatory levels. 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 five times the method detection limit.

12.2.3 Matrix spike recoveries are calculated by the following formula:

% R (%Recovery)= 100 (Xs-Xu)/K

where: XS= measured value for the spiked sample.
XU=measured value for the unspiked sample.
K= known value of the spike in the sample.

12.3 TCLP extractions, preparations and analyses must be conducted within the following time periods:

From Field Collection to TCLP Extraction (Filtration)

Volatiles	14 days
Semivolatiles	14 days
Mercury	28 days
Metals, except mercury	180 days

From TCLP Extraction to Preparation Extraction

Volatiles	Not Applicable
Semivolatiles	7 days
Mercury	Not Applicable
Metals, except mercury	Not Applicable

From Preparation Extraction to Analysis

Volatiles	14 days
Semivolatiles	40 days
Mercury	28 days
Metals, except mercury	180 days

Total Elapsed Time

Volatiles	28 days
Semivolatiles	61 days
Mercury	56 days
Metals, except mercury	360 days

TITLE: TOXICITY CHARACTERISTIC LEACHING PROCEDURE (TCLP)

SUPERCEDES: Revision 7 – AWC-1311-21

13.0 CALIBRATION AND STANDARDIZATION

13.1. The pH meter is calibrated every day prior to use. The calibration is performed with a 7.00 pH buffer solution and a 4.01 pH buffer solution, then checked against a 10.01 pH buffer solution. The check must be accurate within ±0.05. If the check fails, repeat the calibration until a passing check has been achieved.

13.2. Analytical balances are calibrated every 6 months and checked daily to ensure calibration is maintained.

14.0 PROCEDURE

The TCLP preparation procedure can be broken down into four sections:

- Preliminary Sample Evaluation
- Preparation of Extraction Fluids
- TCLP Extraction Procedure for Nonvolatile Analytes
- TCLP Extraction Procedure for Volatile Compounds

14.1. Preliminary Sample Evaluation

14.1.1. A preliminary evaluation of the samples is performed prior to TCLP extraction. The results of the evaluation determine how the extraction is conducted and how the results are reported. The preliminary evaluation includes the following:

- Determination of percent solids.
- Determination if the waste contains insignificant amount of solid material, and is therefore the TCLP extract after filtration.
- Determination if the solid part of the waste needs particle size reduction.
- Determination of the extraction fluid to be used for the nonvolatile extractions, based on the pH of the waste.

14.1.2. Percent Solids Determination- Percent solids is defined as that fraction of a waste sample from which no liquid may be forced out by an applied pressure. The samples are filtered under pressure through glass Microfiber filters.

14.1.2.1. If the samples contain no obvious liquid phase, and contain pieces of material that exceed 1 cm in diameter, proceed to section 14.1.2 for particle size reduction before continuing. To physically verify it will yield no liquid apply pressure to a small portion of the sample using a clean spatula if no liquid rises to the surface around the spatula proceed to section 14.1.2 for particle size reduction. Document spatula test in logbook.

14.1.2.2. Prior to assembly of the filtration apparatus, clean all parts by washing with soapy water followed by rinsing with deionized water and reagent grade water.

14.1.2.3. Weigh a Whatman glass microfiber filter (grade GF/F) and record the weight in the TCLP preparation logbook.

14.1.2.4. Support the filtration apparatus on the stand.

14.1.2.5. Rinse the glass filter paper and the metal filter screen with deionized water. Place the filter paper on the screen and in the extractor such that the glass fiber filter will be facing the sample.

14.1.2.6. Transfer 100g (to the nearest 0.1 g) of a representative aliquot of the sample into a tared 250 mL beaker, record the total weight in the logbook.

14.1.2.7. Carefully pour or spread the sample onto the filter paper in the cylinder. Material may stick to the sides of the beaker. Quantitatively determine the amount transferred to the filtration apparatus by weighing the filter container and record the weight in the logbook.

NOTE: If the sample is a mixed phase sample, first decant and filter the liquid portion. After the liquid has been filtered, transfer the solid material onto the same filter and repeat the filtering process.

14.1.2.8. Gradually apply vacuum or gentle pressure of 1-10 psi, until air or pressurizing 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 2 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, and if no additional liquid has passed through the filter in any 2 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 does not result in any additional filtrate within any 2 minute period, stop the filtration.

NOTE: Some wastes, such as oily wastes and some paint wastes, will obviously contain some material that appears to be a liquid. Even after applying vacuum or pressure filtration, as outlined in section 14.1.1.7, this material may not filter. If this is the case, the material within the filtration device is defined as a solid. Do not replace the original filter with a fresh filter under any circumstances. Use only one filter.

14.1.2.9. If no liquid is forced from the sample, the sample is considered to be 100% solid waste. Proceed to section 14.1.3.

14.1.2.10. If it is obvious by visually looking at the material on the filter that a significant amount (more than 0.5%) of the material is solid, go to section.

14.1.2.11. If a small amount of residue remains on the filter, carefully remove the filter and dry it at 100 + 2° C for one hour. Weigh the filter and waste. Return the filter to the oven for an additional 15 minutes and reweigh it to demonstrate that constant weight has been reached. Use the following calculation to determine the percent dry solids:

$$\text{Percent Dry Solids} = \frac{(\text{Wt of dry waste + Filter}) - \text{Wt of filter}}{\text{Initial Wt of Waste}} \times 100$$

14.1.2.12. If the percent dry solids exceeds 0.5%, go to section 14.1.1.13. If the percent dry solids is less than 0.5%, the filtrate becomes the TCLP extract. Additional sample may need to be filtered to meet the volume required for analysis. After sufficient sample has been filtered proceed to section 14.3.

14.1.2.13. Weigh the beaker and the filtrate and subtract the initial beaker weight. The filtrate is the liquid phase. The initial sample weight minus the weight of the liquid phase is the solid phase. Percent solids is calculated as follows:

$$\text{Percent solids} \quad \frac{\text{Weight of solids}}{\text{Total weight of waste}} \times 100$$

14.1.2.14. If the percent solids exceeds 0.5%, the liquid. If any, is saved for either future combination with the TCLP extract or for separate analysis.

14.1.3. Particle Size Reduction –The solid portion (0.5% solids) of the sample is evaluated to determine whether particle size reduction is needed.

14.1.3.1. If the solid material in the sample can pass through a 9.5 mm sieve (less than 1 cm in diameter), particle size reduction is unnecessary.

14.1.3.2. If the samples need particle size reduction, crush or grind the sample with a mortar and pestle or whatever means necessary to reduce the sample's particle size. Record in logbook.

14.1.4 pH Analysis to Determine Extraction Fluid Type for Non volatile Analyses- An aliquot of sample is initially tested for pH. The results determine which fluid is used

for nonvolatile TCLP Extraction. Volatile TCLP extraction uses only extraction fluid #1 , prepared with volatile free reagent water.

14.1.4.1 Weigh out 5.0 g of representative sample. Record in logbook.

14.1.4.2 Using a 100-mL graduated cylinder, add 96.5 ml of DiH2O and stir the sample for 5 minutes using a magnetic stirrer

14.1.4.3 Measure the pH of the sample using a calibrated pH meter and record in the logbook. Calibrate the pH meter using buffers, which bracket the pH of the samples and extraction fluid.

14.1.4.4 If pH is less than 5, use extraction fluid #1 for the TCLP sample extraction. Record in logbook.

14.1.4.5 If pH is greater than 5, using a disposable 10mL pipette, add 3.5 ml of 1N HCl and swirl. Warm the sample on a hotplate to 50°C and hold at that temperature for 10 minutes.

14.1.4.6 Allow the solution to cool to room temperature, measure the pH, record in the logbook.

14.1.4.7 If the pH is now less than 5, use extraction fluid #1. If the pH is still greater than 5, use extraction fluid #2.

14.1.4.8 Record the extraction fluid used for each sample in the logbook.

14.2. Preparing Extraction Fluid

The sample extraction fluids should not be stored for more than 48 hours. If the extraction fluid is made more than 24 hours before use, the pH must be checked prior to extraction. Care must be taken to ensure adequate mixing of large volumes to make certain that a stable pH has been reached prior to recording the result

14.2.1. Preparation of Extraction Fluid #1 (pH 4.93 + 0.05)

14.2.1.1 Into a 2-L graduated cylinder, add 1000mL of deionized water. Using a disposable pipette add 11.4 ml of glacial acetic acid and 128.6 ml of 1N NaOH into the 2-L cylinder and dilute to the mark with DiH_2O. To make a carboy (24 L): add 136.8ml glacial acetic acid, 1543 1N NaOH and dilute to the mark with DiH_2O. Invert repeatedly to mix well. Using the pH meter, monitor the pH of the solution. Stir with a magnetic stirrer until the pH stabilizes. The pH must be in the range of 4.93 ± 0.05. If it is not,

it must be remade. Record the pH, lot number of the acids and volumes into the logbook.

14.2.2. Preparation of Extraction Fluid #2 (pH 2.88 + 0.05)

14.2.2.1. Into a 2-L graduated cylinder, add 1500 mL of deionized water. Using a disposable pipette add 11.4ml glacial acetic acid and dilute to the mark with DiH2O. Invert repeatedly to mix well. The pH of this fluid must be 2.88 + 0.05. If it is not, it must be remade. Record the pH, lot number of acid and volume in the logbook.

14.2.3. Preparation of the Volatile free Extraction Fluid for ZHE (pH 4.93 + 0.05)

14.2.3.1. Into a 2-L graduated cylinder, add 1000mL of volatile free water found in the GC/MS laboratory. Using a disposable pipette add 11.4 ml of glacial acetic acid and 128.6 ml of 1N NaOH into the 2-L cylinder and dilute to the mark with DiH2O. To make a carboy (24 L): add 136.8ml glacial acetic acid, 1543 1N NaOH and dilute to the mark with volatile free water. Invert repeatedly to mix well. Using the pH meter, monitor the pH of the solution. Stir with a magnetic stirrer until the pH stabilizes. The pH must be in the range of 4.93 + 0.05. If it is not, it must be remade. Record the pH, lot number of the acids and volume into the logbook.

14.3. TCLP Extraction Procedure- Nonvolatile Samples Metals/Extractables
This procedure describes the TCLP extraction of samples for Extractables (semivolatile, pesticide, herbicide) and metal analysis.

14.3.1. Glassware Preparation

14.3.1.1. All extraction vessels, glassware and utensils used for the extraction procedure must be washed with soapy water, rinsed with dilute nitric acid, rinsed with tap water and then followed by a rinse with reagent grade water.

14.3.2. Extraction Vessel Blanks

14.3.2.1. The nonvolatile extraction vessels are 2-L borosilicate glass bottles with screw caps. Each extraction vessel must be demonstrated to be free of contamination by performing a blank extraction in each vessel. The extraction blanks are set up in vessels, which are rotated as samples as. One blank must be extracted per extraction fluid per rotation. A separate blank is required for ZHE extractions. A separate blank is required for metal extractions done in plastic containers.

14.3.2.2. Using a 2000mL graduated cylinder add extraction fluid #1 (or extraction fluid #2) depending on preliminary sample evaluation, to the vessel. Place the container in the tumbler and secure the lid. Rotate the vessel at 30 ± 2 rpm for 18 ± 2 hours.

14.3.3. LIQUID PHASE SAMPLES

If the samples have been found to contain less than 0.5% dry solids, the filtrate is the TCLP extract.

14.3.3.1 Using a GF/F filter and filter apparatus collect the filtrate into the appropriate container. If metals are to be analyzed, acid wash the filter prior to filtration.

14.3.3.2 If the filtrate is to be analyzed for metals, adjust the pH to less than 2 with 1N metals grade nitric acid. Check an aliquot for precipitation before acidifying the entire extract. If a precipitate does form, do not adjust the pH of the extract. Analyze the extract for metals as soon as possible.

14.3.3.3 The Extractable portion of the extraction is stored at 4° C in main sample cooler. The metals portion of the extract is released to the metals digestion lab.

14.3.4 SOLID PHASE SAMPLES

If the sample is found to be 100% solids, and requires no particle size reduction (or has undergone reduction),

14.3.4.1 Weigh out 100g sample and transfer to the extraction vessel. Record the weight and the vessel number in the logbook.

14.3.4.2 The volume of the extraction fluid used is 20 times the sample weight. For example, 100g sample aliquot to 2000mL of extraction fluid.

14.3.4.3 After the sample and appropriate extraction fluid has been added to the vessel record in the logbook. Cap the vessel and clamp the vessels to the rotator.

14.3.4.4 Rotate the vessel at 30 ± 2 rpm for 18 ± 2 hours. Record the analyst, date, time and temperature at the beginning and end of the extraction. The temperature in the room should be maintained at 23± 2° C and checked using a min/max thermometer.

NOTE: For some types of waste, pressure may build up within the extractor bottle during extraction. To relieve excess pressure, periodically vent the extractor bottle in the hood.

14.3.4.5 When the extractions are complete, remove the extractors from the rotator and separate the liquid and solid phases be filtering through a new glass fiber filter (GF/F).

14.3.4.6 Carefully decant the extraction fluid into the filtration apparatus. Collect the fluid in appropriate containers; glass for extractables and plastic for the metals. Discard the solid material left in the vessel.

14.3.4.7 Measure and record the pH of the final extract.

14.3.4.8 If metals are to be analyzed, the filter must be acid washed and the pH of the extract corrected to < 2 with 1N metals reagent grade nitric acid.

14.3.4.9 Store the extractable portion in the main sample cooler. Release the metals portion to the metals digestion analyst. Release samples in the LIMS System.

14.3.5 MIXED PHASE SAMPLES

14.3.5.1 If the samples are mixed phase, decant and filter the liquid part first as described in section 14.1.1, using sufficient sample to perform the required analysis.

14.3.5.2 After the liquid part of the aliquot has been filtered, pour or spread the solid material onto the same filter. Complete the filtration procedure, and record the weights of the filtrate and the solid material.

14.3.5.3 Hold the liquid portion for future analyses, or for combination with the TCLP extract.

14.3.5.4 Evaluate the solid portion of the sample and transfer to the extraction vessel following the instructions in section 14.3.4.

14.3.5.4.1 When transferring the solid material in the filtration apparatus to the extraction vessel, include the filter. Record all weights in the logbook.

14.3.5.4.2 Use the following calculation to determine the amount of extraction fluid to use:

$$\text{Wt of Fluid} = \frac{20 \times \text{Percent Solids} \times \text{Wt of Waste Filtered}}{100}$$

14.3.5.5 Conduct the extraction of the solid material as described in sections 14.3.4.4 to 14.3.4.8.

14.3.5.6 If the filtered liquid phase of the mixed phase sample is compatible with the liquid extract from the solid phase extraction, combine the phases. This combination is the final mixed phase TCLP extract.

14.3.5.7 If the filtered liquid phase of the mixed phase is not compatible with the liquid extract from the section 14.3.5.2 and 14.3.5.3, do not combine the phases.

14.3.5.7.1 The liquids are analyzed separately, and the results are mathematically combined. The following equation is used to obtain the final analyte concentrations:

$$\text{Final Analyte Concentration} = \frac{(V1)(C1) + (V2)(C2)}{(V1 + V2)}$$

V1 = The volume of the first phase (L)
C1 = Analyte concentration of the first phase (mg/L)
V2 = The volume of the second phase (L)
C2 = Analyte concentration of the second phase (mg/L)

14.3.5.8 Analyze the TCLP extracts according to the appropriate analytical methods.

NOTE: Some wastes, such as oily wastes and some paints, will obviously contain some material that appears to be a liquid. Even after applying vacuum or pressure filtration, as outlined in section 14.3.5, this material may not filter. If this is the case, the material within the filtration device is defined as a solid and is carried through the extraction as a solid. Do not replace the original filter with a fresh filter under any circumstances. Use only one filter.

TITLE: TOXICITY CHARACTERISTIC LEACHING PROCEDURE (TCLP)

SUPERCEDES: Revision 7 – AWC-1311-21

14.4 TCLP Extraction Procedure- Volatile Samples

This method is used for the TCLP extraction of samples for volatile analysis. Care must be taken to minimize the loss of volatiles by limiting the exposure of the samples, the filtrate, and the extracts to the atmosphere. Headspace should not be allowed in any of the extraction or collection containers.

NOTE: If the sample consists of pure oil, paint or solvent, consult the supervisor before proceeding. If the sample matrix is deemed unacceptable for the ZHE extractor, the supervisor is required to contact the Project Manager to notify the client. The project manager and client would then evaluate if a total Volatile analysis of the sample will meet the needs of the project. The analytical test would be updated to reflect a total analysis. The results of this 'Total Analysis' should be considered approximately 20 times higher than a TCLP leaching procedure.

14.4.1 If the samples have been found to contain less than 0.5 percent dry solids, the filtrate is the TCLP extract (section 14.1.1.12). Sample in addition to that used in the initial evaluation may need to be filtered to provide sufficient volume for all requested analyses. If additional sample is needed, proceed as follows.

14.4.1.1 Set up the filtration apparatus as is section 14.1.1 and follow filtration procedure.

14.4.1.2 Collect the filtrate directly into a 40 mL VOA vial or ZHE Tedlar bag, allowing for no headspace to form.

14.4.1.3 Store the vials in the GC/MS sample cooler. Turn in a copy of the batch to the GC/MS supervisor. Release samples in the LIMS System.

14.4.2 Zero Headspace Extractors (ZHE) are used as the extraction vessels for solid material from which volatile analytes are to be analyzed. The ZHE has an internal capacity of 500 mL. Therefore, because of the 20-fold ratio of extraction fluid to sample, the maximum volatile sample size is 25g.

14.4.3 Cleaning and Maintaining the ZHEs.

14.4.3.1 Before the ZHEs are assembled, wash all the parts with hot, soapy water followed by rinsing with tap and deionized water. A sonic bath can be used to clean the more difficult parts. After cleaning, the cylinder, piston, stainless steel screens, and any rubber O-rings can be placed in the 103°C oven for a few hours. Only these parts can be heat treated as the heat damages any part containing a valve. Make sure the parts are room temperature before setting up samples. Make sure the pistons do not get

interchanged. ZHE O-rings must be free of cuts or cracks or the extractors may leak. Before assembly, examine the O-rings for damage.

14.4.4 Assembly of the ZHEs

14.4.4.1 Assuming the vessel is unassembled, first place the Model #3745-ZHE body (3) on the chair (6) with either end up and install the air side flange O-ring (12) in the gland on top of the body.

14.4.4.2 Replace the piston O-rings (12) on the piston (5) by stretching them over the piston and into the gland. NOTE: Be careful not to "roll" the O-rings into the gland. Wet the piston with extraction fluid.

14.4.4.3 Align the piston (5) carefully with the top of the body (3) and gently press it into the body. Continue pressing the piston into the body until it is completely inside the cylinder. NOTE: Care is necessary not to damage the O-rings. It may be helpful to moisten the O-rings with extraction fluid.

14.4.4.4 Locate the air side flange (1) and place it on top of the ZHE. Visually align the holes in the flange and the ZHE body and secure the flange with three knobs (8). Uniformly tighten the knobs.

14.4.4.5 Invert the partially assembly ZHE on the chair such that the air side flange (1) is now down.

14.4.4.6 Place a Filter-Pak O-ring (12) in the gland on top of the cylinder, making sure that it is fully seated.

14.4.4.7 Prepare a new Filter-Pak by placing a glass filter element between the two Stainless steel screens. Set the assembled filter-Pak into its recess on the top of the ZHE body. NOTE: Center the Filter-Pak carefully.

14.4.4.8 Locate the waste side flange (2) and install a Filter-Pak O-ring (12) in the inner gland of the flange. Install the body O-ring (12) in the outer gland of the waste side flange. NOTE: Be careful not to "roll" the O-rings into the glands.

14.4.4.9 Invert the waste side flange (2) and place it on top of the ZHE, aligning the holes at the same time.

14.4.4.10 Place the assembled ZHE on the chair (6) with liquid inlet/outlet valve (14) on the top.

14.4.4.11 Verify that both the liquid inlet/outlet valve (13) and the quick-exhaust/relief valve (16) are open. NOTE: The quick exhaust/relief valve is open when the handle is up and the liquid inlet/outlet valve is open when the arrow points up.

14.4.4.12 Unscrew the three knobs (8) in the waste side flange (2), remove the flange and carefully take out the Filter-Pak (7, 19). NOTE: The Filter-Pak consists of two screens with a 0.7-micron glass fiber filter between them. Set these aside.

14.4.5 Transfer of Sample to the ZHE

14.4.5.1 If the sample matrix is 100% solid material, and particle size reduction is unnecessary, weigh 25g of sample to the nearest 0.1g. Proceed to section 14.4.5.4.

14.4.5.2 If particle size reduction is needed, proceed as in section 14.1.2 before continuing. Record the sample weight in the logbook.

14.4.5.3 If the sample matrix is a mixed phase, use the percent solids information from section 14.1.1 and the following calculation to determine the correct sample size to use:

$$\text{Wt of Waste to Charge (grams) ZHE} = \frac{25 \text{ grams}}{\text{Percent Solids}} \times 100$$

Pour the appropriate weight of the mixed waste slurry into a tared beaker and transfer to the ZHE, quickly attaching the top flange. Reweigh the beaker and record in the weight ion the logbook.

14.4.5.4 Introduce the sample to be extracted into the open top of the ZHE making sure that the piston (5) is far enough into the body (3) to provide sufficient free volume. Weigh out a representative sample of a maximum of 25.00 $\pm$ 0.1 g. Record the weight in the logbook and the number of the vessel being used.

14.4.5.5 Use the following formula to determine how much extraction fluid to add to the ZHE:

$$\text{Wt of the extraction fluid} = \frac{20 \times \text{percent solids} \times \text{Wt of waste filtered}}{100}$$

For example, if the sample has been classified as 100% solid, 500 mL of extraction fluid will have to be injected.

14.4.5.6 Check that the body O-ring (12) is properly seated in its gland and reinstall the Filter-Pak in the top of the ZHE. NOTE: Center the Filter-Pak carefully in its recess.

14.4.5.7 Verify that the Filter-Pak O-ring (12) and the waste side flange O-ring (12) are properly seated in their respective glands. Place the waste side flange (2) back on the unit and uniformly tighten the three knobs to close the vessel.

14.4.5.8 Close the quick-exhaust/ relief valve (16) on the air side flange (1). Do not close the liquid inlet/outlet valve. NOTE: The quick-exhaust/relief valve is closed when the handle is horizontal.

14.4.5.9 Introduce the proper amount of extraction fluid into the vessel using a peristaltic pump. Now, the object is to put the 500 ml of extraction fluid into the vessel without opening it as to not expose the sample inside to the atmosphere. A metering pump is now set up next to the vessel. A graduated cylinder (that can hold at least 500 ml) with a stopcock at the bottom is set up on a ring stand next to the vessel. Tubing (tygon) is run from the bottom of the stopcock, around the pump wheel, to the Luer fitting on top of the vessel.

14.4.5.10 First, with the tubing off of the top of the vessel, open the 2-way stainless steel valve and the vent relief valve on the bottom of the vessel. Now, start the metering pump and open the stopcock on the cylinder (containing 500 ml of extraction fluid). When the fluid is almost to the end of the tubing, connect the tubing to the Luer fitting on top of the vessel securely. The fluid should start flowing into the vessel, pushing the piston back down to the bottom of the cylinder. The process may be slow. When all the fluid has been pushed into the vessel, shut the pump off, close the 2-way stainless steel valve, and close the vent relief valve on the bottom of the vessel. Disconnect the tubing and set the pump apparatus aside.

14.4.5.11 Close the liquid inlet/outlet valve (13) and the quick-exhaust/ relief valve (16). Close the valve on the syringe and disconnect it from the ZHE. Physically rotate the vessel end over end 2 or 3 times and place it back on the chair (6) with the liquid inlet/outlet valve on top.

14.4.5.12 Connect the pressure source to the air side flange and set the line pressure at 5-10 psi. Slowly open the liquid inlet/outlet valve (13) to bleed out any headspace that may have been introduced during the addition of extraction fluid and close the liquid inlet/outlet valve at the first sign of

liquid. Allow the pressure gage on the ZHE to stabilize and remove the pressure source. NOTE: 10 psi is recommended.

14.4.5.13 Place the ZHE in the rotary agitator (tumbler) and tumble for 18 $\pm$ 2 hours at 30 $\pm$ 2 RPM, periodically checking the pressure on each unit. The temperature in the room should be maintained at 23 $\pm$ 2° C. Record the analyst, date, time and temperature at the beginning and end of the extraction.

14.4.5.14 When rotation is complete, check that the pressure gauges still read 10 psi. If the vessel is no longer pressurized, repeat the extraction with a new sample.

14.4.5.15 Remove the ZHEs from the rotation apparatus and let stand for two hours to settle.

14.4.6 Removal of the Extract from the ZHE

14.4.6.1 Once the contents of the ZHE have settled, attach a pressure regulated source (set to 0 psi) of filtered compressed air or dry nitrogen to the gas inlet quick disconnect (14) found on the air side flange (1)). Note: Compressed air must be prefiltered (5micron rating) to prevent particulate matter from scoring the walls of the ZHE.

14.4.6.2 Increase the supply pressure to 5-10 psi and note the pressure on the vessel gage (17) is approximately 2-5 psi. This indicates that the piston (5) is in motion and removing the sample headspace. At the first sign of liquid release from the liquid inlet/outlet valve (13) immediately close the valve (13). Disconnect the pressure source and open the quick-exhaust/relief valve (16) to depressurize the ZHE. NOTE: This will take a short period of time depending on the volume of free air remaining on the sample side of the vessel. NOTE: Pressure greater than 10 psi may be necessary.

14.4.6.3 Attach an appropriate filtrate collection container to the liquid inlet/outlet valve and firmly hand tighten the Speed-Nut (10). Close the quick-exhaust/relief valve (16). NOTE: If you are using Tedlar® bags (or glass/PTFE syringes) for sample collection, attach the supplied Stainless Steel Luer-Lok® adapter (9) (or the Teflon® Tedlar® adapter (18) to the liquid inlet/outlet valve (13) and then connect to the Tedlar® bag or glass/PTFE syringe).

14.4.6.4 Reattach the pressure source to the vessel and set the line pressure at 5-10 psi. Slowly open the liquid inlet/outlet valve (13) on the ZHE. Gradually

increase the supply pressure in 10 psi increments (up to the maximum of 50 psi) until no more initial liquid phase is expelled in a two minute interval or until the 40mL vial is full and free of any visible air bubbles. NOTE: The pressure gage (17) on the vessel should be allowed to stabilize at the line pressure setting before beginning the two minute period.

14.4.6.5 Close the liquid inlet/outlet valve(s), disconnect the pressure source and remove the collection device. Depressurize the ZHE by slowly opening the quick-exhaust/relief valve (16). NOTE: Leave the quick-exhaust/relief valve open. Repeat steps from section 14.4.5.

14.4.7 Leak-testing the ZHE

14.4.7.1 Pressurize the ZHE to 50 psi and place in a large container of water. If bubbles escape from the vessel, the seals are leaking.

14.4.7.2 De-pressurize the ZHE and open the side that is leaking and re-wet the O-rings. Recheck for leaks in the seal. The leak check must be performed before every extraction and documented in the logbook.

The ZHE system is comprised of a 316 S.S. heavy-wall 500 mL barrel, mounted on a rigid support base to which is fitted a pressure relief valve, and a quick-release air valve. The base is fitted with 3 rigid support legs. The barrel is sealed to the bottom support with a Viton O-ring (fitted to the bottom of barrel) and to the heavy gauge top plate with another Viton O-ring fitted to a liquid sample outlet valve to which is connected a female luer port. The plate is secured to the barrel with 3 handwheels connected to the 3 extension screens (which enclose the filter) are provided, and these are placed on top O-rings, and a Viton-A "wiper" ring (top edge). The latter is used to push away any particles that might interfere with the lower sealing O-rings. The piston is activated by the air (or gas) entering from the bottom plate, and pushed upwards to force the sample liquid through and out of the filter at the top of the barrel. This design and function obviates the introduction of air into the sample, thus eliminating loss of volatiles.

As a safety precaution, the Millipore ZHE System cannot be pressurized unless completely assembled, thus eliminating the possibility of propelling the piston out of the unit. The vent relief valves will automatically open at 125 psi to prevent excessive pressure build up. They also permit manual venting of the systems. The ZHE unit may be used in a standard rotary extractor (legs intact), or in a box-type extractor (legs removed).

TITLE: TOXICITY CHARACTERISTIC LEACHING PROCEDURE (TCLP)

SUPERCEDES: Revision 7 – AWC-1311-21

20.0 WASTE MANAGEMENT/ POLLUTION PREVENTION

20.1 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."

20.2 Waste Streams Produced by the Method - The following waste streams are produced when this method is carried out.

20.2.1 Acidic waste from sample extract. All acid waste is disposed of in "A" waste satellite containers (except nitric acid waste). When full, the satellite container is transferred to the secure waste storage area and disposed of by appropriately trained laboratory technicians in accordance to all state and federal regulations.

20.2.2 Solid waste from sample extract. The solid waste from the sample extract is dried and disposed of in a "BE" satellite container. When full, the satellite container is transferred to the secure waste storage area and disposed of by appropriately trained laboratory technicians in accordance to all state and federal regulations.

20.2.3 Remaining TCLP extracts. The remaining TCLP extracts are considered "A" waste and are disposed of directly to a 55-gallon "A" waste drum by appropriately trained laboratory technicians in accordance to all state and federal regulations.

21.0 REFERENCES

21.1 Method 1311, "Test Methods for Evaluating Solid Waste", EPA SW846 Third Edition, 12/96

22.0 TABLES, DIAGRAMS, FLOWCHARTS AND VALIDATION DATA

22.1 Figure 1. Tumbler- Rotary Agitation Apparatus

22.2 Figure 2. Zero Headspace Extractor (ZHE)

22.3 Figure 3. ZHE Specifications

22.4 Figure 4. ZHE and Fluid Metering Pump

22.5 Figure 5. Flow chart

22.6 Figure 6. Regulated analytes for Toxicity Characteristic with Regulatory Levels.

TITLE: TOXICITY CHARACTERISTIC LEACHING PROCEDURE (TCLP)

SUPERCEDES: Revision 7 – AWC-1311-21

22.7 Figure 7. Logbook – TCLP Metals/Extractables – Page 1 and Page 2

22.8 Figure 8. Logbook – TCLP VOA – Page 1 and Page 2

23.0 CHANGES FROM PREVIOUS REVISION

23.1 Changed responsible lab area from WC (Wet Chemistry) to SP (Sample Preparation)

23.2 Lab director and Department Manager were updated.

23.3 Section 8.0 updated: Includes Corporate EH&S Safety information.

23.4 Sections 13.1 and 13.2 added.

23.5 Section 20.0 updated: Includes Corporate EH&S Waste Management information.

23.6 Section 22.0: Added Figures 7 & 8

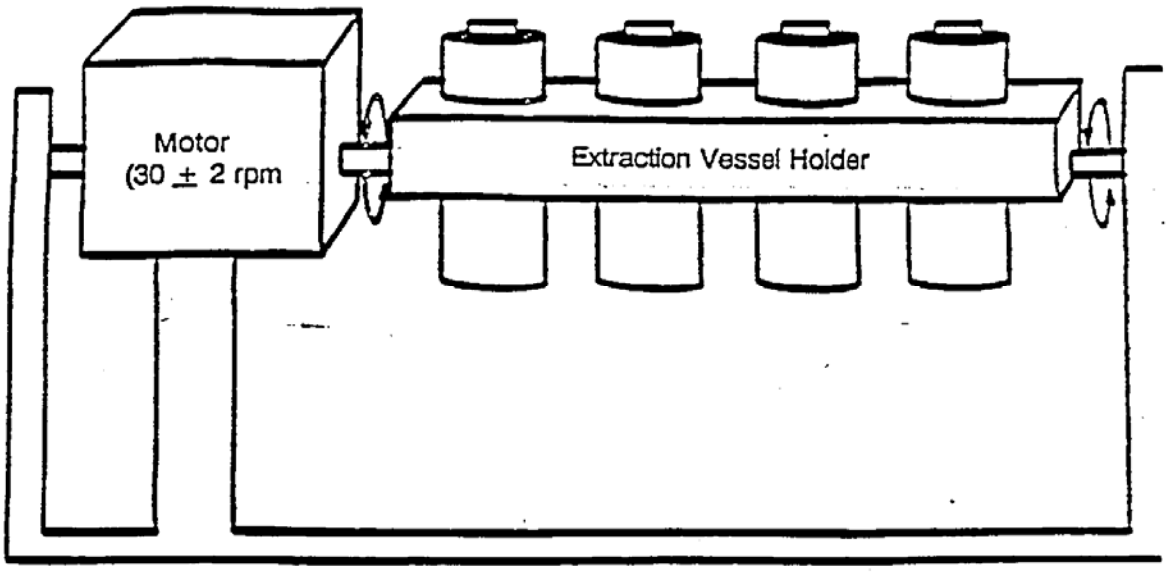


Figure 1. Tumbler- Rotary Agitation Apparatus

SOP No. ASP-1311-21	Revision No. 8	Effective Date October 26, 2005	Page 24 of 33

TITLE: TOXICITY CHARACTERISTIC LEACHING PROCEDURE (TCLP)

SUPERCEDES: Revision 7 – AWC-1311-21

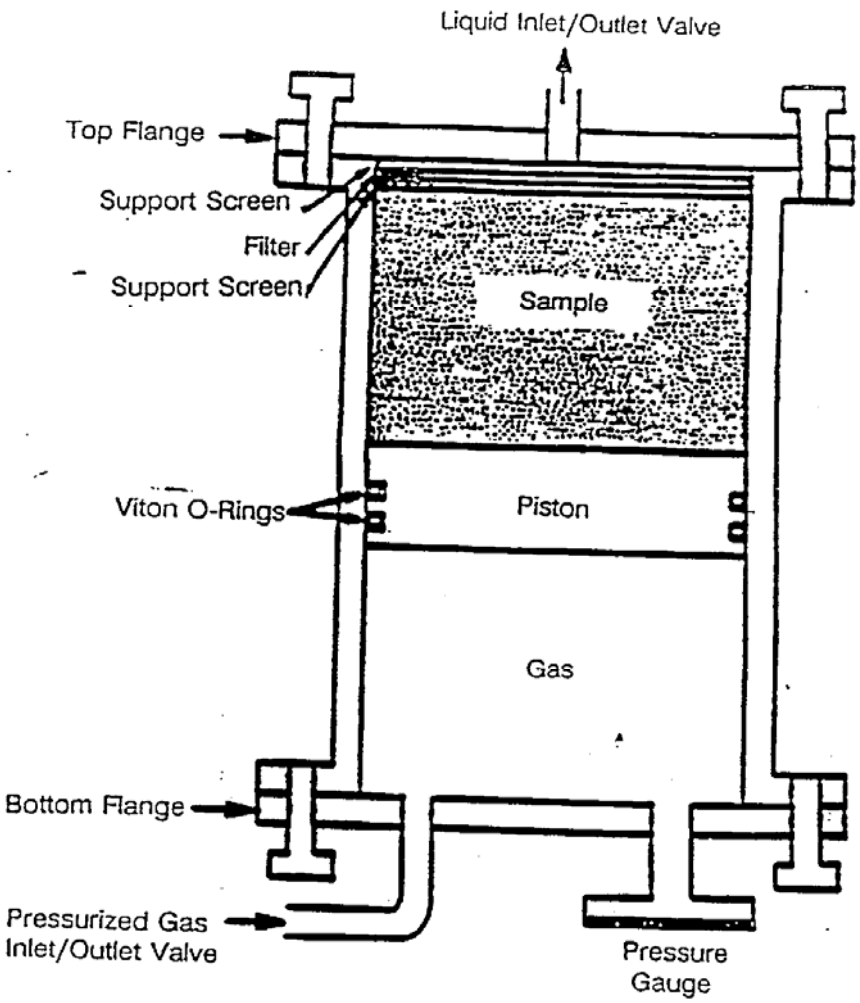
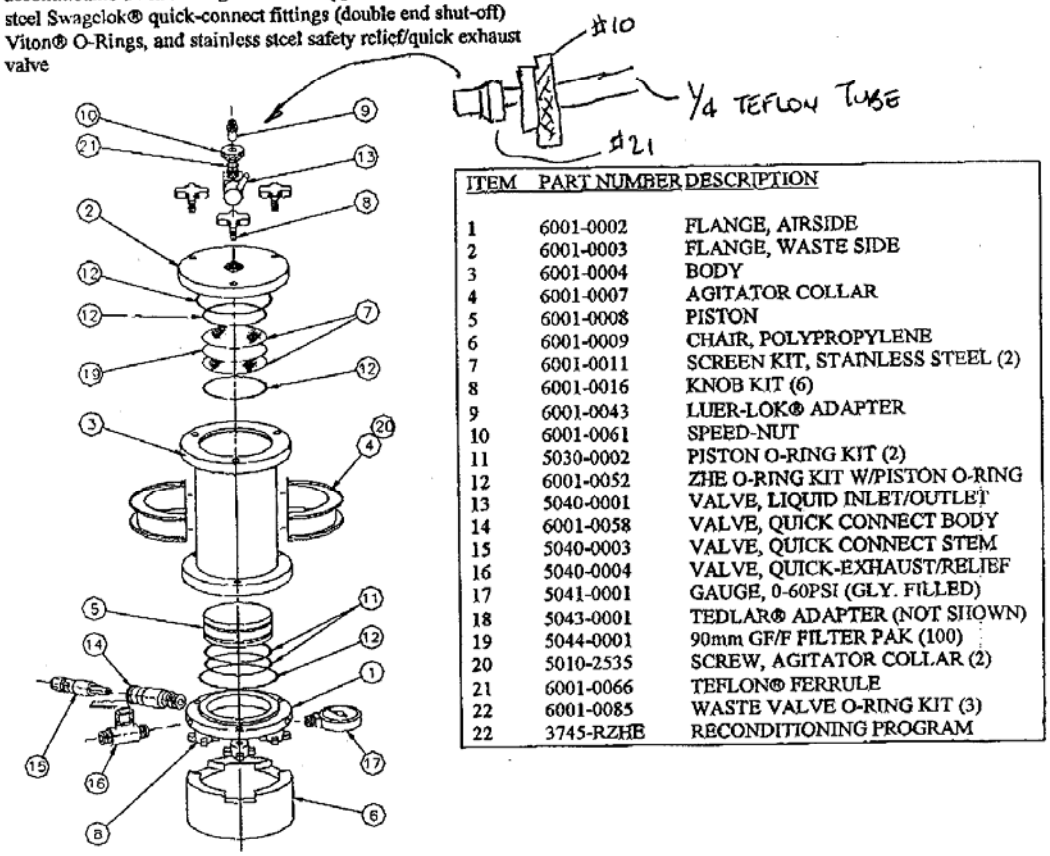


Figure 2. Zero Headspace Extractor (ZHE)

MODEL 3745-ZHE SPECIFICATIONS

MATERIAL: 316 Stainless Steel, all welds inspected per MIL-I-6866. Standard Viton® O-Rings, supplied with an agitator collar, and a polypropylene chair for set-up.
DIMENSIONS: 127.00mm (5.00") dia., 241.30mm (9.50") plus sample fitting. Will easily fit Associated Design's ROTARY AGITATOR MODEL: 3740-4-BRE, 3740-6-BRE, 3740-8-BRE, and 374-12-BRE.
CONNECTIONS: Sample side supplied with 316 stainless Steel Luer-Lok® adapter and a Teflon® bushing adapter to accommodate Tedlar® bags. Airside supplied with stainless steel Swagelok® quick-connect fittings (double end shut-off) Viton® O-Rings, and stainless steel safety relief/quick exhaust valve

PRESSURE: Working pressure 50 psi
FILTER/PREFILTER SIZE: 90mm .7 micron GF/F Filter
EFFECTIVE FILTRATION AREA: 45.8cm²
PISTON BREAKWAY FORCE: 2-5 psi
VOLUME: 550ml (nominal)
GAUGE: 2-1/2" dia., 0-60 psi glycerin filled



ITEM	PART NUMBER	DESCRIPTION
1	6001-0002	FLANGE, AIRSIDE
2	6001-0003	FLANGE, WASTE SIDE
3	6001-0004	BODY
4	6001-0007	AGITATOR COLLAR
5	6001-0008	PISTON
6	6001-0009	CHAIR, POLYPROPYLENE
7	6001-0011	SCREEN KIT, STAINLESS STEEL (2)
8	6001-0016	KNOB KIT (6)
9	6001-0043	LUER-LOK® ADAPTER
10	6001-0061	SPEED-NUT
11	5030-0002	PISTON O-RING KIT (2)
12	6001-0052	ZHE O-RING KIT W/PISTON O-RING
13	5040-0001	VALVE, LIQUID INLET/OUTLET
14	6001-0058	VALVE, QUICK CONNECT BODY
15	5040-0003	VALVE, QUICK CONNECT STEM
16	5040-0004	VALVE, QUICK-EXHAUST/RELIEF
17	5041-0001	GAUGE, 0-60PSI (GLY. FILLED)
18	5043-0001	TEDLAR® ADAPTER (NOT SHOWN)
19	5044-0001	90mm GF/F FILTER PAK (100)
20	5010-2535	SCREW, AGITATOR COLLAR (2)
21	6001-0066	TEFLON® FERRULE
22	6001-0085	WASTE VALVE O-RING KIT (3)
22	3745-RZHE	RECONDITIONING PROGRAM

Figure 3. ZHE Specifications

SOP No. ASP-1311-21	Revision No. 8	Effective Date October 26, 2005	Page 26 of 33

TITLE: TOXICITY CHARACTERISTIC LEACHING PROCEDURE (TCLP)

SUPERCEDES: Revision 7 – AWC-1311-21

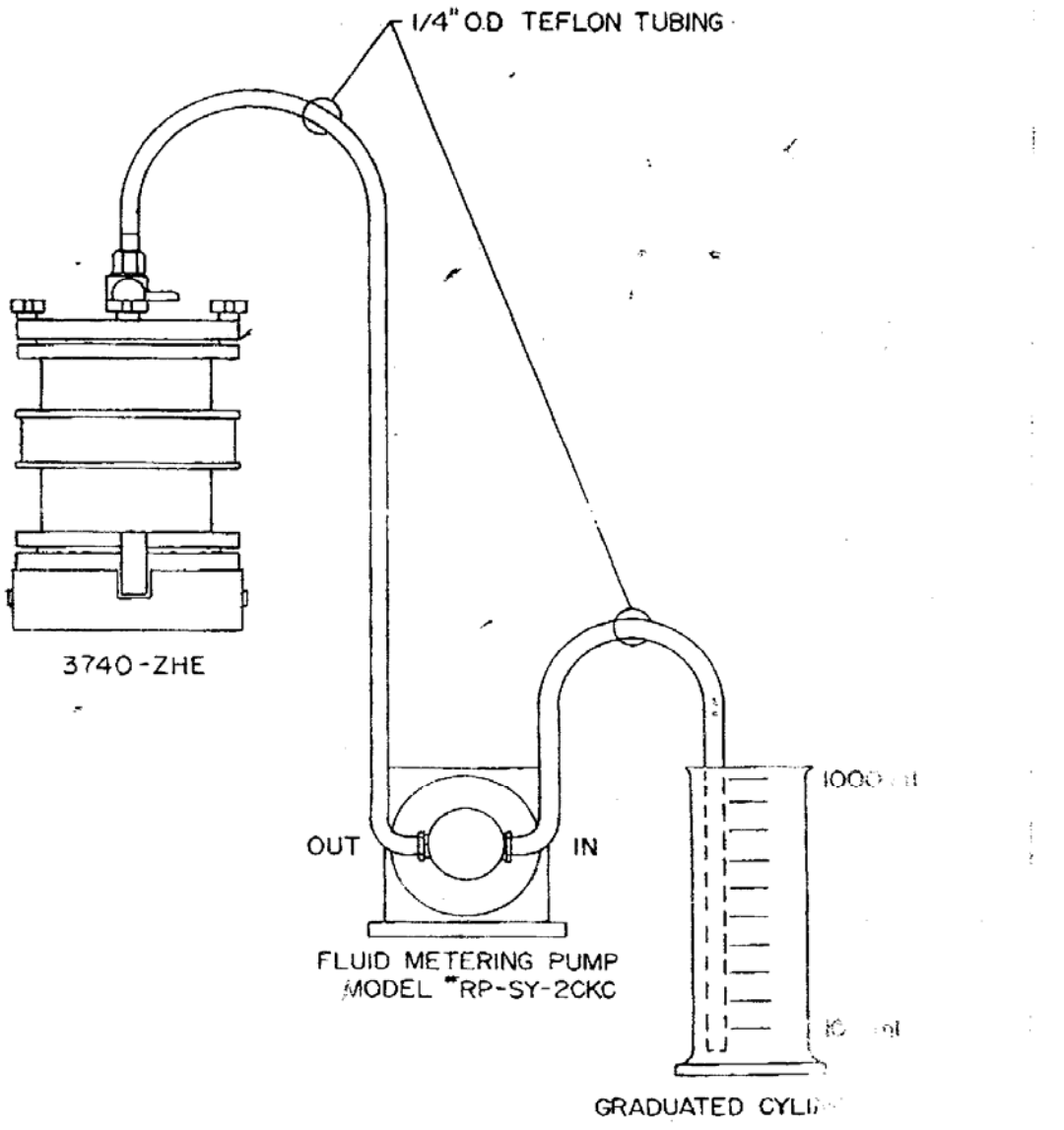


Figure 4.

SOP No. ASP-1311-21	Revision No. 8	Effective Date October 26, 2005	Page 27 of 33

TITLE: TOXICITY CHARACTERISTIC LEACHING PROCEDURE (TCLP)

SUPERCEDES: Revision 7 – AWC-1311-21

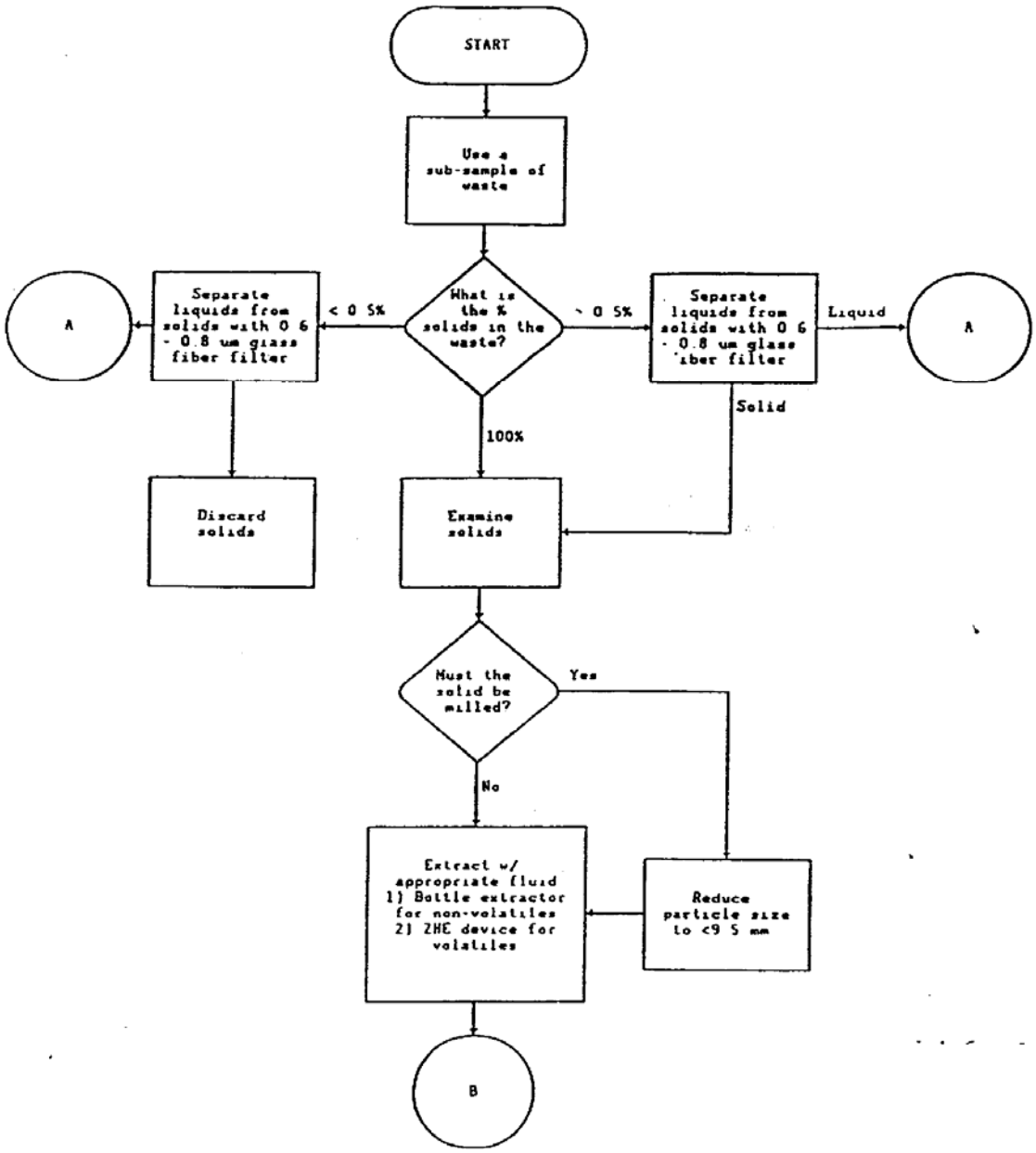


Figure 5 (page 1 of 2)

SOP No. ASP-1311-21	Revision No. 8	Effective Date October 26, 2005	Page 28 of 33

TITLE: TOXICITY CHARACTERISTIC LEACHING PROCEDURE (TCLP)

SUPERCEDES: Revision 7 – AWC-1311-21

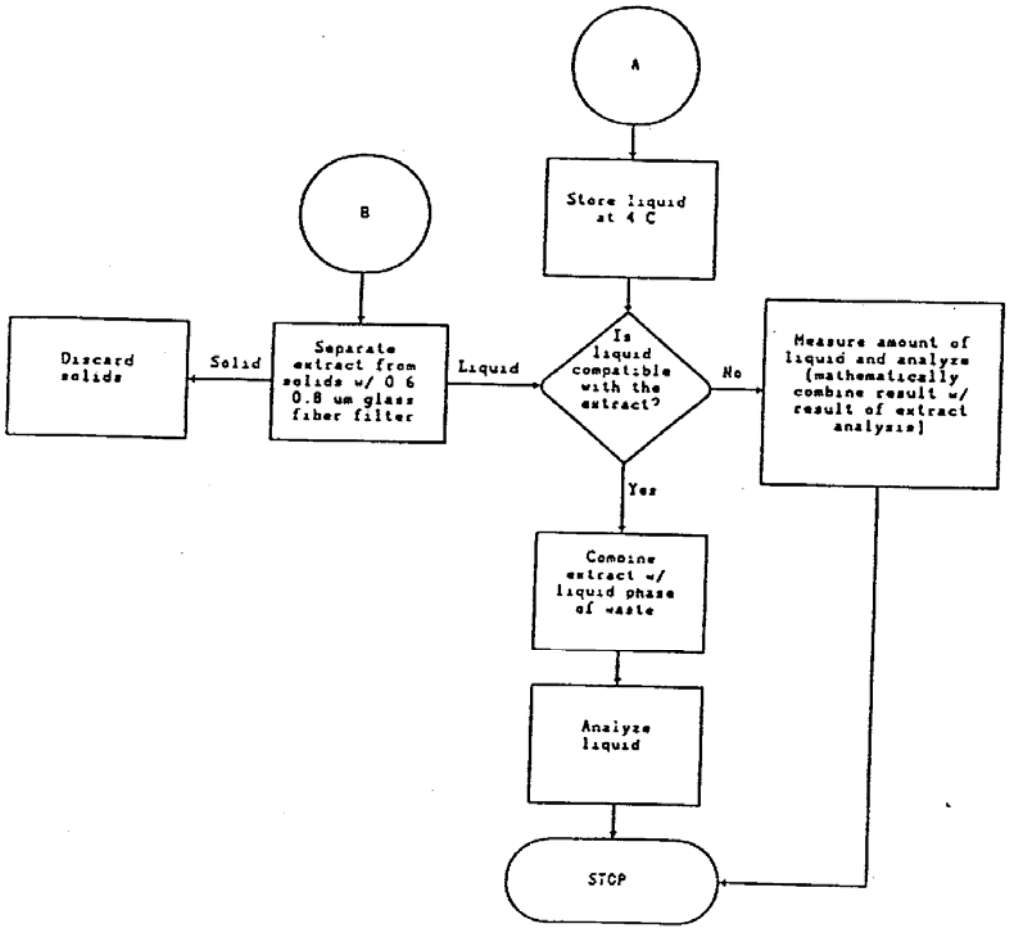


Figure 5 (page 2 of 2)

TITLE: TOXICITY CHARACTERISTIC LEACHING PROCEDURE (TCLP)

SUPERCEDES: Revision 7 – AWC-1311-21

Compound	Regulatory Level (mg/L)
Benzene	0.5
Carbon tetrachloride	0.5
Chlorobenzene	100.0
Chloroform	6.0
1,2-Dichloroethane	0.5
1,1-Dichloroethene	0.7
2-Butanone	200.0
Tetrachloroethene	0.7
Trichloroethene	0.5
Vinyl chloride	0.2

9.2 Semivolatiles

Compound	Regulatory Level (mg/L)
2-Methylphenol	200.0
3-Methylphenol	200.0
4-Methylphenol	200.0
1,4-Dichlorobenzene	7.5
2,4-Dinitrotoluene	0.13
Hexachlorobenzene	0.13
Hexachlorobutadiene	0.5
Hexachloroethane	3.0
Nitrobenzene	2.0
Pentachlorophenol	100.0
Pyridine	5.0
2,4,5-Trichlorophenol	400.0
2,4,6-Trichlorophenol	2.0

9.3 Pesticides

Compound	Regulatory Level (mg/L)
Chlordane	0.03
2,4-D	10.0
Endrin	0.02
Heptachlor	0.008
Heptachlor epoxide	0.008
Lindane (gamma BHC)	0.4
Methoxychlor	10.0
Toxaphene	0.5
2,4,5-TP (Silvex)	1.0

9.4 Metals

Element	Regulatory Level (mg/L)
Arsenic	5.0
Barium	100.0
Cadmium	1.0
Chromium	5.0
Lead	5.0
Mercury	0.2
Selenium	1.0
Silver	5.0

Figure 6. Regulated Analytes for Toxicity Characteristic with Regulatory Levels

TITLE: TOXICITY CHARACTERISTIC LEACHING PROCEDURE (TCLP)

SUPERCEDES: Revision 7 – AWC-1311-21

TCLP Metals Log / Revision 1 / June 2005

Logbook# A05-08-11

STL BUFFALO

TCLP Preparation Logbook for Method 1311 Page 1 of 2

ANALYST: DATE: BATCH#

LABORATORY SAMPLE ID						
EXTRACTION TYPE						
EXTRACTION VESSEL NUMBER						
1. Percent Solids Determination - (check a or b)						
a) Sample will obviously yield no liquid when subjected to pressure filtration (is 100% solids). Verified with Spatula Test. Proceed to Step 3.						
b) Sample is liquid or multiphastic, use pressure filtration to determine percent solids. Proceed to Step 2.						
2. Pressure Filtration-use an acid washed (25% HNO), 0.6 to 0.8 um glass fiber filter						
Weight (g) of Filtrate Container (FC)						
Weight (g) of Subsample (SW) (100 gram minimum)						
Gradually apply vacuum or gentle pressure in increments of 10, 20, 30 ,40 and 50 psi. When filtration does not result in additional filtrate within any 2 minute period, the filtration is done.						
Weight (g) of Filtrate Filled Container (FF)						
Determine Weight (g) of Liquid Phase (LP) : (FF) - (FC)						
Determine Weight (g) of Solid Phase (SP) : (SW) - (LP)						
% Solids = (SP) / (SW) x 100						
If % solids are ≥ 0.5%, the liquid phase is stored at 4°C and Proceed to Step 3 for the solid phase. Liquid phase (LP) will be used in Step 8. If % solids are < 0.5%, the liquid phase is considered the TCLP extract. Enough of the sample should be filtered. Proceed to Step 8.						
3. Particle Size Reduction (yes or no)						
a) Solid phase passes through a 9.5 mm sieve. **YES OR NO.** If no prepare solid portion by crushing, cutting, or grinding to a size that would. Proceed to Step 4.						
4. Extraction Fluid Determination						
Weight of test sample (use 5.0 ± 1.0 grams of solid phase)						
Actual volume of water (add 96.5 ± 1.0 mL)						
After 5 minutes of stirring, record Initial pH If Initial pH is ≤5, use Extraction Fluid I						
If Initial pH is >5, add 3.5 mL of 1N HC1, heat to and hold at 50°C for ten minutes. Record pH after ten minutes.						
Let cool to room temperature and record Final pH If Final pH ≤5 use Fluid I If Final pH > 5 use Fluid II						
Extraction Fluid Used:						

5. Extraction Fluid Preparation:

Glacial Acetic Acid		1.0 N NaOH						
Vol (mls)	Lot#	Vol. (mls)	Lot#	Final Vol.(mls)	Extraction Fluid #	Analyst:	Date:	pH

Comments:_____

Figure 7. Logbook – TCLP Metals/Extractables (page 1 of 2)

TITLE: TOXICITY CHARACTERISTIC LEACHING PROCEDURE (TCLP)

SUPERCEDES: Revision 7 – AWC-1311-21

STL BUFFALO Logbook# A05-08-11

TCLP Preparation Logbook for Method 1311 Page 2 of 2

ANALYST: DATE: BATCH#

LABORATORY SAMPLE ID						
EXTRACTION VESSEL NUMBER						
6. Sample Preparation for Extraction Procedure						
a) If sample is 100% solid, add appropriate extraction fluid.						
Record weight of sample (100 grams minimum)						
Record volume (mL) of extraction fluid added (amount of fluid is 20 X the weight of sample)						
b) If sample is ≥0.5% solid, determine weight of extraction fluid to add using the following equation:						
Record weight (g) of sample (SP) determined in Step 2						
Record volume (mL) of extraction fluid to add Extraction Fluid added = 20 x (SP) weight						
7. TCLP Rotation (rotate for 18 ± 2 hours)						
Record Initial pH of TCLP extract						
Record Start time						
Record Tumbler rpm **(range of 30 ± 2 rpm) YES or NO**						
Record Stop time						
Record min/max temperature **(range of 23 ± 2°C)**						
Record Filtration Complete **Date** and **Time** Use 0.6 to 0.8 um acid washed glass fiber filter						
Record Final pH of TCLP extract						
8. Final TCLP Extract If sample is 100% solids, only c will be used. If sample <0.5 % solids, only a will be used. If sample is multiphasic, both b and c will be used.						
a) Volume (mL) of Liquid Extract from Pressured Filtered Sample (from Step 2) with <0.5% solids						
Record pH						
b) Volume (mL) of Liquid Extract from Pressured Filtered Sample (from Step 2)with ≥ 0.5% solids (LP)						
Record pH						
c) Volume (mL) of Tumbled Extract Obtained in Step 6 from Solid portion of Pressured Filtered sample						
If volumes from b (not tumbled filtered liquid extract) and c (post tumbled filtered liquid extract) are compatible, add together. If volumes are not miscible, treat as separate extracts.						
Record pH of Combined Volume						
Record Combined Volume mL						

Calibration of pH meter for TCLP:
Each electrode must be calibrated at a minimum of two points that bracket the expected pH of the samples or fluids.

Date	Analyst	1.68 WCR-	4.00 WCR-	7.00 WCR-	10:00 WCR-	12.54 WCR-

Date	Analyst	1.68 WCR-	4.00 WCR-	7.00 WCR-	10:00 WCR-	12.54 WCR-

Reviewed By/Date_____

Figure 7. Logbook – TCLP Metals/Extractables (continued: page 2 of 2)

SOP No. ASP-1311-21	Revision No. 8	Effective Date October 26, 2005	Page 32 of 33

TITLE: TOXICITY CHARACTERISTIC LEACHING PROCEDURE (TCLP)

SUPERCEDES: Revision 7 – AWC-1311-21

STL BUFFALO **Logbook#A05-08-12**

TCLP Preparation Logbook for Method 1311 Page 1 of 2

ANALYST:_____ **DATE:**_____ **BATCH#**_____

LABORATORY SAMPLE ID								
EXTRACTION TYPE								
EXTRACTION VESSEL NUMBER								
1. Percent Solids Determination (check a or b)								
a) Sample will obviously yield no liquid when subjected to pressure filtration (is 100% solids). Verified with Spatula Test. Proceed to Step 3.								
b) Sample is liquid or multiphastic, use pressure filtration to determine percent solids. Proceed to Step 2.								
2. Pressure Filtration-use a 0.6 to 0.8 um glass fiber filter								
Weight (g) of Filtrate Container (FC)								
Weight (g) of Subsample (SW) (100 gram minimum)								
Gradually apply vacuum or gentle pressure in increments of 10, 20, 30 ,40 and 50 psi. When filtration does not result in additional filtrate within any 2 minute period, the filtration is done.								
Weight (g) of Filtrate Filled Container (FF)								
Determine Weight (g) of Liquid Phase (LP) : (FF) - (FC)								
Determine Weight (g) of Solid Phase (SP) : (SW) - (LP)								
% Solids = (SP) / (SW) x 100								
If % solids are ≥ 0.5 proceed to Step 3. If % solids are < 0.5%, Proceed to Step 8.								
3. Particle Size Reduction (yes or no)								
Solid phase passes through a 9.5 mm sieve. **YES OR NO.** If no prepare solid portion by crushing, cutting, or grinding to a size that would. Proceed to Step 5.								
4. Extraction Fluid Determination N/A								
Extraction Fluid Used:				**Voa free**	**Voa free**	**Voa free**	**Voa free**	**Voa free**	**Voa free**
5. Extraction Fluid Preparation								
Glacial Acetic Acid		1.0 N NaOH						
Vol (mls)	Lot#	Vol. (mls)	Lot#	Final Vol.(mls)	Extraction Fluid #	Analyst:	Date:	pH

Calibration of pH meter for TCLP:
Each electrode must be calibrated at a minimum of two points that bracket the expected pH of the samples or fluids.

Date	Analyst	1.68 WCR-	4.00 WCR-	7.00 WCR-	10:00 WCR-	12.54 WCR-

Date	Analyst	1.68 WCR-	4.00 WCR-	7.00 WCR-	10:00 WCR-	12.54 WCR-

Figure 8. Logbook – TCLP VOA (page 1 of 2)

SOP No. ASP-1311-21	Revision No. 8	Effective Date October 26, 2005	Page 33 of 33

TITLE: TOXICITY CHARACTERISTIC LEACHING PROCEDURE (TCLP)

SUPERCEDES: Revision 7 – AWC-1311-21

STL BUFFALO Logbook# A05-08-12

TCLP Preparation Logbook for Method 1311 Page 2 of 2

ANALYST:_____ DATE:_____ BATCH#_____

LABORATORY SAMPLE ID						
EXTRACTION VESSEL						
6. Sample Preparation for Extraction Procedure						
a) If sample is 100% solid, add VOA free TCLP Fluid#1.						
Record weight of sample (25 grams maximum for Volatiles)						
Record volume (mL) of extraction fluid added (amount of fluid is 20 X the weight of sample)						
b) If sample is ≥0.5% solid, determine weight of sample (WW) (wet and solid) to add to ZHE using the following equation:						
(WW)=(25 / %solids (from Step2)) x 100 ; record weight						
Add (WW) to ZHE and pressure filter the liquid TCLP portion into a pre-weighed Tedlar bag (record weight (g)) by gently applying pressure in increments of 10, 20, 30, 40, and 50 psi						
Re-weigh Tedlar bag containing liquid TCLP portion						
Determine and record volume (mL) of extraction fluid to add Fluid #1 added = (20 x %solid (from Step 2) x (WW)) / 100						
7. TCLP Rotation (rotate for 18 ± 2 hours)						
ZHE Leak Test performed (before every extraction) **Pass/Fail**						
Record Start time						
Starting pressure						
Record Tumbler rpm **(range of 30 ± 2 rpm) YES or NO**						
Record Stop time						
Record min/max temperature **(range of 23 ± 2°C)**						
Record Filtration Complete **Date** and **Time**						
Record the final Pressure of ZHE. If pressure has been maintained at 10 PSI or greater then **proceed.** If pressure has not been maintained at 10 PSI then **reset** there is a leak.						
8. Final TCLP Extract If sample is 100% solids, only c will be used. If sample <0.5 % solids, only a will be used. If sample is multiphasic, both b and c will be used.						
a) Volume (mL) of Liquid Extract from Pressured Filtered Sample (from Step 2) with <0.5% solids						
b) Volume (mL) of Liquid Extract from Pressured Filtered Sample (from Step 2)with ≥ 0.5% solids (LP)						
c) Volume (mL) of Tumbled Extract Obtained in Step 6 from Solid portion of Pressured Filtered sample						
If volumes from b (not tumbled filtered liquid extract) and c (post tumbled filtered liquid extract) are compatible, add together. If volumes are not miscible, treat as separate extracts.						
Record Combined Volume mL						

Reviewed By/Date_____

Comments:_____

Figure 8. Logbook – TCLP VOA (continued: page 2of 2)

STL Buffalo
LABORATORY STANDARD OPERATING PROCEDURES

SOP No.	Revision No.	Effective Date	Page
ASP-3510-80	10	January 20, 2006	1 of 23

TITLE: METHOD 3510C: AQUEOUS SEPARATORY FUNNEL EXTRACTION PROCEDURE

SUPERCEDES: ASP-3510-80, rev9; ASP-608-65, rev6; ASP-625-68, rev5

REVIEWED & APPROVED BY:	SIGNATURE	DATE
Kathleen Aldrich, Supervisor		
Verl D. Preston, Quality Manager		
Christopher Spencer, Laboratory Director		

1.0 IDENTIFICATION OF TEST METHOD

1.1. Method 3510C, "Separatory Funnel Liquid-Liquid Extraction", Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW-846, Third Edition, Final Update III, December 1996.

1.2. Method 608- Organochlorine Pesticides and PCBs

1.3. Method 625-Base/Neutrals and Acids

2.0 APPLICABLE MATRIX

2.1. Water

3.0 REPORTING LIMIT N/A

4.0 SCOPE AND APPLICATION

4.1. This method describes a procedure to extract a broad range of organic compounds from aqueous samples for analysis by either GC or GCMS. This method also describes concentration techniques, which prepare the extract for the appropriate analysis.

TITLE: METHOD 3510C: AQUEOUS SEPARATORY FUNNEL EXTRACTION PROCEDURE

SUPERCEDES: ASP-3510-80, rev9; ASP-608-65, rev6; ASP-625-68, rev5

5.0 SUMMARY OF THE TEST METHOD

5.1. A measured volume of aqueous sample, approximately 1 liter, is extracted with methylene chloride at a specified pH using a separatory funnel extraction. The extracts are combined, dried through activated anhydrous sodium sulfate and exchanged into a solvent suitable for its cleanup or analysis as necessary.

6.0 DEFINITIONS

6.1. Standard definitions are found in Section 3.0 of the Laboratory Quality Manual.

6.2. Solvent Exchange: The extraction solvent is exchanged to the final volume solvent using Hexane.

7.0 INTERFERENCES

7.1. Method interference may be caused by contaminants in solvents, reagents, glassware and other sample processing hardware that lead to discrete artifacts or elevated baselines in gas chromatograms. All these materials must be routinely demonstrated to be free from interference under the conditions of the analysis, by analyzing reagent blanks.

7.2. Matrix interference may be caused by contaminants that are co-extracted from the sample.

7.3. Glassware used for water extractions is kept separate from soil glassware to prevent cross-contamination of high level contamination.

8.0 SAFETY

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

8.2. The use of separatory funnels to extract aqueous samples with Methylene Chloride creates excessive pressure very rapidly. Initial venting should be done immediately after the sample container has been sealed and inverted. Vent the funnel into the hood away from people and other samples. This is considered a high-risk activity, and a face shield must be worn over safety glasses or goggle when it is performed.

8.3. 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.

TITLE: METHOD 3510C: AQUEOUS SEPARATORY FUNNEL EXTRACTION PROCEDURE

SUPERCEDES: ASP-3510-80, rev9; ASP-608-65, rev6; ASP-625-68, rev5

Material (1)	Hazards	Exposure Limit (2)	Signs and symptoms of exposure
Hexane	Flammable Irritant	500 ppm-TWA	Inhalation of vapors irritates the respiratory tract. Overexposure may cause lightheadedness, nausea, headache, and blurred vision. Vapors may cause irritation to the skin and eyes.
Methylene Chloride	Carcinogen Irritant	25 ppm-TWA 125 ppm-STEL	Causes irritation to respiratory tract. Has a strong narcotic effect with symptoms of mental confusion, light-headedness, fatigue, nausea, vomiting and headache. Causes irritation, redness and pain to the skin and eyes. Prolonged contact can cause burns. Liquid degreases the skin. May be absorbed through skin.
Sulfuric Acid	Corrosive Oxidizer Dehydra-dator	1 mg/m^3	This material will cause burns if comes into contact with the skin or eyes. Inhalation of vapors will cause irritation of the nasal and respiratory system.
Sodium Hydroxide	Corrosive Poison	2 ppm, 5 mg/m^3	This material will cause burns if comes into contact with the skin or eyes. Inhalation of Sodium Hydroxide dust will cause irritation of the nasal and respiratory system.
1 – Always add acid to water to prevent violent reactions.			
2 – Exposure limit refers to the OSHA regulatory exposure limit.			

8.4. All parameters of this extraction must be performed in an operational fume hood or within an extraction apparatus that is ventilated by the fume hood system. The following analytes have been tentatively classified as known or suspected, human or mammalian carcinogens: benzo(a)anthracene, benzidine, 3,3'dichlorobenzindine, benzo(a)pyrene, alpha-BHC, beta-BHC, gamma-BHC, delta-BHC, dibenz(a,h)anthracene, N-nitrosodimethylamine, 4,4'-DDT, and polychlorinated biphenyl compounds. Primary standards of these toxic compounds should be prepared in hood.

8.5. Safety glasses, gloves, and lab coats must be worn at all times. Nitrile gloves should be used when performing this extraction. Latex and vinyl gloves provide no significant protection against the organic solvents used in this SOP, and should not be used.

8.6. All solvents, reagents, and standards must be handled inside a fume hood and with proper personal safety equipment due to their hazardous properties. All samples must be opened inside a fume hood due to their unknown hazardous properties.

TITLE: METHOD 3510C: AQUEOUS SEPARATORY FUNNEL EXTRACTION PROCEDURE

SUPERCEDES: ASP-3510-80, rev9; ASP-608-65, rev6; ASP-625-68, rev5

9.0 EQUIPMENT AND SUPPLIES

9.1. 1 liter graduated cylinder

9.2. 250 mL graduated cylinder

9.3. 2 liter Teflon separatory funnels, stopcocks, and caps

9.4. Syringes

9.5. Turbovaps and turbovap vessels

9.6. 16 oz. French squares

9.7. Powder funnels

9.8. Glasswool

9.9. Pipets

9.10. 2 mL vials and caps (amber or clear depending on application)

9.11. 10 mL vials

9.12. Vial crimpers

9.13. Teflon and plastic caps for 10 mL vials

9.14. Wide range pH paper

9.15. Centrifuge and centrifuge tubes

9.16. Glass wool

9.17. Automatic separatory funnel rotators

9.18. Narrow range pH paper

9.19. Aluminum weigh dishes

TITLE: METHOD 3510C: AQUEOUS SEPARATORY FUNNEL EXTRACTION PROCEDURE

SUPERCEDES: ASP-3510-80, rev9; ASP-608-65, rev6; ASP-625-68, rev5

10.0 REAGENTS AND STANDARDS

10.1. High purity, reagent grade chemicals will be used at all times.

10.1.1. Methylene chloride

10.1.2. 10N sodium hydroxide

10.1.3. Hexane

10.1.4. Anhydrous granular sodium sulfate. Sodium sulfate is baked in a 400°C oven for a minimum of 4 hours and allowed to cool in a dessicator prior to use.

10.1.5. 1:1 sulfuric acid

10.1.6. Deionized water and/or carbon filtered water

10.1.7. Concentrated Sulfuric Acid

10.1.8. Methanol

10.1.9. Acetone

10.1.10. Appropriate spikes and surrogates (See Table 3)

10.1.11. Florisil Cartridges

11.0 SAMPLE COLLECTION, PRESERVATION, SHIPMENT AND STORAGE

11.1. All samples must be stored in a glass amber sample container and stored at 4 degrees Celsius. The sample is stored unpreserved and unfiltered unless otherwise requested by the client.

11.2. Typical method holding time for water samples is seven days from sampling. However, the client may impose a more strict time constraint.

12.0 QUALITY CONTROL

12.1. All batches (20 samples or less) will contain a matrix spike blank (MSB) and method blank (MBLK) when a matrix spike (MS) and matrix spike duplicate (SD) are supplied. When client-specific QC is not assigned or there is not sufficient volume to assign QC samples, a matrix spike blank (MSB), matrix spike blank duplicate (MSBD) and a method blank (MBLK), will be assigned. All reagent blanks, method spike blanks, matrix spikes and matrix spike duplicates will undergo the same procedure as the samples.

TITLE: METHOD 3510C: AQUEOUS SEPARATORY FUNNEL EXTRACTION PROCEDURE

SUPERCEDES: ASP-3510-80, rev9; ASP-608-65, rev6; ASP-625-68, rev5

13.0 CALIBRATION AND STANDARDIZATION N/A

14.0 PROCEDURE

14.1. **Methods 8082, 8081, 608 (three neutral shakes)**

14.1.1. Assemble and pre-rinse 2L separatory funnel, stopcock and stopper, as well as, all other extraction supplies and glassware with DI, methanol and methylene chloride.

14.1.2. Label each separatory funnel with Severn Trent Laboratories vial label that corresponds with the sample I.D. number.

14.1.3. Make a powder funnel by placing a glass wool plug in a powder funnel, fill funnel 2/3 full with activated granular sodium sulfate. Rinse the sodium sulfate, in the funnel, with 20-30ml of methylene chloride and allow draining. Discard this methylene chloride rinse. Place the powder funnel into clean french squares labeled with Severn Trent Laboratories vial labels. Push the end of the funnel through a napkin to serve as a guard against condensing water.

14.1.4. Obtain the designated spike and surrogate solutions (See Table 3), as stated on the Task Assignment sheet, and allow them to come to room temperature.

14.1.5. Obtain the samples from cooler and sign samples out in the sample chain of custody logbook.

14.1.6. Using a disposable pipette and wide range pH paper, test and record the initial pH of the sample.

14.1.6.1. Since the sample pH will be unknown, wide range pH paper may be used for initial process. When defining the actual extraction pH later in this procedure (14.1.17, 14.2.2 & 14.2.4), the use of narrow-range pH paper will be required.

14.1.7. All pH measurements are to be made by the following method:

14.1.7.1. Dip the back end of a glass disposable pipette into the liquid.

14.1.7.2. Tap this end onto a piece of wide-range pH paper.

14.1.7.3. Record the measurement and discard the paper and pipette.

14.1.8. Check each sample for large amounts of sediment on the bottom of the sample bottle.

14.1.9. If there is a large amount of sediment in the sample the sample volume should be measured by pouring the sample into a pre-rinsed graduated cylinder (see rinsing protocol in step 14.1.1). Make sure to try and leave as much of the sediment in the sample bottle as possible. Record the sample volume and then pour the sample into its corresponding labeled separatory funnel.

14.1.10. If the sample is relatively free of sediment then mark the meniscus on the bottle.

14.1.11. For MSB, MSBD, and/or MBLK samples, 1 liter of deionized water will be measured and transferred to its labeled separatory. funnel. This will be treated as all other samples and will go through the entire extraction process.

14.1.12. Add the appropriate surrogate solution to all samples (see Table 3). The surrogate code, expiration date, prep analyst initials and surrogate analyst initials and the syringe number are all recorded in the batch sheets.

14.1.12.1. It is important to mark the labels of each sample and blank accordingly when adding spikes and surrogates to avoid error. Once a surrogate has been added (whether it is to the original sample jar or the separatory funnel should the occasion warrant it), an "X" must be drawn on the label affixed to the separatory funnel. After a spike has been added, circle the "X" immediately.

14.1.13. Samples that have been transferred already to their separatory funnels have the surrogate added directly to the separatory funnel.

14.1.14. Samples remaining in their sample bottles have the surrogate added directly to the sample bottle, recapped and shaken.

14.1.15. Spike all samples designated to receive spikes (see Table 3) and record all necessary information. After a spike has been added, circle the "X" on the label immediately.

14.1.16. Transfer all samples to their corresponding labeled separatory funnels. Record their appearance in the batch work.

14.1.17. Make any pH adjustments as needed for extraction using either 10N sodium hydroxide or 1:1 H2SO4 and narrow range pH paper. Pesticide and PCB analysis require the samples be at a pH between 5 and 9.

14.1.17.1. If any pH adjustment is made the separatory funnel must be capped and shaken for a moment to ensure homogenization of the newly added acid or base. Once shaken, the samples pH can be tested.

14.1.18. Add 60 ml of methylene chloride either to the separatory funnel or the sample bottle, depending on where the surrogate was added. If the surrogate was added directly to the separatory funnel, then add the methylene chloride directly to the separatory funnel. If the surrogate was added to the sample bottle, add the methylene chloride to the sample bottle. If the methylene chloride is added to the sample bottles then proceed to recap, shake, and vent. Then add this to the separatory funnel. Save the sample bottle for volume measurement later.

14.1.18.1. **Note:** The method for the delivery of solvent to the extraction container will be as follows: Obtain Teflon graduated cylinders and add the necessary amount of solvent. Pour this solvent from the graduated cylinder to the extraction vessel. Under no circumstances is the main bottle of solvent to be lifted up to the sample and solvent added directly from the solvent pump.

14.1.19. Seal and shake the separatory funnels, venting frequently. Shake each separatory funnel for 2 minutes. If the automatic separatory funnel rotator is to be used: seal the separatory funnels and rotate a few times, vent all separatory funnels and then rotate for an additional 2 minutes.

14.1.20. Allow the organic layer to separate from the water for a minimum of 10 minutes.

14.1.21. Wet the powder funnel with a little $MeCl_2$ prior to draining. Drain the $MeCl_2$ layer through the powder funnel.

14.1.21.1. If an emulsion occurs so that it is 1/3 the solvent layer, it must be centrifuged.

14.1.21.2. Following centrifugation of the emulsion, place the aqueous layer back in the separatory funnel and pour the $MeCl_2$ layer into the powder funnel.

14.1.21.3. Rinse the centrifuge tube with 5-10 mLs of $MeCl_2$ and add this to the powder funnel to complete the transfer.

14.1.22. Rinse each powder funnel with approximately 20-30ml methylene chloride.

14.1.23. Perform two more extractions with 60ml methylene chloride (added directly to the separatory funnel via the Teflon graduated cylinders designated for solvent delivery), shaking or rotating for 1 minute each time. Rinse the powder funnel with 10 – 20 mLs of $MeCl_2$ after the third drain.

14.1.24. Pour the extracted samples into the satellite "W" waste containers. Adjust the pH of each container to between 5 and 9 and discard in the main W-waste drum.

14.1.25. Fill each of the original sample bottles (that were set aside earlier) with tap water to the meniscus mark. Pour the water into a graduated cylinder, measure, and record the initial volume used on the batch sheet. Discard water into A-waste.

14.1.26. Concentrate the extract using the Turbovaps. Pour approximately 150ml of extract into pre-rinsed, labeled Turbovap vessel and place in the Turbovap. Add the remaining extract volume to the vessel once it has concentrated down enough to add the remainder of the extract. Rinse the french square with 10-20 mLs of $MeCl_2$ to complete the transfer to the turbovap vessel. Keep the Turbovap nitrogen pressure as high as possible without splashing the extract.

14.1.26.1. Splashing of the extracts must be avoided since cross-contamination could occur.

14.1.27. Rinse the french square with methylene chloride and also add this to the Turbovap vessel. Water temperature should be set at 32°C. During concentration rinse the walls of the turbovap with a small amount of $MeCl_2$ periodically.

14.1.28. When the extract reaches the 1ml calibration mark on the turbovap vessel, remove from Turbovap to reduce the amount of volatile compounds lost. If solvent

exchanging is required at this point, add 20-30 ml of the appropriate final solvent and concentrate back to the 1.0ml mark. See Table 1 for appropriate extraction and final solvents.

14.1.29. Homogenize the extract and perform any necessary cleanups (possible Florisil cleanup for pesticides and sulfuric acid cleanup for PCBs) then adjust the extract to its appropriate final volume.

14.1.29.1. For a final volume of 1.0mL: When the extract concentrates down to the calibrated 1-mL mark on the Turbovap vessel, transfer the entire extract into a 2-mL vial using a 9 inch disposable pipette.

14.1.29.2. For a final volume of 10.0mL: When the extract concentrates down to the calibrated 1-mL mark on the Turbovap vessel, add 9.0mL of the solvent to the vessel using a repipetter. Transfer approximately 1.0mL to a 2-mL vial and send for the appropriate analysis.

14.1.29.3. If the final volume is 10.0 mLs, make transfer an additional aliquot of each sample into the 2-mL vials. Discard the remaining extract, send one aliquot to GC for analysis and store the other aliquot in the sample incubator for a period not less than one month.

14.2. **BNA, 8270, 625 extraction (six extractions: 3 at acidic pH: 3 at basic pH)**

14.2.1. Set up and concentration for these tests are exactly the same as the method outlined above in section 14.1. However, carbon-filtered water is used instead of deionized water for the blanks and MSBs (except for 8270 low level extractions). The carbon-filtered water provides a preferable ionic matrix that more closely approximates the samples and results in better recoveries.

14.2.2. Once the funnels have been set, adjust the pH of each sample and blank to < 2 by adding approximately 1.5 to 2.0 mLs of 1:1 H_2SO_4. Test the pH using narrow range pH paper to verify. Add additional 1:1 H_2SO_4 if necessary and recheck the pH until the desired pH is achieved.

14.2.3. Continue with three extractions as outlined above.

14.2.4. After the third extraction, adjust the pH of each sample and blank to > 11 by adding approximately 4 to 5 mLs of 10N NaOH. Test to verify the pH with narrow range

pH paper. Add additional 10N NaOH if necessary and recheck the pH until the desired pH is achieved.

14.2.5. Continue with three more extractions by the same method outlined above.

14.2.6. After the sixth shake the sample extraction can proceed to the concentration step.

14.2.6.1. It is possible to begin concentration after the first three acid shakes and continue concentration when the base shakes are complete. However, this method is generally used for the extraction of semi-volatile compounds. Therefore, one should be careful to limit the time the sample spends in the turbovap to avoid losing target analytes.

14.2.6.2. All turbovap vessels containing semi-volatile extracts should be covered with an aluminum weigh dish during concentration to improve recoveries.

14.2.7. The extracted sample water can be disposed of in the manner outlined above.

14.2.8. Once concentrated to a volume of 1.0 mL, vial the entire concentrated extract into a 2 mL amber vial and send to GC/MS for analysis.

14.2.8.1. It is critical to transfer the entire portion of the 1.0 mL concentrate. Accurate results can only be obtained by the internal standard procedure used in GCMS if the final extract volume is precise. If some of the extract was lost in the transference, you must record the exact volume being sent to GC/MS.

14.3. **Extraction of TCLP Leachates**

14.3.1. TCLP leachate extractions can be performed for either of the two analyte categories (Pest/PCB or BNA) listed above. The only differences are in the sample volume used. Pour exactly 250 mLs of the sample into a 1000 mL graduated cylinder and dilute to the mark with Deionized water. Pour this sample directly into a separatory funnel. Record the volume as 250 mLs.

14.3.2. For this method you cannot add surrogate directly to the bottle, nor can you add the first aliquot of methylene chloride to the sample bottle. Instead, add all spikes and surrogates and $MeCl_2$directly to the sample in the separatory funnel.

14.3.3. Continue the extraction as detailed in 14.1 or 14.2 depending on the analyte category required.

14.4. **Diesel Range Organics (DRO) Extraction**

14.4.1. Follow the same procedure as listed in section 14.1; however, adjust the pH of all samples, blanks and spikes to <2 with 1:1 H_2SO_4.

14.4.2. After the third extraction and drain, and after the all sample water has been poured into the "W" waste satellite containers, add an additional 40 mLs of $MeCl_2$ to the empty separatory funnel.

14.4.3. Shake or rotate the funnel for approximately thirty seconds and drain through the powder funnel and concentrate this along with the previous extraction volume.

14.4.4. Concentrate to exactly 1.0mL and vial in a 2mL amber vial. Send the vial to GC for analysis.

15.0 CALCULATIONS N/A

16.0 METHOD PERFORMANCE

16.1. Acceptable performance is monitored through the use of Method Detection Limit Studies, as well as, recoveries of surrogate and spike compounds.

17.0 DATA ASSESSMENT AND ACCEPTANCE CRITERIA FOR QUALITY CONTROL MEASURES N/A

18.0 CORRECTIVE ACTIONS FOR OUT-OF-CONTROL DATA

18.1. In the event that sample volume is lost, bottles broken or sample is inappropriately processed (mis-spiked), an attempt should be made to locate additional volume and the extraction process performed within holding time.

18.2. If at any time during the extraction, sample or extract is spilled, it must be determined if extra volume exists. If extra volume exists, the sample will immediately be prepped again. If there is no more volume, a comment must be included in the batch comments section, indicating at which point sample or extract was lost.

TITLE: METHOD 3510C: AQUEOUS SEPARATORY FUNNEL EXTRACTION PROCEDURE

SUPERCEDES: ASP-3510-80, rev9; ASP-608-65, rev6; ASP-625-68, rev5

19.0 CONTINGENCIES FOR HANDELING OUT-OF-CONTROL OR UNACCEPTABLE DATA

19.1. A Job Exception Form must be completed and forwarded to the Department Supervisor, Project Manager, and Quality Manager if any of the following occur:

19.1.1. Holding time exceedance

19.1.2. Insufficient volume

19.1.3. Broken volume

19.1.4. Incorrect amount of surrogate or spike added

19.1.5. Sample Matrix does not allow for appropriate extraction

19.1.5.1. pH can not be adjusted

19.1.5.2. Viscosity will not allow appropriate filtering through powder funnel

19.1.5.3. Excessive emulsions that can not be broken

19.2. If at any time during the extraction, sample or extract is spilled, it must be determined if extra volume exists. If extra volume exists, the sample will immediately be prepped again. If there is no more volume, a comment must be included in the batch comments section, indicating at which point sample or extract was lost.

20.0 WASTE MANAGEMENT/ POLLUTION PREVENTION

20.1. 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."

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

20.2.1. Methylene Chloride rinses. (Spent solvents are stored in red satellite "C" waste containers. When full, a designated laboratory technician will transfer solvent material from these satellite "C" waste containers to a grounded 55-gallon drum. These are located in the secured waste area and are disposed of according to all state and federal regulations**).**

20.2.2. Hexane rinses. (Spent solvents are stored in red satellite "C" waste containers. When full, a designated laboratory technician will transfer solvent material from these satellite "C" waste containers to a grounded 55-gallon drum. These are located in the secured waste area and are disposed of according to all state and federal regulations**).**

20.2.3. Vials containing extracts in solvents. (Extract vials are disposed in BV waste drums and stored in a secure waste area. These drums are disposed of according to all state and federal regulations**).**

20.2.4. Extracted water samples. This material must be neutralized before it is discharged. (All extracted water shall be neutralized and dumped into the designated drum marked as "W" waste. When full, the satellite containers will be transferred to the secure waste storage area and disposed of by appropriately trained laboratory technicians in accordance to all state and federal regulations**).**

20.2.5. Extracted aqueous samples contaminated with methylene chloride. This material must be neutralized before it is discharged to a POTW. **(**All extracted water shall be neutralized and dumped into the designated drum marked as "W" waste. When full, the satellite containers will be transferred to the secure waste storage area and disposed of by appropriately trained laboratory technicians in accordance to all state and federal regulations**).**

20.2.6. Used sodium sulfate and glass wool or filter paper contaminated with methylene chloride from the extract drying step. **(**Solid wastes are dried in trays inside a fume hood then transferred to a 5-gallon satellite containers. Lab generated solid wastes (extracted solid waste, sodium sulfate and glass wool or filter paper) are marked as "BC waste. When full, a designated laboratory technician will transfer all of the lab generated solid waste into a 55-gallon drum. This material will be disposed of according to all state and federal regulations.**).**

20.2.7. Assorted flammable solvent waste from various rinses. **(**Spent solvents are stored in red satellite "C" waste containers. When full, a designated laboratory technician will transfer solvent material from these satellite "C" waste containers to a grounded 55-gallon drum. These are located in the secured waste area and are disposed of according to all state and federal regulations**).**

20.2.8. Methylene chloride waste from various rinses. **(**Spent solvents are stored in red satellite "C" waste containers. When full, a designated laboratory technician will transfer solvent material from these satellite "C" waste containers to a grounded 55-gallon drum. These are located in the secured waste area and are disposed of according to all state and federal regulations**).**

20.2.9. Miscellaneous disposable glassware contaminated with acids, caustics, solvents and sample residue. **(**All disposable glassware is dried of all solvents inside a fume hood then disposed of in a recycling bin).

21.0 REFERENCE

21.1. Method 3510C, "Separatory Funnel Liquid-Liquid Extraction", Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW-846, Third Edition, Final Update III, December 1996.

21.2. CFR, Title 40, Protection of Environment, Pt. 136, App.A, Meth. 608, Revised as of July 1, 1996.

21.3. CFR, Title 40, Protection of Environment, Pt. 136, App. A, Meth. 625, Revised as of July1, 1996.

22.0 TABLES, DIAGRAMS, FLOWCHARTS AND VALIDATION DATA

22.1. Table 1 : Extraction and Final Solvents

22.2. Table 2 : 3510C Prep Batch Sheet

22.3. Table 3 : Spike and Surrogate Reference Sheet

23.0 CHANGES FROM PREVIOUS REVISION

23.1. Laboratory Director – signature update

23.2. Merged SOPs for 608 and 625 extractions into this SOP.

23.3. Added 14.2.6.2- covering semi-volatile extracts during concentration.

23.4. Added 9.1.9.

23.5. Section 10.1.4: Removed the term ‘activated’

23.6. Section 14.1.11: Removed the homogenization process for sample/MS/MSD

23.7. Section 14.1.15: Re-worded to clarify addition of spike solution.

TITLE: METHOD 3510C: AQUEOUS SEPARATORY FUNNEL EXTRACTION PROCEDURE

SUPERCEDES: ASP-3510-80, rev9; ASP-608-65, rev6; ASP-625-68, rev5

24.0

TABLE 1

Extraction and Final Solvents		
Analysis Type	**Extraction Solvent**	**Final Solvent**
Pesticides	$MeCl_2$	Hexane
PCBs	$MeCl_2$	Hexane
BNAs	$MeCl_2$	$MeCl_2$
DRO	$MeCl_2$	$MeCl_2$

25.0

TABLE 2

STL Buffalo
Date: 08/26/2003
Time: 01:33:16

Organic Prep Log Book
(3510C) 8270 LOW LEVEL WATERS
A3B09546

Rept: AN0501

SURROGATE A86
Expiration Date: 09-01-03
Prepared by: KEA
Spiked by: CM
Witnessed by: ____ 20 0.00 ul

MATRIX SPIKE A55
Expiration Date: 12-22-03
Prepared by: mm
Spiked by: CM
Witnessed by: ____ 10 0.00 ul

MeCl2: Y27E39
Acetone: ____
Hexane: ____
Na2SO4: 93134384
1:1 H2SO4: 5183
10 N NaOH: 9345

Date Ext/Initials: 08-26-03 CM
Extraction Type: (SEPF) or CLLE (circle one)

AQUEOUS EXTRACTIONS

Date Cleanup/Initials: ____
Date Conc/Initials: 08-26-03 CM

Job Number	Sample ID	Bottle ID	Sample Type	Vial #	Test	Protoc	Method	Surr Code	Spike Code	Appear.	Initial pH	Sample Volume (ml)	Final Volume (ml)
A03-8067	A3806701	A	FS	AW30010675	8270LOW	SW8463	8270LOW	A00148		clear	6	1030	1.0
A03-8067	A3806702	A	FS	AW30010676	8270LOW	SW8463	8270LOW	A00148		yellow	6	1030	
A03-8067	A3806703	A+B	FS	AW30010677	8270LOW	SW8463	8270LOW	A00148		light yellow	6	1000	
A03-8067	A3806703MS	↓	MS	AW30010678	8270LOW	SW8463	8270LOW	A00148	A00147	↓	↓	540	
A03-8067	A3806703SD	↓	SD	AW30010679	8270LOW	SW8463	8270LOW	A00148	A00147	↓	↓	540	
A03-8067	A3806704	A	FS	AW30010680	8270LOW	SW8463	8270LOW	A00148		yellow	6	1055	
A03-8067	A3806705	↓	FS	AW30010681	8270LOW	SW8463	8270LOW	A00148		↓	↓	1000	
A03-8067	A3806706	↓	FS	AW30010682	8270LOW	SW8463	8270LOW	A00148		↓	↓	910	
A03-8067	A3806707	↓	FS	AW30010683	8270LOW	SW8463	8270LOW	A00148		↓	↓	1045	
A3B09546	A3B0954601		MSB	AW30010684	8270LOW	SW8463	8270LOW	A00148	A00147	clear	5	1000	
A3B09546	A3B0954602		MBLK	AW30010685	8270LOW	SW8463	8270LOW	A00148		↓	↓	↓	↓

Comments: ____

KEA 8/25/03

TITLE: METHOD 3510C: AQUEOUS SEPARATORY FUNNEL EXTRACTION PROCEDURE

SUPERCEDES: ASP-3510-80, rev9; ASP-608-65, rev6; ASP-625-68, rev5

Table 3

SPIKE AND SURROGATE REFERENCE SHEET

SURROGATES

A001: 8270 BN/AP SURROGATE

Nitrobenzene-d5	100.00 ng/µL
2-Fluorobiphenyl	100.00 ng/µL
p-Terphenyl	100.00 ng/µL
Phenol-d5	150.00 ng/µL
2-Fluorophenol	150.00 ng/µL
2,4,6-Tribromophenol	150.00 ng/µL

A026: 625 SURROGATE

Nitrobenzene-d5	50.00 ng/µL
2-Fluorobiphenyl	50.00 ng/µL
p-Terphenyl	50.00 ng/µL
Phenol-d5	50.00 ng/µL
2-Fluorophenol	50.00 ng/µL
2,4,6-Tribromophenol	50.00 ng/µL

A027: 8015B DRO SURROGATE

o-Terphenyl	20.0 ng/µL

A028: CLP 3/90 SVOA SURROGATE

Nitrobenzene-d5	50.00 ng/µL
2-Fluorobiphenyl	50.00 ng/µL
p-Terphenyl	50.00 ng/µL
1,2-Dichlorobenzene	50.00 ng/µL
Phenol-d5	75.00 ng/µL
2-Fluorophenol	75.00 ng/µL
2,4,6-Tribromophenol	75.00 ng/µL
2-Chlorophenol-d4	75.00 ng/µL

A033: 8151 HERBICIDE SURROGATE

Dichlorophenyl Acetic Acid	5.00 ng/µL

A035: PCB, PESTICIDE SURROGATE

Tetrachloro-m-xylene	0.20 ng/µL
Decachlorobiphenyl	0.20 ng/µL

TITLE: METHOD 3510C: AQUEOUS SEPARATORY FUNNEL EXTRACTION PROCEDURE

SUPERCEDES: ASP-3510-80, rev9; ASP-608-65, rev6; ASP-625-68, rev5

A093: CLP PEST/PCB SURROGATE

Tetrachloro-m-xylene	0.40 ng/µL
Decachlorobiphenyl	0.40 ng/µL

A0148: 8270 LOW LEVEL SURROGATE

Use 100.0µL of A0026

A0151: 608, 8082 LOW LEVEL WATER SURROGATE

Use 100.0 µL of A0035

A0181 AND A0233: BASE ONLY 8270 SURROGATES

Use 1000.0 µL of A0001

A0277: BASE ONLY 8270 LOW LEVEL SURROGATE

Use 200.0 µL of A0026

SPIKES

A0047: 8151 HERBICIDE SPIKE

2,4-D	2.0 ng/µL
Dalapon	2.0 ng/µL
Dinoseb	2.0 ng/µL
Pentachlorophenol	2.0 ng/µL
Picloram	2.0 ng/µL
2,4,5-TP (Silvex)	2.0 ng/µL
2,4,5-T	2.0 ng/µL
2,4-DB	2.0 ng/µL
Dicamba	2.0 ng/µL
Dinoseb	2.0 ng/µL
Dichloroprop	2.0 ng/µL

A0049: CLP PEST/PCB SPIKE

gamma-BHC (Lindane)	0.5 ng/µL
Heptachlor	0.5 ng/µL
Aldrin	0.5 ng/µL
Dieldrin	1.0 ng/µL
Endrin	1.0 ng/µL
4,4'-DDT	1.0 ng/µL

A0051: 8081 PESTICIDE SPIKE

gamma-BHC (Lindane)	1.0 ng/µL
alpha-BHC	1.0 ng/µL
Heptachlor	1.0 ng/µL

Aldrin	1.0 ng/µL
Beta-BHC	1.0 ng/µL
Dieldrin	1.0 ng/µL
Endrin	1.0 ng/µL
4,4'-DDD	1.0 ng/µL
4,4'-DDT	1.0 ng/µL
4,4'-DDE	1.0 ng/µL
Endosulfan I	1.0 ng/µL
Endosulfan II	1.0 ng/µL
Endrin Aldehyde	1.0 ng/µL
Endosulfan Sulfate	1.0 ng/µL
Heptachlor epoxide	1.0 ng/µL
Methoxychlor	1.0 ng/µL
Endrin Ketone	1.0 ng/µL

A0055: 8270 BN/AP SPIKE

Phenol	100.0 ng/µL
2-Chlorophenol	100.0 ng/µL
1,4-Dichlorobenzene	100.0 ng/µL
N-Nitroso-Di-n-propylamine	100.0 ng/µL
1,2,4-Trichlorobenzene	100.0 ng/µL
4-Chloro-3-methylphenol	100.0 ng/µL
Acenaphthene	100.0 ng/µL
4-Nitrophenol	100.0 ng/µL
2,4-Dinitrotoluene	100.0 ng/µL
Pentachlorophenol	100.0 ng/µL
Pyrene	100.0 ng/µL

A0056: 625 SPIKE

Use 500.0 µL of A0193

A0057: CLP 3/90 SVOA SPIKE

Phenol	75.0 ng/µL
2-Chlorophenol	75.0 ng/µL
1,4-Dichlorobenzene	50.0 ng/µL
N-Nitroso-Di-n-propylamine	50.0 ng/µL
1,2,4-Trichlorobenzene	50.0 ng/µL
4-Chloro-3-methylphenol	75.0 ng/µL
Acenaphthene	50.0 ng/µL
4-Nitrophenol	75.0 ng/µL
2,4-Dinitrotoluene	50.0 ng/µL
Pentachlorophenol	75.0 ng/µL
Pyrene	50.0 ng/µL

TITLE: METHOD 3510C: AQUEOUS SEPARATORY FUNNEL EXTRACTION PROCEDURE

SUPERCEDES: ASP-3510-80, rev9; ASP-608-65, rev6; ASP-625-68, rev5

A0060: CUSTOM CHLOROPYRIDINES SPIKE

2-Chloropyridine	100.0 ng/µL
3-Chloropyridine	100.0 ng/µL
2,6-Dichloropyridine	100.0 ng/µL
p-Fluoroaniline	100.0 ng/µL

A0061: CECOS CONSENT 1.6 BN/AP SPIKE

1,4-Dichlorobenzene	50.0 ng/µL
N,N'-Dimethylacetamide	50.0 ng/µL
Methylaniline N.O.S.	50.0 ng/µL
Pyridine	50.0 ng/µL

A0062: 8270 TCLP SPIKE

1,4-Dihlorobenzene	100.0 ng/µL
2,4-Dinitrotoluene	100.0 ng/µL
Hexachlorobenzene	100.0 ng/µL
Hexachlorobutadiene	100.0 ng/µL
Hexachloroethane	100.0 ng/µL
2-Methylphenol	100.0 ng/µL
3-Methylphenol	200.0 ng/µL
4-Methylphenol	200.0 ng/µL
Nitrobenzene	100.0 ng/µL
Pentachlorophenol	100.0 ng/µL
Pyridine	100.0 ng/µL
2,4,5-Trichlorophenol	100.0 ng/µL
2,4,6-Trichlorophenol	100.0 ng/µL

A0095: DIESEL FUEL #2 DRO AND PETRO SPIKE

Diesel Fuel #2	1500.0 ng/µL

A0113: DECHLORANE PLUS

Dechlorane Plus	1.0 ng/µL

A0143: 8082 PCB SPIKE (USE 100.0µL)

Aroclor 1254	50.0 ng/µL

A0147: 8270 LOW LEVEL SPIKE

Use 100.0 µL of A0055

A00152: 608 PESTICIDE AND PESTICIDE/PCB SPIKE

Use 50.0 µL A0051

TITLE: METHOD 3510C: AQUEOUS SEPARATORY FUNNEL EXTRACTION PROCEDURE

SUPERCEDES: ASP-3510-80, rev9; ASP-608-65, rev6; ASP-625-68, rev5

A00153: 608 PCB SPIKE

Use 100.0 µL of A0213

A00184: DRO 10 COMPONENT SPIKE

Diesel Range Organics	200.0 ng/µL

A00193: 8270 FULL LIST SPIKE

Acenaphthene	100.0 ng/µL
Aniline	100.0 ng/µL
Acenaphthylene	100.0 ng/µL
Anthracene	100.0 ng/µL
Benzo(a)anthracene	100.0 ng/µL
Benzo(b)fluoranthene	100.0 ng/µL
Benzo(k)fluoranthene	100.0 ng/µL
Benzo(ghi)perylene	100.0 ng/µL
Benzo(a)pyrene	100.0 ng/µL
Benzoic Acid	250.0 ng/µL
Benzyl alcohol	100.0 ng/µL
Bis(2-chloroethoxy) methane	100.0 ng/µL
Bis(2-chloroethyl) ether	100.0 ng/µL
2,2'-Oxybis(1-Chloropropane)	100.0 ng/µL
Bis(2-ethylhexyl)phthalate	100.0 ng/µL
4-Bromophenyl phenyl ether	100.0 ng/µL
Butyl benzyl phthalate	100.0 ng/µL
4-Chloroaniline	100.0 ng/µL
4-Chloro-3-methylphenol	100.0 ng/µL
2-Chloronaphthalene	100.0 ng/µL
2-Chlorophenol	100.0 ng/µL
4-Chlorophenyl phenyl ether	100.0 ng/µL
Chrysene	100.0 ng/µL
Dibenzo(a,h)anthracene	100.0 ng/µL
Dibenzofuran	100.0 ng/µL
Di-n-butyl phthalate	100.0 ng/µL
1,2-Dichlorobenzene	100.0 ng/µL
1,3-Dichlorobenzene	100.0 ng/µL
1,4-Dichlorobenzene	100.0 ng/µL
3,3'-Dichlorobenzidine	100.0 ng/µL
2,4'-Dichlorophenol	100.0 ng/µL
Diethyl phthalate	100.0 ng/µL
2,4-Dimethylphenol	100.0 ng/µL
Dimethyl phthalate	100.0 ng/µL
4,6-Dinitro-2-methylphenol	100.0 ng/µL
2,4-Dinitrophenol	100.0 ng/µL

2,4-Dinitrotoluene	100.0 ng/µL
2,6-Dinitrotoluene	100.0 ng/µL
Di-n-octyl phthalate	100.0 ng/µL
Fluoranthene	100.0 ng/µL
Fluorene	100.0 ng/µL
Hexachlorobenzene	100.0 ng/µL
Hexachlorobutadiene	100.0 ng/µL
Hexachlorocyclopentadiene	100.0 ng/µL
Hexachloroethane	100.0 ng/µL
Indeno(1,2,3-cd)pyrene	100.0 ng/µL
Isophorone	100.0 ng/µL
2-Methylnaphthalene	100.0 ng/µL
2-Methylphenol	100.0 ng/µL
4-Methylphenol	100.0 ng/µL
Naphthalene	100.0 ng/µL
2-Nitroaniline	100.0 ng/µL
3-Nitroaniline	100.0 ng/µL
4-Nitroaniline	100.0 ng/µL
Nitrobenzene	100.0 ng/µL
2-Nitrophenol	100.0 ng/µL
4-Nitrophenol	100.0 ng/µL
N-Nitrosodiphenylamine	100.0 ng/µL
N-Nitroso-Di-n-prpoylamine	100.0 ng/µL
Pentachlorophenol	100.0 ng/µL
Phenanthrene	100.0 ng/µL
Phenol	100.0 ng/µL
Pyrene	100.0 ng/µL
1,2,4-Trichlorobenzene	100.0 ng/µL
2,4,5-Trichlorophenol	100.0 ng/µL
2,4,6-Trichlorophenol	100.0 ng/µL

A0213: 8082 PCB SPIKE

Aroclor 1242	5.0 ng/µL

A0222: 8082 PCB SPIKE

Aroclor 1016	5.0 ng/µL
Aroclor 1260	5.0 ng/µL

A0251: AFCEE 8081 PESTICIDE SPIKE

gamma-BHC (Lindane)	1.0 ng/µL
alpha-BHC	1.0 ng/µL
Heptachlor	1.0 ng/µL
Aldrin	1.0 ng/µL

Beta-BHC	1.0 ng/µL
Dieldrin	1.0 ng/µL
Endrin	1.0 ng/µL
4,4'-DDD	1.0 ng/µL
4,4'-DDT	1.0 ng/µL
4,4'-DDE	1.0 ng/µL
Endosulfan I	1.0 ng/µL
Endosulfan II	1.0 ng/µL
Endrin Aldehyde	1.0 ng/µL
Endosulfan Sulfate	1.0 ng/µL
Heptachlor epoxide	1.0 ng/µL
Methoxychlor	1.0 ng/µL
Endrin Ketone	1.0 ng/µL
alpha-Chlordane	1.0 ng/µL
gamma-Chlordane	1.0 ng/µL

A0234: 8270 1,4-DIOXANE ONLY SPIKE

1,4-Dioxane	100.0 ng/µL

A0251: 8082 LOW LEVEL WATER PCB SPIKE (USE 100.0µL)

Aroclor 1254	5.0 ng/µL

A0281: 8082 LOW LEVEL WATER PCB SPIKE

Use 100.0 µL of A0222

STL Buffalo
LABORATORY STANDARD OPERATING PROCEDURES

SOP No.	Revision No.	Effective Date	Page
ASP-3550B-93	11	February 28, 2006	Page 1 of 22

TITLE: METHOD 3550B: ULTRASONIC EXTRACTION OF SOILS AND WIPES

SUPERCEDES: Revision 10

REVIEWED & APPROVED BY:	SIGNATURE	DATE
Kathleen E. Aldrich, Supervisor		
Verl D. Preston, Quality Manager		
Christopher Spencer, Laboratory Director		

1.0 IDENTIFICATION OF TEST METHOD

1.1 This operating procedure refers to the extraction methods for CLP pesticide/PCB, CLP SVOA, and method 3550B soil samples.

2.0 APPLICABLE MATRIX

2.1 Soils, sediments and wipes

3.0 REPORTING LIMIT N/A

4.0 SCOPE AND APPLICATION

4.1 This method is used for the extraction of nonvolatile and semivolatile organic compounds from solids and wipes. The ultrasonic process used ensures thorough contact of the sample with the extraction solvent.

5.0 SUMMARY OF THE TEST METHOD

5.1 Low Level

5.1.1 A 30 gram sample is mixed with anhydrous sodium sulfate. This is solvent extracted three times using ultrasonic extraction. The extract is then filtered and concentrated. The extract may then be subject to clean-up procedures or sent directly for analysis.

TITLE: METHOD 3550B: ULTRASONIC EXTRACTION OF SOILS AND WIPES

SUPERCEDES: Revision 10

5.2 Medium/High Level

5.2.1 A 2 gram sample is mixed with anhydrous sodium sulfate and solvent extracted once using ultrasonic extraction. A portion of the extract is removed for cleanup and/or analysis.

5.3 Wipes

5.3.1 A wipe sample is mixed with anhydrous sodium sulfate and solvent extracted once using ultrasonic extraction. A portion of the extract is removed for cleanup and/or analysis.

6.0 DEFINITIONS

6.1 Standard definitions are found in Section 3.0 of the Laboratory Quality Manual.

7.0 INTERFERENCES

7.1 Method interference may be caused by contaminants in solvents, reagents, glassware and other sample processing hardware that lead to discrete artifacts or elevated baselines in gas chromatograms. All these materials must be routinely demonstrated to be free from interference under the conditions of the analysis, by analyzing reagent blanks.

7.2 Matrix interference may be caused by contaminants that are co-extracted from the sample.

7.3 Major organic interferences may be removed during cleanup procedures.

8.0 SAFETY

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

8.2 All parameters of this extraction must be performed in an operational fume hood or within an extraction apparatus that is ventilated by the fume hood system.

8.3 Any excess unextracted sample (including dry weights) waste will be disposed of in "BE" waste. Solid waste generated in the extraction process will be disposed of in "BC" waste. All solvent and extract waste is disposed of in "C" waste.

8.4 Safety glasses, gloves, and lab coats must be worn at all times. Nitrile gloves should be used when performing this extraction. Latex and vinyl gloves provide no significant protection against the organic solvents used in this SOP and should not be used.

8.5 All solvents, reagents, and standards must be handled inside a fume hood and with proper personal safety equipment due to their hazardous properties. All samples must be opened inside a fume hood due to their unknown hazardous properties.

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

Material (1)	Hazards	Exposure Limit (2)	Signs and symptoms of exposure
Methylene Chloride	Carcinogen Irritant	25 ppm-TWA 125 ppm-STEL	Causes irritation to respiratory tract. Has a strong narcotic effect with symptoms of mental confusion, light-headedness, fatigue, nausea, vomiting and headache. Causes irritation, redness and pain to the skin and eyes. Prolonged contact can cause burns. Liquid degreases the skin. May be absorbed through skin.
Acetone	Flammable	1000 ppm-TWA	Inhalation of vapors irritates the respiratory tract. May cause coughing, dizziness, dullness, and headache.
Hexane	Flammable Irritant	500 ppm-TWA	Inhalation of vapors irritates the respiratory tract. Overexposure may cause lightheadedness, nausea, headache, and blurred vision. Vapors may cause irritation to the skin and eyes.
1 – Always add acid to water to prevent violent reactions.			
2 – Exposure limit refers to the OSHA regulatory exposure limit.			

8.6 ***Noise Levels and Hearing Protection***: Ultrasonic disrupters can produce high intensity noise and must be used in an area with adequate noise protection, which includes box-style enclosures (with doors), hood sashes and/or ear plugs/muffs. STL Buffalo operates two styles of ultrasonic disruptors; standard one-inch (dual horn), and micro-tip units. Noise level surveys have been conducted for each style during the normal course of their operation.

8.6.1 Standard One-inch dual horn units were surveyed for noise levels with their box doors completely closed and the hood sash opened approximately 18". Noise measurements yielded acceptable levels and additional hearing protection is not required. The same units were measured with their box doors open and the hood

sash completely closed. Again, noise measurements yielded acceptable levels and additional hearing protection is not required.

8.6.2 Micro-tip units do not have enclosures and were surveyed for noise levels with the hood sash opened approximately 18" and again with the hood sash fully closed. Noise measurements exceeded acceptable levels with the hood sash opened to 18". Noise measurements yielded acceptable levels with the hood sash fully closed. Therefore, **the hood sash MUST BE FULLY CLOSED when operating any micro-tip ultrasonic extractor.** Following this scenario, additional hearing protection is not required. It should be noted that the hood sash has been labeled with an operator warning.

9.0 EQUIPMENT AND SUPPLIES

9.1 Aluminum Dishes, Foil

9.2 Metal spatula or disposable, wood tongue depressor

9.3 Toploader Balance, capable of accurately measuring to 0.1g

9.4 Syringes

9.5 ¾ in. dual horn Sonicators® with Sonabox® acoustic enclosures

9.6 16 oz. french squares, disposable

9.7 Ovens - 104°C and 400°C

9.8 16 oz. wide mouth jars, disposable

9.9 Turbovap concentrators and vessels

9.10 Stainless steel filter funnels

9.11 Graduated cylinders

9.12 Ear Protection

9.13 2,10 and 24ml vials, septa and caps

9.14 Disposable pipets and pipet bulbs

9.15 18.5 cm #41 filter paper

9.16 Microtip horn Sonicators® with Sonabox® acoustic enclosures

10.0 REAGENTS AND STANDARDS

10.1 All solvents are pesticide grade or equivalent.

10.1.1 Hexane

10.1.2 Compressed Nitrogen

10.1.3 Anhydrous granular sodium sulfate, previously baked in a 400°C oven for a minimum of 4 hours, cooled and dried in a dessicator, and rinsed with methylene chloride.

10.1.4 Methylene Chloride

10.1.5 Acetone

10.1.6 Surrogate and spike solutions appropriate to the final determinative procedures as assigned by test profile (See Table 2).

10.1.7 De-ionized water (DI)

11.0 SAMPLE COLLECTION, PRESERVATION, SHIPMENT AND STORAGE

11.1 All samples must be stored in a glass amber sample container and stored at 4°C. The sample is stored unpreserved and unfiltered unless otherwise requested by the client.

11.2 Typical method holding time for SW-846, third edition soil samples is fourteen days from sampling. However, the client may impose a more strict time constraint.

11.3 Typical method holding time for USEPA Contract Laboratory Program (CLP) soil samples is ten days from receipt.

12.0 QUALITY CONTROL

12.1 All batches (20 samples or less) will contain a matrix spike blank (MSB) and method blank (MBLK) when a matrix spike (MS) and matrix spike duplicate (SD) are supplied. When client-specific QC is not assigned or there is not sufficient volume to assign QC samples, a matrix spike blank (MSB), matrix spike blank duplicate (MSBD) and a method blank (MBLK), will be assigned. All reagent blanks and matrix spike duplicates will undergo the same procedure as the samples.

13.0 CALIBRATION AND STANDARDIZATION

13.1 The top loader balance will be calibrated every 6 months and checked daily prior to use to ensure calibration is maintained.

14.0 PROCEDURE

Note: All samples must be signed out of the sample chain of custody logbook and signed back in when returned. If the entire sample is to be used for the extraction, write "disc" for discard in the "TIME IN" column.

14.1 Low Level Extraction:

14.1.1 Decant and discard any standing water on sample. On the batch sheet, mark a "Y" for "yes" or an "N" for "no" in the "Decant" column. Discard any sticks, leaves, rocks or other foreign matter.

14.1.2 Tare a labeled 16oz wide mouth jar and transfer at least 90 to 100 grams of the sample to the jar. Homogenize the sample thoroughly. Transfer all but 30 grams of the sample back to the original sample jar.

14.1.3 Mark aluminum dish on bottom with the last three digits of the STL vial number. Weigh and record the weight of the dish.

14.1.4 Weigh 5 – 9 grams of recently homogenized sample into the dish and record the combined sample plus dish weight. Place in the dry weight oven (104°C) for at least four hours prior to dry weight determination.

14.1.5 Add granular sodium sulfate to the 30g sample and blend with a spatula or wood tongue depressor until the sample is free flowing.

14.1.6 If the sample is excessively wet or needs to be decanted prior to homogenization, add the sodium sulfate to the sample, mix and let the sample sit for ten minutes. This time allows the sodium sulfate to absorb the water from the sample, however it will also harden the sample. After the sample sits, it will be necessary to break up the sample with the spatula until a free flowing consistency is again achieved.

14.1.6 Add surrogate (See Table2) to the samples using the appropriate surrogate as designated on the batch sheet. Write an "X" on the label after adding the surrogate.

14.1.8 Add appropriate spike (See Table2) to samples designated MS, MSD, MSB, MSBD. (The spike code to be used appears on the preparation batch logbook sheets.) Circle the "X" after adding the appropriate spike.

14.1.9 For blank samples (MSB, MSBD, or MBLK), approximately 30g of sodium sulfate will be used in lieu of soil and shall be taken through the entire analytical procedure.

14.1.10 Add 100mls of appropriate solvent to the sample; the solvent for determinative methods is as follows:
All CLP methods – 1:1 methylene chloride/acetone
8080,8081,8082 - 1:1 acetone/hexane
8270 – Methylene chloride
DRO - Methylene chloride
*8270 soils for specific clients (NiSource) and USACE projects will be extracted with 1:1 Methylene chloride/Acetone

14.1.11 Fold an 18.5cm filter paper into quarters and place it in a stainless steel filter funnel.

14.1.12 Place this funnel in a labeled french square bottle, wrapped in a napkin to prevent condensing water from entering the sample.

14.1.13 Before use, clean the sonication horns with DI water, acetone, and the extraction solvent. Wipe the horns thoroughly with paper towels after the DI water rinse.

14.1.14 Place the 16oz. wide mouth jar under the sonication horn so it is submerged ½ inch. Ideally, the sonicator horn is to be submerged into the solvent ½ inch and still above the soil sample by the ½ inch. In the case of excessively wet samples that needed a great deal of $MeCl_2$, more solvent may be added and the position of the sonicator jar adjusted to the ideal parameters.

14.1.15 Sonicate for 3 minutes at out put setting 10, pulsed mode, 50% duty cycle, using ¾ inch horn.

14.1.16 Collect the extract in a labeled french square jar by first decanting the extract through the filter funnel containing the 18.5 cm filter paper folded inside. When using solvents with acetone, add a little sodium sulfate to the filter paper to reduce the amount of water in the extract.

14.1.17 Add 100ml of appropriate solvent to the sample.

14.1.18 Repeat steps 14.1.13, 14.1.14, 14.1.15, and 14.1.16

14.1.19 Repeat steps 14.1.13, 14.1.14, and 14.1.15

14.1.20 After the third sonication, rinse the contents of the sonication jar into the funnel.

14.1.21 After sample has drained, rinse down the funnel with 20-30mLs of the extraction solvent being used. Allow the sample to drain completely inside a fume hood.

14.1.22 Clean the sonicator horn between samples as describe in 14.1.13.

14.2 Concentration Procedure

14.2.1 Pour the extract into a labeled turbovap vessel that is pre-rinsed with $MeCl_2$, rinse the french square with the appropriate solvent and add this to the turbovap vessel.

14.2.2 Place the vessel in the turbovap at a water temp of 32°C and turn on the nitrogen to concentrate the extract to approximately 1ml. During concentration, the turbovap vessel should be periodically rinsed with the extraction solvent.

14.2.3 For 8270 and DROs, bring the volume to exactly 1ml using the calibrated 1.0ml mark on the turbovap vessel. Transfer this to a 2ml vial using a disposable 9-inch pipette. The entire 1.0mL volume must be transferred to the vial. If any sample is spilled during the transfer to the vial, it must be noted in the comment section of the batch sheets. 8270 samples can be relinquished to GC/MS for analysis and DRO samples can be relinquished to GC for analysis.

14.2.3 For 8080, 8081, 8082 the extract is ready for cleanup or analysis, depending on the extent of interfering co-extractives. If proceeding directly to analysis, bring the volume to 1.0ml using the calibrated 1.0ml mark on the turbovap vessel then adjust the final volume to 10.0ml by adding 9.0ml of Hexane to the turbovap vessel with a repipetter. Transfer 1ml to a 2ml vial using a disposable pipette, mark the meniscus on the vial and relinquish to GC for analysis. Vial a second one mL aliquot and store this in the sample incubator with the necessary label for future reference. This needs to be stored for a period no less than one month. The remaining 8 mLs can now be disposed of by trained personnel. If cleanup is required, follow the appropriate SOPs at this point.

14.2.4 For all CLP method soils, GPC cleanup is required. Bring the volume to 1.0ml using the calibrated 1.0ml mark on the turbovap vessel then adjust the final volume to 10.0ml by adding 9.0ml of Methylene chloride to the turbovap vessel with a repipetter. Transfer to a 24mL vial. Cap and set aside in a $4^oC \pm 2^o C$ incubator for later clean up by GPC. The GPC procedure can be found in ASP-3640A-96.

14.2.4.1 After GPC, SVOA samples can be transferred to pre-rinsed turbovap vessels, concentrated to 0.5mL, and vialed in amber vials. These SVOA samples can then be relinquished to GC/MS for analysis.

14.2.4.2 After GPC, pesticide/PCB samples must be solvent exchanged to hexane and florisiled as stated in ASP-3620A-95. After florisil cleanup, the samples can be transferred to pre-rinsed turbovap vessels and concentrated to 5.0mL. These samples will then be transferred to labeled 24mL clear

vials and 2 aliquots will be transferred to clear 2mL vials. One aliquot will be saved and stored in the extra volume refrigerator and one aliquot will be relinquished to GC for analysis. The remaining extract in the 24mL vial can be discarded into C-waste.

14.3 MEDIUM LEVEL EXTRACTION:

14.3.1 Decant and discard any standing water on sample. On the batch sheet, mark a "Y" for "yes" or an "N" for "no" in the "Decant" column. Discard any sticks, leaves, rocks or other foreign matter.

14.3.2 Homogenize the sample by transferring all contents of the sample jar into a clean disposable 16oz wide mouth jar and mixing thoroughly. Transfer 2 grams of the sample into a tared 24 mL extraction vial. Return the remaining sample back to the sample jar and discard the 16oz wide mouth jar.

14.3.3 Mark aluminum dish on bottom with the last three digits of the STL vial number. Weigh and record the weight of the dish.

14.3.4 Weigh 5 – 9 grams of recently homogenized sample into the dish and record the combined sample plus dish weight. Place in the dry weight oven for at least four hours prior to dry weight determination.

14.3.5 Add granular sodium sulfate to the 2g sample and blend with a spatula or disposable tongue depressor until the sample is free flowing.

14.3.6 If the sample is excessively wet or needs to be decanted prior to homogenization, add the sodium sulfate to the sample and let the sample sit for ten minutes. This time allows the sodium sulfate to absorb the water from the sample, however it will also harden the sample. After the sample sits, it will be necessary to break up the sample with the spatula until a free flowing consistency is again achieved.

14.3.6 Add surrogate to the samples using the appropriate surrogate (See Table 2) as designated on the batch sheet. Write an "X" on the label after adding the surrogate.

14.3.8 Add appropriate spike (See Table 2) to samples designated MS, MSD, MSB, MSBD. (The spike code to be used appears on the preparation batch logbook sheets.) Circle the "X" after adding the appropriate spike.

14.3.9 For blank samples (MSB, MSBD, or MBLK), approximately 2 g of sodium sulfate will be used in lieu of soil and shall be taken through the entire analytical procedure.

14.3.10 Add 10.0 mLs of hexane to the sample.

14.3.11 Before use, clean the sonication horns with DI water, acetone, and hexane. Wipe the horns thoroughly with paper towels after the DI water rinse.

14.3.12 Sonicate each sample once for 3 minutes on pulse mode of half power using a microtip sonicating horn.

14.3.13 Decant the sample into a 24mL vial that is pre labeled with the appropriate vial number. Add 10 mLs of concentrated H_2SO_4, cap and shake for 2 minutes. Allow the solvent layer to separate from the acid layer (at least 10 minutes) and remove an aliquot from the top layer, vial in a 2mL vial and relinquish to GC for analysis. Remove a second aliquot and save in the extra volume incubator. The remaining 9 mLs can now be disposed of by trained personnel.

14.3.14 Clean the sonicator horn between samples as describe in 14.3.11.

14.4 Wipe Extraction

14.4.1 Place entire sample into a labeled 16oz. wide-mouth jar.

14.4.2 Add anhydrous granular sodium sulfate.

14.4.3 Add 1ml of surrogate (See Table 2) appropriate to the final determinative procedure to the sample (the surrogate code to be used will be printed out on the preparation batch logbook sheets).

14.4.4 Add appropriate spike (See Table2) to samples designated MS, MSD, MSB, MSBD. (The spike code to be used appears on the preparation batch logbook sheets.)

14.4.5 For blank samples (MSB, MSBD, and MBLK), approximately 30g of sodium sulfate will be used in lieu of soil and shall be taken through the entire analytical procedure.

14.4.6 Add 100mls of hexane to the sample.

14.4.7 Fold an 18.5cm filter paper into quarters and place it in a stainless steel filter funnel.

14.4.8 Place this funnel in a labeled french square bottle with a napkin wrapped around the outside to prevent condensing water from entering the extract.

14.4.9 Before use, clean the sonication horns with DI water, acetone, hexane. Wipe the horns thoroughly with paper towels after the DI water rinse.

14.4.10 Place the 16oz. wide mouth jar under the sonication horn so it is submerged ½ inch. Ideally, the sonicator horn is to be submerged into the solvent ½ inch and still above the soil sample by the ½ inch.

14.4.11 Sonicate for 3 minutes at out put setting 10, pulsed mode, 50% duty cycle, using ¾ inch horn.

14.4.12 Pour off the solvent and transfer the wipe to the funnel. Rinse the sonicator jar with 10 – 20 mLs of hexane and transfer rinse also to the funnel.

14.4.13 After sample has drained, rinse down the funnel with 20-30mLs of the extraction solvent being used Allow the sample to drain thoroughly inside a fume hood.

14.4.14 Clean the sonicator horn between samples as described in 14.4.9.

14.4.15 For the wipes concentration procedure, follow 14.2.

15.0 CALCULATIONS N/A

16.0 METHOD PERFORMANCE

16.1 Acceptable performance is monitored through the use of Method Detection Limit Studies, as well as, recoveries of surrogate and spike compounds.

17.0 DATA ASSESSMENT AND ACCEPTANCE CRITERIA FOR QUALITY CONTROL MEASURES N/A

TITLE: METHOD 3550B: ULTRASONIC EXTRACTION OF SOILS AND WIPES

SUPERCEDES: Revision 10

18.0 CORRECTIVE ACTIONS FOR OUT-OF-CONTROL DATA

18.1 A job exception will be filed if any of the following occur.

18.1.1 Holding time is exceeded.
18.1.2 Insufficient sample volume.
18.1.3 Any matrix problems that prevent the extraction from being completed.

18.2 If at any time during the extraction, sample or extract is spilled, it must be determined if extra volume exists. If extra volume exists, the sample will immediately be prepped again. If there is no more volume, a comment must be included in the batch comments section, indicating at which point sample or extract was lost.

19.0 CONTINGENCIES FOR HANDLING OUT-OF-CONTROL OR UNACCEPTABLE DATA N/A

20.0 WASTE MANAGEMENT/ POLLUTION PREVENTION

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

20.1.1 Waste Hexane in vials. (Spent solvents are stored in red satellite "C" waste containers. When full, a designated laboratory technician will transfer solvent material from these satellite "C" waste containers to a grounded 55-gallon drum. These are located in the secured waste area and are disposed of according to all state and federal regulations.**).**

20.1.2 Waste Methylene Chloride. (Spent solvents are stored in red satellite "C" waste containers. When full, a designated laboratory technician will transfer solvent material from these satellite "C" waste containers to a grounded 55-gallon drum. These are located in the secured waste area and are disposed of according to all state and federal**).**

20.1.3 Waste solid material from the extraction process. (Solid wastes are separated into 5-gallon satellite containers. Lab generated solid wastes (extracted solid waste) are marked as "BC waste" and extra solid sample volumes (dry weights and other unextracted solid waste) are marked as "BE waste". When full, a designated laboratory technician will transfer all of the lab generated solid waste into a 55-gallon drum.)

20.1.4 Used sodium sulfate and glass wool or filter paper contaminated with methylene chloride/acetone or acetone/hexane from the extract drying step. (Solid wastes are separated into 5-gallon satellite containers. Lab generated solid wastes (extracted solid waste) are marked as "BC waste" and extra solid sample volumes (dry weights and other unextracted solid waste) are marked as "BE waste". When

full, a designated laboratory technician will transfer all of the lab generated solid waste into a 55-gallon drum.**).**

20.1.5 Assorted flammable solvent waste from various glassware rinses. (Spent solvents are stored in red satellite "C" waste containers. When full, a designated laboratory technician will transfer solvent material from these satellite "C" waste containers to a grounded 55-gallon drum. These are located in the secured waste area and are disposed of according to all state and federal regulations**).**

20.1.6 Methylene chloride waste from various glassware rinses. (Spent solvents are stored in red satellite "C" waste containers. When full, a designated laboratory technician will transfer solvent material from these satellite "C" waste containers to a grounded 55-gallon drum. These are located in the secured waste area and are disposed of according to all state and federal regulations**).**

20.1.7 Miscellaneous disposable glassware contaminated with solvents and sample residue. (All disposable glassware contaminated with solvent is air dried inside an operational fume hood then disposed in the recycling receptacle**).**

20.2 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."

21 REFERENCE

21.1 USEPA Test Methods for Evaluating Solid Waste, Physical/Chemical Methods; SW-846, Third Edition; Revision 2, December 1996; Method 3550B.

21.2 USEPA Contract Laboratory Program, Statement of Work for Organics Analysis, Multi-Media, Multi-Concentration, OLMO4.2.

22 TABLES, DIAGRAMS, FLOWCHARTS AND VALIDATION DATA

22.2 Table 1: Organic prep worksheet

22.3 Table 2: Spike and Surrogate Reference Sheet

TITLE: METHOD 3550B: ULTRASONIC EXTRACTION OF SOILS AND WIPES

SUPERCEDES: Revision 10

23.0 CHANGES FROM PREVIOUS REVISION

23.1. Merged the SOP, ASP-CLPSONC-75, with this SOP. This is reflected in Sections 1.2, 7.3, 11.3, 14.1.10, 14.2.4, and 21.2.

23.2. Section 9.0, included designation of glassware as disposable where applicable.

23.3. Section 10.1.7, added DI water.

23.4. Added 13.1, top loader balance requirements.

23.5. Changed wording in sections 14.1.2, 14.1.4, 14.1.5, 14.1.10, 14.1.11, 14.1.21, 14.1.22, 14.4.7, and 14.4.14 for clarification.

23.6. Changed 14.3.10 to reflect the addition of 10mL of hexane rather than 9.0mL.

23.7. Added 14.1.13, 14.3.11, 14.3.14, and 14.4.9 to specify required clean-up procedure for the sonication horns.

SOP No.	Revision No.	Effective Date	Page
ASP-3550B-93	11	February 28, 2006	Page 15 of 22

TITLE: METHOD 3550B: ULTRASONIC EXTRACTION OF SOILS AND WIPES

SUPERCEDES: Revision 10

Table 1

STL Buffalo
Date: 08/28/2003
Time: 17:31:24

Organic Prep Log Book
(3550B) 8270 RUSH SOILS
A3B09714

Rept: AN0501

SURROGATE: A01
Expiration Date: 1-14-04
Prepared by: mm
Spiked by: MR 1cc 0.00 ul
Witnessed by: ____

MATRIX SPIKE: A55
Expiration Date: 12/23/03
Prepared by: mm
Spiked by: MR 1cc 0.00 ul
Witnessed by: ____

MeCl2: Y27E38
Acetone: ____
Hexane: ____
Na2So4: [illegible]
Conc.H2SO4: ____

Date Ext/Initials: 8/28/03 MR
Preconc Date/Initials: ____
Cleanup Date/Initials: ____ ____
SOLID EXTRACTIONS
Final Conc Date/Initials: 8/28/03 MR

Job Number	Sample ID	BT ID	Samp Type	Vial #	Test	Protoc	Method	Surr Code	Spike Code	Sample Weight (g)	Clean Up	Final Volume (ml)	Dish Wght	Comb Wet	Comb Dry	D*
A03-8302	A3830201	A	FS	AS30015699	8270STAR	SW8463	8270	A00001	____	30.98	____	1.0	1.27	5.34	4.56	N
A03-8302	A3830201MS	↓	MS	AS30015700	8270STAR	SW8463	8270	A00001	A00055	30.04	____	↓	____	____	____	↓
A03-8302	A3830201SD	✓	SD	AS30015701	8270STAR	SW8463	8270	A00001	A00055	30.15	____	____	____	____	____	____
A3B09714	A3B0971401	__	MSB	AS30015702	8270STAR	SW8463	8270	A00001	A00055	30.92	____	____	____	____	____	____
A3B09714	A3B0971402	__	MBLK	AS30015703	8270STAR	SW8463	8270	A00001	____	30.14	____	✓	____	____	____	✓

Comments: ____

D* = Decanted (Y/N)

KSA 9/1/03

TITLE: METHOD 3550B: ULTRASONIC EXTRACTION OF SOILS AND WIPES

SUPERCEDES: Revision 10

Table 2
SPIKE AND SURROGATE REFERENCE SHEET

SURROGATES

A001: 8270 BN/AP SURROGATE

Nitrobenzene-d5	100.00 ng/µL
2-Fluorobiphenyl	100.00 ng/µL
p-Terphenyl	100.00 ng/µL
Phenol-d5	150.00 ng/µL
2-Fluorophenol	150.00 ng/µL
2,4,6-Tribromophenol	150.00 ng/µL

A026: 625 SURROGATE

Nitrobenzene-d5	50.00 ng/µL
2-Fluorobiphenyl	50.00 ng/µL
p-Terphenyl	50.00 ng/µL
Phenol-d5	50.00 ng/µL
2-Fluorophenol	50.00 ng/µL
2,4,6-Tribromophenol	50.00 ng/µL

A027: 8015B DRO SURROGATE

o-Terphenyl	20.0 ng/µL

A028: CLP 3/90 SVOA SURROGATE

Nitrobenzene-d5	50.00 ng/µL
2-Fluorobiphenyl	50.00 ng/µL
p-Terphenyl	50.00 ng/µL
1,2-Dichlorobenzene	50.00 ng/µL
Phenol-d5	75.00 ng/µL
2-Fluorophenol	75.00 ng/µL
2,4,6-Tribromophenol	75.00 ng/µL
2-Chlorophenol-d4	75.00 ng/µL

A033: 8151 HERBICIDE SURROGATE

Dichlorophenyl Acetic Acid	5.00 ng/µL

A035: PCB, PESTICIDE SURROGATE

Tetrachloro-m-xylene	0.20 ng/µL
Decachlorobiphenyl	0.20 ng/µL

A093: CLP PEST/PCB SURROGATE

Tetrachloro-m-xylene	0.40 ng/µL
Decachlorobiphenyl	0.40 ng/µL

TITLE: METHOD 3550B: ULTRASONIC EXTRACTION OF SOILS AND WIPES

SUPERCEDES: Revision 10

A0148: 8270 LOW LEVEL SURROGATE
Use 100.0µL of A0026

A0151: 608, 8082 LOW LEVEL WATER SURROGATE
Use 100.0 µL of A0035

A0181 AND A0233: BASE ONLY 8270 SURROGATES
Use 1000.0 µL of A0001

A0277: BASE ONLY 8270 LOW LEVEL SURROGATE
Use 200.0 µL of A0026

SPIKES

A0047: 8151 HERBICIDE SPIKE

2,4-D	2.0 ng/µL
Dalapon	2.0 ng/µL
Dinoseb	2.0 ng/µL
Pentachlorophenol	2.0 ng/µL
Picloram	2.0 ng/µL
2,4,5-TP (Silvex)	2.0 ng/µL
2,4,5-T	2.0 ng/µL
2,4-DB	2.0 ng/µL
Dicamba	2.0 ng/µL
Dichloroprop	2.0 ng/µL

A0049: CLP PEST/PCB SPIKE

gamma-BHC (Lindane)	0.5 ng/µL
Heptachlor	0.5 ng/µL
Aldrin	0.5 ng/µL
Dieldrin	1.0 ng/µL
Endrin	1.0 ng/µL
4,4’-DDT	1.0 ng/µL

A0051: 8081 PESTICIDE SPIKE

gamma-BHC (Lindane)	1.0 ng/µL
alpha-BHC	1.0 ng/µL
Heptachlor	1.0 ng/µL
Aldrin	1.0 ng/µL
Beta-BHC	1.0 ng/µL
Dieldrin	1.0 ng/µL
Endrin	1.0 ng/µL
4,4’-DDD	1.0 ng/µL
4,4’-DDT	1.0 ng/µL

4,4'-DDE	1.0 ng/µL
Endosulfan I	1.0 ng/µL
Endosulfan II	1.0 ng/µL
Endrin Aldehyde	1.0 ng/µL
Endosulfan Sulfate	1.0 ng/µL
Heptachlor epoxide	1.0 ng/µL
Methoxychlor	1.0 ng/µL
Endrin Ketone	1.0 ng/µL

A0055: 8270 BN/AP SPIKE

Phenol	100.0 ng/µL
2-Chlorophenol	100.0 ng/µL
1,4-Dichlorobenzene	100.0 ng/µL
N-Nitroso-Di-n-propylamine	100.0 ng/µL
1,2,4-Trichlorobenzene	100.0 ng/µL
4-Chloro-3-methylphenol	100.0 ng/µL
Acenaphthene	100.0 ng/µL
4-Nitrophenol	100.0 ng/µL
2,4-Dinitrotoluene	100.0 ng/µL
Pentachlorophenol	100.0 ng/µL
Pyrene	100.0 ng/µL

A0056: 625 SPIKE

Use 500.0 µL of A0193

A0057: CLP 3/90 SVOA SPIKE

Phenol	75.0 ng/µL
2-Chlorophenol	75.0 ng/µL
1,4-Dichlorobenzene	50.0 ng/µL
N-Nitroso-Di-n-propylamine	50.0 ng/µL
1,2,4-Trichlorobenzene	50.0 ng/µL
4-Chloro-3-methylphenol	75.0 ng/µL
Acenaphthene	50.0 ng/µL
4-Nitrophenol	75.0 ng/µL
2,4-Dinitrotoluene	50.0 ng/µL
Pentachlorophenol	75.0 ng/µL
Pyrene	50.0 ng/µL

A0060: CUSTOM CHLOROPYRIDINES SPIKE

2-Chloropyridine	100.0 ng/µL
3-Chloropyridine	100.0 ng/µL
2,6-Dichloropyridine	100.0 ng/µL
p-Fluoroaniline	100.0 ng/µL

TITLE: METHOD 3550B: ULTRASONIC EXTRACTION OF SOILS AND WIPES

SUPERCEDES: Revision 10

A0061: CECOS CONSENT 1.6 BN/AP SPIKE

1,4-Dichlorobenzene	50.0 ng/µL
N,N'-Dimethylacetamide	50.0 ng/µL
Methylaniline N.O.S.	50.0 ng/µL
Pyridine	50.0 ng/µL

A0062: 8270 TCLP SPIKE

1,4-Dihlorobenzene	100.0 ng/µL
2,4-Dinitrotoluene	100.0 ng/µL
Hexachlorobenzene	100.0 ng/µL
Hexachlorobutadiene	100.0 ng/µL
Hexachloroethane	100.0 ng/µL
2-Methylphenol	100.0 ng/µL
3-Methylphenol	200.0 ng/µL
4-Methylphenol	200.0 ng/µL
Nitrobenzene	100.0 ng/µL
Pentachlorophenol	100.0 ng/µL
Pyridine	100.0 ng/µL
2,4,5-Trichlorophenol	100.0 ng/µL
2,4,6-Trichlorophenol	100.0 ng/µL

A0095: DIESEL FUEL #2 DRO AND PETRO SPIKE

Diesel Fuel #2	1500.0 ng/µL

A0113: DECHLORANE PLUS

Dechlorane Plus	1.0 ng/µL

A0143: 8082 PCB SPIKE (USE 100.0µL)

Aroclor 1254	50.0 ng/µL

A0147: 8270 LOW LEVEL SPIKE

Use 100.0 µL of A0055

A00152: 608 PESTICIDE AND PESTICIDE/PCB SPIKE

Use 50.0 µL A0051

A00153: 608 PCB SPIKE

Use 100.0 µL of A0213

A00184: DRO 10 COMPONENT SPIKE

Diesel Range Organics	200.0 ng/µL

TITLE: METHOD 3550B: ULTRASONIC EXTRACTION OF SOILS AND WIPES

SUPERCEDES: Revision 10

A00193: 8270 FULL LIST SPIKE

Acenaphthene	100.0 ng/µL
Aniline	100.0 ng/µL
Acenaphthylene	100.0 ng/µL
Anthracene	100.0 ng/µL
Benzo(a)anthracene	100.0 ng/µL
Benzo(b)fluoranthene	100.0 ng/µL
Benzo(k)fluoranthene	100.0 ng/µL
Benzo(ghi)perylene	100.0 ng/µL
Benzo(a)pyrene	100.0 ng/µL
Benzoic Acid	250.0 ng/µL
Benzyl alcohol	100.0 ng/µL
Bis(2-chloroethoxy) methane	100.0 ng/µL
Bis(2-chloroethyl) ether	100.0 ng/µL
2,2'-Oxybis(1-Chloropropane)	100.0 ng/µL
Bis(2-ethylhexyl)phthalate	100.0 ng/µL
4-Bromophenyl phenyl ether	100.0 ng/µL
Butyl benzyl phthalate	100.0 ng/µL
4-Chloroaniline	100.0 ng/µL
4-Chloro-3-methylphenol	100.0 ng/µL
2-Chloronaphthalene	100.0 ng/µL
2-Chlorophenol	100.0 ng/µL
4-Chlorophenyl phenyl ether	100.0 ng/µL
Chrysene	100.0 ng/µL
Dibenzo(a,h)anthracene	100.0 ng/µL
Dibenzofuran	100.0 ng/µL
Di-n-butyl phthalate	100.0 ng/µL
1,2-Dichlorobenzene	100.0 ng/µL
1,3-Dichlorobenzene	100.0 ng/µL
1,4-Dichlorobenzene	100.0 ng/µL
3,3'-Dichlorobenzidine	100.0 ng/µL
2,4'-Dichlorophenol	100.0 ng/µL
Diethyl phthalate	100.0 ng/µL
2,4-Dimethylphenol	100.0 ng/µL
Dimethyl phthalate	100.0 ng/µL
4,6-Dinitro-2-methylphenol	100.0 ng/µL
2,4-Dinitrophenol	100.0 ng/µL
2,4-Dinitrotoluene	100.0 ng/µL
2,6-Dinitrotoluene	100.0 ng/µL
Di-n-octyl phthalate	100.0 ng/µL
Fluoranthene	100.0 ng/µL
Fluorene	100.0 ng/µL
Hexachlorobenzene	100.0 ng/µL

Hexachlorobutadiene	100.0 ng/µL
Hexachlorocyclopentadiene	100.0 ng/µL
Hexachloroethane	100.0 ng/µL
Indeno(1,2,3-cd)pyrene	100.0 ng/µL
Isophorone	100.0 ng/µL
2-Methylnaphthalene	100.0 ng/µL
2-Methylphenol	100.0 ng/µL
4-Methylphenol	100.0 ng/µL
Naphthalene	100.0 ng/µL
2-Nitroaniline	100.0 ng/µL
3-Nitroaniline	100.0 ng/µL
4-Nitroaniline	100.0 ng/µL
Nitrobenzene	100.0 ng/µL
2-Nitrophenol	100.0 ng/µL
4-Nitrophenol	100.0 ng/µL
N-Nitrosodiphenylamine	100.0 ng/µL
N-Nitroso-Di-n-prpoylamine	100.0 ng/µL
Pentachlorophenol	100.0 ng/µL
Phenanthrene	100.0 ng/µL
Phenol	100.0 ng/µL
Pyrene	100.0 ng/µL
1,2,4-Trichlorobenzene	100.0 ng/µL
2,4,5-Trichlorophenol	100.0 ng/µL
2,4,6-Trichlorophenol	100.0 ng/µL

A0213: 8082 PCB SPIKE

Aroclor 1242	5.0 ng/µL

A0222: 8082 PCB SPIKE

Aroclor 1016	5.0 ng/µL
Aroclor 1260	5.0 ng/µL

A0251: AFCEE 8081 PESTICIDE SPIKE

gamma-BHC (Lindane)	1.0 ng/µL
alpha-BHC	1.0 ng/µL
Heptachlor	1.0 ng/µL
Aldrin	1.0 ng/µL
Beta-BHC	1.0 ng/µL
Dieldrin	1.0 ng/µL
Endrin	1.0 ng/µL
4,4'-DDD	1.0 ng/µL
4,4'-DDT	1.0 ng/µL
4,4'-DDE	1.0 ng/µL

Endosulfan I	1.0 ng/µL
Endosulfan II	1.0 ng/µL
Endrin Aldehyde	1.0 ng/µL
Endosulfan Sulfate	1.0 ng/µL
Heptachlor epoxide	1.0 ng/µL
Methoxychlor	1.0 ng/µL
Endrin Ketone	1.0 ng/µL
alpha-Chlordane	1.0 ng/µL
gamma-Chlordane	1.0 ng/µL

A0234: 8270 1,4-DIOXANE ONLY SPIKE

1,4-Dioxane	100.0 ng/µL

A0251: 8082 LOW LEVEL WATER PCB SPIKE (USE 100.0µL)

Aroclor 1254	5.0 ng/µL

A0281: 8082 LOW LEVEL WATER PCB SPIKE

Use 100.0 µL of A0222

STL Buffalo
LABORATORY STANDARD OPERATING PROCEDURES

SOP No.	Revision No.	Effective Date	Page
AWC-1010-46	7	December 12, 2005	1 of 9

TITLE: METHOD 1010 – FLASH POINT

Supersedes: Revision 6

REVIEWED AND APPROVED BY:	SIGNATURE	DATE
Verl D. Preston, Quality Manager		
Christopher Spencer, Laboratory Director		
Peggy Gray-Erdmann, Supervisor		

0.0 IDENTIFICATION OF TEST METHOD

1.1 SW 846 3rd edition, Method 1010 Flash point and ASTM D93-80

0.0 APPLICABLE MATRIX

2.1 These methods determine the flash point of fuels, oils, suspension of solids, liquids including those that tend to form a surface film under test conditions and other liquids of similar viscosity.

0.0 REPORTING LIMIT

3.1 If no flash occurs record the result as ">200°F".

0.0 SCOPE AND APPLICATION

0.0 These test methods cover the determination of flash point by Pensky-Martens closed cup tester to determine the flash point of all types of liquid and soil samples.

4.2 Flash point measures tendency of the sample to form a flammable mixture with air under controlled laboratory conditions. It is only one of a number of properties, which must be considered in assessing the overall flammability hazard of a material.

4.3 Flash point is used in shipping and safety regulations to define flammable and combustible materials. One should consult the particular regulation involved for precise definitions of classes.

TITLE: METHOD 1010 – FLASH POINT

Supersedes: Revision 6

4.4 Flash point can indicate the possible presence of highly volatile and flammable materials in a relatively nonvolatile or nonflammable material.

5.0 SUMMARY OF THE TEST METHOD

5.1 Sample is heated at a slow, constant rate with continual stirring if sample is aqueous. A small flame is directed into the sample cup at regular intervals with simultaneous interruption of stirring if applicable. The flash point is the lowest temperature at which application of the test flame ignites the vapor above the sample. The sample is deemed to have flashed when a large flame appears and instantaneously propagates itself over the surface of the sample. Occasionally, the application of the test flame will cause a blue halo or an enlarged flame. This generally occurs near the actual flash point but in some cases, especially with halogenated hydrocarbons and admixtures, can occur at any temperature. These phenomena are not to be considered true flash points.

6.0 DEFINITIONS

6.1 Flash point- the lowest temperature corrected to a barometric pressure, at which application of a test flame causes the vapor of a specimen to ignite under specified conditions of test.

5.1 Standard definitions are found in section 3.0 in the Laboratory Quality Manual.

6.0 INTERFERENCES

6.0 Low boiling oils or neat liquids may boil over at temperatures below the 200 degree Fahrenheit limit. If such a sample fails to flash below its boiling point, a result of "< (b.p. Temperature)" may be assigned to the sample.

8.0 SAFETY

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

8.1 SPECIFIC SAFETY CONCERNS OR REQUIREMENTS

In the event a sample ignites in the test apparatus do not attempt to remove the sample. Turn off the apparatus and flame. The flame should go out when the cup is closed. If this does not happen the flame may be extinguished by covering the sample with a non-flammable material. After the apparatus has cooled the sample may be removed.

8.2 PRIMARY MATERIALS USED

The following is a list of the materials used in this method, which have a serious or significant hazard rating. **NOTE: This list does not include all materials used in the method. The table contains a summary of the primary hazards listed in the MSDS for each of the materials listed in the table.** A complete list of materials used in the method can be found in the reagents

and materials section. Employees must review the information in the MSDS for each material before using it for the first time or when there are major changes to the MSDS.

Material (1)	Hazards	Exposure Limit (2)	Signs and symptoms of exposure
Methylene Chloride	Carcinogen Irritant	25 ppm-TWA 125 ppm-STEL	Causes irritation to respiratory tract. Has a strong narcotic effect with symptoms of mental confusion, light-headedness, fatigue, nausea, vomiting and headache. Causes irritation, redness and pain to the skin and eyes. Prolonged contact can cause burns. Liquid degreases the skin. May be absorbed through skin.
Xylene	Flammable Irritant	100 ppm-TWA	Inhalation of vapors may be irritating to the nose and throat. Inhalation of high concentrations may result in nausea, vomiting, headache, ringing in the ears, and severe breathing difficulties, which may be delayed in onset. High vapor concentrations are anesthetic and central nervous system depressants. Skin contact results in loss of natural oils and often results in a characteristic dermatitis. May be absorbed through the skin. Vapors cause eye irritation. Splashes cause severe irritation, possible corneal burns and eye damage.
1 – Always add acid to water to prevent violent reactions.			
2 – Exposure limit refers to the OSHA regulatory exposure limit.			

7.2 The test must be performed under a hood that is free of clutter.

7.2 A suitable means of fire suppression (fire extinguisher and/ or blanket) must be in the immediate vicinity of the test.

7.2 The sample cup (brass) must be cooled to ambient laboratory temperature between each sample analysis.

7.2 Suspected flammable materials (samples) or those of unknown chemical composition must be initially pre-screened for flammability (if you are unsure, the default is to prescreen).

7.2 Take appropriate safety precautions during the initial application of the test flame, since samples containing low-flash material can give an abnormally strong flash when the test flame is first applied.

9.0 EQUIPMENT AND SUPPLIES

9.1 Pensky-Martens closed cup flash tester

9.2 Propane and lighter

9.3 Thermometer: Pensky-Martens low range thermometer having a range of 20 to 230°F

8.3 Stirrer motor, calibrated annually

TITLE: METHOD 1010 – FLASH POINT

Supersedes: Revision 6

10.0 REAGENTS AND STANDARDS

10.1 p-Xylene; Flash point = 81 ± 2°F (27.2 + 1.1°C)

11.0 SAMPLE COLLECTION, PRESERVATION, AND STORAGE

11.1 Samples are to be preserved by cooling to 4 ±2°C and stored in glass containers. Do not store samples in plastic container, since volatile material may diffuse through the walls of the enclosure.

11.2 Erroneously high flash points may be obtained if precautions are not taken to avoid the loss of volatile material. Do not open containers unnecessarily and make a transfer unless the sample temperature is at least the equivalent of 18°F below the expected flash point. Do not use samples from leaky containers for these test methods.

12.0 QUALITY CONTROL

12.1 Begin and end each run of 20 samples or fewer with analysis of p-Xylene LCS. The obtained value for p-Xylene should be within 79-83°F.

12.2 Run at least one duplicate every 20 samples or fewer. The relative percent difference between duplicate analyses should be <10%.

12.3 All samples exhibiting a Flash point must be run in duplicate.

13.0 CALIBRATION AND STANDARDIZATION

13.1 Determine flash point of p-Xylene as described in section 11.0. A value of 81 ± 2°F should be obtained.

14.0 PROCEDURE

14.1 Thoroughly clean and dry all parts of the cup and its accessories before starting the test, being sure to remove any solvent.

14.2 Obtain a reading of the barometric pressure in mm/Hg. Record in the excel spreadsheet located in the (F) drive under F: LabNY /Share/Wet Chemistry Spreadsheets/ Flashpoint and save the spreadsheet by date.

14.3 Set up Pensky-Martens closed cup flash tester in the hood and allow propane flame to stabilize.

14.4 Fill tester cup to the fill line with sample, set in the stove, secure the lid, and insert thermometer. Sample should initially be at a temperature of approximately 60°F or 20°F lower than the estimated flash point, whichever is lower. Record the temperature in the logbook.

14.5 Adjust the flame to 4mm in diameter. Supply the heat at such a rate that the temperature as indicated by the thermometer increases 2 to 3°F /min. Turn the stirrer 90 to 120 rpm, stirring in a downward direction if the sample is aqueous.

14.6 Apply the test flame by operating the mechanism on the cover that controls the shutter and test flame burner so that the flame is lowered into the vapor space of the cup. Leave in its lowered position for 1 second and quickly raise it to its high position. Do not stir the sample while applying the test flame.

14.7 Record the temperature at the time the test flame application causes a distinct flash in the interior of the cup. Do not confuse the true flash with the bluish halo that sometimes surrounds the test flame at applications preceding the one that causes the actual flash. If flash occurs, analyze a duplicate of the sample to confirm flash. If no flash occurred, record the result as ">200°F".

15.0 CALCULATION

15.1 Observe and record the ambient barometric pressure at the time of the test. When the pressure differs from 101.3kPa (760 mm Hg), correct the flash point as follows:

Corrected flash point = F + 0.06 (760-P)

Where F = uncorrected flash points, °F
P = ambient barometric pressure, mm/Hg

14.1 Record the corrected flash point to the nearest 1°F.

15.3 The barometric pressure used in this calculation is the ambient pressure of the laboratory at the time of the test. This barometer is not pre corrected for sea level, which is verified by the manufacturer.

15.4 Relative Percent Difference (RPD):

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

where:

x_1 = analytical % recovery
x_2 = replicate % recovery

16.0 METHOD PERFORMANCE

15.0. A one–time initial demonstration of performance must be generated.

15.0. Training Qualifications

TITLE: METHOD 1010 – FLASH POINT

Supersedes: Revision 6

15.0.0. The supervisor has the responsibility to ensure that an analyst who has been properly trained in its use and has the required experience performs this procedure.

15.0.0. The following analyst validation information is maintained for this method in the laboratory QA files.

15.0.0.0. The analyst must read and understand this SOP.

15.0.0.0. The analyst must complete a DOC or successfully analyze PT samples annually.

15.0.0.0. The analyst must complete the STL Quality Assurance Training.

17.0 DATA ASSESSMENT AND ACCEPTANCE CRITERIA FOR QUALITY CONTROL MEASURES

16.0 LCS: p-Xylene must be 81 $\pm$ 2° F

16.0 Duplicate: RPD $\leq$ 10%

16.0 Any sample that produces a flash, must be analyzed a second time.

18.0 CORRECTIVE ACTIONS FOR OUT OF CONTROL DATA

18.1 If LCS is not between 79° F - 83°F, rerun samples.

17.1 If RPD is > 10%, rerun samples.

19.0 CONTINGENCIES FOR HANDLING OUT-OF-CONTROL OR UNACCEPTABLE DATA

19.1 A Job Exception Form must be completed and filed with the Project Manager and QA Manager for any of the following conditions:

18.0.0. Holding times exceeded

18.0.0. Insufficient sample volume for re-analysis

20.0 WASTE MANAGEMENT / 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.”

TITLE: METHOD 1010 – FLASH POINT

Supersedes: Revision 6

20.1 Waste Streams Produced by the Method

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

- Aqueous acidic samples are discarded in the "A" waste containers.
- Aqueous alkaline samples are discarded in the "D" waste containers.
- Excess flammable samples and waste solvents are discarded in the "C" waste containers.
- All soil samples are discarded in the soil collection tray for later disposal.

21.0 REFERENCES

21.1 D93-80 Test Methods for Flash points by Pensky-Martens closed tester, American Society for Testing and Materials. 1916 Race St., Philadelphia, PA 19103, 04.99, 1986.

21.2 Method 1010, "Pensky-Martens Closed Cup Method for Determining Ignitability", Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, Final Update II, September 1994.

22.0 TABLES, DIAGRAMS, FLOWCHARTS AND VALIDATION DATA

22.1 Analytical Sequence

22.2 Analytical Batch

22.3 Wet Chemistry Batch Summary

23.0 CHANGES FROM PREVIOUS REVISION

23.1 Combined SOP #880 'Ignitability of Soils' with #118 'Ignitability of Liquids'. Archived SOP #880

22.1 Analytical Sequence

LCS
SAMPLE
SAMPLE
SAMPLE
SAMPLE
SAMPLE
SAMPLE
SAMPLE
SAMPLE
SAMPLE
SAMPLE
SAMPLE
SAMPLE
SAMPLE

TITLE: METHOD 1010 – FLASH POINT

Supersedes: Revision 6

SAMPLE
SAMPLE
SAMPLE
SAMPLE
SAMPLE
SAMPLE
SAMPLE
SAMPLE DUP
LCS

21.1 Analytical Batch

STL Buffalo

Laboratory Bench Sheet
Flashpoint
Method 1010

Flashpoint_Template
Revision 1
September, 2005

Analyst:	SH	BATCH #	A5B13426		
Date:	9/1/2005	p-Xylene	CHA-101-J		
		Acceptance Range=	79-83 °F		
Barometer Instrument ID	25548	Thermometer ID:	61091-001		
Pressure in inHg from Barometer		29.30			

Job#	Sample ID	Start Temp	Uncorrected Flashpoint	Ambient Barometric Pressure	Corrected Flashpoint	Comment
		°F	°F	mm Hg	°F	
LCS	P-XYLENE	65.0	80.00	744.22	80.9	
9517	1	60	>200	744.22	#VALUE!	
9479	1	61	>200	744.22	#VALUE!	
9475	1	65	>200	744.22	#VALUE!	
	1 MD	70	>200	744.22	#VALUE!	
LCS	P-XYLENE	65	80.0000	744.22	80.9	
				744.22	0.9	
				744.22	0.9	
				744.22	0.9	
				744.22	0.9	
				744.22	0.9	
				744.22	0.9	
				744.22	0.9	
				744.22	0.9	
				744.22	0.9	
				744.22	0.9	
				744.22	0.9	
				744.22	0.9	
				744.22	0.9	
				744.22	0.9	

Page 1 of 2

TITLE: METHOD 1010 – FLASH POINT

Supersedes: Revision 6

15.3 Wet Chemistry Batch Summary

WET CHEMISTRY BATCH SUMMARY

PARAMETER_____ **METHOD**_____ **BATCH** _____

COMMENTS	JOB NUMBER
WC Reporting Limit < STL Quant Limit	
WC Historical confirms within Hold Time	
WC Historical NO confirm & RE outside of HT	
WC Hold Time Exceedance-Dilution required	
WC Hold Time Exceedance-Instrument Failure	
WC Holding Time Exceedance by Date	
WC Holding Time Exceedance by Hours	
WC LCS within ERA limits outside internal	
WC LCS high recovery, sample ND	
WC MBLK hit but samples > 10X blank value	
WC RPD Exceedance for MS / SD	
WC Spike Failure HIGH MS only	
WC Spike Failure LOW MS only	
WC Spike Failure MS and SD	
WC BOD HT met- Oxygen depleted-RE out HT	
WC Carbonate Alkalinity, LCS/MBLK	
WC Reactivity Qualification	
WC TDS/Conductivity ratio outside of range	
WC TOX Breakthrough- no volume for redo	
WC TOX samples were centrifuged	
Other	

DILUTION CODES	REASON
002	Sample matrix effects
003	Excessive foaming
004	High levels of non-target compounds
008	High concentration of target analytes
009	Sample turbidity
010	Sample color
011	Insufficient volume for lower dilution
012	Sample viscosity
013	other

ICAL Compliant? YES NO NA IF NO, Why? _____
LCS/CCV Compliant? YES NO NA IF NO, Why? _____
CCB Compliant? YES NO NA IF NO, Why? _____
RPD Compliant? YES NO NA IF NO, Why? _____
ERA Compliant? YES NO NA IF NO, Why? _____

NUMBER of REANALYSIS FOR THIS BATCH:_____

Analyst_____ Date_____

Time Critical Batch Review_____ Date_____

Secondary Review & Closure_____ Date_____ WC Summary Rev 4 / 5-2005

STL Buffalo
LABORATORY STANDARD OPERATING PROCEDURES

SOP No.	Revision No.	Effective Date	Page
AWC-310.2-18	4	February 26, 2007	1 of 16

TITLE: ALKALINITY Method 310.2 (Colorimetric, Automated)

SUPERCEDES: Revision 3

REVIEWED & APPROVED BY:	SIGNATURE	DATE
Verl D. Preston, Quality Manager		
Christopher Spencer, Laboratory Director		
Peggy Gray-Erdmann, Supervisor		

1.0 IDENTIFICATION OF TEST METHOD

1.1 This method is taken from EPA Method 310.2.

2.0 APPLICABLE MATRIX

2.1 This method is applicable to drinking, surface, and saline waters, domestic and industrial wastes.

2.2 Soil samples may also be analyzed using this method after the ASTM Leaching procedure has been completed.

3.0 REPORTING LIMIT

3.1 The reporting limit has been determined to be 10.0 mg/L.

4.0 SCOPE AND APPLICATION

4.1 The range of the curve is 10 - 100 mg/L. Samples greater then 100mg/l will require dilutions.

TITLE: ALKALINITY Method 310.2 (Colorimetric, Automated)

SUPERCEDES: Revision 3

5.0 SUMMARY OF THE TEST METHOD

5.1 Methyl orange is used as a color reagent for this method because its pH range is the same as the pH of the equivalence point for the total alkalinity titration. The methyl orange indicator is in a dilute pH 3.1 buffer which is just below its color change pH. When an alkaline sample is injected, the poorly buffered methyl orange changes color in proportion to the alkalinity of the sample.

6.0 DEFINITIONS

6.1 LCS: Laboratory Control Sample

6.2 Standard definitions are used in this document as defined by the STL Corporate Quality Assurance Plan.

6.3 ICV/ICB: Initial Calibration Verification/Blank

6.4 Konelab: Automated multi-Chemistry Analyzer

7.0 INTERFERENCES

7.1 Turbidity and color will interfere. Turbidity can be removed by filtration.

7.2 The pH of samples is tested prior to analysis. Samples over a pH of 8.3 are run titrametrically (method 310.1).

8.0 SAFETY

8.1 The toxicity or carcinogenicity of each reagent used in this method has not been fully established. Each chemical should be regarded as a potential health hazard and exposure should be as low as reasonably achievable. Cautions are included for known extremely hazardous materials.

8.2 The following chemicals have the potential to be highly toxic or hazardous; for detailed explanations consult the MSDS.

8.2.1 Hydrochloric Acid
8.3.2 Methyl Orange

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

TITLE: ALKALINITY Method 310.2 (Colorimetric, Automated)

SUPERCEDES: Revision 3

8.4 SPECIFIC SAFETY CONCERNS OR REQUIREMENTS

There are no specialized safety concerns associated with this method.

8.4.1 PRIMARY MATERIALS USED

There are no materials used in this method that have a serious or significant hazard rating. **NOTE: This list does not include all materials used in the method.** 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.

9.0 EQUIPMENT AND SUPPLIES

9.1 Analytical Balance capable of accurately weighing to the nearest 0.0001g.

9.2 Glassware- Class A volumetric flasks and pipettes or plastic containers as required. Samples may be stored in plastic or glass.

9.3 pH strips measuring from 6.5 to 9.0.

9.7 Konelab

10.0 REAGENTS AND STANDARDS

10.1 *PREPARATION OF REAGENTS FOR THE KONELAB*

10.1.1 **Methyl Orange Solution**: Dissolve 0.0125 g Methyl Orange in 100ml of carbon dioxide free di water

10.1.2 **PH 3.1 Buffer solution:** Dissolve 0.51047 of Potassium acid phthalate in carbon dioxide free di water. Add 8.76ml of 0.1N HCl and dilute to 100ml with carbon dioxide free di water. Pour into a glass storage bottle and prepare fresh weekly.

10.1.3 **Methyl Orange Buffered Indicator:** Add 25 mls of the pH 3.1 Buffer with 5 mls of the Methyl Orange Solution. Mix well. Pour into a glass storage bottle and prepare fresh daily.

10.2 PREPARATION OF STANDARDS FOR THE KONELAB

10.2.1 **Sodium Carbonate Primary STD, 1000 ppm:** dissolve 0.1060 g of anhydrous Sodium carbonate (oven dried at 250°C for 4 hours) in carbon dioxide free di water. Dilute to 100ml. 1.0ml=1.0 mg NaC03. A pre made Sodium Carbonate Primary STD, 1000 ppm can also be used.

TITLE: ALKALINITY Method 310.2 (Colorimetric, Automated)

SUPERCEDES: Revision 3

10.2.2 **Sodium Carbonate 100ppm Std:** Add 5.0 ml of the 1000ppm sodium Carbonate Std (10.4.1) and dilute to 50 mls using carbon dioxide free di water. Alkalinity curve is set up in series. A 100ppm standard is placed in the Kone and a series of curve points are diluted from the 100ppm standard. The curve consists of the following points: 100ppm, 50ppm, 25ppm, 10ppm and 5ppm. The alkalinity curve on the Kone must be done at a minimum of once every three months.

10.2.3 **Sodium Carbonate 50ppm Std:** Add 5 mls of a 1000ppm sodium carbonate from a separate source (other than the curve) to 100 mls of carbon dioxide free di water.

10.2.4 **Sodium Carbonate 20 ppm Spike:** Add 100ug of a 1000 ppm sodium carbonate std to 5 ml of sample, mix and analyze.

11.0 SAMPLE COLLECTION, PRESERVATION, SHIPMENT AND STORAGE

11.1 Samples should be collected in plastic or glass bottles. All bottles must be thoroughly cleaned and rinsed with reagent water. The volume collected should be sufficient to ensure a representative sample, allow for replicate analysis (if required), and minimize waste disposal.

11.2 Samples should be refrigerated at 4ºC and determined as soon as practical. Do not open sample bottle before analysis, The maximum holding time is 14 days.

12.0 QUALITY CONTROL

12.1 ICAL: A calibration curve must be analyzed once every three months at a minimum. Acceptance criteria for the calibration curve is a correlation coefficient (R value) $\geq$0.990.

12.2 Initial Calibration Verification (ICV): (ppm) The ICV must be prepared from a separate source from the calibration curve and must be analyzed immediately after the curve. Obtained values must be $\pm$ 10% of the true value.

12.3 Initial Calibration Blank (ICB): The ICV must be analyzed immediately after the curve. The ICB must exhibit values less than the STL Buffalo Quantitation limit.

12.4 Laboratory Control Sample (LCS): (ppm) The LCS must be analyzed at the beginning and end of the analytical procedure and after every ten samples. Obtained values of the LCS must be $\pm$ 10% of the true value.

12.5 Method Blanks: To determine freedom from contamination, Method Blanks are prepared at the beginning of the analytical procedure as well as after every ten samples and at the end of the analytical procedure. The Method Blank goes through the same treatment as the samples and standards. The Method Blank must exhibit values less than the STL Buffalo Quantitation limit.

12.5.1 All blanks associated with DOD QSM and AFCEE samples should be less than half of the reporting limit.

TITLE: ALKALINITY Method 310.2 (Colorimetric, Automated)

SUPERCEDES: Revision 3

12.6 Sample Duplicate (MD): Sample duplicates should be analyzed at least once for every group of twenty or fewer samples. Samples should agree within 20% RPD. Samples that fail to meet these criteria should be reanalyzed.

12.7 Sample Spikes (MS): Sample spikes should be analyzed at least once for every group of twenty or fewer samples.

13.0 CALIBRATION AND STANDARDIZATION

13.1 Prepare a series of standards, covering the desired range and a blank by diluting suitable volumes of standard solutions.

13.2 Calibrate Kone as described in Section 14.1.5.

13.3 Prepare the standard calibration curve by plotting instrument response against concentration values. The curve for the Konelab is a linear (1^{st} Order) curve. The calibration curve may be fitted to the calibration solution concentration/response data using the computer. Acceptance or control limits should be established using the difference between the measured values of the calibration solution and the "true value" concentration. Acceptance criteria for the calibration curve is a correlation coefficient (R value) $\geq$0.990.

13.4 After the calibration has been established, it must be verified by the analysis of a second source standard (ICV). If measurements exceed +/- 10% of the established ICV value, the analysis should be terminated and the instrument recalibrated. The new calibration must be verified before continuing analysis.

13.5 A Calibration curve must be done at a minimum of once every three months or sooner on the Kone.

14.0 PROCEDURE

14.1 PROCEDURE FOR KONELAB

14.1.1 **Start up procedure every day:** Turn on the Konelab first then switch the PC on. Enter the software by pressing Ctr, Alt and Delete. Log onto the software when prompted. The Username is **administrator** and the Password is **Klab1sUPER.** Wait until the yellow banner appears on the screen stating "start up". Press F1 on the Konelab main screen. Press OK when prompted to perform operation. The analyzer powers up the lamp and chopper motor, and perform a washing function. Start up takes approximately three minutes. After the start up, the main screen will display a "ready" flag.

14.1.2 **Checking the Water Blank**: Each day it is necessary to check the quality of the water blank. This is done after the start up procedure. From the main screen, select F8 [more], then F2 [Instrument Actions]. Select F8 [more] again, then F1 [check water blank]. When complete 9 approximately three

minutes), you will be given a graphical representation of the water blank. Make sure that all results are within +/-2mA (milliabsorbance). If not rerun this procedure.

14.1.3 **To load the reagents:** to load the reagents on the instrument using the provided 20 ml plastic reagent vials. Click on REAGENTS at the top of the screen. Click on INSERT REAGENT. Then choose your first reagent and click OK. The instrument will prompt you when to insert the reagent. Follow this procedure for each reagent.

14.1.4 **To load a new calibration**: Click on SAMPLES, then F8 MORE, then SAMPLE SEGMENT. When a template appears on the screen, choose what tray you want to use, then choose from the pick list the appropriate standard(s) and ICV/ICB you need. When this is done, physically using that same number tray and the 2 ml plastic sample cups, place your items in the tray. On the screen, click on INSERT SEGMENT and the instrument will prompt you when to place the tray on the instrument. There are 6 trays with 14 slots each that you can place on the instrument at one time. Also, when selecting calibrators for your template, if you make a mistake, you must click on REMOVE ALL SAMPLES and start over.

14.1.5 **To run a new calibration:** Once the desired concentrations are inserted into the instrument and the template has been set up, you can then click on F6 CALIBRATION/QC SELECTION. Choose the parameters you are calibrating for. NOTE: you MUST choose the Sulfate Kinetic as well as Sulfate when calibrating for that parameter. Click F1 CALIBRATE, then click MAIN at the top right of the screen. Then press the green button on the keyboard. Your curves have begun being generated. It takes approximately 15 minutes to generate any given calibration. You can check time left by clicking F8 MORE twice and then PENDING REQUESTS whereas the time left will be posted at the top of the screen. When a calibration is completed, the instrument will prompt you to view and then accept it. Check your correlation coefficient (required 0.9950 or better) and the recovery of your ICV (90-110%) as well as the result of your ICB (less than the detection limit for that parameter). If these requirements are not met, you must still click on ACCEPT CALIBRATION, then restart for a new calibration keeping in mind a solution or a standard may need remade prior to recalibrating. Print calibrations that you will use for the day.

14.1.6 **To load samples for analysis:** Click on SAMPLES, then F8 MORE, then SAMPLE SEGMENT. When a template appears on the screen, you can choose the number, then type in your sample IDs, keeping in mind that if you make a mistake, you must click on REMOVE ALL SAMPLES and start over. Make sure you analyze an ERA with your run, you can assign all appropriate tests to the one ERA sample. For every 20 samples, analyze a duplicate sample that you can assign all tests to. Also analyze a spike for each parameter being analyzed for. You must have a separate volume for each parameter being analyzed for and spike them separately with 100 microliters of the corresponding 1000ppm spike solution to 5 ml of sample and place these in 5 ml test tubes. *NOTE: You do not have to place a LCS/MBLK every ten slots, the instrument is programmed to take all required LCS/MBLK volumes from one placing for each and run them at a frequency of every ten samples.* Once you get your templates ready onscreen, you can then take your corresponding sample trays and physically create them using the 2 ml

sample cups. Use the 10 ml test tubes for the LCS/MBLK for your run as the instrument will need the additional volume. It is a good idea to place these LCS/MBLK tubes for each parameter being run at the beginning of your trays, preferably in slots 1-6 of your tray #1 (Alk 50ppm LCS, Alk blank MBLK, Cl 50ppm LCS, Cl blank MBLK, Sulfate 30ppm LCS, Sulfate blank MBLK) When using test tubes, take a black marker and draw a thick line down the side of the tube so when placed in the instrument it will recognize it as being a 5 ml tube as opposed to a 2 ml sample cup. You will see the recognition when you view your sample segments onscreen after you place them on the instrument.

14.1.7 **To run samples for analysis:** Once you have your templates created and your samples loaded on the instrument following your template set-up, you are now ready to tell the instrument what to do with those samples. Click on SAMPLES at the top of your screen. Click on F8 MORE, then click on SAMPLE SEGMENT. Starting at your first template, click on the first sample after your CCV/CCB placings on template #1. A screen will appear that will now allow you to choose what tests to assign to each sample. You will not be able to assign tests to CCV/CCB samples. There are groups created at the far right to minimize errors. You can also pick each test individually if you like. The sample you are choosing for will be bordered in blue and overall the samples will look like a small version of your templates so you can recognize placings. The ID will also be posted at the top of the page for each one as you click on them. Go ahead and assign your tests. Once you assign all your tests to each sample for each tray you have set up. Click MAIN at the top right of your screen, then press the green button on your keyboard. Your samples are now analyzing. You can check time left by clicking F8 MORE twice and then PENDING REQUESTS whereas the time left will be posted at the top of the screen. The instrument is programmed to make a one time automatic dilution of any sample that goes over the highest standard on your curve. If this dilution is not enough, see **reviewing data** for the next step in this process.

14.1.8 **Reviewing data:** Once a run is complete, you can then review it for compliancy. Make sure all your CCVs are 90-110% and your blanks are less than the detection limit for their corresponding parameters. Make sure your ERA check standard falls within the desired range designated by Environmental Standards, but preferably 90-110%. Check your spike recoveries (section 17.2). Check your duplicate analyses (section 17.3). Check to see that all results, and ones with their corresponding auto-dilutions fall within the curve range, if not, highlight the samples one by one and click on RERUN WITH DILUTION. You will be prompted to type in a desired dilution and the sample. Once you send all required ones back for secondary dilutions, you may need to click on MAIN, then press the green button on the keyboard to start the process. Wait until this secondary run ends to finalize your data review. You can then send back for further dilution if necessary when this run ends or click on ACCEPT DATA. Once you accept all data, print out data by clicking on F4 REPORTS, change “samples” to “tests”, and change “exclude rejected” to “include rejected”. Highlight one test at a time to print.

14.1.9 **To shut down for the day:** Click on SAMPLES. F8 MORE, then SAMPLE SEGMENT and click on REMOVE SEGMENT. Remove each segment and click on REMOVE ALL SAMPLES after you remove each one. Click on REAGENTS and then click on each reagent and

remove each one as instrument prompts, cap and place in the fridge until next use. Then click on F8 MORE, MANAGEMENT, then F7 to CLEAR DAILY FILES. Choose all samples. Then click on MAIN, F8 MORE, INSTRUMENT ACTIONS, and click on PERFORM WATER WASH to clean out instrument for the day. When finished running, click MAIN, then F2 STAND BY. The instrument will prompt you to open the cover and insert the 2ml cup of washing solution. Close the cover and wait until the instrument prompts you to remove the washing solution (you may just lift and close the cover and leave the solution on the instrument refilling as needed each day). Once STAND BY procedure is completed you can then shut down the instrument by clicking F8 MORE, F3 MANAGEMENT, F8 MORE, then F3 EXIT.

14.2 The pH of samples must be 8.3 or less. If analyzing samples for Bicarbonate, Carbonate or Hydroxide Alkalinity the samples must be pH tested prior to analysis. Samples over a pH of 8.3 must be analyzed titrametrically (method 310.1).

14.3 Soils are analyzed using the same method after they have undergone the ASTM Leachate procedure. Data is then entered into the LIMs system, including the weight and volume of the sample used in the ASTM Leachate procedure. The LIMS system will automatically calculate the result and adjust the reporting limit accordingly.

15.0 CALCULATIONS

15.1 Calibration is done by injecting standards. The data system will then prepare a calibration curve by plotting response versus standard concentration. Sample concentration is calculated from the regression equation.

15.2 Report only those values that fall between the lowest and the highest calibration standards. Samples exceeding the highest standard should be diluted and reanalyzed. Report results in mg CaCO3/L.

Percent Recovery for Analyses Involving Spikes:

$$\%\,\text{Recovery} = \left[\frac{(SSR - SR)}{SA}\right] \text{ x } 100$$

where:

SSR = spiked sample result
SR = sample result
SA = spike added

TITLE: ALKALINITY Method 310.2 (Colorimetric, Automated)

SUPERCEDES: Revision 3

Relative Percent Difference (RPD):

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

where:

x_1 = analytical % recovery
x_2 = replicate % recovery

Dry Weight of Sample:

Sample conc. (µg/kg) as dry weight = [sample conc. µg/kg]/D

where:

D = (100 –% moisture)/100

Measured Concentration by Linear Regression:

$$x = \frac{a - b}{m}$$

where:

a = area counts for analyte to be measured
m = slope
x = concentration
b = intercept

and

$$m = \frac{\sum x_i a_i}{\sum x_i^2}$$

$$b = Y_{ave} - b x_{ave}$$

Percent Recovery for LCS:

$$\%\ \text{Recovery (LCS)} = 100\left(\frac{E}{C}\right)$$

where:

E = obtained (experimental) value
C = true value

TITLE: ALKALINITY Method 310.2 (Colorimetric, Automated)

SUPERCEDES: Revision 3

Final Concentration of Analyte in a Solid Sample (on a dry weight basis) and Waste (on a wet weight basis). This calculation is done automatically in the LIMS system by entering the weight and volume of the sample:

$$\text{Concentration (mg/kg)} = (\text{ir})\left(\frac{v_1}{w_s}\right)\left(\frac{u}{D}\right)$$

where:

ir = instrument result (mg/L or µg/mL)
v_1 = final digestate volume
w_s = weight of sample(g)
u = conversion factor for units calculation

$$\left(\frac{1\,\text{L}}{1000\,\text{mL}} \text{ x } \frac{1000\,\text{g}}{1\,\text{kg}}\right)$$

D = (100 – % moisture)/100

16.0 METHOD PERFORMANCE

16.1. Method Detection Limit: A valid method detection limit for each analyte of interest must be generated. The MDL must be below the reporting limit for each analyte. The procedure for determination of the method detection limit is given in 40 CFR Part 136, Appendix B. See STL SOP S-Q-003, "Method Detection Limit Studies," current revision, for further guidance. Current STL Buffalo MDLs are maintained the QA department and are easily viewed in the laboratory LIMs system.

16.2. A one–time initial demonstration of performance for each individual method must be generated.

16.2.1. This requires quadruplicate analysis of a mid–level check standard containing all of the standard analytes for the method using the same procedures used to analyze samples, including sample preparation.

16.2.2. Calculate the average recovery and standard deviation of the recovery for each analyte of interest.

16.2.3. Compare these results with the acceptance criteria given in the Method or to laboratory historical limits (if available).

16.2.4. Repeat the test for any analyte that does not meet the acceptance criteria. Repeated failure for any analyte indicates the need for the laboratory to evaluate the analytical procedure and take corrective action.

TITLE: ALKALINITY Method 310.2 (Colorimetric, Automated)

SUPERCEDES: Revision 3

16.3. Training Qualifications

16.3.1. The supervisor has the responsibility to ensure that this procedure is performed by an analyst who has been properly trained in its use and has the required experience.

16.3.2. The following analyst validation information is maintained for this method in the laboratory QA files.

16.3.2.1. The analyst must complete the laboratory safety orientation training that includes, but is not limited to, chemicals, PPE requirements, and electrical safety.

16.3.2.2. The analyst must read and understand this SOP.

16.3.2.3. The analyst must read and understand the Method used as reference for this SOP.

16.3.2.4. The analyst must complete a DOC or successfully analyze PT samples annually.

16.3.2.5. The analyst must complete the STL Quality Assurance Training.

17.0 DATA ASSESSMENT AND ACCEPTANCE CRITERIA FOR QUALITY CONTROL MEASURES

17.1 ICAL: Acceptance criteria for the calibration curve is a correlation coefficient (R value) $\geq$0.990.

17.2 ICV (second source): Within $\pm$10% of true value

17.3 LCS: Within $\pm$10% of true value

17.4 Method Blank:
17.4.1 Detected concentrations < PQL or
17.4.2 Detected concentrations < 10X amount in associated samples

17.5 MS/MSD: Sample spike recovery acceptance limits are calculated yearly and maintained for easy reference and/or inspection.

18.0 CORRECTIVE ACTIONS FOR OUT-OF-CONTROL DATA

18.1 ICAL: Analysis cannot begin without an acceptable calibration curve. Instrument maintenance may be required.

18.2 ICV: Reanalyze calibration curve if unacceptable ICV is obtained.

TITLE: ALKALINITY Method 310.2 (Colorimetric, Automated)

SUPERCEDES: Revision 3

18.3 LCS:

18.3.1 If below limits: Re-extract all samples associated with an unacceptable MSB

18.3.2 If above limits: Re-extract all samples with detections of DRO. Re-extraction not required if samples are ND.

18.4 Method Blank: Reanalyze all samples associated with an unacceptable method blank unless the detected concentrations are <10X amount in associated samples.

18.5 MS/MSD:

18.5.1 Matrix interference can be assumed and corrective action is not required if both of the following conditions are met:

18.5.1.1MSB recovery is acceptable

18.5.1.2Recoveries in both MS and MSD are consistent (%RSD<30)

18.5.2 If MSB is unacceptable – re-extraction of batch is required.

18.5.3 If recoveries in MS/MSD are different (e.g.: one high, one low) further evaluation should be made. Matrix interference cannot be assumed in this case. Discussion with the department supervisor, operations manager or QA manager should be included in the final decision process prior to releasing data.

19.0 CONTINGENCIES FOR HANDLING OUT-OF-CONTROL OR UNACCEPTABLE DATA

19.1 Job exception forms are to be filled out in the event of unknown positives or sample matrix, which present the analyst with questionable data, the project manager shall be notified so the client may be contacted and involved in the decision process and course of action or if there is a holding time exceedance.

20.0 WASTE MANAGEMENT/POLLUTION PREVENTION

20.1 Instrument waste produced must be placed in an "A" waste disposal container as detailed in STL's Laboratory Safety Manual, Chemical Hygiene Plan and SOP AWM-HAZ.MG-01.

20.2 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."

20.3 Waste Streams Produced by the Method

20.4 There are no special waste streams associated with this method.

TITLE: ALKALINITY Method 310.2 (Colorimetric, Automated)

SUPERCEDES: Revision 3

21.0 REFERENCE

21.1 Alkalinity Method 310.1, Methods for Chemical Analysis of Water and Wastes, EPA-600/4-712-020, March 112123.

21.2 Alkalinity Method 310.2, Methods for Chemical Analysis of Water and Wastes, EPA-600/4-712-020, March 11283.

21.3 EST Analytical Methodologies for Konelab instrumentation.

22.0 TABLES, DIAGRAMS, FLOWCHARTS AND VALIDATION DATA

22.1 Analytical Run sequence for the Konelab.

22.2 Analytical Batch Sequence for the Lachat

22.3 Analytical Batch Sequence for the Konelab.

22.4 Wet Chemistry Batch Summary

23.0 CHANGES FROM PREVIOUS REVISION

23.1 Section 14.2: clarified what type of Alkalinity must be pH checked prior to analysis.

22.1 Analytical run sequence

ICAL: minimum of once every three months.
ICV: Second source check standard
ICB: Blank

LCS: 50 ppm
MBLK
SAMPLE
SAMPLE
SAMPLE
SAMPLE
SAMPLE
SAMPLE
SAMPLE
SAMPLE
SAMPLE
SAMPLE

TITLE: ALKALINITY Method 310.2 (Colorimetric, Automated)

SUPERCEDES: Revision 3

LCS: 50 ppm
MBLK
SAMPLE DUP
SAMPLE SPIKE
SAMPLE
SAMPLE
SAMPLE
SAMPLE
SAMPLE
SAMPLE
SAMPLE
SAMPLE
LCS: 50 ppm
MBLK

22.2 pH screening logbook

STL Buffalo
pH Screening for Alkalinity Method 310.2 000021
Logbook # A03-14-13

DATE	ANALYST	JOB #	SAMPLE ID	pH	COMMENTS
19 Feb 2004	LRM	1272	A4127201	6.5	
		1173	A4117301	6.5	
		1178	A4117801	6.5	
		1190	A4119001	6.5	
			02		
			03		
			04		
			05		
			06		
			07		
			08		
			09		
		1196	A4119601	6.5	
		1197	A4119701	6.5	
		1207	A4120701	6.5	
			02		
			03		
			04		

LRM 19 Feb 2004

REWD BY/ DATE LRM 19Feb 2004 pH Narrow range Paper Lot # OC303517

TITLE: ALKALINITY Method 310.2 (Colorimetric, Automated)

SUPERCEDES: Revision 3

22.5 Analytical Batch for Kone

Result Report — Konelab 6.0.5 — Page: 1

STL Buffalo
PGE, KG, KK, CS

BATCH: A4B06953

Date : 2004-03-15
Time : 20.17

Test: Alkalinity
Unit: mg/l

Sample ID:	Result	Resp.	Blank	Dilut	Date and Time	
Alk CCV	46.411	-0.091	0.524		2004-03-15 15.41	92.8%
Alk CCB	1.916	-0.045	0.527		2004-03-15 15.41	<5
207102	2.357	-0.046	0.530		2004-03-15 15.41	
207103		-0.386	0.530		-	
207104		-0.403	0.531		-	
207105 → pH >8.3		-0.429	0.533		-	
207106 → pH >8.3		-0.419	0.533		-	
207107	97.545	-0.144	0.535		2004-03-15 15.41	
207108	98.068	-0.144	0.536		2004-03-15 15.41	
207109		-0.417	0.536		-	
207110		-0.346	0.535		-	
207111		-0.354	0.533		-	
Alk CCV	47.209	-0.092	0.524		2004-03-15 16.03	94.4%
Alk CCB	2.837	-0.046	0.526		2004-03-15 16.03	<5
207112		-0.374	0.528		-	
207113		-0.448	0.529		-	
207114	47.584	-0.092	0.530		2004-03-15 16.03	>2.3% RPD
207114FD	48.691	-0.093	0.532		2004-03-15 16.03	
207115		-0.381	0.533		-	
207116		-0.442	0.534		-	
207117	15.798	-0.060	0.535		2004-03-15 16.03	>4.0% RPD
207117FD	16.439	-0.060	0.535		2004-03-15 16.03	
208601	25.263	-0.069	0.534		2004-03-15 16.03	
208602	17.770	-0.062	0.529		2004-03-15 16.03	
Alk CCV	47.735	-0.092	0.525		2004-03-15 16.11	95.5%
Alk CCB	3.950	-0.047	0.527		2004-03-15 16.11	<5
208602 dup	19.210	-0.063	0.528		2004-03-15 16.11	7.8% RPD
208602 alk20	37.985	-0.082	0.529		2004-03-15 16.11	101%
208603	13.838	-0.058	0.531		2004-03-15 16.11	
208604	4.182	-0.048	0.532		2004-03-15 16.11	
208701	78.873	-0.124	0.532		2004-03-15 16.11	
208702	79.257	-0.125	0.535		2004-03-15 16.11	
208703	71.700	-0.117	0.535		2004-03-15 16.11	
208704	5.335	-0.049	0.536		2004-03-15 16.11	
212601		-0.333	0.535		-	
212602		-0.183	0.533		-	
Alk CCV	49.443	-0.094	0.526		2004-03-15 16.14	98.9%
Alk CCB	4.381	-0.048	0.527		2004-03-15 16.14	<5
212603	70.448	-0.116	0.528		2004-03-15 16.14	
213901		-0.407	0.529		-	
213902		-0.330	0.530		-	
213903		-0.378	0.531		-	
213905		-0.424	0.532		-	
213906		-0.396	0.534		-	
213909		-0.312	0.536		-	
213910		-0.350	0.534		-	
213911		-0.389	0.535		-	
213911 dup		-0.394	0.533		-	
Alk CCV	47.340	-0.092	0.518		2004-03-15 16.19	94.7%
Alk CCB	3.235	-0.047	0.525		2004-03-15 16.19	<5
213911 alk20		-0.386	0.525		-	
era 1/2	30.905	-0.075	0.524		2004-03-15 16.19	ACTUAL 30.8 (RANGE) 27.9-32.9
Alk CCV	48.172	-0.093	0.517		2004-03-15 16.20	96.3% 100%

INITIAL ANALYSIS

STL Buffalo
LABORATORY STANDARD OPERATING PROCEDURES

SOP No.	Revision No.	Effective Date	Page
AWC-310.2-18	4	February 26, 2007	16 of 16

TITLE: ALKALINITY Method 310.2 (Colorimetric, Automated)

SUPERCEDES: Revision 3

22.5 Wet Chemistry Batch Summary

WET CHEMISTRY BATCH SUMMARY

PARAMETER_____ **METHOD**_____ **BATCH** _____

COMMENTS	JOB NUMBER
WC Reporting Limit < STL Quant Limit	
WC Historical confirms within Hold Time	
WC Historical NO confirm & RE outside of HT	
WC Hold Time Exceedance-Dilution required	
WC Hold Time Exceedance-Instrument Failure	
WC Holding Time Exceedance by Date	
WC Holding Time Exceedance by Hours	
WC LCS within ERA limits outside internal	
WC LCS high recovery, sample ND	
WC MBLK hit but samples > 10X blank value	
WC RPD Exceedance for MS / SD	
WC Spike Failure HIGH MS only	
WC Spike Failure LOW MS only	
WC Spike Failure MS and SD	
WC BOD HT met- Oxygen depleted-RE out HT	
WC Carbonate Alkalinity, LCS/MBLK	
WC Reactivity Qualification	
WC TDS/Conductivity ratio outside of range	
WC TOX Breakthrough- no volume for redo	
WC TOX samples were centrifuged	
Other	

DILUTION CODES	REASON
002	Sample matrix effects
003	Excessive foaming
004	High levels of non-target compounds
008	High concentration of target analytes
009	Sample turbidity
010	Sample color
011	Insufficient volume for lower dilution
012	Sample viscosity
013	other

ICAL Compliant? YES NO NA IF NO, Why? _____
LCS/CCV Compliant? YES NO NA IF NO, Why? _____
CCB Compliant? YES NO NA IF NO, Why? _____
RPD Compliant? YES NO NA IF NO, Why? _____
ERA Compliant? YES NO NA IF NO, Why? _____

NUMBER of REANALYSIS FOR THIS BATCH:_____

Analyst_____ Date_____

Time Critical Batch Review_____ Date_____

Secondary Review & Closure_____ Date_____ WC Summary Rev 4 / 5-2005

STL Buffalo
LABORATORY STANDARD OPERATING PROCEDURES

SOP No.	Revision No.	Date	Page
AWC-350.1-19	6	May 26, 2006	1 of 22

TITLE: AMMONIA NITROGEN Method 350.1 –Automated Phenate

Supersedes: Revision 5

REVIEWED & APPROVED BY:	Signature	Date
Verl Preston , Quality Manager		
Christopher Spencer, Laboratory Director		
Peggy Gray-Erdmann, Supervisor		

1.0 IDENTIFICATION OF TEST METHODS

1.1 Ammonia Nitrogen 350.1 – Automated Phenate

2.0 APPLICABLE MATRIX

2.1 Water, industrial wastes and soil (leachate). Soils can be analyzed from leachates prepared using ASTM method D3987.

3.0 REPORTING LIMIT

3.1 The laboratory's reporting limit is 0.02 mg/L for Lachat and Konelab.

4.0 SCOPE AND APPLICATION

4.1 This method is used for the determination of ammonia in drinking, surface and saline waters and domestic and industrial wastes.

4.2 Soils can be analyzed from leachates prepared using ASTM Method D 3987.

TITLE: AMMONIA NITROGEN Method 350.1 –Automated Phenate

Supersedes: Revision 5

5.0 SUMMARY OF TEST METHOD

5.1 Ammonia reacts with alkaline phenol and hypochlorite to form indophenol blue in an amount that is proportional to the ammonia concentration. The blue color is intensified with sodium nitroferricyanide. The absorbence is measured at 630nm for Lachat and 660nm for Konelab.

6.0 DEFINITIONS

6.1 Standard definitions are used in this document as defined by the STL Buffalo Laboratory Quality Manual.

7.0 INTERFERENCES

7.1 Calcium and magnesium ions may precipitate if present in sufficient concentration. EDTA is added to the sample in-line, by the instrument, in order to prevent these problems.

7.2 Color, turbidity and certain organic species may interfere. Turbidity is removed by manual filtration. Dirty or improperly washed glassware may cause interference.

8.0 SAFETY

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

8.2 SPECIFIC SAFETY CONCERNS OR REQUIREMENTS

Sodium Nitroferricyanide will generate Hydrogen Cyanide (HCN) gas if combined with strong acids. Inhalation of CN gas can cause irritation, dizziness, nausea, unconsciousness, and potentially death.

8.3 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 this 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.

TITLE: AMMONIA NITROGEN Method 350.1 –Automated Phenate

Supersedes: Revision 5

Material (1)	Hazards	Exposure Limit (2)	Signs and symptoms of exposure
Phenol	Corrosive	5 ppm-TWA	Breathing vapor, dust or mist results in digestive disturbances. Will irritate, possibly burn respiratory tract. Rapidly absorbed through the skin with systemic poisoning effects to follow. Discoloration and severe burns may occur, but may be disguised by a loss in pain sensation. Eye burns with redness, pain, blurred vision may occur. May cause severe damage and blindness.
Sodium Hydroxide	Corrosive	2 Mg/M3-Ceiling	Severe irritant. Effects from inhalation of dust or mist vary from mild irritation to serious damage of the upper respiratory tract, depending on severity of exposure. Symptoms may include sneezing, sore throat or runny nose. Contact with skin can cause irritation or severe burns and scarring with greater exposures. Causes irritation of eyes, and with greater exposures it can cause burns that may result in permanent impairment of vision, even blindness.
Sodium Nitroferri-cyanide	Poison	5 mg/m^3 as HCN gas	This material may cause irritation if it comes into the contact with the skin. The materials will give off HCN gas if combined with strong acids. Inhalation of HCN gas can cause irritation, dizziness, nausea, unconsciousness and potentially death.
Sulfuric Acid	Corrosive Oxidizer Dehydrator Poison Carcinogen	1 Mg/M3-TWA	Inhalation produces damaging effects on the mucous membranes and upper respiratory tract. Symptoms may include irritation of the nose and throat, and labored breathing. Symptoms of redness, pain, and severe burn can occur. Contact can cause blurred vision, redness, pain and severe tissue burns. Can cause blindness.
1 – Always add acid to water to prevent violent reactions.			
2 – Exposure limit refers to the OSHA regulatory exposure limit.			

9.0 EQUIPMENT AND SUPPLIES

Lachat Quikchem 8000, Konelab Aqua20, or Konelab 20XT

9.1 Sampler

9.2 Multi-channel proportioning pump

9.3 Reaction Unit or Manifold

9.4 Colorimetric Detector

9.5 Balance – Analytical, capable of accurately weighing to the nearest 0.00001g

9.6 Glassware – Class A volumetric flasks and pipettes or plastic containers as required.

9.7 Eppendorf and Finntip – range 10 microliters to 5 milliliters.

9.8 Sample cups (2ml) – Opaque plastic or glass

9.9 Glass Culture Tubes – 5 and 10 ml

10.0 REAGENTS AND STANDARDS

10.1 REAGENTS AND STANDARDS FOR LACHAT

10.1.1 Documentation of standards is recorded in the "standards and reagents logbook"

10.1.2 Alkaline phenol: Add 40 ml 10 N NaOH (or 16.0g NaOH pellets) + 44 ml 88% liquefied phenol to ~250 ml DiH_2O in a 500 ml volumetric flask. Dilute to 500 ml with DiH_2O. Make weekly.

10.1.3 Sodium hypochlorite: 500 ml volumetric flask, add 250 ml 5% sodium hypochlorite. Dilute to the mark with DiH_2O. Make daily for a final concentration of 2.5% Sodium Hypochlorite. Make daily.

10.1.4 EDTA Buffer: In an IL volumetric flask, add 50.0 g disodium ethylenediamine tetraacetate dihydrate ($Na_2EDTA\ 2H_2O$) + 20 ml 10 N NaOH (or 9.0g NaOH). Dilute to the mark with DiH_2O and stir with magnetic stirrer until dissolved. De-gas with helium. For a final concentration of 5% EDTA Buffer.

10.1.5 Sodium nitroferricyanide: In an IL volumetric flask, dissolve 3.50 g sodium nitroferricyanide in ~500 ml DiH_2O. Dilute to the mark with DiH_2O. Make monthly.

10.1.6 0.2% Sulfuric acid carrier/blank: In an IL volumetric flask, add 2 ml conc H_2SO^4 into 900 ml DiH_2O. Dilute to 1000 ml with DiH_2O.

10.1.7 Sodium Hydroxide, 0.1N: Dissolve 4 g of NAOH in reagent water and dilute to 1 L.

10.1.8 Sodium Tetraborate Solution, 0.025M: Dissolve 5 g of Na2B4O7 [anhydrous] or 9.5g of Na2B4O7 x 5H2O and dilute to 1 L with reagent water. This is made when the Borate buffer solution (10.1.9) is made and not purchased pre-made.

10.1.9 Borate Buffer Solution: Add 88 mL of 0.1N NaOH to 500 ml of 0.025M Sodium Tetraborate and dilute to 1L with reagent water. This is also available as a commercially prepared solution from various vendors.

10.1.10 Ammonia Nitrogen 1000 mg/l STD: Dilute 4.7168 g of ammonium chloride to 1000 ml with DiH_2O. Preserve with 2 drops of chloroform. This is also available as a commercially prepared standard from various vendors.

10.1.11 Ammonia Nitrogen 1000 mg/l SRM: Dilute 4.7168 g of ammonium chloride to 1000 ml with DiH_2O. Preserve with 2 drops of chloroform. This is also available as a commercially prepared standard from various vendors.

10.1.12 Intermediate 100 mg/l Ammonia Nitrogen STD: To a 100 ml volumetric flask, add 10 ml of stock ammonia nitrogen standard to ~50 ml DiH_2O. Add 0.2 ml conc H_2SO^4 and dilute to the mark.

10.1.13 Calibration Standards

2.0 ppm:	2.0 ml NH_3 INT STD + 0.20 ML CONC H_2SO^4
1.0 ppm:	1.0 ml NH_3 INT STD + 0.20 ML CONC H_2SO^4
0.50 ppm:	0.50 ml NH_3 INT STD + 0.20 ML CONC H_2SO^4
0.20 ppm:	0.20 ml NH_3 INT STD + 0.20 ML CONC H_2SO^4
0.05 ppm:	0.05 ml NH_3 INT STD + 0.20 ML CONC H_2SO^4
0.020ppm:	0.02 ml NH_3 INT STD + 0.20 ML CONC H_2SO^4
0 ppm:	0 ml NH_3 INT STD + 0.20 ML CONC H_2SO^4

*Dilute up to 100 ml with DiH_2O.

10.1.14 LCS at 0.75 ppm: to a 100 ml volumetric flask, add 0.75 ml of stock ammonia nitrogen to ~50 ml DiH_2O. Add 0.20 ml conc H_2SO^4 and dilute to the mark with DiH20.

10.1.14 ICV at 0.375 ppm: to a 100 ml volumetric flask, add 0.375 ml of stock ammonia nitrogen to ~50ml DiH2O. Add 0.20 ml conc. H_2SO^4 and dilute to the mark with DiH20. This is to be analyzed once after every calibration curve and is to be made from a separate source than the calibration standards.

10.1.15 Matrix Spike at 0.2 ppm: to prepare sample spikes, add 0.02 ml of the 100ppm Ammonia Nitrogen standard to

10.1.16 Distilled LCS at 1.0 ppm: to a 50 ml volumetric flask, add 0.50 ml of stock ammonia nitrogen to ~30 ml DiH_2O. Add 0.20 ml conc H_2SO^4 and dilute to the mark with DiH20. Distill as you would samples (14.1) then analyze.

10.1.17 Distilled Matrix Spikes at 0.5 ppm: to a 50 ml volumetric flask, add 0.25 ml of stock ammonia nitrogen to ~30 ml DiH_2O. Add 0.20 ml conc H_2SO^4 and dilute to the mark with DiH20. Distill as you would samples (14.1) then analyze.

10.2 REAGENTS AND STANDARDS FOR KONELAB

10.2.1 All standards, reagents, and solutions are documented in the appropriate logbook. Items that should be included for Konelab analysis are as follows:

10.2.2 (Reagent) Sodium Phenolate: add 9.3ml liquid phenol to 50ml reagent DiH2O. Slowly add 3.2g NaOH and mix to dissolve. Cool and dilute to 100ml with DiH2O. This reagent may be purchased pre-made from EST.

10.2.3 (Reagent) Sodium Hypochlorite: Dilute 50ml Chlorox to 100ml with DiH2O. This reagent may be purchased pre-made from EST.

10.2.4 (Reagent) EDTA Buffer: Dissolve 50g EDTA (disodium salt) and 9g of NaOH into 500ml DiH2O, cool and dilute to 1000ml with DiH2O. This reagent may be purchased pre-made from EST.

10.2.5 (Reagent) Sodium Nitroferricyanide: Dissolve 0.05g of Sodium Nitroferricyanide in 50ml DiH20, dilute to 100ml with DiH20.

10.2.6 (Reagent) Ammonia Diluent: dilute 200 microliters of conc. Sulfuric Acid to 100 ml with DiH20. This solution is used for the ICB and CCB also.

10.2.7 Stock Ammonia: Dissolve 0.3819g anhydrous Ammonium Chloride (dried at 105 degrees Celsius) in DiH2O and dilute to 100ml (conc. = 1000ppm). A 1000ppm stock ammonia solution may be purchased pre-made from EST.

10.2.8 100 ppm Intermediate Standard: 10ml of 1000ppm, add 200microliters conc. H2SO4, dilute to 100ml with DiH2O.

10.2.9 ICV (0.375ppm): 0.375 ml of 100ppm, add 200microliters conc. H2SO4, dilute to 100ml with DiH2O.

10.2.10 LCS (0.750ppm): 0.75 ml of 100ppm, add 200microliters conc. H2SO4, dilute to 100ml with DiH2O

10.2.11 1ppm Standard (for curve calibration): 1ml of 100ppm, add 200microliters conc. H2SO4, dilute to 100ml with DiH2O.

TITLE: AMMONIA NITROGEN Method 350.1 –Automated Phenate

Supersedes: Revision 5

11.0 SAMPLE COLLECTION, PRESERVATION, SHIPMENT AND STORAGE

11.1 Samples are preserved by adding 2ml of conc. Sulfuric acid per liter of sample for a pH less than 2. Then stored at 4C. Samples must be analyzed within 28 days of collection.

12.0 QUALITY CONTROL

12.1 To create a run:

12.1.1 Begin and end every batch with an LCS and MBLK.

12.1.2 Analyze a LCS and MBLK after every 10 samples for both distilled and undistilled ammonia batches.

12.1.3 Analyze a sample duplicate (matrix duplicate) and sample spike (matrix spike) with every 20 samples for both distilled and undistilled ammonia samples.

12.1.4 Acceptance criteria for accuracy and precision are calculated on an annual basis using the previous year's results.

13.0 CALIBRATION AND STANDARDIZATION

13.1 For Lachat, prepare a series of standards, covering the desired range, and a blank by diluting suitable volumes of standard solution. For Konelab, prepare the high point 1.0ppm as well as the ICV/ICB.

Calibrate the instrument according to the desired methodology:

13.2 For Lachat, prepare standard curve by plotting instrument response against concentration values. A calibration curve may be fitted to the calibration solution concentration/response data using the computer. Acceptance criteria for the correlation coefficient is a correlation coefficient (R value) $\geq$ 0 .995.

13.3 For Konelab, the standard curve is plotted by the instrument using instrument response against concentration values. Acceptance criteria for the calibration curve is a correlation coefficient (R^2 value) $\geq$ 0.990. The coefficient of detection listed on the Kone curve data is a R^2 value and must be that evaluated using the $\geq$ 0.990 criteria.

13.4 After the calibration has established, it must be verified by the analysis of a suitable quality control sample (ICV). If measurements exceed +/- 10% of the established QCS value, the analysis should be terminated and the instrument re-calibrated. The new calibration must be verified before continuing analysis.

TITLE: AMMONIA NITROGEN Method 350.1 –Automated Phenate

Supersedes: Revision 5

14.0 PROCEDURE

14.1 DISTILLATION:

14.1.1 Steam out the distillation glassware using 50mls of reagent water with a pinch of boiling chips until no trace of ammonia can be detected.

14.1.2 To 50mL of sample add 1N NaOH dropwise until the pH reaches 9.5.

14.1.3 To the receiver tube, add 5ml of 0.2% Sulfuric Acid. The tip of the long stem must be below the sulfuric acid. Set the temperature to 160C and watch for bumping of the sample to alter the temperature.

14.1.3 Collect a minimum of 30ml of distillate.

14.1.4 Dilute the distillate to the 50ml graduation mark with ammonia free water.

14.1.5 The next run can be started when the heater temperature falls below 100C

14.1.6 One LCS and MBLK must be distilled for every batch of twenty samples or less.

14.1.7 One sample duplicate must be distilled for every batch of twenty samples or less.

14.1.8 One Matrix Spike must be distilled for every batch of twenty samples or less.

14.2 CALIBRATION PROCEDURE for Lachat

14.2.1 Prepare reagent and standards

14.2.2 Set up manifold

14.2.3 Input data system parameters

14.2.4 Pump DI water through all reagent lines and check for leaks and smooth flow. Switch to reagents and allow the system to equilibrate until a stable baseline is achieved.

14.2.5 Place samples and/or standards in the autosampler. Input the information required by the data system, such as concentration, replicates and QC scheme.

14.2.6 Calibrate the instrument by injecting the standards. The data system will then associate the concentrations with the instrument responses for each standard.

TITLE: AMMONIA NITROGEN Method 350.1 –Automated Phenate

Supersedes: Revision 5

14.3 ANALYSIS FOR LACHAT

14.3.1 If samples are preserved and determined without distillation, the level of preservation acid is critical.

14.3.2 For information on system maintenance and troubleshooting, refer to the Troubleshooting Guide in the System Operation Manual. This guide is also available on request from Lachat.

14.3.3 Allow 15 min for heating unit to warm up to 60°C.

14.3.4 If baseline drifts, peaks are too wide or other problems with precision arise, clean the manifold by the following procedure.

A. Place all reagent lines in deionized water and pump to clear reagents (2 to 5 min).

B. Place all reagent lines in 1 M hydrochloric acid (1 volume concentrated HCl added to 11 volumes of deionized water) and pump for several minutes.

C. Place all reagent lines in deionized water and pump until the HCl is thoroughly washed out.

D. Resume pumping reagents.

14.4 PROCEDURE FOR KONELAB:

14.4.1 **Start up procedure every day:** Turn on the Konelab first then switch the PC on. The Username is **administrator** and the Password is **Klab1sUPER.** Wait until the yellow banner appears on the screen stating "start up". Press F1 on the Konelab main screen. Press OK when prompted to perform operation. The analyzer powers up the lamp and chopper motor, and perform a washing function. Start up takes approximately three minutes. After the start up, the main screen will display a "ready" flag.

14.4.2 **Checking the Water Blank**: Each day it is necessary to check the quality of the water blank. This is done after the start up procedure. From the main screen, select F8 [more], then F2 [Instrument Actions]. Select F8 [more] again, then F1 [check water blank]. When complete 9 approximately three minutes), you will be given a graphical representation of the water blank. Make sure that all results are within +/-1mA (milliabsorbance). If not rerun this procedure.

14.4.3 **To load the reagents:** to load the reagents on the instrument using the provided 20 ml plastic reagent vials. Click on REAGENTS at the top of the screen. Click on INSERT REAGENT. Then choose your first reagent and click OK. The instrument will prompt you when to insert the reagent. Follow this procedure for each reagent.

14.4.4 **To load a new calibration**: Click on SAMPLES, then F8 MORE, then SAMPLE SEGMENT. When a template appears on the screen, choose what tray you want to use, then choose from the pick list the appropriate standard(s) and ICV/ICB you need. When this is done, physically using

that same number tray and the 2 ml plastic sample cups, place your items in the tray. On the screen, click on INSERT SEGMENT and the instrument will prompt you when to place the tray on the instrument. There are 6 trays with 14 slots each that you can place on the instrument at one time. Also, when selecting calibrators for your template, if you make a mistake, you must click on REMOVE ALL SAMPLES and start over.

14.4.5 **To run a new calibration:** Once the desired concentrations are inserted into the instrument and the template has been set up, you can then click on F6 CALIBRATION/QC SELECTION. Choose the parameters you are calibrating for. Click F1 CALIBRATE, then click MAIN at the top right of the screen. Then press the green button on the keyboard. Your curves have begun being generated. It takes approximately 15 minutes to generate any given calibration. You can check time left by clicking F8 MORE twice and then PENDING REQUESTS whereas the time left will be posted at the top of the screen. When a calibration is completed, the instrument will prompt you to view and then accept it. Check your correlation coefficient (required 0.9950 or better) and the recovery of your ICV (90-110%) as well as the result of your ICB (less than the detection limit for that parameter). If these requirements are not met, you must still click on ACCEPT CALIBRATION, then restart for a new calibration keeping in mind a solution or a standard may need remade prior to recalibrating. Print calibrations that you will use for the day.

14.4.6 **To load samples for analysis:** Click on SAMPLES, then F8 MORE, then SAMPLE SEGMENT. When a template appears on the screen, you can choose the number, then type in your sample IDs, keeping in mind that if you make a mistake, you must click on REMOVE ALL SAMPLES and start over. Make sure you analyze an ERA with your run, you can assign all appropriate tests to the one ERA sample. For every 20 samples, analyze a duplicate sample that you can assign all tests to. Also analyze a spike for each parameter being analyzed for. You must have a separate volume for each parameter being analyzed for and spike them separately with 100 microliters of the corresponding 1000ppm spike solution to 5 ml of sample and place these in 5 ml test tubes. *NOTE: You do not have to place a CCV/CCB every ten slots, the instrument is programmed to take all required CCV/CCB volumes from one placing for each and run them at a frequency of every ten samples.* Once you get your templates ready onscreen, you can then take your corresponding sample trays and physically create them using the 2 ml sample cups. Use the 5 ml test tubes for the CCV/ CCB for your run as the instrument will need the additional volume. It is a good idea to place these CCV/CCB tubes for each parameter being run at the beginning of your trays, preferably in slots 1-6 of your tray #1 . When using test tubes, take a black marker and draw a thick line down the side of the tube so when placed in the instrument it will recognize it as being a 5 ml tube as opposed to a 2 ml sample cup. You will see the recognition when you view your sample segments onscreen after you place them on the instrument.

14.4.7 **To run samples for analysis:** Once you have your templates created and your samples loaded on the instrument following your template set-up, you are now ready to tell the instrument what to do with those samples. Click on SAMPLES at the top of your screen. Click on F8 MORE, then click on SAMPLE SEGMENT. Starting at your first template, click on the first sample after your CCV/CCB placings on template #1. A screen will appear that will now allow you to choose what tests to assign to each sample. You will not be able to assign tests to CCV/CCB samples. There

are groups created at the far right to minimize errors. You can also pick each test individually if you like. The sample you are choosing for will be bordered in blue and overall the samples will look like a small version of your templates so you can recognize placings. The ID will also be posted at the top of the page for each one as you click on them. Go ahead and assign your tests. Once you assign all your tests to each sample for each tray you have set up. Click MAIN at the top right of your screen, then press the green button on your keyboard. Your samples are now analyzing. You can check time left by clicking F8 MORE twice and then PENDING REQUESTS whereas the time left will be posted at the top of the screen. The instrument is programmed to make a one time automatic dilution of any sample that goes over the highest standard on your curve. If this dilution is not enough, see **reviewing data** for the next step in this process.

15.0 CALCULATIONS

15.1 Sample results are calculated from the calibration curve by using linear regression.

15.2 In the case of any dilution, the results have the dilution factor automatically calculated.

15.3 For liquid samples, the result is expressed in sulfate as SO_4^{-2} mg/l.

15.4 For solid samples, the result is expressed as sulfate as SO_4^{-2} mg/kg on a dry basis.

15.4.1 To convert the mg/l result obtained from the calibration curve to mg/kg use the following equation:

mg/kg (wet) = [mg/l X final vol. of leached sample] / grams sample used

mg/kg (dry) = mg/kg (wet) / decimal dry weight

15.5 Percent Recovery for Analyses Involving Spikes:

$$\%\,\text{Recovery} = \left[\frac{(SSR - SR)}{SA}\right] \times 100$$

where:

SSR = spiked sample result
SR = sample result
SA = spike added

15.6 Relative Percent Difference (RPD):

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

where:

x_1 = analytical % recovery
x_2 = replicate % recovery

15.7 Measured Concentration by Linear Regression:

$$x = \frac{a - b}{m}$$

where:

a = area counts for analyte to be measured
m = slope
x = concentration
b = intercept

and

$$m = \frac{\sum x_i a_i}{\sum x_i^2}$$

$$b = Y_{ave} - bx_{ave}$$

15.8 Percent Recovery for LCS:

$$\%\ \text{Recovery (LCS)} = 100\left(\frac{E}{C}\right)$$

where:

E = obtained (experimental) value C = true value

15.9 Once a run is complete, you can then review it for compliancy. Make sure all your CCVs are 90-110% and your blanks are less than the detection limit for their corresponding parameters. Make sure your ERA check standard falls within the desired range designated by Environmental Standards, but preferably 90-110%. Check your spike recoveries. Check your duplicate analyses. Check to see that all results, and ones with their corresponding auto-dilutions fall within the curve range, if not, highlight the samples one by one and send them back for a manual dilution that would place the result within the curve range.

.

TITLE: AMMONIA NITROGEN Method 350.1 –Automated Phenate

Supersedes: Revision 5

16.0 METHOD PERFORMANCE

16.1. Method Detection Limit: A valid method detection limit for each analyte of interest must be generated. The MDL must be below the reporting limit for each analyte. The procedure for determination of the method detection limit is given in 40 CFR Part 136, Appendix B. See STL SOP S-Q-003, "Method Detection Limit Studies," current revision, for further guidance. Current STL Buffalo MDLs are maintained the QA department and are easily viewed in the laboratory LIMs system.

16.2. A one–time initial demonstration of performance for each individual method for both soils and water matrices must be generated.

16.2.1. This requires quadruplicate analysis of a mid–level check standard containing all of the standard analytes for the method using the same procedures used to analyze samples, including sample preparation.

16.2.2. Calculate the average recovery and standard deviation of the recovery for each analyte of interest.

16.2.3. Compare these results with the acceptance criteria given in the Method or to laboratory historical limits (if available).

16.2.4. Repeat the test for any analyte that does not meet the acceptance criteria. Repeated failure for any analyte indicates the need for the laboratory to evaluate the analytical procedure and take corrective action.

16.3. Training Qualifications

16.4. The supervisor has the responsibility to ensure that this procedure is performed by an analyst who has been properly trained in its use and has the required experience.

16.5. The following analyst validation information is maintained for this method in the laboratory QA files.

16.6. The analyst must complete the laboratory safety orientation training that includes, but is not limited to, chemicals, PPE requirements, and electrical safety.

16.7. The analyst must read and understand this SOP.

16.8. The analyst must read and understand the Method used as reference for this SOP.

16.9. The analyst must complete a DOC or successfully analyze PT samples annually.

16.10. The analyst must complete the STL Quality Assurance Training.

TITLE: AMMONIA NITROGEN Method 350.1 –Automated Phenate

Supersedes: Revision 5

17.0 DATA ASSESSMENT AND ACCEPTANCE CRITERIA FOR QUALITY CONTROL MEASURES

17.1 ICAL: calibration factor >0.995, must be analyzed at a minimum of once every three months.

17.2 ICV (second source)/ LCS: Obtained values must be within ±10% of true value.

17.3 Method Blank:

17.3.1 Detected concentrations < PQL or

17.3.2 Detected concentrations < 10X amount in associated sample

17.3.3 All blanks associated with DOD QSM and AFCEE must be less than half the reporting limit.

17.4 Matrix Spike: Acceptance limits for sample spike recovery are based on the historical data and are statistically derived annually. They are maintained in the LIMs system.

18.0 CORRECTIVE ACTIONS FOR OUT-OF-CONTROL DATA

18.1 ICAL: Analysis cannot begin without an acceptable calibration curve. Instrument maintenance may be required. Please refer to STL Corporate Policy for information on the proper selection of calibration points.

18.2 ICV: Reanalyze calibration curve if unacceptable ICV is obtained.

18.3 CCV: Reanalyze the CCV.

18.3.1 If 2nd analysis is acceptable, analytical sequence can continue, however the previous 10 samples must be reanalyzed.

18.3.2 If 2nd analysis is unacceptable, analyze a new ICAL.

18.3.3 Method Blank: Reanalyze all samples associated with an unacceptable method blank unless:

18.3.3.1 Detected concentrations < PQL or

18.3.3.2 Detected concentrations < 10X amount in associated sample

18.3.4 Matrix Spike: Matrix interference can be assumed and corrective action is not required if both of the following conditions are met:

18.3.4.1 LCS recovery is acceptable

18.3.4.2 Recoveries in both MS and MSD are consistent (%RSD<30)

18.3.4.3 If LCS is unacceptable – re-analysis is required.

18.3.4.4 If recoveries in MS/MSD are different (e.g.: one high, one low) further

evaluation should be made. Matrix interference can not be assumed in this case. Discussion with the department supervisor, operations manager or QA manager should be included in the final decision process prior to releasing data.

19.0 CONTINGENCIES FOR HANDLING OUT-OF-CONTROL OR UNACCEPTABLE DATA

19.1 A Job Exception Form must be completed and filed with the Project Manager and QA Manager for any of the following conditions:

19.1.1. Holding times exceeded

19.1.2. Insufficient sample volume for reanalysis

19.1.3. In the event of unknown positives or sample matrix which present the analyst with questionable data, the project manager shall be notified so the client may be contacted and involved in the decision process and course of action

19.2 Sample will be redistilled and reanalyzed when necessary

20.0 WASTE MANAGEMENT / POLLUTION PREVENTION

20.1 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".

20.2 Waste streams produced by the method shall be disposed of in "A" waste. Reagents shall be disposed according to STL waste disposal rules.

21.0 REFERENCE

21.1 U.S. Environmental Protection Agency, **Methods for Chemical Analysis of Water and Wastes**, EPA-600/4-79-020, Revised March 1983, Method 350.1.

21.2 U.S. Environmental Protection Agency, 40 CFR, Part 36 Table 1B, footnote 6, 1994.

21.3 Lachat Quik Chem Method 10-107-06-1-B Revision Date March 13, 1998

21.4 EST Analytical Konelab Methodolgy

TITLE: AMMONIA NITROGEN Method 350.1 –Automated Phenate

Supersedes: Revision 5

22.0 TABLES, DIAGRAMS, FLOWCHARTS AND VALIDATION DATA

22.1 Ammonia Manifold Program

22.2 Data System Parameters for Quikchem 8000

22.3 Konelab Water Check

22.4 Konelab Ammonia Curve

22.5 Konelab Analytical Batch

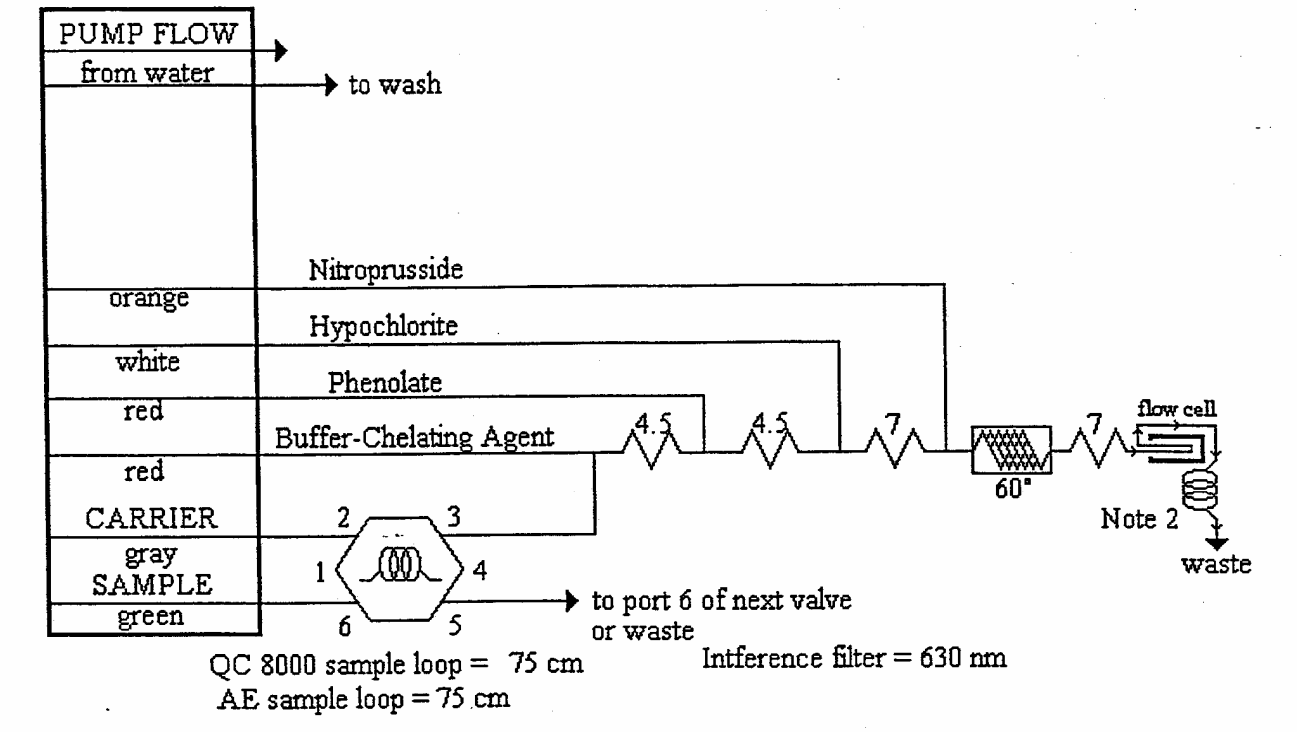
22.6 Wet Chemistry Batch Summary Sheet

23.0 CHANGES FROM PREVIOUS REVISION

23.1 Updated Section 10.1, 15.0, 16.0, 17.0, 18.0 and 19.0.

23.2 Updated QC terminology in section 12.0

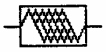
22.1 **Ammonia Manifold Program**



CARRIER is Reagent 5.

Manifold tubing is **0.8 mm (0.032 in) i.d.** This is **5.2 uL/cm.**

4.5	is	**70**	cm of tubing on a 4.5 cm coil support
7	is	**135**	cm of tubing on a 7 cm coil support

APPARATUS: The  indicates 650 cm of tubing wrapped around the heater block at the specified temperature.

Note 1: TYGON PUMP TUBES MUST BE USED FOR THIS METHOD

Note 2: 200 cm x 0.022" i.d. backpressure loop.

22.2 **Data System Parameters for Quikchem 8000**

The timing values listed below are approximate and will need to be optimized using graphical events programming.

Sample throughput:	60 samples/h, 60 s/sample
Pump seed:	35
Cycle Period:	60

Analyte Data:

Concentration Units:	mg N/L
Peak Base Width:	27.0 s
% Width Tolerance:	100
Threshold:	10000
Inject to Peak Start:	41.8 s
Chemistry:	Direct

TITLE: AMMONIA NITROGEN Method 350.1 –Automated Phenate

Supersedes: Revision 5

Calibration Data:

Level	1	2	3	4	5	6	7
Concentration (mg/L)	5.00	3.75	3.50	1.25	0.50	0.10	0.00

Calibration Fit Type: 1st Order Polynomial

Calibration Rep. Handling: Average

Weighting Method: None

Concentration Scaling: None

Force Through Zero: No

Sampler Timing:

Min. Probe in Wash Period: 5.0 s

Probe in Sample Period: 24 s

Valve Timing:

Load Time: 0.0 s

Load Period: 15 s

Inject Period: 45 s

TITLE: AMMONIA NITROGEN Method 350.1 –Automated Phenate

Supersedes: Revision 5

22.3 **Konelab Water Check**

Check water blank Konelab 6.0.5 Page: 1

STL Buffalo
Konelab 1
Analyst:

13.02.2005 15:48

Performed 13.02.2005 09:04
MAX acceptable SD 2.0 mA

Wavelength	Abs (mA)	SD (mA)	SignGain	RefGain	Voltage (V)
380 nm	-161.7	0.8	5	5	6.2
420 nm	-199.8	0.6	5	5	6.2
450 nm	-232.2	0.5	4	4	5.9
480 nm	-240.7	0.4	3	3	6.0
510 nm	-248.6	0.4	2	2	6.2
520 nm	-245.3	0.4	2	2	6.2
540 nm	-250.9	0.5	2	2	6.2
575 nm	-248.6	0.6	2	2	6.2
600 nm	-246.2	0.4	2	2	6.2
620 nm	-246.0	0.5	2	2	5.7
660 nm	-247.8	0.3	2	2	5.9
700 nm	-246.9	0.4	1	1	6.2
880 nm	-233.1	0.2	1	1	6.2

TITLE: AMMONIA NITROGEN Method 350.1 –Automated Phenate

Supersedes: Revision 5

22.4 Konelab Ammonia Curve

Calibration results AquaKem 6.0.7 Page: 1

STL Buffalo
Konelab 2
Analyst:

13.01.2005 14:40

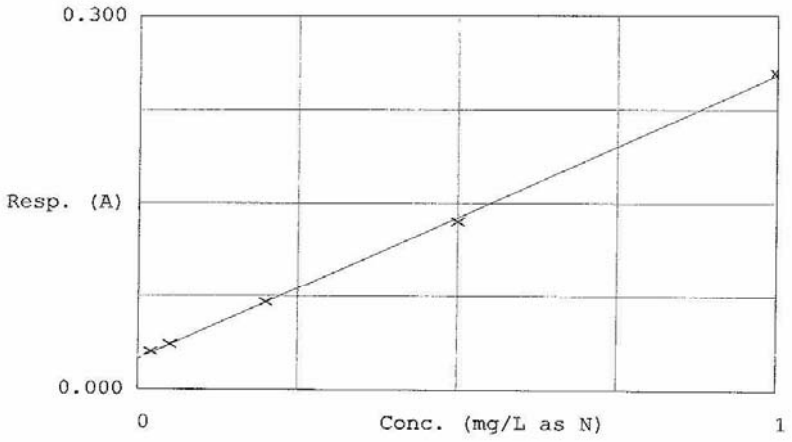
Test Ammonia

Accepted 13.01.2005 13:42

Factor 4.389
Bias 0.025

Coeff. of det. 0.999570

Errors Batch incomplete



	Calibrator	Response	Calc. con.	Conc.	Errors
1	NH3 1.0pp	0.030	0.0256	0.0200	
2	NH3 1.0pp	0.036	0.0514	0.0500	
3	NH3 1.0pp	0.070	0.2004	0.2000	
4	NH3 1.0pp	0.135	0.4857	0.5000	
5	NH3 1.0pp	0.254	1.0069	1.0000	
6	NH3 ICB(control			0.0000	Aut. rejected
7	NH3 ICV(control			0.3750	Aut. rejected

22.5 Konelab Analytical Batch

Result Report — Konelab 6.0.5 — Page: 1

STL Buffalo
Konelab 1

Date : 2005-01-06
Time : 18.16

Test: Ammonia
Unit: mg/l

Sample ID:	Result	Resp.	Blank	Dilut	Date and Time
NH3 CCV	0.406	0.096	-0.000		2005-01-06 14.45
NH3 CCB	-0.017	0.011	-0.000		2005-01-06 14.45
NH3 BLANK	-0.019	0.011	0.000		2005-01-06 14.45
NH3 MDL1 0.05	-0.247	0.012	0.005	1+19.0	2005-01-06 14.55
NH3 MDL2 0.05	0.294	0.018	-0.000	1+19.0	2005-01-06 14.56
NH3 MDL3 0.05	0.260	0.017	-0.000	1+19.0	2005-01-06 14.56
NH3 MDL4 0.05	0.258	0.017	0.000	1+19.0	2005-01-06 14.56
NH3 MDL5 0.05	0.275	0.017	-0.000	1+19.0	2005-01-06 14.56
NH3 MDL6 0.05	0.278	0.017	-0.000	1+19.0	2005-01-06 14.56
NH3 MDL7 0.05	0.273	0.017	-0.000	1+19.0	2005-01-06 15.00
NH3 CCV	0.406	0.096	-0.000		2005-01-06 15.03
NH3 CCB	-0.018	0.011	-0.000		2005-01-06 15.03
NH3 CCV	0.394	0.094	0.000		2005-01-06 15.28
NH3 CCB	-0.014	0.012	-0.000		2005-01-06 15.28
NH3 1.0	1.033	0.222	-0.000		2005-01-06 15.28
NH3 1.0	1.036	0.222	-0.000		2005-01-06 15.28
NH3 1.0	1.039	0.223	-0.000		2005-01-06 15.28
NH3 1.0	1.037	0.223	-0.000		2005-01-06 15.28
NH3 1.0	1.053	0.226	-0.000		2005-01-06 15.28
NH3 CCV	0.408	0.096	-0.000		2005-01-06 15.32
NH3 CCB	-0.017	0.011	-0.000		2005-01-06 15.32
NH3 CCV	0.698	0.155	-0.000		2005-01-06 16.37
NH3 CCB	-0.011	0.012	0.000		2005-01-06 16.37
NH3 MDL1 0.05	0.043	0.023	-0.000		2005-01-06 16.37
NH3 MDL2 0.05	0.040	0.022	-0.000		2005-01-06 16.37
NH3 MDL3 0.05	0.040	0.023	-0.000		2005-01-06 16.37
NH3 MDL4 0.05	0.039	0.022	-0.000		2005-01-06 16.37
NH3 MDL5 0.05	0.040	0.023	-0.000		2005-01-06 16.37
NH3 MDL6 0.05	0.039	0.022	-0.000		2005-01-06 16.37
NH3 MDL7 0.05	0.039	0.022	-0.000		2005-01-06 16.37
NH3 BLANK	-0.016	0.011	0.000		2005-01-06 16.37
NH3 CCV	0.745	0.164	-0.000		2005-01-06 16.42
NH3 CCB	-0.014	0.012	0.000		2005-01-06 16.42
NH3 CCV	0.707	0.157	-0.000		2005-01-06 17.17
NH3 CCB	-0.013	0.012	-0.000		2005-01-06 17.17
NH3 MDL1 0.05	0.042	0.023	-0.000		2005-01-06 17.17
NH3 MDL2 0.05	0.042	0.023	-0.000		2005-01-06 17.17
NH3 MDL3 0.05	0.040	0.023	-0.000		2005-01-06 17.17
NH3 MDL5 0.05	0.040	0.023	-0.000		2005-01-06 17.18
NH3 MDL7 0.05	0.039	0.022	-0.000		2005-01-06 17.18
NH3 MDL4 0.05	0.045	0.024	-0.000		2005-01-06 17.25
NH3 MDL6 0.05	0.045	0.024	0.000		2005-01-06 17.25
NH3 CCV	0.737	0.162	-0.000		2005-01-06 17.25
NH3 CCB	-0.008	0.013	-0.000		2005-01-06 17.25
NH3 BLANK	-0.012	0.012	-0.000		2005-01-06 17.25
NH3 CCV	0.770	0.169	-0.000		2005-01-06 17.37
NH3 CCB	-0.007	0.013	0.000		2005-01-06 17.37
NH3 CCV	0.740	0.163	-0.000		2005-01-06 17.53
NH3 CCB	-0.012	0.012	-0.000		2005-01-06 17.53
NH3 CCV	0.739	0.163	-0.000		2005-01-06 18.09
NH3 CCB	-0.012	0.012	-0.000		2005-01-06 18.10
NH3 CCV	0.749	0.165	-0.000		2005-01-06 18.15
NH3 CCB	-0.013	0.012	-0.000		2005-01-06 18.15

WORKED!

TITLE: AMMONIA NITROGEN Method 350.1 –Automated Phenate

Supersedes: Revision 5

22.6 Wet Chemistry Batch Summary Sheet

WET CHEMISTRY BATCH SUMMARY

PARAMETER_____ METHOD_____ BATCH _____

COMMENTS	JOB NUMBER
WC Reporting Limit < STL Quant Limit	
WC Historical confirms within Hold Time	
WC Historical NO confirm & RE outside of HT	
WC Hold Time Exceedance-Dilution required	
WC Hold Time Exceedance-Instrument Failure	
WC Holding Time Exceedance by Date	
WC Holding Time Exceedance by Hours	
WC LCS high recovery, sample ND	
WC MBLK hit but samples > 10X blank value	
WC RPD Exceedance for MS / SD	
WC Spike Failure HIGH MS only	
WC Spike Failure LOW MS only	
WC Spike Failure MS and SD	
WC BOD HT met- Oxygen depleted-RE out HT	
WC Carbonate Alkalinity, LCS/MBLK	
WC Reactivity Qualification	
WC TOX Breakthrough- no volume for redo	
WC TOX samples were centrifuged	
Other	

DILUTION CODES	REASON
002	Sample matrix effects
003	Excessive foaming
004	High levels of non-target compounds
008	High concentration of target analytes
009	Sample turbidity
010	Sample color
011	Insufficient volume for lower dilution
012	Sample viscosity
013	other

ICAL Compliant? YES NO NA IF NO, Why? _____
LCS/CCV Compliant? YES NO NA IF NO, Why? _____
CCB Compliant? YES NO NA IF NO, Why? _____
RPD Compliant? YES NO NA IF NO, Why? _____
ERA Compliant? YES NO NA IF NO, Why? _____

NUMBER of REANALYSIS FOR THIS BATCH:_____

Analyst_____ Date_____

Time Critical Batch Review_____ Date_____

Secondary Review & Closure_____ Date_____

WC Summary Rev 2 / 2-2005

STL Buffalo
LABORATORY STANDARD OPERATING PROCEDURES

SOP No.	Revision No.	Effective Date	Page
AWC-353.2-24	8	September 16, 2006	1 of 22

TITLE: NITRATE+NITRITE NITROGEN, NITRITE NITROGEN AND NITRATE NITROGEN METHOD 353.2 – AUTOMATED CADMIUM REDUCTION METHOD

SUPERCEDES: Revision 7

REVIEWED & APPROVED BY:	SIGNATURE	DATE
Verl D. Preston, Quality Manager		
Christopher Spencer, Laboratory Director		
Peggy Gray-Erdmann, Supervisor		

1.0 IDENTIFICATION OF TEST METHOD

1.1 This method is taken from EPA method 353.2 and Standard Methods, method 4500-NO_3-F.

2.0 APPLICABLE MATRIX

2.1 This method is used for the determination of the nitrate and nitrite (singly or total) in drinking, surface and saline waters and domestic and industrial wastes.

2.2 Soils can be analyzed from leachates prepared using ASTM Method D 3987, "Shake Extraction of Solid Waste with Water".

3.0 REPORTING LIMIT

3.1 The reporting limit has been determined to be 0.05 mg/l.

4.0 SCOPE AND APPLICATION

4.1 This method is used for the determination of the nitrate and nitrite (singly or total) in drinking, surface and saline waters and domestic and industrial wastes.

TITLE: NITRATE+NITRITE NITROGEN, NITRITE NITROGEN AND NITRATE NITROGEN METHOD 353.2 – AUTOMATED CADMIUM REDUCTION METHOD

SUPERCEDES: Revision 7

5.0 SUMMARY OF THE TEST METHOD

5.1 Nitrate is reduced quantitatively to nitrite in the presence of cadmium. The nitrite thus formed plus any originally present in the sample is determined as an azo dye at 520 nm following its diazotization with sulfanilamide and subsequent coupling with N(-1-naphthyl)ethylenediamine dihydrochloride. Without the introduction of the sample to the cadmium column, nitrite singly is determined. A Nitrate only value may be calculated by subtracting the nitrite from the Total Nitrite+Nitrate value.

6.0 DEFINITIONS

6.1 Standard definitions are used in this document as defined by the STL Corporate Quality Assurance Plan.

7.0 INTERFERENCES

7.1 Sample color, particulates or turbidity may interfere. Turbid samples or samples with suspended solids must be filtered prior to analysis through a 0.45 pore diameter filter.

7.2 Some metals in high concentration will cause various interferences. EDTA is added to eliminate these interferences.

7.3 Acidic samples are to be adjusted to a pH of 5 to 9 with a dilute solution of ammonium hydroxide.

7.4 Residual Chlorine can interfere by oxidizing the cadmium column and must be removed.

8.0 SAFETY

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

8.1 SPECIFIC SAFETY CONCERNS OR REQUIREMENTS

None

8.2 PRIMARY MATERIALS USED

The following is a list of the materials used in this method, which have a serious or significant hazard rating. **NOTE: This list does not include all materials used in the method. The table contains a summary of the primary hazards listed in the MSDS for each of the materials listed in the table.** A complete list of materials used in the method can be found in the reagents

and materials section. Employees must review the information in the MSDS for each material before using it for the first time or when there are major changes to the MSDS.

Material (1)	Hazards	Exposure Limit (2)	Signs and symptoms of exposure
Ammonium Hydroxide	Corrosive Poison	50 ppm-TWA	Vapors and mists cause irritation to the respiratory tract. Causes irritation and burns to the skin and eyes.
Chloroform	Carcinogen Irritant	50 ppm Ceiling	Acts as a relatively potent anesthetic. Irritates respiratory tract and causes central nervous system effects, including headache, drowsiness, dizziness. Causes skin irritation resulting in redness and pain. Removes natural oils. May be absorbed through skin. Vapors cause pain and irritation to eyes. Splashes may cause severe irritation and possible eye damage.
Phosphoric Acid	Corrosive	1 Mg/M3 TWA	Inhalation is not an expected hazard unless misted or heated to high temperatures. May cause redness, pain, and severe skin burns. May cause redness, pain, blurred vision, eye burns, and permanent eye damage.
Potassium Nitrate	Oxidizer	None	Causes irritation to the respiratory tract, skin and eyes. Symptoms may include coughing, shortness of breath. Symptoms include redness, itching, and pain.
1 – Always add acid to water to prevent violent reactions.			
2 – Exposure limit refers to the OSHA regulatory exposure limit.			

9.0 EQUIPMENT AND SUPPLIES

9.1 LACHAT autoanalyzer equipped with nitrate/nitrite manifold and cadmium column. The cadmim column is a disposable acrylic column packed with cadmium with seals on either end to prevent leaks and water evaporation during storage.

9.2 Class A volumetric and graduated glassware and calibrated eppendorfs.

9.3 Miscellaneous disposable supplies, such as culture tubes, pipets, parafilm, etc.

TITLE: NITRATE+NITRITE NITROGEN, NITRITE NITROGEN AND NITRATE NITROGEN METHOD 353.2 – AUTOMATED CADMIUM REDUCTION METHOD

SUPERCEDES: Revision 7

10.0 REAGENTS AND STANDARDS

10.1. All chemicals shall conform to American Chemical Society specifications or equivalent.

10.1.1. For best results, all reagents should be filtered prior to on-line use.

10.2. Reagent Water

10.3. Concentrated Ammonium Hydroxide

10.4 1:4 Ammonium Hydroxide Solution (200 ml): Add 50 ml of conc. ammonium hydroxide to 150 ml of reagent water and mix well. This has a shelf life of six months.

10.5 Stock Ammonium Chloride-EDTA Buffer Solution (2 L): Dissolve 170 g of ammonium chloride and 2.0 g of disodium EDTA in about 1800 ml of reagent water. Adjust the pH to 8.5 with 1:4 NH_4OH. Dilute to 2000 ml with reagent water and mix well. This has a shelf life of one month.

10.6 Concentrated Phosphoric Acid

10.7 Color Reagent (1 L): While stirring, add 100 mL of conc. H_3PO_4 to about 700 mL of reagent water. Dissolve 40 g of sulfanilamide and 1 g of N(-1-naphthyl)ethylenediamine dihydrochloride in the acid solution. Dilute to 1 L with reagent water and mix well. Store reagent in an amber bottle and keep in the dark when not in use. This reagent is stable for one month.

10.8 Sampler Wash Solution: Reagent water.

10.9 Chloroform

10.10 Stock 1000 mg/L **Nitrate** Nitrogen Standard purchased from two separate vendors.

10.10.1 Intermediate 100 mg/L Nitrate Nitrogen Solution (100 ml): Add 10.0 ml of the 1st source stock nitrate nitrogen solution (1000 mg/L) to about 80 ml of reagent water. Dilute to 100 ml with reagent water and mix well. This solution is to be prepared daily.

10.10.1.1 The calibration curve and matrix spike solution will be prepared from the primary source

TITLE: NITRATE+NITRITE NITROGEN, NITRITE NITROGEN AND NITRATE NITROGEN METHOD 353.2 – AUTOMATED CADMIUM REDUCTION METHOD

SUPERCEDES: Revision 7

10.10.2 ICV/CCV 2.5 mg/L Nitrate Nitrogen Solution (100ml): Add 0.25 ml of the 2nd source stock nitrate nitrogen solution (100 mg/L) to about 80 ml of reagent water. Dilute to 100 ml with reagent water and mix well. This solution is to be prepared daily. This solution is used to check the reduction efficiency of the cadmium column and as the continuing quality control check for Nitrates.

10.10.3 Prepare **Nitrate** calibration standards by adding the appropriate amount of intermediate standard nitrate nitrogen solution (100 mg/L) (see table below for recommended calibrants) to about 80 ml of reagent water. Dilute to 100 ml with reagent water and mix well. These solutions are to be prepared once a month.

10.11 Stock 1000 mg/L **Nitrite** Nitrogen Standard purchased from two separate vendors.

10.11.1 Intermediate 100 mg/L Nitrite Nitrogen Solution (100 ml): Add 10.0 ml of the 1st source stock nitrite nitrogen solution (1000 mg/L) to about 80 ml of reagent water. Dilute to 100 ml with reagent water and mix well. This solution is to be prepared daily.

10.11.1.1 The calibration curve and matrix spike solution will be prepared from the primary source.

10.11.2 High Level ICV/CCV 2.5 mg/L Nitrite Nitrogen Solution (100ml): Add 0.25 ml of the 2nd source stock nitrite nitrogen solution (100 mg/L) to about 80 ml of reagent water. Dilute to 100 ml with reagent water and mix well. This solution is to be prepared daily. This solution is used to check the reduction efficiency of the cadmium column on the NO2-NO3 analytical batch.

10.11.3 Low Level ICV/CCV 1.0 mg/L Nitrite Nitrogen Solution (100 ml): Add 0.10 ml of the 2nd source stock nitrite nitrogen solution (1000 mg/L) to about 80 ml of reagent water. Dilute to 100 ml with reagent water and mix well. This solution is to be prepared daily. This solution is used as quality control check for Nitrites.

10.11.4 Prepare **Nitrite** calibration standards by adding the appropriate amount of intermediate standard nitrite nitrogen solution (100 mg/L) (see table below for recommended calibrants) to about 80 ml of reagent water. Dilute to 100 ml with reagent water and mix well. These solutions are to be prepared daily.

TITLE: **NITRATE+NITRITE NITROGEN, NITRITE NITROGEN AND NITRATE NITROGEN METHOD 353.2 – AUTOMATED CADMIUM REDUCTION METHOD**

SUPERCEDES: Revision 7

CALIBRANTS

Nitrate Calibrant Concentration (mg/L)	Volume of Intermediate Solution (ml)
0 (blank)	0
0.050	0.050
0.20	0.20
0.50	0.50
1.0	1.0
3.0	3.0
5.0	5.0

CALIBRANTS

Nitrite Calibrant Concentration (mg/L)	Volume of Intermediate Solution (ml)
0 (blank)	0
0.050	0.050
0.20	0.20
0.50	0.50
1.0	1.0
3.0	3.0

10.12 Sodium Thiosulfate solution (Dechlorinating solution): Dissolve 3.5 g sodium thiosulfate ($Na_2S_2O_3 \cdot 5H_2O$) in water and dilute to 1 Liter. Prepare fresh weekly. Use 1ml reagent to remove 1mg/l residual chlorine in 500 ml sample.

11.0 SAMPLE COLLECTION, PRESERVATION AND STORAGE

11.1. **Nitrite** only samples must be unpreserved and be analyzed within 48 hours of sample time. They require refrigeration at 4°C.

11.2. **Nitrate** only samples must be unpreserved and be analyzed within 48 hours of sample time. They require refrigeration at 4°C.

11.3. **Total Nitrate + Nitrite** samples may be preserved or unpreserved. For a 28-day holding time, samples must be preserved with 2-ml conc. H_2SO_4/L to a pH < 2.0 and refrigerated at 4°C. If samples are unpreserved they require refrigeration at 4°C and must be analyzed within 48 hours of sample time. **Nitrite can not be analyzed on samples after they have been preserved**, as preservation

converts all available nitrite to nitrate. Therefore, once a sample has been preserved, only combined nitrate-nitrite can be tested for.

12.0 QUALITY CONTROL

12.1. Initial Calibration Verification (ICV) and Continuing Calibration Verification (CCV): The ICV is prepared from a source other than that used to prepare the calibration curve and is analyzed at start of run. The CCVs are analyzed after every 10 samples and at the end of the analytical sequence. The ICV/CCV is equivalent to an LCS for this method.

12.1.1. The results of the analyses of the ICV and CCVs must be within +/-10% of the true value. If unacceptable results are obtained, all samples analyzed since the last acceptable CCV must be re-analyzed.

12.2. An Initial Calibration Blank (ICB) is analyzed after the ICV and a Continuing Calibration Blank (CCB) is analyzed with each analytical batch of 10 samples or less and is carried through the entire analytical procedure. These blanks must not exhibit Nitrate (or Nitrite) at concentrations greater than the STL Buffalo quantitation limit. If unacceptable results are achieved, all samples associated with that blank must be re-analyzed.

12.2.1. All blanks associated with USACE samples should be less than 1/2 the STL Buffalo quantitation limit for Nitrate/Nitrite.

12.3. Matrix Spike (MS) and Matrix Duplicate (MD): A MS/MD set is performed for each sample batch or once every 20 samples, whichever is more frequent. One of the samples in the batch is prepared in triplicate, with the second and third aliquots being the MS and MD. The MS is fortified with Intermediate Nitrate Nitrogen Solution or Intermediate Nitrite Nitrogen Solution, depending on analyses performing. The MD is analyzed neat.

12.3.1. The calculated RPD of the sample and MD should be < 20%. If results fall outside of the QC limits but all other QC criteria for the analytical batch have been achieved, re-analysis may not be required.

12.3.2. Results of the MS analysis should be compared to the in-house % recovery limits. These limits are statistically derived based upon historical data and are updated annually. If the lab calculated limits are wider than the method limits, the method limits of 90-110% are used to evaluate matrix spike acceptance. The sample matrix may affect accuracy, therefore if results fall outside QC limits but all other QC criteria for the analytical batch have been achieved, re-analysis may not be required.

TITLE: NITRATE+NITRITE NITROGEN, NITRITE NITROGEN AND NITRATE NITROGEN METHOD 353.2 – AUTOMATED CADMIUM REDUCTION METHOD

SUPERCEDES: Revision 7

12.4. The acceptance criteria for the reduction efficiency for the cadmium column are 90-110%. If criteria are not met a new column must be used.

13.0 CALIBRATION AND STANDARDIZATION

13.1. A calibration curve containing *at least* 5 points is analyzed monthly. STL Buffalo currently uses the 6-7 point curves described in Section 10.10 and 10.11. These curves consist of the following concentrations: 0.0, 0.050, 0.20, 0.50, 1.0, and 3.0, with a 5.0 mg/l point added for Nitrate.

13.2. Prepare standard curve by plotting instrument response against concentration values. The curve is a linear (1st Order) curve. A calibration curve may be fitted to the calibration solution concentration/response data using the computer. Acceptance or control limits should be established using the difference between the measured values of the calibration solution and the "true value" concentration. Acceptance criteria for the calibration curve is a correlation coefficient (R value) $\geq$0.995.

14.0 PROCEDURE

14.1. Prepare reagents and standards as described in Section 10.0.

14.2. Set up manifold as shown in Section 22.

14.3. Pump DI water through all reagent lines and check for leaks and smooth flow. Switch to reagents and allow system to equilibrate.

14.4 Input the sample identification required by the data system. Before placing samples in the auto sampler, check all samples for residual chlorine. If samples test positive for residual chlorine it must be removed by adding sodium thiosulfate solution (dechlorinating reagent 10.12). Use 1ml reagent to remove 1mg/l residual chlorine in 500 ml sample. If samples are preserved, add 1:4 NH_4OH drop wise until a pH of 5-9 is achieved.

14.5 **Total Nitrate + Nitrite:** Switch valve to turn cadmium column on. The nitrate in the sample is reduced to nitrite as it passes through the cadmium column. Start instrument analysis, checking to be certain that baseline is steady, checking standards are compliant, and no further dilutions are required.

14.6 **Nitrite singly**: Switch valve to turn cadmium column off. The nitrite method is identical in setup to the nitrate/nitrite method, with the exception of the cadmium column. By not allowing the sample to pass through the cadmium column only nitrite values will be acquired.

TITLE: NITRATE+NITRITE NITROGEN, NITRITE NITROGEN AND NITRATE NITROGEN METHOD 353.2 – AUTOMATED CADMIUM REDUCTION METHOD

SUPERCEDES: Revision 7

14.7 **Nitrate singly:** Nitrate only values are calculated by subtracting the nitrite only value from the combined Nitrite+ Nitrate value. If the Total Nitrite + Nitrate value is below the Quantitation limit, it is unnecessary to analyze the Nitrite only value.

15.0 CALCULATIONS

15.1. The concentration of each sample will be calculated by the program based on peak area from the calibration curve.

15.1.1. If nitrate nitrogen singly is to be determined, using the calculated concentrations of the total nitrate/nitrite nitrogen and nitrite nitrogen, then use the following formula:

$$NO_3^- \text{ singly} = [\text{Total } NO_3^- + NO_2^-] - [NO_2^- \text{ singly}]$$

15.1.2 If the nitrate nitrogen is singly to be determined and the total nitrate/nitrite nitrogen ($NO_3 + NO_2$) is non detect then the nitrate nitrogen (NO_2) does not need to be run

15.2. The reduction efficiency of the cadmium column can be calculated by the following formula and should be within 90-100%, if not a new column must be used:

$$\% \text{ Reduction Efficiency} = \frac{NO_3^- \text{ peak height}}{NO_2^- \text{ peak height}} \text{ X } 100$$

15.3 Results must also be printed off showing area counts. To do this, the current curve must be deleted, both in the Review screen, and in the Analyte Table. After deleting the curve, hit the Analyze button. **Do not at any point save the method**. This will completely delete the current curve, and the instrument will need to be re-calibrated before the next run. Once you have printed area counts, exit out the Omnion program, being sure to hit ***No***, when asked if you want to save any changes to the method.

16.0 METHOD PERFORMANCE

16.1 The applicable range of this method is 0.05 - 3.0 mg/L. The range can be extended by sample dilution.

16.2 As specified in EPA Method 353.2, Revision 2.0, method detection limit studies are performed every 6 months. The MDL determination process is performed in accordance with 40 CFR, part 136, Appendix B and must demonstrate the ability to report a detection limit of 0.05 mg/L.

TITLE: NITRATE+NITRITE NITROGEN, NITRITE NITROGEN AND NITRATE NITROGEN METHOD 353.2 – AUTOMATED CADMIUM REDUCTION METHOD

SUPERCEDES: Revision 7

17.0 DATA ASSESSMENT AND ACCEPTANCE CRITERIA FOR QUALITY CONTROL

17.1. Obtained ICV and CCV values must be within 90–110% of the true value.

17.2. Sample spike recovery acceptance limits are calculated yearly and maintained for easy reference and/or inspection.

17.3. Sample duplicates should have a calculated RPD ≤ 20.0%.

17.4. Blanks (ICB, CCB) must be less than the STL Buffalo quantitation limit or the sample concentration must be greater than 10X the amount in the blank. If these criteria are not met, all associated samples must be reanalyzed.

17.4.1. All blanks associated with USACE samples should be less than ½ the STL Buffalo quantitation limit for Nitrate / Nitrite.

18.0 CORRECTIVE ACTIONS FOR OUT-OF-CONTROL OR UNACCEPTABLE DATA

18.1 If acceptance criteria are exceeded, all samples and check standards must be repeated.

19.0 CONTINGENCIES FOR HANDLING OUT-OF-CONTROL OR UNACCEPTABLE DATA

19.1 Job exception forms are to be filled out and turned in to the appropriate project manager for final approval.

20.0 WASTE MANAGEMENT/POLLUTION PREVENTION

20.1. 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."

20.2. Waste Streams Produced by the Method: The following waste streams are produced when this method is carried out.

20.2.1. Acidic waste generated by the Lachat auto-analyzer. Dispose of this waste in the "A" waste container.

20.2.2. Acidic sample waste generated by sample preparation. Dispose of this waste in the "A" waste container.

TITLE: NITRATE+NITRITE NITROGEN, NITRITE NITROGEN AND NITRATE NITROGEN METHOD 353.2 – AUTOMATED CADMIUM REDUCTION METHOD

SUPERCEDES: Revision 7

20.2.3. Contaminated disposable glassware utilized for the analysis. Empty the contests of the glassware into the "A" waste and dispose of the glassware in the recycling bins located throughout the lab.

21.0 REFERENCE

21.1 Lachat QuikChem® Method 10-107-04-1-C, Determination of Nitrate/Nitrite in surface and wastewaters by flow injection analysis, 1999.

21.2 Methods for Chemical Analysis of Water and Wastes - Method 353.2.

21.3 Standard Methods, 19th Edition, method 4500-NO_3-F.

22.0 TABLES, DIAGRAMS, FLOWCHARTS AND VALIDATION DATA

22.1 Nitrate/Nitrite Manifold Diagram

22.2 Data System Parameters for QuickChem AE

22.3 Data System Parameters for QuickChem 8000

22.4 Analytical Run Sequence

22.5 Analytical Batch

22.6 Wet Chemistry Batch Summary & Data Review Checklist

23.0 CHANGES FROM PREVIOUS REVISION

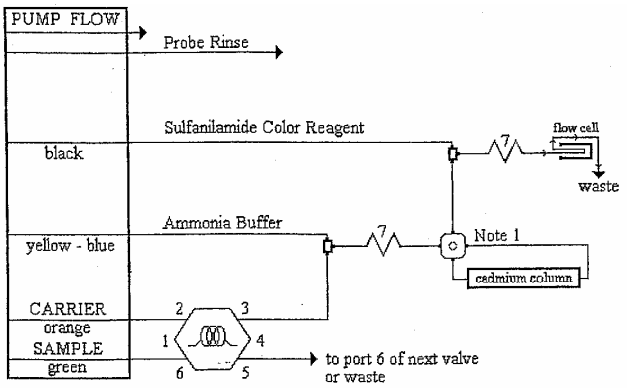
23.1 Updated section 1.1 and 21.3 to include method reference to Standard Methods.

SOP No.	Revision No.	Effective Date	Page
AWC-353.2-24	8	September 16, 2006	12 of 22

TITLE: NITRATE+NITRITE NITROGEN, NITRITE NITROGEN AND NITRATE NITROGEN METHOD 353.2 – AUTOMATED CADMIUM REDUCTION METHOD

SUPERCEDES: Revision 7

22.1 NITRATE/NITRITE MANIFOLD DIAGRAM



Sample Loop = 17 cm X 0.8 mm
QC8000 Sample Loop = 22.5 cm X 0.8 mm

Interference Filter = 520 nm

CARRIER is Helium Degassed DI water

Manifold tubing is **0.8 mm (0.032 in)** i.d. This is 5.2 uL/cm.

7 is **135** cm of tubing on a 7 cm coil support

APPARATUS: An injection valve, a 10 mm path length flow cell , and a colorimetric detector module is required..

Note 1: This is a 2 state switching valve used to place the cadmium column in-line with the manifold.

SOP No.	Revision No.	Effective Date	Page
AWC-353.2-24	8	September 16, 2006	13 of 22

TITLE: **NITRATE+NITRITE NITROGEN, NITRITE NITROGEN AND NITRATE NITROGEN METHOD 353.2 – AUTOMATED CADMIUM REDUCTION METHOD**

SUPERCEDES: Revision 7

22.2 **DATA SYSTEM PARAMETERS FOR QUIKCHEM AE**

Sample Throughput: 90 samples/h; 40 s/sample

Pump Speed: 35

Cycle Period: 40 s

Nitrate + Nitrite

Inject to start of peak: 22 s

Inject to end of peak: 60 s

Nitrite (no column)

Inject to start of peak: 15 s

Inject to end of peak: 53 s

Parameter, Data Window:

Top Scale Response: 0.50 abs

Bottom Scale Response: 0.00 abs

Note: The nitrate concentration can be determined in an RDF using the following equation:

[(nitrate + nitrite) - nitrite]/column efficiency.

SOP No. AWC-353.2-24	Revision No. 8	Effective Date September 16, 2006	Page 14 of 22

TITLE: **NITRATE+NITRITE NITROGEN, NITRITE NITROGEN AND NITRATE NITROGEN METHOD 353.2 – AUTOMATED CADMIUM REDUCTION METHOD**

SUPERCEDES: Revision 7

22.3 **DATA SYSTEM PARAMETERS FOR QUIKCHEM 8000**

The timing values listed below are approximate and will need to be optimized using graphical events programming.

Sample throughput: 55 samples/h, 65 s/sample
Pump speed: 35
Cycle Period: 65

Analyte Data:

Concentration Units: mg N/L
Peak Base Width: 25 s
% Width Tolerance: 100
Threshold: 5000
Inject to Peak Start: 22 s
Chemistry: Direct

Calibration Data:

Level	1	2	3	4	5	6	7
Concentration mg/L	2.00	0.80	0.20	0.05	0.02	0.01	0.00

Calibration Fit Type: 1st Order Polynomial
Calibration Rep. Handling: Average
Weighting Method: None
Concentration Scaling: None
Force Through Zero: No

Sampler Timing:

Min. Probe in Wash Period: 12 s
Probe in Sample Period: 32 s

Valve Timing:

Load Time: 0.0 s
Load Period: 28 s
Inject Period: 37 s

TITLE: **NITRATE+NITRITE NITROGEN, NITRITE NITROGEN AND NITRATE NITROGEN METHOD 353.2 – AUTOMATED CADMIUM REDUCTION METHOD**

SUPERCEDES: Revision 7

22.4 Analytical Run Sequence

Total Nitrate / Nitrite sequence

LCS/CCV NO2 (2.5 mg/l)
LCS/CCV NO3 (2.5 mg/l)
ICB
Sample
Sample
Sample
Sample
Sample
Sample
Sample
Sample
Sample
Sample
CCV (2.5 mg/l)
CCB
Sample
Sample
Sample
Sample
Sample
Sample
Sample
Sample
Sample
Sample duplicate
Sample spike
CCV (2.5 mg/l)
CCB

Total Nitrite sequence

LCS/CCV NO2 (1.0 mg/l)
ICB
Sample
Sample
Sample
Sample
Sample
Sample
Sample
Sample
Sample
Sample
CCV (1.0 mg/l)
CCB
Sample
Sample
Sample
Sample
Sample
Sample
Sample
Sample
Sample
Sample duplicate
Sample spike
CCV (1.0 mg/l)
CCB

TITLE: NITRATE+NITRITE NITROGEN, NITRITE NITROGEN AND NITRATE NITROGEN METHOD 353.2 – AUTOMATED CADMIUM REDUCTION METHOD

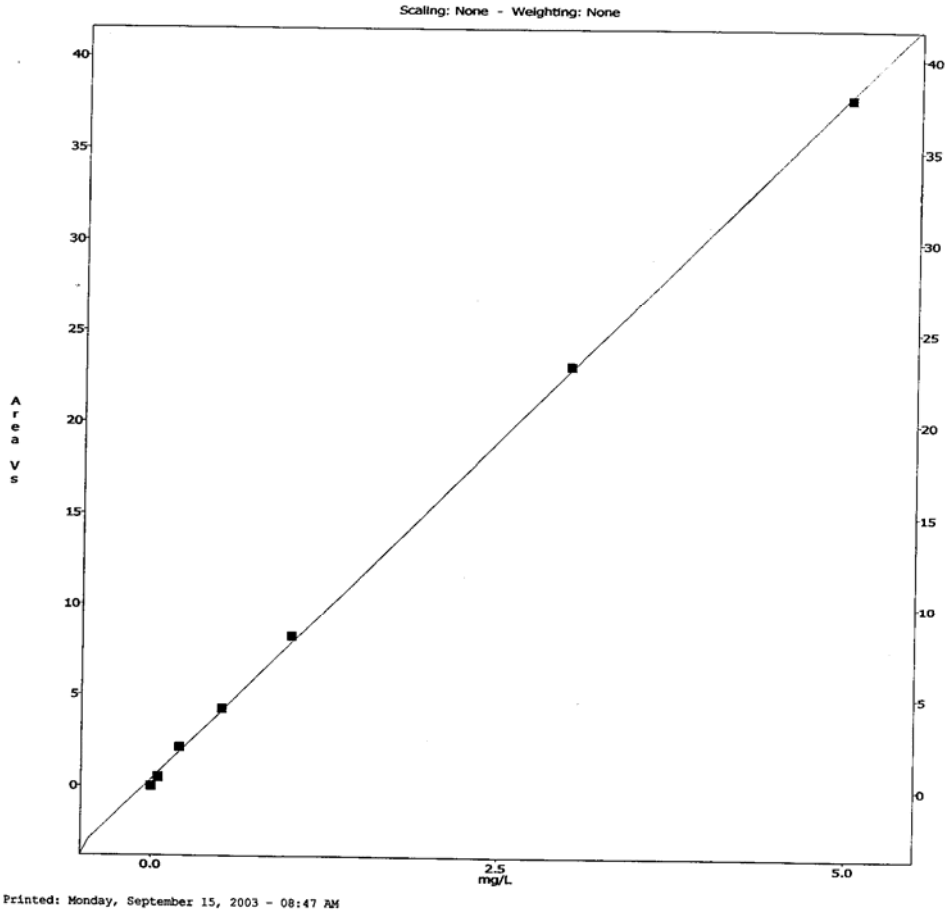
SUPERCEDES: Revision 7

22.5 Analytical Batch

NO2-NO3 Curve Date: 9/9/03

Lvl	Area	mg/L	Rep 1	Rep 2	Rep 3	Rep 4	Rep 5	Replic STD	Replic % RSD	Residual 1st Poly
1	37811432	5.00	37811432					0.0	0.0	0.5
2	23112960	3.00	23112960					0.0	0.0	-0.7
3	8258765	1.00	8258765					0.0	0.0	-4.6
4	4261991	0.50	4261991					0.0	0.0	-3.0
5	2138803	0.20	2138803					0.0	0.0	-16.4
6	479360	0.05	479360					0.0	0.0	75.4
7	0	0.00	0					0.0	0.0	

1st Order Poly
Conc = 1.329e-007 Area - 5.139e-002
r = 0.9998



Printed: Monday, September 15, 2003 - 08:47 AM

TITLE: **NITRATE+NITRITE NITROGEN, NITRITE NITROGEN AND NITRATE NITROGEN METHOD 353.2 – AUTOMATED CADMIUM REDUCTION METHOD**

SUPERCEDES: Revision 7

KAH 9/13/03

DATE M: A3B103602
START: 1230
END: 1325
METHOD: 353.2

NO2/NO3

STL-BUFFALO (NO2)
OPERATOR: katie
ACQ. TIME: Sep 13, 2003 12:33:36
DATA FILENAME: C:\OMNION\DATA\091303A.FDT
METHOD FILENAME:

Multi-Channel Table
Type: Calibration Standards
Channel Range: 1 to 8 – Cup Range: 1 to 50

Cup	Sample ID	# of Reps	NO2-NO3 (uv-s)

NO SAMPLE INFO FOR THE SELECTED CUP RANGE

Method - Ch. 2 (NO2-NO3)

CALIBRATION DATA:
Levels:
1 : 5.000 2 : 3.000 3 : 1.000 4 : 0.500
5 : 0.200 6 : 0.050 7 : 0.000

Calibration Rep Handling:	Replace
Calibration Fit Type:	1st Order Poly
Force Though Zero:	No
Weighting Method:	None
Concentration Scaling:	None

SAMPLER TIMING:

Method Cycle Period:	65.0
Min. Probe in Wash Period:	20.0
Probe in Sample Period:	32.0

*** Prep Sequence Not Enabled ***

Multi-Channel Table
Type: Unknowns
Channel Range: 2 to 2 – Cup Range: 1 to 15

Cup	Sample ID	Sampling Time	# of Reps	NO2-NO3 (mg/L)	Auto Dil Factor	NO2	NO3
1	no2 icv@2.5ppm	12:33:40	1	2.621	1.00		
2	no3 icv@2.5ppm	12:34:43	1	2.608	1.00 104%		
3	icb	12:35:47	1	-0.051	1.00 0.005		
4	a3871108	12:37:50	1	-0.051	1.00		
5	a3876001	12:38:53	1	-0.051	1.00		
6	a3876002	12:39:55	1	0.070	1.00	0	0.070
7	a3876003	12:40:58	1	-0.035	1.00		
8	a3876004	12:42:00	1	-0.028	1.00		
9	a3876101	12:43:03	1	-0.051	1.00		
10	a3876102	12:44:06	1	0.557	1.00	0	0.557
11	a3876103	12:45:08	1	0.043	1.00	0.065	0
12	a3876104	12:46:11	1	0.214	1.00	0	0.214
13	a3876105	12:47:12	1	-0.051	1.00		
14	ccv	12:48:14	1	2.608	1.00 104%		
15	ccb	12:49:15	1	-0.051	1.00 / 0.05		

NOTE: All samples tested negative for residual chlorine.

Reduction Efficiency: $\frac{2.608}{2.621} \times 100 = 100\%$

Sample mation/Re - Ch. 1 INACTIVE

TITLE: NITRATE+NITRITE NITROGEN, NITRITE NITROGEN AND NITRATE NITROGEN METHOD 353.2 – AUTOMATED CADMIUM REDUCTION METHOD

SUPERCEDES: Revision 7

STL-BUFFALO (NO2)
OPERATOR: katie
ACQ. TIME: Sep 13, 2003 12:33:36
DATA FILENAME: C:\OMNION\DATA\091303A.FDT
METHOD FILENAME:

Multi-Channel Table
Type: Unknowns
Channel Range: 1 to 8 -- Cup Range: 16 to 45

Cup	Sample ID	Sampling Time	# of Reps	NO2-NO3 (mg/L)	Auto Dil Factor	NO_2	NO_3
16	a3876106	12:50:19	1	-0.051	1.00		
17	a3876107	12:51:23	1	-0.024	1.00		
18	a3876108	12:52:26	1	0.615	1.00	0	0.615
19	a3876201	12:53:30	1	0.938	1.00	0	0.938
20	a3876202	12:54:33	1	-0.036	1.00		
21	a3876202FD	12:55:37	1	-0.025	1.00 RPD=0		
22	a3876203	12:56:40	1	-0.038	1.00		
23	a3876204	12:57:42	1	0.445	1.00	0.047	0.398
24	a3876205	12:58:45	1	0.856	1.00	0	0.856
25	a3876205spk	12:59:47	1	1.910	1.00 105% @ 1.0ppm		
26	ccv	13:00:50	1	2.610	1.00 104%		
27	ccb	13:01:52	1	-0.051	1.00 < 0.05		
28	a3876207	13:02:55	1	-0.040	1.00		
29	a3876301	13:03:56	1	0.013	1.00	0.053	0
38	ccv	13:06:12	1	2.615	1.00 105%		
39	ccb	13:07:15	1	-0.051	1.00 < 0.05		

STL Buffalo
LABORATORY STANDARD OPERATING PROCEDURES

SOP No.	Revision No.	Effective Date	Page
AWC-353.2-24	8	September 16, 2006	19 of 22

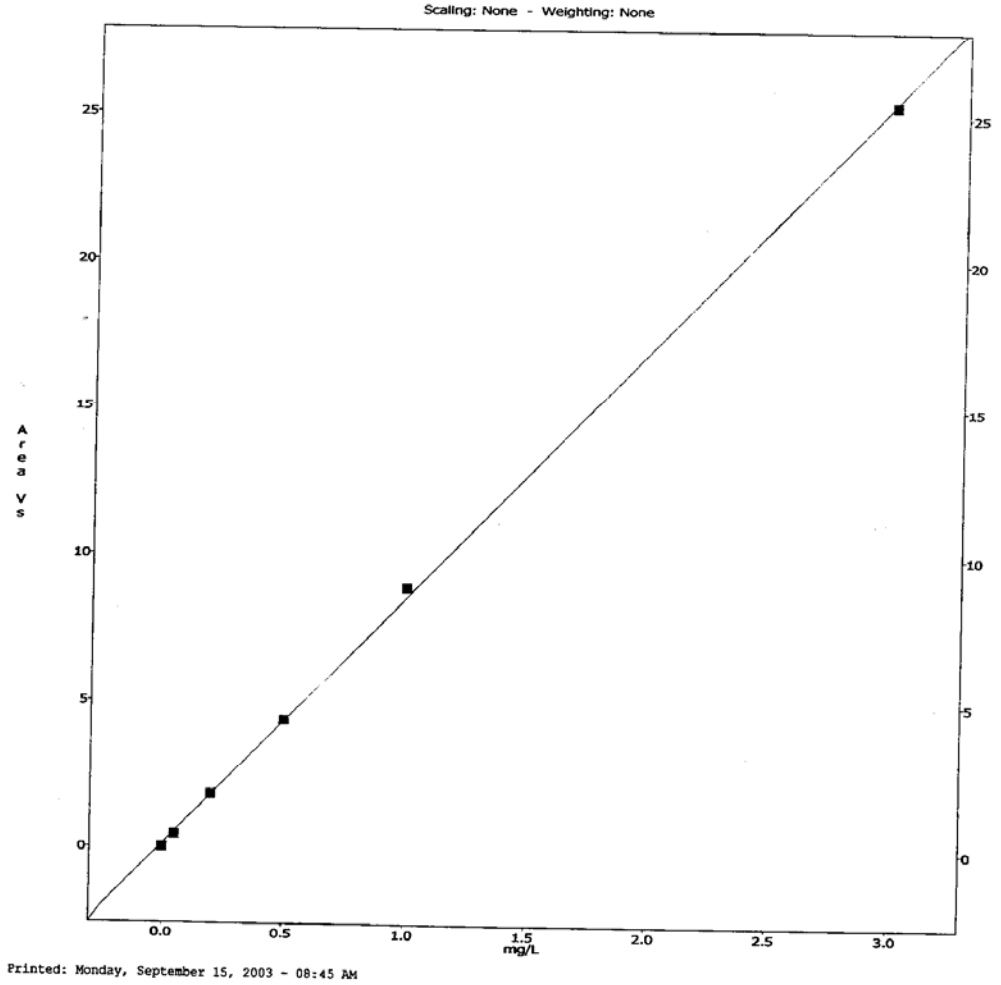
TITLE: NITRATE+NITRITE NITROGEN, NITRITE NITROGEN AND NITRATE NITROGEN METHOD 353.2 – AUTOMATED CADMIUM REDUCTION METHOD

SUPERCEDES: Revision 7

NO2 Curve Date: 9/9/03

Lvl	Area	mg/L	Rep 1	Rep 2	Rep 3	Rep 4	Rep 5	Replic STD	Replic % RSD	Residual 1st Poly
1	25375718	3.00	25375718					0.0	0.0	0.5
2	8898407	1.00	8898407					0.0	0.0	-3.8
3	4376500	0.50	4376500					0.0	0.0	-0.6
4	1820851	0.20	1820851					0.0	0.0	-0.4
5	434240	0.05	434240					0.0	0.0	26.5
6	0	0.00	0					0.0	0.0	

1st Order Poly
Conc = 1.183e-007 Area - 1.461e-002
r = 0.9998



Printed: Monday, September 15, 2003 - 08:45 AM

STL Buffalo
LABORATORY STANDARD OPERATING PROCEDURES

SOP No.	Revision No.	Effective Date	Page
AWC-353.2-24	8	September 16, 2006	20 of 22

TITLE: NITRATE+NITRITE NITROGEN, NITRITE NITROGEN AND NITRATE NITROGEN METHOD 353.2 – AUTOMATED CADMIUM REDUCTION METHOD

SUPERCEDES: Revision 7

STL-BUFFALO (NO2)
OPERATOR: katie
ACQ. TIME: Sep 13, 2003 13:09:42
DATA FILENAME: C:\OMNION\DATA\091303B.FDT
METHOD FILENAME: C:\OMNION\METHODS\NO2A.MET

NO2

Multi-Channel Table
Type: Calibration Standards
Channel Range: 1 to 8 -- Cup Range: 1 to 50

Cup	Sample ID	# of Reps	NO2 (uv-s)

NO SAMPLE INFO FOR THE SELECTED CUP RANGE

Method - Ch. 2 (NO2)

CALIBRATION DATA:
Levels:
1 : 3.000 2 : 1.000 3 : 0.500 4 : 0.200
5 : 0.050 6 : 0.000

Calibration Rep Handling:	Replace
Calibration Fit Type:	1st Order Poly
Force Though Zero:	No
Weighting Method:	None
Concentration Scaling:	None

Multi-Channel Table
Type: Unknowns
Channel Range: 2 to 2 -- Cup Range: 1 to 22

Cup	Sample ID	Sampling Time	# of Reps	NO2 (mg/L)	Auto Dil Factor
1	no2 icv@1.0ppm	13:09:46	1	1.099	1.00 110%
3	icb	13:10:49	1	-0.015	1.00 (0.05
6	a3876002	13:11:52	1	-0.003	1.00
10	a3876102	13:12:55	1	-0.015	1.00
11	a3876103	13:13:57	1	0.065	1.00
12	a3876104	13:15:00	1	-0.015	1.00
18	a3876108	13:16:03	1	-0.015	1.00
19	a3876201	13:17:06	1	-0.009	1.00
20	a3876201dup	13:18:10	1	-0.007	1.00 RPD=0
22	a3876301	13:19:13	1	0.053	1.00

STL Buffalo
LABORATORY STANDARD OPERATING PROCEDURES

SOP No.	Revision No.	Effective Date	Page
AWC-353.2-24	8	September 16, 2006	21 of 22

TITLE: NITRATE+NITRITE NITROGEN, NITRITE NITROGEN AND NITRATE NITROGEN METHOD 353.2 – AUTOMATED CADMIUM REDUCTION METHOD

SUPERCEDES: Revision 7

STL-BUFFALO (NO2)
OPERATOR: katie
ACQ. TIME: Sep 13, 2003 13:09:42
DATA FILENAME: C:\OMNION\DATA\091303B.FDT
METHOD FILENAME: C:\OMNION\METHODS\NO2A.MET

Multi-Channel Table
Type: Unknowns
Channel Range: 2 to 2 -- Cup Range: 23 to 51

Cup	Sample ID	Sampling Time	# of Reps	NO2 (mg/L)	Man Dil Factor	Auto Dil Factor
23	a3876204	13:20:16	1	0.047	1.0	1.00
24	a3876205	13:21:19	1	-0.013	1.0	1.00
25	a3876205spk	13:22:21	1	1.095	1.0	1.00 10% @ 1ppm
26	ccv	13:23:24	1	1.074	1.0	1.00 107%
27	ccb	13:24:26	1	-0.015	1.0	1.00 <0.05

SOP No.	Revision No.	Effective Date	Page
AWC-353.2-24	8	September 16, 2006	22 of 22

TITLE: NITRATE+NITRITE NITROGEN, NITRITE NITROGEN AND NITRATE NITROGEN METHOD 353.2 – AUTOMATED CADMIUM REDUCTION METHOD

SUPERCEDES: Revision 7

22.6 Wet Chemistry Batch Summary

WET CHEMISTRY BATCH SUMMARY

PARAMETER_____ METHOD_____ BATCH _____

COMMENTS	JOB NUMBER
WC Reporting Limit < STL Quant Limit	
WC Historical confirms within Hold Time	
WC Historical NO confirm & RE outside of HT	
WC Hold Time Exceedance-Dilution required	
WC Hold Time Exceedance-Instrument Failure	
WC Holding Time Exceedance by Date	
WC Holding Time Exceedance by Hours	
WC LCS high recovery, sample ND	
WC MBLK hit but samples > 10X blank value	
WC RPD Exceedance for MS / SD	
WC Spike Failure HIGH MS only	
WC Spike Failure LOW MS only	
WC Spike Failure MS and SD	
WC BOD HT met- Oxygen depleted-RE out HT	
WC Carbonate Alkalinity, LCS/MBLK	
WC Reactivity Qualification	
WC TOX Breakthrough- no volume for redo	
WC TOX samples were centrifuged	
Other	

DILUTION CODES	REASON
002	Sample matrix effects
003	Excessive foaming
004	High levels of non-target compounds
008	High concentration of target analytes
009	Sample turbidity
010	Sample color
011	Insufficient volume for lower dilution
012	Sample viscosity
013	other

ICAL Compliant? YES NO NA IF NO, Why? _____
LCS/CCV Compliant? YES NO NA IF NO, Why? _____
CCB Compliant? YES NO NA IF NO, Why? _____
RPD Compliant? YES NO NA IF NO, Why? _____
ERA Compliant? YES NO NA IF NO, Why? _____

NUMBER of REANALYSIS FOR THIS BATCH:_____

Analyst_____ Date_____

Time Critical Batch Review_____ Date_____

Secondary Review & Closure_____ Date_____

WC Summary Rev 2 / 2-2005

STL Buffalo
LABORATORY STANDARD OPERATING PROCEDURES

SOP No.	Revision No.	Effective Date	Page
AWC-365.2-55	3	April 22, 2005	1 of 15

TITLE: METHOD 365.2 – TOTAL AND ORTHO PHOSPHORUS

SUPERCEDES: Revision 2

REVIEWED & APPROVED BY:	SIGNATURE	DATE
Verl D. Preston, Quality Manager		
Christopher Spencer, Laboratory Director		
Peggy Gray-Erdmann, Supervisor		

1.0 IDENTIFICATION OF TEST METHOD

1.1 This method is taken from EPA Method 365.2 for ortho-phosphorus and HACH method for Total Phosphorus.

2.0 APPLICABLE MATRIX

2.1 This method covers the determination of specified forms of phosphorus in drinking, surface and saline waters, and domestic and industrial wastes.

3.0 REPORTING LIMIT

3.1 The reporting limit for this method is 0.01mg P/L.

4.0 SCOPE AND APPLICATION

4.1 The method is based on reactions that are specific for the orthophosphate ion, depending on the prescribed pre-treatment of the sample. The forms are defined in Section 6.

4.1.1 Except for in-depth and detailed studies, the most commonly measured forms are phosphorous and dissolved phosphorous and orthophosphate and dissolved orthophosphate. Hydrolyzable phosphorus is normally found only in sewage type samples and insoluble forms of phosphorus are determined by calculation.

TITLE: METHOD 365.2 – TOTAL AND ORTHO PHOSPHORUS

SUPERCEDES: Revision 2

5.0 SUMMARY OF THE TEST METHOD

5.1 Ammonium molybdate and antimony potassium tartrate react in an acid medium with dilute solutions of phosphorus to form a heteropoly acid-phosphomolybdic acid- that is reduced to an intensely colored molybdem blue by ascorbic acid.

5.2 Orthophosphate forms a blue color in this test. Polyphosphates (and some organic phosphorous compounds) may be converted to the orthophosphate form by sulfuric acid hydrolysis. Organic phosphorus compounds may be converted to the orthophosphate form by persulfate digestion.

6.0 DEFINITIONS

6.1 Total Phosphorus - all of the phosphorus present in the sample, regardless of form, as measured by the persulfate digestion procedure.

6.1.1 Total Orthophosphate - inorganic phosphorus [(PO4)-3] in the sample as measured by the direct colorimetric analysis procedure.

6.1.2 Total Organic Phosphorus - phosphorous in the sample measured by the persulfate digestion procedure and minus hydrolyzable phosphorus and orthophosphate.

6.2 Dissolved Phosphorus -all of the phosphorous present in the filtrate of a sample filtered through a 0.45-micron membrane filter. Membrane filters must be soaked in distilled water before use so as not to contribute significant amounts of phosphorus to samples.

6.2.1 Dissolved Orthophosphate - as measured by the direct colorimetric analysis procedure.

6.2.2 Dissolved Organic Phosphorus- as measured by the persulfate digestion procedure and minus dissolved hydrolyzable phosphorus and orthophosphate.

6.3 Standard definitions can be found in section 3.0 of the STL Buffalo laboratory Quality Manual.

7.0 INTERFERENCES

7.1 Arsenates react with the molybdate reagent to produce a blue color similar to that formed with phosphate.

7.2 Hexavalent chromium and NO interfere to give results about 3% low at concentrations of 1 mg/L and 10 to 15% low at 10 mg/L.

TITLE: METHOD 365.2 – TOTAL AND ORTHO PHOSPHORUS

SUPERCEDES: Revision 2

7.3 Sulfide and silicate do not interfere at concentrations of 1.0 and 10 mg/L.

7.4 If samples are turbid, absorbency blank may be used.

8.0 SAFETY

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

8.1 SPECIFIC SAFETY CONCERNS OR REQUIREMENTS None

8.2 PRIMARY MATERIALS USED

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

Material (1)	Hazards	Exposure Limit (2)	Signs and symptoms of exposure
Potassium Persulfate	Oxidizer	None	Causes irritation to the respiratory tract. Symptoms may include coughing, shortness of breath. Causes irritation to skin and eyes. Symptoms include redness, itching, and pain. May cause dermatitis, burns, and moderate skin necrosis.
Sodium Hydroxide	Corrosive	2 Mg/M3-Ceiling	Severe irritant. Effects from inhalation of dust or mist vary from mild irritation to serious damage of the upper respiratory tract, depending on severity of exposure. Symptoms may include sneezing, sore throat or runny nose. Contact with skin can cause irritation or severe burns and scarring with greater exposures. Causes irritation of eyes, and with greater exposures it can cause burns that may result in permanent impairment of vision even blindness.
Sulfuric Acid	Corrosive Oxidizer Dehydrator Poison Carcinogen	1 Mg/M3-TWA	Inhalation produces damaging effects on the mucous membranes and upper respiratory tract. Symptoms may include irritation of the nose and throat, and labored breathing. Symptoms of redness, pain, and severe burn can occur. Contact can cause blurred vision, redness, pain and severe tissue burns. Can cause blindness.
1 – Always add acid to water to prevent violent reactions.			
2 – Exposure limit refers to the OSHA regulatory exposure limit.			

TITLE: METHOD 365.2 – TOTAL AND ORTHO PHOSPHORUS

SUPERCEDES: Revision 2

9.0 EQUIPMENT AND SUPPLIES

9.1 Spectrophotometer suitable for measurements at 880 nm for ortho-phosphorus and 890nm for total phosphorus with a light path of 1 cm or longer.

9.2 Acid-washed glassware: all glassware used should be washed with hot 1:1 HCl and rinsed with distilled water to remove the last traces of phosphorus that might be adsorbed on the glassware. Commercial detergents should never be used.

9.3 COD reactor.

9.4 Glass test tubes for ortho-phosphorus analyses.

10.0 REAGENTS AND STANDARDS

10.1 Ortho-Phosphorus

10.1.1 Sulfuric acid solution, 5N: Dilute 70 ml of conc. H2SO4, to 500 ml distilled water.

10.1.2 Antimony potassium tartrate solution: Dissolve 1.3715g Antimony Potassium Tartrate, Trihydrate Powder in 500ml distilled water. This solution is stable for six months if stored at 4 C. Reagent can also be purchased pre-made.

10.1.3 Ammonium molybdate solution: Dissolve 20.00g Ammonium Molybdate in 500ml distilled water. This solution is stable for six months if stored at 4 C. Reagent can also be purchased pre-made.

10.1.4 Ascorbic acid, 0.1M: Dissolve 1.76g of ascorbic acid in 100ml distilled water. The solution is stable for about a week if stored at 4 C.

10.1.5 Combined Reagent, Mix the above reagents in the following proportions for 100ml of the combined reagent: 50ml of 5N H2SO4 (7.1), 5ml antimony potassium tartrate solution (7.,2), 15ml of ammonium molybdate solution (7.3), and 30ml ascorbic acid solution. Mix after addition of each reagent. Let all reagents reach room temperature before they are mixed and mix in the order given. If turbidity forms in the combined reagent, shake and let stand for a few minutes until turbidity disappears before proceeding. The reagent is stable for four hours.

10.2 Total Phosphorus

10.2.1 Total and Acid hydrolyzable test and tube reagent set purchased by HACH containing: PhosVer 3 phosphate reagent powder pillows, Potassium persulfate

powder pillows, Sodium hydroxide solution (1.54 N), and Total and acid hydrolyzed test vials.

10.3 Two different Phosphorus stock standards (1000 ppm) purchased from Ultra Scientific and SCP Science.

10.4 Standard phosphorus solutions (1000 ppm), dilute 10 ml of stock standards (see 10.3) into 1000 ml of DiH2O. One is used for the standard and the other is used for the SRM.

11.0 SAMPLE COLLECTION, PRESERVATION, SHIPMENT AND STORAGE

11.1 Sample containers may be of plastic material or of Pyrex glass.

11.2 Total phosphorus

11.2.1 If the analysis cannot be performed the day of the collection, the sample should be preserved by the addition of 2 ml conc. H2SO4 per liter and refrigeration at 4° C.

11.2.1 In accordance with EPA Methods for Chemical Analysis of Water and Wastes, CFR136, analysis is to be performed within 28 days of sample collection.

11.3 Orthophosphate

11.3.1 In accordance with EPA Methods for Chemical Analysis of Water and Wastes, CFR136, samples must be refrigerated at 4°C and analyzed within 48 hours of collection.

11.3.2 Samples must be filtered before analysis.

12.0 QUALITY CONTROL

12.1 Initial Calibration Verification (ICV) and Continuing Calibration Verification (CCV): The ICV is prepared from a source other than that used to prepare the calibration curve and is a different concentration than the CCV's. The CCVs are analyzed after every 10 samples. ICV is at 0.5 ppm and CCV is at 1.0 ppm.

12.1.1 The results of the analyses of the ICV and CCVs must be within +/- 10% of the true value. If unacceptable results are achieved, all samples analyzed since the last acceptable CCV must be re-analyzed.

12.2 An initial Calibration Blank (ICB) is analyzed after the ICV and a Continuing Calibration Blank (CCB) is analyzed with each analytical batch of 10 samples or less and is carried through the entire analytical procedure. These blanks must not exhibit total phosphorus at

concentrations greater than the reporting limit. If unacceptable results are achieved, all samples associated with the blank must be re-analyzed.

12.3 Matrix spike and Matrix Duplicate: A MS/MD is performed for each sample batch or once every 20 samples, whichever is more frequent. One of the samples in the batch is prepared in triplicate, with the second and third aliquots being the MD and MS. The MD is analyzed neat. The MS is fortified with standard phosphorus solution.(10.3.)

12.3.1 Results of the Matrix Duplicate analysis should be compared to the laboratory calculated RPD limits however, the sample matrix may affect the precision. If the results fall outside QC limits but all other QC criteria for the analytical batch have been achieved, re-analysis may not be required.

12.3.2 Results of the Matrix Spike analysis should be compared to the laboratory calculated recovery limits however; the sample matrix may affect accuracy. If results fall outside QC limits but all other QC criteria for the analytical batch have been achieved, re-analysis may not be required.

13.0 CALIBRATION AND STANDARDIZATION

13.1 A calibration curve and a blank are run every three months for Total Phosphorus. A calibration curve and a blank are run daily for ortho-phosphorus.

13.2 The instrument must be zeroed on the blank before analysis and may be rezeroed upon appearance of appreciable drift.

13.3 Prepare the calibration standards by diluting stock phosphorus solution (10.3).

TOTAL PHOSPHORUS

ml of stock phosphorus solution (10.4)	ml diH2O	Conc. mg/l
0.00	5.0	0.00
0.05	4.95	0.01
0.10	4.9	0.02
0.3	4.7	0.06
0.5	4.5	0.10
1.0	4.0	0.20
2.0	3.0	0.40
3.0	2.0	0.60
4.0	1.0	0.80
5.0	0.0	1.0

ORTHO-PHOSHORUS

ml of stock phosphorus solution (10.4)	ml diH2O	Conc. mg/l
0	10	0.00
0.10	9.9	0.01
0.20	9.8	0.02
.60	9.4	0.06
1.00	9.0	0.10
2.00	8.0	0.20
4.00	6.0	0.40
6.00	4.0	0.60
8.00	2.0	0.80
10.00	0.0	1.00

13.4 The curve coefficient must be greater than 0.995. If the value is less than 0.995, the calibration standards must be re-made and a new curve analyzed.

14.0 PROCEDURE

14.1 Total Phosphorus

14.1.1 Pipet 5.0 ml of sample into a total and hydrolyzed test vial.

14.1.2 Add the contents of one Potassium persulfate powder pillow into each vial. Cap tightly and shake to dissolve.

14.1.3 Heat vials for 30 minutes @ 150 degrees C on a pre-heated COD reactor.

14.1.4 Carefully remove the vials from the reactor and allow to cool to room temperature.

14.1.5 Pipet 2 ml of 1.54N Sodium hydroxide to each vial, cap and mix.

14.1.6 Add the contents of one PhosVer3 Phosphate reagent pillow to each vial, cap tightly and shake for 10-15 seconds.

14.1.7 Let stand for 8 to 10 minutes, then read on spectrophotometer at 890 nm.
NOTE: If sample is turbid, read on spec after 14.1.5 and record value, then continue on to remaining steps.

14.2 Orthophosphate

14.2.1 Add 10 ml of sample to test tube. Add 1.60 ml of combined reagent (10.1.5) to sample and mix thoroughly. After a minimum of ten minutes, but no longer than

thirty minutes, measure the color absorbance of each sample at 880nm with a spectrophotometer, using the reagent blank as the reference solution.

15.0 CALCULATIONS

15.1 Obtain concentration value of sample directly from prepared standard curve. Report results as P, mg/l.

15.2 When phosphate is requested as Phosphate as PO4, analyze the sample as you would total phosphorus and multiply the result by 3.065. This becomes the phosphate result.

15.3 If absorbance correction was done, take absorbance of sample minus the absorbance blank to obtain actual absorbance and then get concentration value directly from curve.

15.4 If absorbance is above the highest point on curve, a dilution may be necessary.

16.0 METHOD PERFORMANCE

16.1 Method detection limit studies are performed annually in accordance with 40 CFR, part 136, Appendix B and must demonstrate the ability to report to a detection limit of 0.01 mg P /l.

17.0 DATA ASSESSMENT AND ACCEPTANCE CRITERIA FOR QUALITY CONTROL MEASURES

17.1 Obtained ICV and CCV values must be within 90–110% of the true value.

17.2 Sample spike recovery and RPD acceptance limits are calculated annually and maintained in the LIMS system for easy reference and/or inspection.

17.3 ICB and CCB values must be less than the reporting limit.

18.0 CORRECTIVE ACTIONS FOR OUT-OF-CONTROL DATA

18.1 If acceptance criteria are exceeded for any QC element, all related samples and check standards must be repeated.

19.0 CONTINGENCIES FOR HANDLING OUT-OF-CONTROL OR UNACCEPTABLE DATA

19.1 Job exception forms are to be filled out and turned in to the appropriate project manager for final approval.

19.2 Historical data review may be used to evaluate sample results.

TITLE: METHOD 365.2 – TOTAL AND ORTHO PHOSPHORUS

SUPERCEDES: Revision 2

20.0 WASTE MANAGEMENT/ POLLUTION PREVENTION

20.1 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."

20.2 Waste Streams Produced by the Method

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

- Acidic sample waste generated by the analysis. Dispose of this waste in the "A" waste container.
- Contaminated disposable glass or plastic materials utilized in the analysis. Empty the contents of the glassware into the "A" waste and dispose of the glassware in the recycling bins located throughout the lab.

21.0 REFERENCE

21.1 Standard Methods for the Examination of Water and Wastewater, 19th Edition 4500-P

21.2 EPA Methods for Chemical Analysis of Water and Wastes, Method 365.2

22.0 TABLES, DIAGRAMS, FLOWCHARTS AND VALIDATION DATA

22.1 Analytical Run Sequence

22.2 Analytical Batch

22.3 Wet Chemistry Batch Summary

23.0 CHANGES FROM PREVIOUS REVISION

23.1 Updated Laboratory Director Name/Signature

23.2 Section 8: added specific safety information developed by STL Corporate EH&S

23.3 Section 20: added specific waste management information developed by STL corporate EH&S

23.4 Added Attachments 22.1, 22.2 and 22.3.

23.5 Updated Total Phosphorous curve concentrations

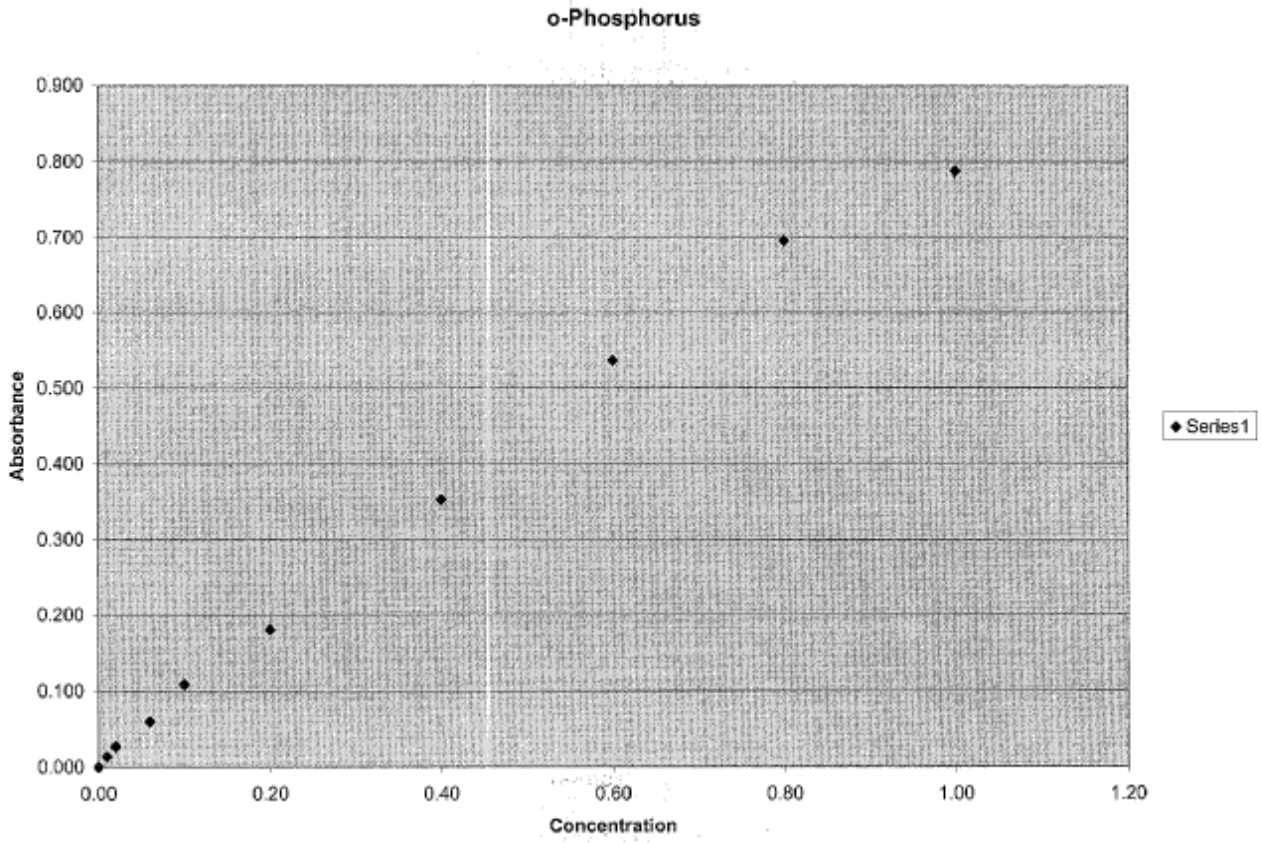
23.6 Revised 10.1.2 and 10.1.3.

23.7 Added section 15.2 to report Phosphate as PO4.

22.1 Analytical Run Sequence For Ortho and Total

ICV (.2mg/L)
ICB
Sample
Sample
Sample
Sample
Sample
Sample
Sample
Sample
Sample
Sample
CCV (.5mg/L)
CCB
Sample
Sample
Sample
Sample
Sample
Sample
Sample
Sample
Sample duplicate
Samples spike
CCV (.5mg/L)
CCB

22.2 Analytical Batch



SOP No.	Revision No.	Effective Date	Page
AWC-365.2-55	3	April 22, 2005	12 of 15

TITLE: METHOD 365.2 – TOTAL AND ORTHO PHOSPHORUS

SUPERCEDES: Revision 2

Laboratory Bench Sheet
ortho Phosphorous
Revision 1 - May 2003

STL Buffalo

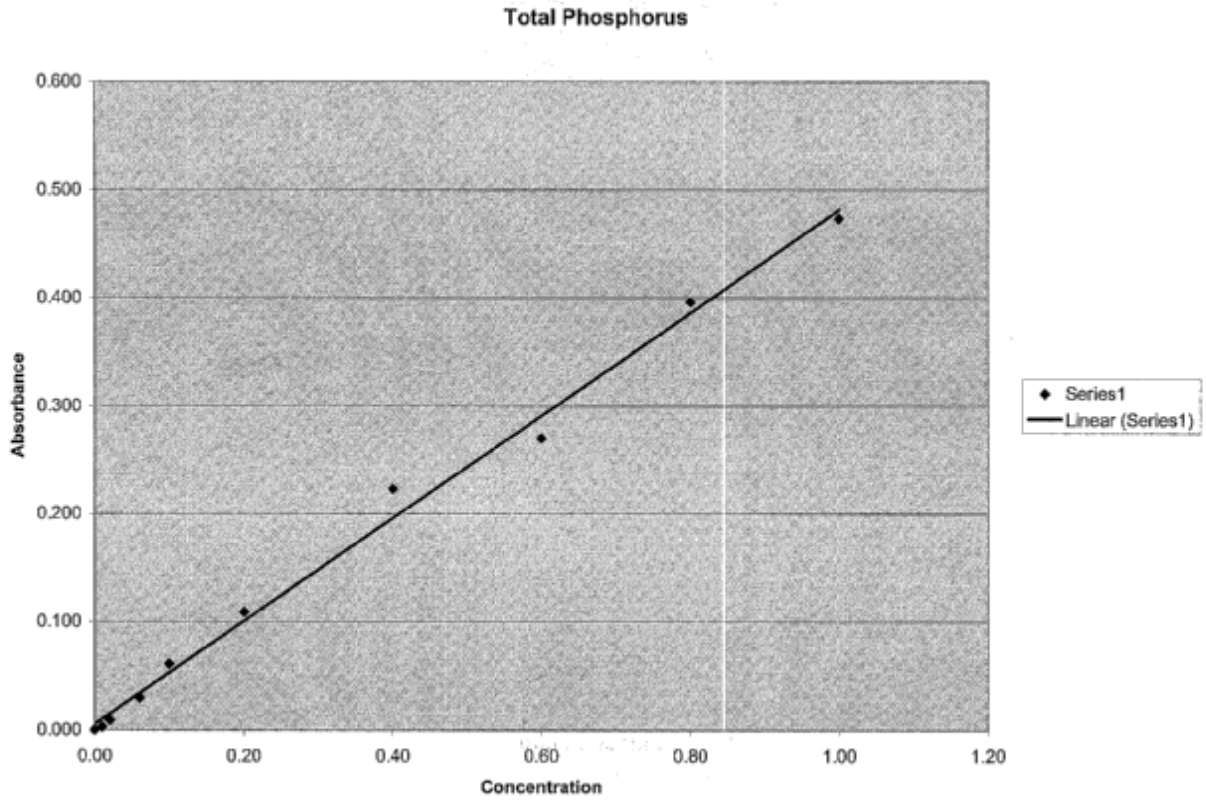
Analyst:	KW		Calibration Curve Information						BATCH:	A5B00435	
Start Date:	1/11/2005		Conc.(mg/L)		ABS.				Instrument Information		
Start Time:	18:30		STD1	0.00	0.000				Instrument:	Odyssey	
End Date:	1/11/2005		Std. 2	0.01	0.014				Wavelength:	880	
End Time:	20:20		Std. 3	0.02	0.027				Parameter:	O-Phos	
DATE OF CURVE=			Std. 4	0.06	0.060				Corr. Coef:	0.99731	
SOP Information			Std. 5	0.10	0.109				Slope:	0.81701	
Number:	AWC-365.2-55		Std. 6	0.20	0.181				Intercept:	0.01587	
Revision:	2		Std. 7	0.40	0.353						
MDL:	0.007	mg/L	Std. 8	0.60	0.537				Phos Tube source	NA	
RV:	0.010	mg/L	Std. 9	0.80	0.696				Lot#		
EQL :	0.010	mg/L	STD 10	1.00	0.788				Expiration date:		
ICV Information: TV = 75 mg/L					CCV Information: TV = 75/mg/L				Matrix Spike Information: TV = 50 mg/L		
Lot #	8-83-E				Lot #	8-83-D			Lot#:	8-83-D	
Prep Date:	01/04/05				Prep Date:	01/04/05			Prep date:	01/04/05	
Concentration (mg/L)	1 ppm				Concentration (mg/L):	1 ppm			Concentration (mg/L):	1 ppm	
Expiration Date:	07/04/05				Expiration Date:	07/04/05			Expiration Date:	07/04/05	
ICV	True value:		0.20		CCV	True value	0.50		MS	True Value	1

Job #	Sample ID	Vial #	Sample Amount (mL)	Sample ABS.	Blank ABS.	Conc. (mg/L-mg/kg)	Prep D.F.	Anal. D.F.	Final Conc. (mg/L-mg/kg)	% Rec
ICV	ICV		10.00	0.185	0.000	0.20701	1	1	0.207	104%
ICB	ICB		10.00	0.000	0.000	-0.01943	1	1	ND	
O235	A5023501		5.00	0.594	0.021	0.68191	2	1	1.364	
	2		10.00	0.809	0.093	0.85694	1	1	0.857	
	3		10.00	0.881	0.072	0.97077	1	1	0.971	
	4		5.00	0.827	0.028	0.95853	2	1	1.917	
	5		1.00	0.120	0.000	0.12745	10	1	1.274	
	6		1.00	0.390	0.000	0.45792	10	1	4.579	
	7		1.00	0.706	0.000	0.84470	10	1	8.447	
	7DUP		1.00	0.715	0.000	0.85571	10	1	8.557	
	7SPK		0.50	0.802	0.000	0.96220	10	1	9.622	
CCV	CCV		10.00	0.420	0.000	0.49464	1	1	0.495	99%
CCB	CCB		10.00	0.000	0.000	-0.01943	1	1	ND	
	2		5.00	0.517	0.034	0.57175	2	1	1.144	
	3		5.00	0.541	0.021	0.61704	2	1	1.234	
	4		2.00	0.280	0.000	0.29880	5	1	1.494	
CCV	CCV		10.00	0.423	0.000	0.49831	1	1	0.498	100%
CCB	CCB		10.00	0.006	0.000	-0.01209	1	1	ND	
							1	1		
							1	1		
							1	1		
							1	1		
							1	1		
							1	1		
							1	1		
							1	1		

SOP No.	Revision No.	Effective Date	Page
AWC-365.2-55	3	April 22, 2005	13 of 15

TITLE: METHOD 365.2 – TOTAL AND ORTHO PHOSPHORUS

SUPERCEDES: Revision 2



SOP No.	Revision No.	Effective Date	Page
AWC-365.2-55	3	April 22, 2005	14 of 15

TITLE: METHOD 365.2 – TOTAL AND ORTHO PHOSPHORUS

SUPERCEDES: Revision 2

Laboratory Bench Sheet
Total Phosphorous
Revision 1 - May 2003

STL Buffalo

Analyst:	sh	Calibration Curve Information			Reactor Temperatures			BATCH #	a5b02951	
Start Date	3-8-05		Conc.(mg/L)	ABS.				Instrument Information		
Start Time:	12:00	STD1	0.00	0.000	Reactor #1	150		Instrument:	Odyssey	
End Date:	3/8/2005	Std. 2	0.01	0.003	Reactor #2			Wavelength:	890	
End Time:	15:15	Std. 3	0.02	0.009	Reactor #3			Parameter:	Total - Phos	
DATE OF CURVE=	02/20/05	Std. 4	0.06	0.030	Reactor #4			Corr. Coef:	0.99703	
SOP Information		Std. 5	0.10	0.051				Slope:	0.47539	
Number:	AWC-365.2-55	Std. 6	0.20	0.109				Intercept:	0.00575	
Revision:	2	Std. 7	0.40	0.223						
		Std. 8	0.60	0.270				Phos Tube source	Hach	
MDL:	0.007 mg/L	Std. 9	0.80	0.396				Lot#	CHA 68E	
RV:	0.010 mg/L	STD 10	1.00	0.473				Expiration date:	07/01/09	
EQL:	0.010 mg/L									
ICV Information					CCV Information			Matrix Spike Information		
Lot #	8-83-d				Lot #	8-83-e		Lot#:	8-83-e	
Prep Date:	01/04/05				Prep Date:	01/04/05		Prep date:	01/04/05	
Concentration (mg/L)	1 ppm				Concentration (mg/L):	1 ppm		Concentration (mg/L):	1 ppm	
Expiration Date:	07/04/05				Expiration Date:	07/04/05		Expiration Date:	07/04/05	
ICV	True value:		0.20		CCV	True value	0.50	MS	True Value	0.50

Job #	Sample ID	Vial #	Sample Amt (mL)	Sample ABS.	Blank ABS.	Conc. (mg/L-mg/kg	Prep D.F.	Anal. D.F.	Final Conc. (mg/L-mg/kg)	% Rec.
ICV	ICV	1	5.00	0.103		0.20457	1	1	0.205	102%
ICB	ICB	2	5.00	0.000		-0.01209	1	1	ND	
1980	1	3	5.00	0.050		0.09308	10	1	0.931	
1982	1	4	5.00	0.041		0.07415	5	1	0.371	
1975	1	5	5.00	0.103		0.20457	20	1	4.091	
	2	6	5.00	0.085		0.16671	20	1	3.334	
	3	7	5.00	0.150		0.30343	20	1	6.069	
	4	8	5.00	0.128		0.25716	20	1	5.143	
1981	1	9	5.00	0.152		0.30764	20	1	6.153	RPD=6.65
	1dup	10	5.00	0.151		0.30554	20	1	6.111	54.6% @ 5.00
	1spk	11	5.00	0.217		0.44437	20	1	8.887	
	ccv	12	5.00	0.250		0.51379	1	1	0.514	103%
	ccb	13	5.00	0.000		-0.01209	1	1	ND	
		14	5.00				1	1	#VALUE!	
		15	5.00				1	1	#VALUE!	
		16	5.00				1	1	#VALUE!	
		17	5.00				1	1	#VALUE!	
		18	5.00				1	1	#VALUE!	
		19	5.00				1	1	#VALUE!	
		20	5.00				1	1	#VALUE!	
		21	5.00				1	1	#VALUE!	
		22	5.00				1	1	#VALUE!	
		23	5.00				1	1	#VALUE!	
		24	5.00				1	1	#VALUE!	
		25	5.00				1	1	#VALUE!	
		26	5.00				1	1	#VALUE!	
		27	5.00				1	1	#VALUE!	

Page 1 of 4

TITLE: METHOD 365.2 – TOTAL AND ORTHO PHOSPHORUS

SUPERCEDES: Revision 2

22.3 Wet Chemistry Batch Summary Sheet

WET CHEMISTRY BATCH SUMMARY

PARAMETER_____ METHOD_____ BATCH _____

COMMENTS	JOB NUMBER
WC Reporting Limit < STL Quant Limit	
WC Historical confirms within Hold Time	
WC Historical NO confirm & RE outside of HT	
WC Hold Time Exceedance-Dilution required	
WC Hold Time Exceedance-Instrument Failure	
WC Holding Time Exceedance by Date	
WC Holding Time Exceedance by Hours	
WC LCS high recovery, sample ND	
WC MBLK hit but samples > 10X blank value	
WC RPD Exceedance for MS / SD	
WC Spike Failure HIGH MS only	
WC Spike Failure LOW MS only	
WC Spike Failure MS and SD	
WC BOD HT met- Oxygen depleted-RE out HT	
WC Carbonate Alkalinity, LCS/MBLK	
WC Reactivity Qualification	
WC TOX Breakthrough- no volume for redo	
WC TOX samples were centrifuged	
Other	

DILUTION CODES	REASON
002	Sample matrix effects
003	Excessive foaming
004	High levels of non-target compounds
008	High concentration of target analytes
009	Sample turbidity
010	Sample color
011	Insufficient volume for lower dilution
012	Sample viscosity
013	other

ICAL Compliant? YES NO NA IF NO, Why? _____
LCS/CCV Compliant? YES NO NA IF NO, Why? _____
CCB Compliant? YES NO NA IF NO, Why? _____
RPD Compliant? YES NO NA IF NO, Why? _____
ERA Compliant? YES NO NA IF NO, Why? _____

NUMBER of REANALYSIS FOR THIS BATCH:_____

Analyst_____ Date_____

Time Critical Batch Review_____ Date_____

Secondary Review & Closure_____ Date_____

WC Summary Rev 2 / 2-2005

STL Buffalo
LABORATORY STANDARD OPERATING PROCEDURES

SOP No.	Revision No.	Date	Page 1 of 13
AWC-376.2-130	4	November 22, 2005	

TITLE: **SULFIDE Method 376.2**

SUPERCEDES: **Revision 3**

REVIEWED & APPROVED BY:	Signature	Date
Verl D. Preston, Quality Manager		
Christopher Spencer, Laboratory Director		
Peggy Gray-Erdmann, Supervisor		

1.0 IDENTIFICATION OF TEST METHODS

1.1 Method 376.2.

2.0 APPLICABLE MATRIX

2.1 Method is applicable to drinking, surface and saline waters domestic and industrial wastes.

3.0 REPORTING LIMIT

3.1 The reporting limit has been determined to be 0.1 mg/l.

4.0 SCOPE AND APPLICATION

4.1 This method is suitable for the measurement of total and dissolved sulfide in concentrations up to 20 mg/L

4.2 Acid insoluble sulfides are not measured by the use of this test. Copper sulfide is the only common sulfide in this class.

5.0 SUMMARY OF TEST METHOD

5.1 Sulfide reacts with dimethyl-p-phenylenediamine (p-aminodimethyl aniline) in the presence of ferric chloride to produce methylene blue, a dye, which is measured at a 625 nm.

SOP No.	Revision No.	Date	Page 2 of 13
AWC-376.2-130	4	November 22, 2005	

TITLE: **SULFIDE Method 376.2**

SUPERCEDES: **Revision 3**

6.0 DEFINITIONS

6.1 Refer to STL Buffalo Laboratory Quality Manual.

7.0 INTERFERENCES

7.1 Strong reducing agents, such as thiosulfate and sulfite, interfere by preventing formation of the blue color. Thiosulfate at concentrations about 10 mg/L may retard or prevent it.

7.2 Ferrocyanide produces a blue color

7.3 Sulfide itself prevents reaction if its concentration is very high, in the range of several hundred milligrams per liter. To avoid the possibility of false negative results, the antimony test may be used to obtain a qualitative result in industrial wastes likely to contain sulfur but giving no color by the Methylene Blue method (see *Standard Methods for the Examination of Water and Wastewater, 19th Ed.*, 1995, p 4-122).

7.4 Samples must be taken with a minimum of aeration.

7.4.1 Sulfide may be volatilized by aeration.

7.4.2 Any oxygen added to the sample may convert the sulfide to an unmeasurable form.

7.5 If the sample is not preserved with zinc acetate and sodium hydroxide, the analysis must be started immediately.

7.6 The measurement of dissolved sulfide must be commenced immediately.

8.0 SAFETY

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

8.2 *Specific Concerns or Recommendations*
Sodium Sulfide will form Hydrogen Sulfide (HS) gas if combined with water moisture or strong acids. **Inhalation of HS gas may be fatal**.

8.3 *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.

SOP No. AWC-376.2-130	Revision No. 4	Date November 22, 2005	Page 3 of 13

TITLE: **SULFIDE Method 376.2**

SUPERCEDES: **Revision 3**

Material (1)	Hazards	Exposure Limit (2)	Signs and symptoms of exposure
Hydrochloric Acid	Corrosive Poison	5 ppm-Ceiling	Inhalation of vapors can cause coughing, choking, inflammation of the nose, throat, and upper respiratory tract, and in severe cases, pulmonary edema, circulatory failure, and death. Can cause redness, pain, and severe skin burns. Vapors are irritating and may cause damage to the eyes. Contact may cause severe burns and permanent eye damage.
Sodium Hydroxide	Corrosive	2 Mg/M3-Ceiling	Severe irritant. Effects from inhalation of dust or mist vary from mild irritation to serious damage of the upper respiratory tract, depending on severity of exposure. Symptoms may include sneezing, sore throat or runny nose. Contact with skin can cause irritation or severe burns and scarring with greater exposures. Causes irritation of eyes, and with greater exposures it can cause burns that may result in permanent impairment of vision even blindness.
Sodium Sulfide	Corrosive	10 ppm-TWA 15 ppm-STEL	Will form Hydrogen Sulfide (HS) gas if combined with strong acids. **Inhalation of HS gas may be fatal**. Symptoms include painful conjunctivitis, headache, nausea, dizziness, coughing and, in extreme cases, pulmonary edema and possible death. Irritant. Contact with skin can produce serious caustic burns with painful inflammation and possible destruction of tissue. Inflammation, tearing and pain may be expected. Severe contact can cause destruction of tissue.
1 – Always add acid to water to prevent violent reactions.			
2 – Exposure limit refers to the OSHA regulatory exposure limit.			

8.3 Waste Disposal

8.3.2.2 Preserved samples and sulfide stock solutions must be disposed of in "D" waste containers.

8.4 Spill Response

8.4.1 Any spills should be cleaned as soon as possible.

8.4.2 Neutralize acid spills before cleaning.

8.5 Special Attention or Instructions

8.5.1 Sulfide solutions release noxious hydrogen sulfide gas in acidic solutions, thus it is imperative to dispose of sulfide stock solutions in "D" waste receptacles.

9.0 EQUIPMENT AND SUPPLIES

9.1 Standardization of sulfide stock

9.1.1 250-ml Erlenmeyer flasks

9.1.2 Burette

9.1.3 100-ml graduated cylinder

9.1.4 Eppendorfs calibrated for 5 ml and 200 µl

9.1.5 Transfer pipettes (disposable)

9.1.6 10-ml graduated pipette (disposable)

9.2 Colorimetric analysis

9.2.1 Spectrophotometer for use at 625 nm

9.2.2 Matched test tubes suitable for use as spectrophotometer cells

9.2.3 Pasteur pipette (disposable)

9.2.4 10-ml graduated pipettes (disposable)

9.2.5 Eppendorfs for delivering (2) 500 µl and (1) 1,600 µl

10.0 REAGENTS AND STANDARDS

10.1 Standardization of sulfide stock

10.1.1 Hydrochloric acid, 6 N

10.1.2 Starch indicator

10.1.3 Sodium thiosulfate solution, 0.0250 N

10.1.4 VWR Iodine solution, 0.025N.

10.1.5 Sulfide stock solution, 5000 µg/ml: Dissolve 6.40 g sodium sulfide in 200 ml of deionized water.

10.1.6 Second source sulfide stock solution, purchased from ERA.

10.2 Colorimetric analysis

10.2.1 Amino-sulfuric acid stock solution: Dissolve 27 g N,N-dimethyl-p-phenylenediamine oxalate (p-aminodimethylalinine) in a cold mixture of 50 ml conc. H_2SO_4 and 20 ml deionized water in a 100-ml volumetric flask. Cool and dilute to the mark with deionized water. Store in a dark glass bottle.

10.2.2 Sulfuric acid solution, H_2SO_4 1+1

10.2.3 Amino-sulfuric acid reagent: Dissolve 2.5 ml Amino-sulfuric acid stock solution (10.2.1) in 97.5 ml 1+1 H_2SO_4 (10.2.2). This solution should be clear.

10.2.4 Ferric chloride solution: Dissolve 100 g $FeCl_3 \cdot 6H_2O$ in 40 ml deionized water.

10.2.5 Diammonium hydrogen phosphate (ammonium phosphate, dibasic) solution: Dissolve 40 g $(NH_4)_2HPO_4$ in 80 ml deionized water.

11.0 SAMPLE COLLECTION, PRESERVATION, SHIPMENT AND STORAGE

11.1 Samples should be taken with a minimum of aeration.

11.1.1 Sulfide may be volatilized by aeration.

11.1.2 Any oxygen added to the sample may convert the sulfide to an unmeasurable form.

11.2 Preserve samples with zinc acetate and sodium hydroxide. Fill bottles completely and stopper. Store at 4 degrees.

11.3 Samples must be analyzed within 7 days of collection.

12.0 QUALITY CONTROL

12.1 Calibration Curve must be done every three months at a minimum. The curve is a linear regression curve.

12.2 Initial Calibration Verification (ICV): Prepare a ICV using a separate source from the calibration curve. An ICV must be run after each new curve.

12.3 Initial Calibration Blank (ICB): One blank going through the same analytical process as the samples and standards must be run after each new curve.

12.4 Laboratory Control Sample (LCS): (0.50 ppm) Prepare a calibration standard from the standardized sodium sulfide solution. Analyze one CCV after every ten sample analyses. An ERA certified standard may be use as a second source. Obtained values must be +/- 10% of the true value.

12.5 Laboratory Control Sample (LCS): (0.75 ppm) Prepare a calibration from the ERA certified standard. Analyze one ICV after ever curve in order to confirm the curve values. Obtained values must be +/-10% of its true value.

12.3 Method Blank (MBLK): To determine freedom from contamination, analyze one method blank at the beginning and end of the analytical batch and after every ten samples. A method blank consists of reagent water carried through the entire analytical procedure.

12.5.1 All blanks associated with DOD QSM and AFCEE samples must be less than half of the reporting limit.

12.6 Sample Duplicate (MD): Sample duplicates should be analyzed at least once for every twenty samples or less.

12.5 Sample Spike (MS): Sample spikes must be analyzed every twenty samples or less. Samples must fall within the established range that is calculated yearly and available in AIMS. Sample results associated with a spike recovery that fails should be evaluated to se if reanalysis is required.

12.6 Due to the instability of sulfide solutions LCS and sample spikes should be prepared from freshly standardized sodium sulfide stock solution (10.1.5)

13.0 CALIBRATION AND STANDARDIZATION

13.1 Curve standards: Prepare seven (7) curve standards in the range from 0.10 ppm to 1.00 ppm from standardized sodium sulfite (0.10ppm, 0.25 ppm, 0.40 ppm, 0.50 ppm, 0.65 ppm, 0.80 ppm, 1.00 ppm). Also, prepare a blank. Analyze curve standards according to the procedure herein.

13.2 A calibration curve must be run at a minimum every three (3) months.

13.3 Curve correlation (r) must be 0.995 or better.

14.0 PROCEDURE

14.1 Standardization of Sulfide Solution: The sulfide stock is standardized every time analysis is done. The standardization identified the concentration of the sulfide stock in order to calculate the amount of sulfide required to make the LCS concentrations of .75 and .50 ppm.

14.1.1 Add 5.0ml of I_2 into a 250ml flask
14.1.2 Add 5 ml of 6N HCl
14.1.3 Add 0.200 ml of Na_2S below surface of I_2.
14.1.4 Dilute to approximately 100 ml with reagent water.
14.1.5 Add dropper of starch indicator.
14.1.6 Titrate until blue solution turns clear.

14.2 Color Development:

14.2.1 Transfer 7.5 ml of sample to each of two matched test tubes (A and B) using a wide mouthed pipette.

14.2.2 To tube A add 0.5 ml amino-sulfuric acid reagent (10.2.3) and 3 drops (0.15 ml) $FeCl_3$ solution (10.2.4).

14.2.3 Mix immediately by slowly inverting the tube only once.

14.2.4 To tube B add 0.5 ml 1+1 H_2SO_4 (10.2.2) and 3 drops (0.15 ml) $FeCl_3$ solution (10.2.4) and mix.

14.2.5 Color will develop in tube A in the presence of sulfide. Color development is usually complete in about one minute, but a longer time is often required for the fading of the initial pink color.

14.2.6 Wait 3 to 5 minutes.

14.2.7 Add 1.6 ml $(NH_4)_2HPO_4$ solution (10.2.5) to each tube.

14.2.8 Mix immediately by slowly inverting the tube only once

14.2.9 Wait 3 to 5 minutes and make color comparisons. If Zinc acetate was used as a preservative, wait at least 10 minutes**.**

14.3 Photometric Color Comparison

14.3.1 Set spectrophotometer to 625 nm.

14.3.2 Zero instrument on tube B.

14.3.3 Read absorbance of tube A, and compare with calibration curve.

SOP No.	Revision No.	Date	Page 8 of 13
AWC-376.2-130	4	November 22, 2005	

TITLE: **SULFIDE Method 376.2**

SUPERCEDES: **Revision 3**

15.0 CALCULATIONS

15.1 Standardization of sulfide stock (10.1.5) in order to find the amount of sulfide required to make the LCS concentrations.

mg/l sulfide = [(A x B) - (C x D)] x 16,000 / E

Where:

A = Volume of iodine (ml)
B = Normality of iodine
C = Volume of $Na_2S_2O_3$
D = Normality of $Na_2S_2O_3$
E = Volume of sulfide stock

16.0 METHOD PERFORMANCE

16.1 The method detection limit (MDL) is to be performed on an annual basis in accordance with the specifications in 40 CFR 136, appendix B, and must demonstrate the ability to report a detection limit below 0.10 mg/l.

17.0 DATA ASSESSMENT AND ACCEPTANCE CRITERIA FOR QUALITY CONTROL MEASURES

17.1 ICAL: Correlation Coefficient > 0.995

17.2 LCS: Obtained values must be within 90 - 110% of the true value.

17.3 Method Blank: Detections < PQL

17.4 All duplicate samples must have RPD values less than 20%

17.5 All sample spike values are calculated yearly and available in AIMS.

18.0 CORRECTIVE ACTIONS FOR OUT-OF-CONTROL DATA

18.1 If acceptance criteria are exceeded, all related sample analyses must be repeated.

19.0 CONTINGENCIES FOR HANDLING OUT-OF-CONTROL OR UNACCEPTABLE DATA

19.1 Job exception forms are filled out and turned into the project managers for client input.

SOP No.	Revision No.	Date	Page 9 of 13
AWC-376.2-130	4	November 22, 2005	

TITLE: **SULFIDE Method 376.2**

SUPERCEDES: **Revision 3**

20.0 WASTE MANAGEMENT/ POLLUTION PREVENTION

20.1. 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."

20.2. Waste Streams Produced by the Method

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

Aqueous waste generated during procedure is disposed of in the "A" waste containers.

Preserved samples and sulfide stock solutions must be disposed of in "D" waste containers.

21.0 REFERENCE

21.1 Methods for Chemical Analysis of Water and Wastes, U.S. Environmental Protection Agency, 376.2-1.

21.2 Standard Methods for the Examination of Water and Wastewater, 19th Edition, 4500-S2-, pp 4-122 -- 4-125.

22.0 TABLES, DIAGRAMS, FLOWCHARTS AND VALIDATION DATA

22.1 Analytical Sequence

22.2 Analytical Run log

22.3 Wet Chemistry Batch Summary

23.0 CHANGES FROM PREVIOUS REVISION

23.1 Updated Attachments 22.1, 22.2, 22.3

23.2 Laboratory Director change, signature updated.

23.3 Updated terminology in section 12.0

23.4 Section 17.0: Acceptance criteria for ICAL and method blank added

23.5 Section 9.0: Designated disposable equipment

22.1 Analytical Sequence

LCS
MBLK
Sample
Sample
Sample
Sample
Sample
Sample
Sample
Sample
Sample Duplicate
Sample Spike
LCS
MBLK

SOP No. AWC-376.2-130	Revision No. 4	Date November 22, 2005	Page 11 of 13

TITLE: **SULFIDE Method 376.2**

SUPERCEDES: **Revision 3**

22.2 Analytical Run Log

Laboratory Bench Sheet
SULFIDE
Method 376.2
Revision 0 - Nov. 2005

STL Buffalo

Analyst:		**Curve Information**			**Sulfide Stock Standardization**			**BATCH #**	
Start Date:		Conc.(mg/L)		ABS.	Standard	Normality	Volume (mls	**Instrument Information**	
Start Time		STD1	0	0.021				Instrument:	Odyssey
		Std. 2	0.1	0.086	Iodine	0.025	5	Wavelength:	625
		Std. 3	0.25	0.209	Na2SO	0.025	2.7	Parameter:	Sulfide
DATE OF CURVE=	10/30/05	Std. 4	0.4	0.311	Sulfide Stock		0.2	**Corr. Coef:**	**0.99941**
EQL: 0.100	mg/L	Std. 5	0.5	0.399				**Slope:**	**0.79189**
		Std. 6	0.65	0.520	S2 Standardization mg/l =		4600	**Intercept:**	**0.00838**
		Std. 7	0.8	0.637					
		Std 8	1	0.814					

Solutions:	
Amino sulfuric acid Stock Solution	
Amino sulfuric acid Stock	
Ferric Chloride Solution	
Sulfuric Acid 1+1 Solution	

Solutions:	
Iodine	
Sulfide Stock	
NaSO4	
HCL 1+1 Solution	
Starch	

LCS Information:		**LCS Information:**		**Matrix Spike Information:**		
Solution #		Solution #		Solution #		
Concentration (mg/l	0.75	Concentration (mg/L):	0.50	Concentration (mg/L):		0.50
ICV True Value	0.75	CCV True Value	0.50	MS	True Value	0.50

Job #	Sample ID	Sample Amount (ml)	Sample ABS.	Conc. (mg/L-mg/kg)	Dilution	Final Conc. (mg/L-mg/kg)	% Rec.
	LCS	7.5		-0.011	1	-0.011	-1.4%
	MBLK	7.5		-0.011	1	-0.011	
		7.5			1	#VALUE!	
		7.5			1	#VALUE!	
		7.5			1	#VALUE!	
		7.5			1	#VALUE!	
		7.5			1	#VALUE!	
		7.5			1	#VALUE!	
		7.5			1	#VALUE!	
		7.5			1	#VALUE!	
		7.5			1	#VALUE!	
		7.5			1	#VALUE!	
		7.5			1	#VALUE!	
		7.5			1	#VALUE!	
	LCS	7.5		-0.011	1	-0.011	-2.1%
	MBLK	7.5		-0.011	1	-0.011	
		7.5			1	#VALUE!	
		7.5			1	#VALUE!	
		7.5			1	#VALUE!	
		7.5			1	#VALUE!	
		7.5			1	#VALUE!	
		7.5			1	#VALUE!	
		7.5			1	#VALUE!	
		7.5			1	#VALUE!	
		7.5			1	#VALUE!	
		7.5			1	#VALUE!	
	LCS	7.5		-0.011	1	-0.011	-2.1%
	MBLK	7.5		-0.011	1	-0.011	

Page 1 of 1

SOP No. AWC-376.2-130	Revision No. 4	Date November 22, 2005	Page 12 of 13

TITLE: SULFIDE Method 376.2

SUPERCEDES: Revision 3

22.3 Wet Chemistry Batch Summary Sheet

WET CHEMISTRY BATCH SUMMARY

PARAMETER_____ **METHOD**_____ **BATCH** _____

COMMENTS	JOB NUMBER
WC Reporting Limit < STL Quant Limit	
WC Historical confirms within Hold Time	
WC Historical NO confirm & RE outside of HT	
WC Hold Time Exceedance-Dilution required	
WC Hold Time Exceedance-Instrument Failure	
WC Holding Time Exceedance by Date	
WC Holding Time Exceedance by Hours	
WC LCS within ERA limits outside internal	
WC LCS high recovery, sample ND	
WC MBLK hit but samples > 10X blank value	
WC RPD Exceedance for MS / SD	
WC Spike Failure HIGH MS only	
WC Spike Failure LOW MS only	
WC Spike Failure MS and SD	
WC BOD HT met- Oxygen depleted-RE out HT	
WC Carbonate Alkalinity, LCS/MBLK	
WC Reactivity Qualification	
WC TDS/Conductivity ratio outside of range	
WC TOX Breakthrough- no volume for redo	
WC TOX samples were centrifuged	
Other	

DILUTION CODES	REASON
002	Sample matrix effects
003	Excessive foaming
004	High levels of non-target compounds
008	High concentration of target analytes
009	Sample turbidity
010	Sample color
011	Insufficient volume for lower dilution
012	Sample viscosity
013	other

ICAL Compliant? YES NO NA IF NO, Why? _____
LCS/CCV Compliant? YES NO NA IF NO, Why? _____
CCB Compliant? YES NO NA IF NO, Why? _____
RPD Compliant? YES NO NA IF NO, Why? _____
ERA Compliant? YES NO NA IF NO, Why? _____

NUMBER of REANALYSIS FOR THIS BATCH:_____

Analyst_____ Date_____

Time Critical Batch Review_____ Date_____

Secondary Review & Closure_____ Date_____ WC Summary Rev 4 / 5-2005

SOP No.	Revision No.	Date	Page
AWC-IC-05	10	July 31, 2006	1 of 22

TITLE: Ion Chromatography: Methods 300.0 / 9056 / SM 4110B

SUPERCEDES: Revision 9

REVIEWED & APPROVED BY:	Signature	Date
Christopher Spencer, Laboratory Director		
Verl D. Preston, Quality Manager		
Peggy Gray-Erdmann, Supervisor		

1.0 IDENTIFICATION OF TEST METHODS

1.1. This method is taken from EPA method 300.0 and 9056 and Standard Methods for the Examination of Water and Wastewater method 4110 B.

2.0 APPLICABLE MATRIX

2.1. This method is applicable to surface water, groundwater, wastewater, drinking waters and soils.

3.0 REPORTING LIMIT

3.1. The reporting limits for each anion are listed below

3.1.1. Fluoride 0.05 ppm.

3.1.2. Chloride 0.5 ppm.

3.1.3. Bromide 0.2 ppm.

3.1.4. Nitrate 0.05 ppm.

3.1.5. Sulfate 2.0 ppm.

3.2. MDLs are calculated every six months in accordance with method specification and kept on file with the QA department.

SOP No.	Revision No.	Date	Page
AWC-IC-05	10	July 31, 2006	2 of 22

TITLE: Ion Chromatography: Methods 300.0 / 9056 / SM 4110B

SUPERCEDES: Revision 9

4.0 SCOPE AND APPLICATION

4.1. Ion Chromatography provides a single instrumental technique that may be used for the measurement in water samples of common anions such as bromide, chloride, fluoride, nitrate, and sulfate.

4.2. This procedure can also be applied to soil samples (14.2.2).

5.0 SUMMARY OF TEST METHOD

5.1. A filtered aqueous sample is injected into an ion chromatograph with the use of an automated sampler. The sample merges with an eluent stream and is pumped through the system. The ion exchanger separates the anions of interest. Ions are separated based on their affinity for the exchange sites of the resin. The separated anions in their acid form are measured using an electrical conductivity cell. Anions are identified based on their retention times compared to known standards. Quantitation is accomplished by measuring the peak height or area and comparing it to a calibration curve generated from known standards.

5.2 An extraction procedure must be performed to use this method for soils (14.2.2),

6.0 DEFINITIONS

6.1. Standard definitions can be found in section 3.0 of the STL Buffalo Laboratory Quality Manual.

7.0 INTERFERENCES

7.1. Interferences can be caused by substances with retention times that are similar to and overlap those of the anion of interest. Anions of high concentrations can interfere with the peak resolution of an adjacent anion. Overlap can be minimized by diluting the sample.

7.2. Method interferences may be caused by contaminants in the reagent water, reagents, glassware and other sample processing apparatus that lead to discrete artifacts or an elevated baseline in the ion chromatograms.

7.3.1 All samples must be pre-filtered through a 0.2um filter before injection. If particles contaminate the guard or analytical columns, follow the manufacturer's suggestions for cleaning, or simply replace the column.

8.0 SAFETY

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

8.2. SPECIFIC SAFETY CONCERNS OR REQUIREMENTS

8.2.1. Sodium Fluoride is Highly Toxic.

8.2.2. Exercise caution when using syringes with attached filter assemblies. Application of excessive force has, upon occasion, caused a filter disc to burst during the process.

SOP No.	Revision No.	Date	Page
AWC-IC-05	10	July 31, 2006	3 of 22

TITLE: Ion Chromatography: Methods 300.0 / 9056 / SM 4110B

SUPERCEDES: Revision 9

9.0 EQUIPMENT AND SUPPLIES

9.1. Ion chromatograph complete with all required accessories:

9.1.1. Anion separator column capable of resolving bromide, chloride, fluoride, nitrate, and sulfate.

9.1.2. Guard column to protect the separator column from fouling by particles.

9.1.3. Conductivity detector with temperature control and separate working and reference electrodes.

9.1.4. Pump able to deliver 1.2 ml/min of constant flow rate.

9.1.5. Data collection and analysis system.

9.1.6. Automated sampler.

9.1.7. Reagent Fee Controller

9.1.8. Column Temperature Stabilizer

9.1.9. Carbonate Removal Device (4mm)

9.2. Various laboratory glassware such as Class A graduated cylinders, syringes, volumetric flasks and pipettes.

9.3. 10 ml syringes and 0.2 um syringe filters for colored samples

9.4. Analytical balance, capable of weighing to the nearest 0.0001g.

9.5. Filter caps for clean samples purchased from Dionex

9.6. 5 ml sample vials purchased from Dionex

10.0 REAGENTS AND STANDARDS

10.1. Sample bottles: Glass or polyethylene bottles of sufficient volume to allow replicate analyses of anions of interest.

10.2. Reagent water: Distilled or deionized water free of the anions of interest. Water should contain particles no larger than 0.20 microns.

10.3. Eluent Concentrate (0.17 M $NaHCO_3$, 0.18 M Na_2CO_3): dissolve 18.5475 g Sodium Carbonate and 4.20 g Sodium Bicarbonate in reagent water and dilute to 500 ml.

10.3.1. Eluent Solution: Dilute 10.0 ml of the Eluent Concentrate (10.3) to 1 liter with reagent water or 100 ml to 10 L.

10.4 Elugen Cartridge Potassium Hydroxide purchased from Dionex.

TITLE: Ion Chromatography: Methods 300.0 / 9056 / SM 4110B

SUPERCEDES: Revision 9

10.5 Multi Element Ion Chromatography (IC) Standards purchased from AccuStandard (F 100mg/l, Cl l000mg/l, Br 400mg/l, NO3 100mg/l, SO4 1000 mg/l). One standard source is used for the calibration curve (10.6) and one standard source is used for the ICV/ CCV/LCS Solutions.

10.6 Multi Element Ion Chromatography (IC) Standards purchased from Ultra Scientific (F 100mg/l, Cl l000mg/l, Br 400mg/l, NO3 100mg/l, SO4 1000 mg/l). One standard source is used for the calibration curve (10.6) and one standard source is used for the ICV/ CCV/LCS Solutions.

10.7 Individual Anion Standards are used for Matrix Spikes. All standards are purchased from various vendors. They are purchased at the following concentrations: Fluoride at a concentration of 100 mg/l, Chloride at a concentration of 1000 mg/l, Bromide at a concentration of 1000 mg/l. and Sulfate at a concentration of 1000 mg/l. The Nitrate is a custom made mix at a concentration of 100 mg/l.

10.8 Calibration standards: all are made from dilutions of the Multi Element IC Standards in reagent water. (If the Ultra Scientific IC standard is used for the calibration curve, the Accustandard IC standard is used for the CCV/LCS Solutions.)

10.8.1 Prepare the calibration standards for a 7-point curve by measuring the following volumes into a 100 ml Class A volumetric. Bring to the final volume of 100 ml with eluent.

	Level 1	Level 2	Level 3	level 4	level 5	level 6	level 7
Stock solution (See 10.5/10.6 for concentrations)	0	50ul	100ul	500ul	2ml	5ml	5ml
Final Volume	100ml	100ml	100ml	100ml	100ml	100ml	50ml

10.8.2 The final concentrations of each anion in the 7 calibration points are summarized below.

	Level 1 (mg/L)	Level 2 (mg/L)	Level 3 (mg/L)	Level 4 (mg/L)	Level 5 (mg/L)	Level 6 (mg/L)	Level 7 (mg/L)
Fl-	0	0.05	0.1	0.5	2	5	10
Cl-	0	0.5	1	5	20	50	100
Br-	0	0.2	0.4	2	8	20	40
NO3-	0	0.05	0.1	0.5	2	5	10
SO4-	0	0.5	1	5	20	50	100

SOP No.	Revision No.	Date	Page
AWC-IC-05	10	July 31, 2006	5 of 22

TITLE: Ion Chromatography: Methods 300.0 / 9056 / SM 4110B

SUPERCEDES: Revision 9

10.9 ICV lower and LCS lower solution: 10ml of the Multi Element IC Standard diluted to 1000ml with eluent. The final concentration for each anion in the LCS Lower is as follows:

INSTRUMENT #1	ICV /LCS LOWER
FL-	1.0 mg/L
Cl-	10 mg/l
Br-	4.0 mg/l
NO3-	1.0 mg/l
SO4-	10 mg/l

10.10 LCS upper and LCS upper solution: 80ml of Multi Element IC Standard diluted to 1000ml with reagent water. The final concentration for each anion in the LCS Upper is as follows:

INSTRUMENT #1	ICV /LCS UPPER
FL-	8.0 mg/l
Cl-	80 mg/l
Br-	32 mg/l
NO3-	8.0 mg/l
SO4-	80 mg/l

10.11 ICV and LCS solution for use with the RFC on instrument #2: 20ml of Multi Element IC Standard diluted to 1000ml with reagent water. The final concentration for each anion in the LCS is as follows:

INSTRUMENT #2 RFC	ICV /LCS RFC
Fl_	2.0 mg/l
Cl-	20 mg/l
Br-	8.0 mg/l
NO3-	2.0 mg/l
SO4-	20 mg/l

10.12 Matrix spikes are prepared using the Multi Element Ion Chromatography Standards (section 10.7). Prepare matrix spikes at the following concentrations

Analyte	Conc.	Volume
Bromide	10 mg/l	0.05 ml of IC 1000mg/l STD (sect. 10.7) to a final volume of 5 mls of sample
Chloride	25 mg/l	0.125 ml of IC 1000mg/l STD (sect. 10.7) to a final volume of 5 mls of sample
Sulfate	25 mg/l	0.125 ml of IC 1000 mg/l STD (sect. 10.7) to a final volume of 5 mls of sample
Fluoride	2.5mg/l	0.0125 ml of IC 100 mg/l STD (sect. 10.7) to a final volume of 5 mls of sample
Nitrate	2.5 mg/l	0.125 ml of IC 100 mg/l Custom Mix (sect. 10.7) to a final volume of 5 mls of sample

SOP No.	Revision No.	Date	Page
AWC-IC-05	10	July 31, 2006	6 of 22

TITLE: Ion Chromatography: Methods 300.0 / 9056 / SM 4110B

SUPERCEDES: Revision 9

11.0 SAMPLE COLLECTION, PRESERVATION, SHIPMENT AND STORAGE

11.1. Samples should be shipped and stored in plastic bottles at 4 ± 2 degrees C. Samples should be analyzed for fluoride, chloride, bromide and sulfate within 28 days of collection. Samples should be analyzed for nitrate within 48 hours of collection.

11.2. Soil Leachates will follow the same preservation and holding times as the water samples, starting from the time of extraction.

12.0 QUALITY CONTROL

12.1. Before analyzing samples, the laboratory must establish a method detection limit (MDL). The MDL is repeated every six months.

12.2. Each group of sample analyses must be bracketed by an acceptable calibration verification sample and calibration blank. All quality control data should be maintained and available for easy reference or inspection.

12.3. Initial and Continuing Calibration Blank (ICB, CCB): To determine freedom from contamination, prepare one calibration blank (ICB) at the beginning of the analytical procedure and another (CCB) after every ten samples and at the end of the analytical procedure. The blank consists of 5 ml reagent water that gets the same treatment as the samples and standards. The blanks must be free of the analytes of concern at levels less than the STL Buffalo quantitation limit.

12.3.1. All blanks associated with USACE samples should be less than half the STL Buffalo quantitation limit for each anion.

12.4. Continuing Calibration Verification/Laboratory Control Sample (CCV/LCS): Prepare a lower and an upper CCV/LCS at the beginning of the analytical procedure and additional lower and upper CCV/LCS after every ten samples and again at the end of the procedure. The recovery of the CCV/LCS must be within 90-110% of the true value.

12.5. Sample Duplicate:

12.5.1. Method 300.0 - must be run for every batch of twenty or fewer samples

12.5.2. Method 9056 – must be run for every group of ten samples

12.6. Sample spikes are to be run after every 10 or fewer samples. Deviations may occur due to specific client, state, or protocol requirements.

12.7. The Retention Time shift in each CCV must be within ± 10% of the beginning daily CCV check.

SOP No.	Revision No.	Date	Page
AWC-IC-05	10	July 31, 2006	7 of 22

TITLE: Ion Chromatography: Methods 300.0 / 9056 / SM 4110B

SUPERCEDES: Revision 9

13.0 CALIBRATION AND STANDARDIZATION

13.1. Prepare standard curve by plotting instrument response against concentration values.

13.1.1. The curve is a cubic curve for instrument #1 and a linear curve for instrument #2 (RFC).

13.1.2. A calibration curve may be fitted to the calibration solution concentration/response data using the computer.

Acceptance or control limits should be established using the difference between the measured values of the calibration solution and the "true value" concentration. The recovery of the CCV/LCS must be within 90-110% of the true value.

13.1.3. Acceptance criteria for the calibration curve is a correlation coefficient (R value) $\geq$0.995. If the R-value is less than 0.995, the calibration standards must be remade and a new curve analyzed.

13.1.4. New calibration curves must be run at a minimum of once every three months.

13.1.5. The curves should each consist of at least six different concentrations for each anion to be measured and a blank.

13.2. Initial Calibration Verification Solutions (ICV Lower and ICV Upper), prepared from a different standard source, are analyzed immediately after the calibration curve to verify the accuracy of the curve. The recovery of the ICV must be within 90-110%.

14.0 PROCEDURE

14.1. System Equilibrium:

14.1.1. Set up the ion chromatograph as specified in the manufacturer's instructions.

14.1.2 Turn on and prime the pump.

14.1.3 Adjust the eluent flow rate to 1.2 $\pm$ 0.1 ml/min. for instrument #1 and 1.0 $\pm$ 0.1 ml/min. for instrument #2 RFC.

14.1.4 Allow the system to come to equilibrium (15-20 minutes). A stable baseline indicates system equilibrium.

14.2. Sample analysis:

14.2.1. For dirty samples filter sample through a pre-washed 0.2um pore diameter membrane filter. If sample is clean no filtration is required.

14.2.2. For soil samples the following extraction should be used. Add 5 grams of soil sample to 50ml of deionized water. This slurry is mixed for ten minutes using a stirring device. Filter the resulting slurry using a 0.45µ membrane filter. Once filtered this sample is ready to be loaded onto the autosampler.

14.2.3. Fill autosampler vials with the sample to the fill line marked on the vial body (approximately 5 ml). Place vial cap into vial.

14.2.4. Place the filled vial into the sampler cassette and fully insert the cap using the insertion tool.

14.2.5. Place the filled cassettes into the automated sampler and start the run.

14.2.6. Check data for any needed dilutions and calculate percent recovery of check standards and sample spikes. Any data from samples that were diluted will have to be multiplied by the dilution factor before reporting.

14.3 Retention time (migration time) is the expected time retention time or migration time in minutes for the component. If the retention time is unknown, enter any number greater then zero. The correct retention time can be determined later from the first calibration run, and the component table then updated. In subsequent calibrations, PeakNet will automatically update the retention time. The Update Retention Time setting must be selected in the calibration Parameters dialog box.

15.0 CALCULATIONS

15.1. Using the computer and software packages, prepare a cubic calibration curve for each analyte by plotting instrument response against standard concentration. Compute sample concentration by comparing sample response with the standard curve. The response factor produced from a cubic equation best fits a detector's third order response. The equation used is shown below.

$$Y = K_0 + K_1 \times X + K_2 \times X^2 + K_3 \times X^3$$

At least four points are needed to fit the equation: thus, the calibration must have at least four levels for all components.

The following values, used to calculate component amount, are determined automatically by the Method Editor and cannot be edited.

X= area

K_0 indicates the Y intercept of the calibration curve.

K_1 is the coefficient for the first-degree variable. When the fit type is linear, K1 indicates the slope of the calibration curve for the selected calibration level.

K_2 is the coefficient for the second-degree variable in a quadratic equation.

K_3 is the coefficient for the third-degree variable in a cubic fit.

The equation for the calibration curve fit used to calculate the component amount is displayed at the bottom of the replicate page. The r2 value (Coefficient of Determination) for the component is shown at the bottom of the replicate page.

15.2. The analyst corrects the results for and dilution factors:

$$Xf = Xj * \text{Dilution Factor}$$

Where:
Xf = Final sample concentration
Xj = calculated concentration of sample at instrument

15.3. Report only those values that are less than the highest calibration standards. Samples exceeding the highest standard should be diluted and reanalyzed.

15.4 For solid samples, the result is expressed as mg/kg on a dry basis.

15.4.1 To convert the mg/l result obtained from the calibration curve to mg/kg use the following equation:

mg/kg (wet) = [mg/l X final vol. of leached sample] / grams sample used

mg/kg (dry) = mg/kg (wet) / decimal dry weight

15.5 **Percent Recovery for Analyses Involving Spikes:**

$$\%\,\text{Recovery} = \left[\frac{(SSR - SR)}{SA}\right] \text{ x } 100$$

where:
SSR = spiked sample result
SR = sample result
SA = spike added

15.6 **Relative Percent Difference (RPD):**

$$RPD = \frac{|x_1 - x_2|}{\left(\frac{x_1 + x_2}{2}\right)} \text{ x } 100$$

where:
x_1 = analytical % recovery
x_2 = replicate % recovery

15.7 **Percent Recovery for LCS:**

$$\%\,\text{Recovery (LCS)} = 100\left(\frac{E}{C}\right)$$

where:
E = obtained (experimental) value C = true value

SOP No.	Revision No.	Date	Page
AWC-IC-05	10	July 31, 2006	10 of 22

TITLE: Ion Chromatography: Methods 300.0 / 9056 / SM 4110B

SUPERCEDES: Revision 9

16.0 METHOD PERFORMANCE

16.1. The method detection limit (MDL) is to be performed every six months in accordance with the specifications in 40 CFR 136, appendix B, and must demonstrate the ability to quantitate at or below the reporting limit for each anion. The current MDL is on file with the department supervisor and the QA Department.

16.2 A one–time initial demonstration of performance for each individual method for water matrices must be generated.

16.1.1. This requires quadruplicate analysis of a mid–level check standard containing all of the standard analytes for the method using the same procedures used to analyze samples, including sample preparation.

16.1.2. Calculate the average recovery and standard deviation of the recovery for each analyte of interest.

16.1.3. Compare these results with the acceptance criteria given in the Method or to laboratory historical limits (if available).

16.1.4. Repeat the test for any analyte that does not meet the acceptance criteria. Repeated failure for any analyte indicates the need for the laboratory to evaluate the analytical procedure and take corrective action.

16.2. Training Qualifications

16.3. The supervisor has the responsibility to ensure that this procedure is performed by an analyst who has been properly trained in its use and has the required experience.

16.4. The following analyst validation information is maintained for this method in the laboratory QA files.

16.5. The analyst must complete the laboratory safety orientation training that includes, but is not limited to, chemicals, PPE requirements, and electrical safety.

16.6. The analyst must read and understand this SOP.

16.7. The analyst must read and understand the Method used as reference for this SOP.

16.8. The analyst must complete a DOC or successfully analyze PT samples annually.

16.9. The analyst must complete the STL Quality Assurance Training.

17.0 DATA ASSESSMENT AND ACCEPTANCE CRITERIA FOR QUALITY CONTROL MEASURES

17.1. ICAL: calibration factor >0.995.

17.2. Obtained ICV and CCV/LCS values must be within 90-110% of the true value.

17.3. Acceptance limits for sample spike recovery are based on the historical data and are statistically derived annually. They are maintained in the laboratory LIMs system. If the lab calculated limits are wider than the method limits, the method limits of 80-120% are used for evaluation of sample spike acceptance.

17.4. Sample duplicates are required to have a calculated RPD $\leq$ 20.

17.5. ICB and CCB values must be less than the STL quantitation limit.

17.5.1. All blanks associated with USACE samples should be less than ½ the STL Buffalo quantitation limit for each anion.

17.6 Matrix Spike: Acceptance limits for sample spike recovery are based on the historical data and are statistically derived annually. They are maintained in the LIMS system.

18.0 CORRECTIVE ACTIONS FOR OUT-OF-CONTROL DATA

18.1 ICAL: Analysis cannot begin without an acceptable calibration curve. Instrument maintenance may be required. Please refer to STL Corporate Policy for information on the proper selection of calibration points.

18.2 ICV: Reanalyze calibration curve if unacceptable ICV is obtained.

18.3 CCV: Reanalyze the CCV.

18.3.1 If 2nd analysis is acceptable, analytical sequence can continue, however the previous 10 samples must be reanalyzed.

18.3.2 If 2nd analysis is unacceptable, analyze a new ICAL.

18.3.3 Method Blank: Reanalyze all samples associated with an unacceptable method blank unless:

18.3.3.1 Detected concentrations < PQL or

18.3.3.2 Detected concentrations < 10X amount in associated sample

18.3.4 Matrix Spike: Matrix interference can be assumed and corrective action is not required if both of the following conditions are met:

18.3.4.1 LCS recovery is acceptable

18.3.4.2 Recoveries in both MS and MSD are consistent (%RSD<30)

18.3.4.3 If LCS is unacceptable – re-analysis is required.

18.3.4.4 If recoveries in MS/MSD are different (e.g.: one high, one low) further evaluation should be made. Matrix interference can not be assumed in this case. Discussion with the department supervisor, operations manager or QA manager should be included in the final decision process prior to releasing data.

19.0 CONTINGENCIES FOR HANDLING OUT-OF-CONTROL OR UNACCEPTABLE DATA

19.1 A Job Exception Form must be completed and filed with the Project Manager and QA Manager for any of the following conditions:

19.1.1. Holding times exceeded

19.1.2. Insufficient sample volume for reanalysis

19.1.3. In the event of unknown positives or sample matrix which present the analyst with questionable data, the project manager shall be notified so the client may be contacted and involved in the decision process and course of action

20.0 WASTE MANAGEMENT/ POLLUTION PREVENTION

20.1. 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."

20.2. Waste Streams Produced by the Method –The following waste streams are produced when this method is carried out.

20.2.1. Dispose of instrument waste in the "D" waste container.

20.2.2. Contaminated plastic materials such as IC syringes, filters, caps and vials utilized for sample preparation. All plastic materials should be disposed of in the recycling containers located throughout the lab.

21.0 REFERENCE

21.1. Method 300.0, "Determination of Inorganic Anions by Ion Chromatography", Environmental Monitoring Systems Laboratory, Office of Research and Development, U.S. EPA, Cincinnati, Ohio, Revision 2.1, August 1993

21.2. Method 4110, Standard Methods for the Examination of Water and Wastewater, 19th Edition, 1995

21.3. Method 9056, "Determination of Inorganic Anions by Ion Chromatography", SW-846, Revision 0, 1994 and all applicable updates.

22.0 TABLES, DIAGRAMS, FLOWCHARTS AND VALIDATION DATA

22.1. Analytical Run sequence

22.2. Analytical Batch (a few pages from a typical batch)

22.3. Wet Chemistry Batch Summary & Data Review Checklist

SOP No.	Revision No.	Date	Page
AWC-IC-05	10	July 31, 2006	13 of 22

TITLE: Ion Chromatography: Methods 300.0 / 9056 / SM 4110B

SUPERCEDES: Revision 9

23.0 CHANGES FROM PREVIOUS REVISION

23.1 Section 5.2 and 14.2.2: revised to include extraction procedure for soils.

22.1 Analytical Run Sequence for instrument #1

Method 300.0	Method 9056
LCS/CCV (lower sect. 10.7)	LCS/CCV (lower sect. 10.7)
LCS/CCV (upper sect. 10.8)	LCS/CCV (upper sect. 10.8)
ICB	ICB
Sample	Sample
Sample	Sample
Sample	Sample
Sample	Sample
Sample	Sample
Sample	Sample
Sample	Sample
Sample	Sample
Sample	Sample duplicate
Sample Spike	Sample spike
CCV (lower sect. 10.7)	CCV (lower sect. 10.7)
CCV (upper sect. 10.8)	CCV (upper sect. 10.8)
ICB	ICB
Sample	Sample
Sample	Sample
Sample	Sample
Sample	Sample
Sample	Sample
Sample	Sample
Sample	Sample
Sample	Sample
Sample	Sample duplicate
Sample Spike	Sample spike
CCV (lower sect. 10.7)	CCV (lower sect. 10.7)
CCV (upper sect. 10.8))	CCV (upper sect. 10.8)
CCB	CCB

SOP No.	Revision No.	Date	Page
AWC-IC-05	10	July 31, 2006	14 of 22

TITLE: Ion Chromatography: Methods 300.0 / 9056 / SM 4110B

SUPERCEDES: Revision 9

22.2 Analytical Run Sequence for instrument #2 with the RFC

Method 300.0	**Method 9056**
LCS/CCV	LCS/CCV
MBLK	MBLK
Sample	Sample
Sample	Sample
Sample	Sample
Sample	Sample
Sample	Sample
Sample	Sample
Sample	Sample
Sample	Sample
Sample	Sample duplicate
Sample Spike	Sample spike
CCV)	CCV
MBLK	MBLK
Sample	Sample
Sample	Sample
Sample	Sample
Sample	Sample
Sample	Sample
Sample	Sample
Sample	Sample
Sample	Sample
Sample	Sample duplicate
Sample Spike	Sample spike
CCV	CCV
MBLK	MBLK

SOP No.	Revision No.	Date	Page
AWC-IC-05	10	July 31, 2006	15 of 22

TITLE: Ion Chromatography: Methods 300.0 / 9056 / SM 4110B

SUPERCEDES: Revision 9

22.3 Analytical Run (A few pages from a typical batch)

ABB10524

Schedule File: C:\PeakNet\schedule\2003\Sept 2003\09-17-03-1.sch

ne	Sample	Sample Type	Level	Method	Data File	Volume
	lower icv	Sample		inst#1 fl,cl,br,no3,so4.met	c:\peaknet\data\sept 2003\09-17-03-1	1
	upper icv	Sample		inst#1 fl,cl,br,no3,so4.met	c:\peaknet\data\sept 2003\09-17-03-1	1
	icb	Sample		inst#1 fl,cl,br,no3,so4.met	c:\peaknet\data\sept 2003\09-17-03-1	1
	885712	Sample		inst#1 fl,cl,br,no3,so4.met	c:\peaknet\data\sept 2003\09-17-03-1	1
	858703	Sample		inst#1 fl,cl,br,no3,so4.met	c:\peaknet\data\sept 2003\09-17-03-1	1
	858721	Sample		inst#1 fl,cl,br,no3,so4.met	c:\peaknet\data\sept 2003\09-17-03-1	1
	858725 1:2	Sample		inst#1 fl,cl,br,no3,so4.met	c:\peaknet\data\sept 2003\09-17-03-1	1
	872701 1:10	Sample		inst#1 fl,cl,br,no3,so4.met	c:\peaknet\data\sept 2003\09-17-03-1	1
	872702 1:5	Sample		inst#1 fl,cl,br,no3,so4.met	c:\peaknet\data\sept 2003\09-17-03-1	1
0	872703 1:50	Sample		inst#1 fl,cl,br,no3,so4.met	c:\peaknet\data\sept 2003\09-17-03-1	1
1	872704 1:20	Sample		inst#1 fl,cl,br,no3,so4.met	c:\peaknet\data\sept 2003\09-17-03-1	1
2	872704 1:2000	Sample		inst#1 fl,cl,br,no3,so4.met	c:\peaknet\data\sept 2003\09-17-03-1	1
3	872704 1:2000 SPK SO4	Sample		inst#1 fl,cl,br,no3,so4.met	c:\peaknet\data\sept 2003\09-17-03-1	1
4	lower ccv	Sample		inst#1 fl,cl,br,no3,so4.met	c:\peaknet\data\sept 2003\09-17-03-1	1
5	upper ccv	Sample		inst#1 fl,cl,br,no3,so4.met	c:\peaknet\data\sept 2003\09-17-03-1	1
6	ccb	Sample		inst#1 fl,cl,br,no3,so4.met	c:\peaknet\data\sept 2003\09-17-03-1	1
7	872705 1:5	Sample		inst#1 fl,cl,br,no3,so4.met	c:\peaknet\data\sept 2003\09-17-03-1	1
8	872705 1:50	Sample		inst#1 fl,cl,br,no3,so4.met	c:\peaknet\data\sept 2003\09-17-03-1	1
9	860601	Sample		inst#1 fl,cl,br,no3,so4.met	c:\peaknet\data\sept 2003\09-17-03-1	1
0	860602	Sample		inst#1 fl,cl,br,no3,so4.met	c:\peaknet\data\sept 2003\09-17-03-1	1
1	860607	Sample		inst#1 fl,cl,br,no3,so4.met	c:\peaknet\data\sept 2003\09-17-03-1	1
2	860608	Sample		inst#1 fl,cl,br,no3,so4.met	c:\peaknet\data\sept 2003\09-17-03-1	1
3	862201 1:5	Sample		inst#1 fl,cl,br,no3,so4.met	c:\peaknet\data\sept 2003\09-17-03-1	1
4	862202 1:10	Sample		inst#1 fl,cl,br,no3,so4.met	c:\peaknet\data\sept 2003\09-17-03-1	1
5	862202 1:10 DUP	Sample		inst#1 fl,cl,br,no3,so4.met	c:\peaknet\data\sept 2003\09-17-03-1	1
6	862202 1:10 SPK BR	Sample		inst#1 fl,cl,br,no3,so4.met	c:\peaknet\data\sept 2003\09-17-03-1	1
7	lower ccv	Sample		inst#1 fl,cl,br,no3,so4.met	c:\peaknet\data\sept 2003\09-17-03-1	1
8	upper ccv	Sample		inst#1 fl,cl,br,no3,so4.met	c:\peaknet\data\sept 2003\09-17-03-1	1
9	ccb	Sample		inst#1 fl,cl,br,no3,so4.met	c:\peaknet\data\sept 2003\09-17-03-1	1
0	862203 1:10	Sample		inst#1 fl,cl,br,no3,so4.met	c:\peaknet\data\sept 2003\09-17-03-1	1
1	862501	Sample		inst#1 fl,cl,br,no3,so4.met	c:\peaknet\data\sept 2003\09-17-03-1	1
2	862502	Sample		inst#1 fl,cl,br,no3,so4.met	c:\peaknet\data\sept 2003\09-17-03-1	1
3	862503 1:5	Sample		inst#1 fl,cl,br,no3,so4.met	c:\peaknet\data\sept 2003\09-17-03-1	1
4	862503 1:10	Sample		inst#1 fl,cl,br,no3,so4.met	c:\peaknet\data\sept 2003\09-17-03-1	1
5	863202 1:100	Sample		inst#1 fl,cl,br,no3,so4.met	c:\peaknet\data\sept 2003\09-17-03-1	1
6	864601 1:200	Sample		inst#1 fl,cl,br,no3,so4.met	c:\peaknet\data\sept 2003\09-17-03-1	1
7	864706	Sample		inst#1 fl,cl,br,no3,so4.met	c:\peaknet\data\sept 2003\09-17-03-1	1
8	873301	Sample		inst#1 fl,cl,br,no3,so4.met	c:\peaknet\data\sept 2003\09-17-03-1	1
9	873301 SPK CL	Sample		inst#1 fl,cl,br,no3,so4.met	c:\peaknet\data\sept 2003\09-17-03-1	1
0	lower ccv	Sample		inst#1 fl,cl,br,no3,so4.met	c:\peaknet\data\sept 2003\09-17-03-1	1
1	upper ccv	Sample		inst#1 fl,cl,br,no3,so4.met	c:\peaknet\data\sept 2003\09-17-03-1	1
2	ccb	Sample		inst#1 fl,cl,br,no3,so4.met	c:\peaknet\data\sept 2003\09-17-03-1	1
3	873302	Sample		inst#1 fl,cl,br,no3,so4.met	c:\peaknet\data\sept 2003\09-17-03-1	1
4	873303	Sample		inst#1 fl,cl,br,no3,so4.met	c:\peaknet\data\sept 2003\09-17-03-1	1
5	873304	Sample		inst#1 fl,cl,br,no3,so4.met	c:\peaknet\data\sept 2003\09-17-03-1	1
6	873305	Sample		inst#1 fl,cl,br,no3,so4.met	c:\peaknet\data\sept 2003\09-17-03-1	1
7	873306	Sample		inst#1 fl,cl,br,no3,so4.met	c:\peaknet\data\sept 2003\09-17-03-1	1
8	873307	Sample		inst#1 fl,cl,br,no3,so4.met	c:\peaknet\data\sept 2003\09-17-03-1	1
9	873308 1:2	Sample		inst#1 fl,cl,br,no3,so4.met	c:\peaknet\data\sept 2003\09-17-03-1	1
0	873309 1:2	Sample		inst#1 fl,cl,br,no3,so4.met	c:\peaknet\data\sept 2003\09-17-03-1	1
1	873309 1:2 DUP	Sample		inst#1 fl,cl,br,no3,so4.met	c:\peaknet\data\sept 2003\09-17-03-1	1
2	873309 1:2 SPK CL	Sample		inst#1 fl,cl,br,no3,so4.met	c:\peaknet\data\sept 2003\09-17-03-1	1
3	lower ccv	Sample		inst#1 fl,cl,br,no3,so4.met	c:\peaknet\data\sept 2003\09-17-03-1	1
4	upper ccv	Sample		inst#1 fl,cl,br,no3,so4.met	c:\peaknet\data\sept 2003\09-17-03-1	1
5	ccb	Sample		inst#1 fl,cl,br,no3,so4.met	c:\peaknet\data\sept 2003\09-17-03-1	1
6	873311	Sample		inst#1 fl,cl,br,no3,so4.met	c:\peaknet\data\sept 2003\09-17-03-1	1
7	873312	Sample		inst#1 fl,cl,br,no3,so4.met	c:\peaknet\data\sept 2003\09-17-03-1	1
8	873313	Sample		inst#1 fl,cl,br,no3,so4.met	c:\peaknet\data\sept 2003\09-17-03-1	1
9	873501	Sample		inst#1 fl,cl,br,no3,so4.met	c:\peaknet\data\sept 2003\09-17-03-1	1
0	873502	Sample		inst#1 fl,cl,br,no3,so4.met	c:\peaknet\data\sept 2003\09-17-03-1	1
1	873504	Sample		inst#1 fl,cl,br,no3,so4.met	c:\peaknet\data\sept 2003\09-17-03-1	1
2	873506	Sample		inst#1 fl,cl,br,no3,so4.met	c:\peaknet\data\sept 2003\09-17-03-1	1
3	873509	Sample		inst#1 fl,cl,br,no3,so4.met	c:\peaknet\data\sept 2003\09-17-03-1	1
4	873801	Sample		inst#1 fl,cl,br,no3,so4.met	c:\peaknet\data\sept 2003\09-17-03-1	1
5	873801 SPK CL	Sample		inst#1 fl,cl,br,no3,so4.met	c:\peaknet\data\sept 2003\09-17-03-1	1
6	lower ccv	Sample		inst#1 fl,cl,br,no3,so4.met	c:\peaknet\data\sept 2003\09-17-03-1	1
7	upper ccv	Sample		inst#1 fl,cl,br,no3,so4.met	c:\peaknet\data\sept 2003\09-17-03-1	1
8	ccb	Sample		inst#1 fl,cl,br,no3,so4.met	c:\peaknet\data\sept 2003\09-17-03-1	1
9	873801 1:5	Sample		inst#1 fl,cl,br,no3,so4.met	c:\peaknet\data\sept 2003\09-17-03-1	1
0	873901	Sample		inst#1 fl,cl,br,no3,so4.met	c:\peaknet\data\sept 2003\09-17-03-1	1
1	874001	Sample		inst#1 fl,cl,br,no3,so4.met	c:\peaknet\data\sept 2003\09-17-03-1	1
2	874003	Sample		inst#1 fl,cl,br,no3,so4.met	c:\peaknet\data\sept 2003\09-17-03-1	1
3	874004	Sample		inst#1 fl,cl,br,no3,so4.met	c:\peaknet\data\sept 2003\09-17-03-1	1
4	874006	Sample		inst#1 fl,cl,br,no3,so4.met	c:\peaknet\data\sept 2003\09-17-03-1	1
5	874007 1:2	Sample		inst#1 fl,cl,br,no3,so4.met	c:\peaknet\data\sept 2003\09-17-03-1	1
6	874008	Sample		inst#1 fl,cl,br,no3,so4.met	c:\peaknet\data\sept 2003\09-17-03-1	1
7	874008 DUP	Sample		inst#1 fl,cl,br,no3,so4.met	c:\peaknet\data\sept 2003\09-17-03-1	1

STL BUFFALO
Method(s): IC (300.0)
Analyst: [signature] Date: 9/17/03
Reviewed By: [signature] Date: 9-18-03

9/17/03 8:54:52 AM

SOP No.	Revision No.	Date	Page
AWC-IC-05	10	July 31, 2006	16 of 22

TITLE: Ion Chromatography: Methods 300.0 / 9056 / SM 4110B

SUPERCEDES: Revision 9

Schedule File: C:\PeakNet\schedule\2003\Sept 2003\09-17-03-1.sch

Sample	Sample Type	Level	Method	Data File	Volume
874008 SPK CL	Sample		inst#1 fl,cl,br,no3,so4.met	c:\peaknet\data\sept 2003\09-17-03-1	1
lower ccv	Sample		inst#1 fl,cl,br,no3,so4.met	c:\peaknet\data\sept 2003\09-17-03-1	1
upper ccv	Sample		inst#1 fl,cl,br,no3,so4.met	c:\peaknet\data\sept 2003\09-17-03-1	1
ccb	Sample		inst#1 fl,cl,br,no3,so4.met	c:\peaknet\data\sept 2003\09-17-03-1	1
874009	Sample		inst#1 fl,cl,br,no3,so4.met	c:\peaknet\data\sept 2003\09-17-03-1	1
874010 1:2	Sample		inst#1 fl,cl,br,no3,so4.met	c:\peaknet\data\sept 2003\09-17-03-1	1
874011	Sample		inst#1 fl,cl,br,no3,so4.met	c:\peaknet\data\sept 2003\09-17-03-1	1
874012	Sample		inst#1 fl,cl,br,no3,so4.met	c:\peaknet\data\sept 2003\09-17-03-1	1
874013 1:2	Sample		inst#1 fl,cl,br,no3,so4.met	c:\peaknet\data\sept 2003\09-17-03-1	1
874014	Sample		inst#1 fl,cl,br,no3,so4.met	c:\peaknet\data\sept 2003\09-17-03-1	1
874015 1:2	Sample		inst#1 fl,cl,br,no3,so4.met	c:\peaknet\data\sept 2003\09-17-03-1	1
874017 1:2	Sample		inst#1 fl,cl,br,no3,so4.met	c:\peaknet\data\sept 2003\09-17-03-1	1
87417 1:2 SPK CL	Sample		inst#1 fl,cl,br,no3,so4.met	c:\peaknet\data\sept 2003\09-17-03-1	1
lower ccv	Sample		inst#1 fl,cl,br,no3,so4.met	c:\peaknet\data\sept 2003\09-17-03-1	1
upper ccv	Sample		inst#1 fl,cl,br,no3,so4.met	c:\peaknet\data\sept 2003\09-17-03-1	1
ccb	Sample		inst#1 fl,cl,br,no3,so4.met	c:\peaknet\data\sept 2003\09-17-03-1	1
STOP	Sample		shutdown.met	c:\peaknet\data\sept 2003\09-17-03-1	1

STL BUFFALO
Method(s): IC (300)
Analyst: GH Date: 9/17/03
Reviewed By: _____ Date: _____

PeakNet 5.1 9/17/03 8:54:52 AM

SOP No.	Revision No.	Date	Page
AWC-IC-05	10	July 31, 2006	17 of 22

TITLE: Ion Chromatography: Methods 300.0 / 9056 / SM 4110B

SUPERCEDES: Revision 9

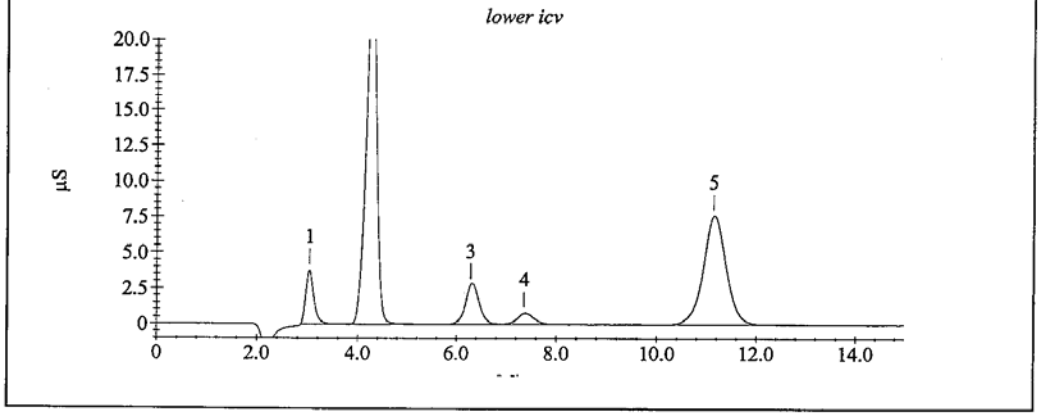
Sample Analysis Report

Sample Name : lower icv
Data File Name : C:\PeakNet\data\Sept 2003\09-17-03-1_001.DXD

Method File Name : c:\peaknet\method\inst#1 fl,cl,br,no3,so4.met
Date Time Collected : 9/17/03 3:13:16 PM
System Operator : GH

Peak Information : All Peaks

Peak #	Component Name	Retention Time	Amount (mG/L)	Peak Area	Peak Height
1	fluoride	3.03	0.94	427263	37722
2	chloride	4.25	9.37 94%	3768071	262365
3	bromide	6.28	4.08 103%	550884	27555
4	nitrate	7.37	1.18	188122	7928
5	sulfate	11.14	9.68 97%	2442652	75729



: PeakNet 5.1 Page 1 of 1 Current Date : 9/17/03
Current Time : 15:30:50

SOP No.	Revision No.	Date	Page
AWC-IC-05	10	July 31, 2006	18 of 22

TITLE: Ion Chromatography: Methods 300.0 / 9056 / SM 4110B

SUPERCEDES: Revision 9

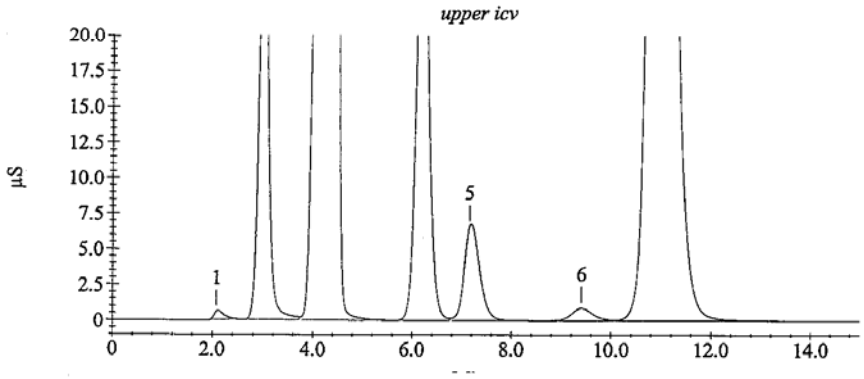
Sample Analysis Report

Sample Name : upper icv
Data File Name : C:\PeakNet\data\Sept 2003\09-17-03-1_002.DXD

Method File Name : c:\peaknet\method\inst#1 fl,cl,br,no3,so4.met
Date Time Collected : 9/17/03 3:30:51 PM
System Operator : GH

Peak Information : All Peaks

Peak #	Component Name	Retention Time	Amount (mG/L)	Peak Area	Peak Height
1		2.08	0.00	86188	5723
2	fluoride	2.95	7.80	4224221	292465
3	chloride	4.33	80.32 100%	38618965	1801964
4	bromide	6.15	30.91 90%	5435682	311702
5	nitrate	7.15	8.07	1455032	65808
6		9.40	0.00	262004	8829
7	sulfate	10.92	78.86 99%	25760885	816207



: PeakNet 5.1

Page 1 of 1

Current Date : 9/17/03
Current Time : 15:48:28

SOP No.	Revision No.	Date	Page
AWC-IC-05	10	July 31, 2006	19 of 22

TITLE: Ion Chromatography: Methods 300.0 / 9056 / SM 4110B

SUPERCEDES: Revision 9

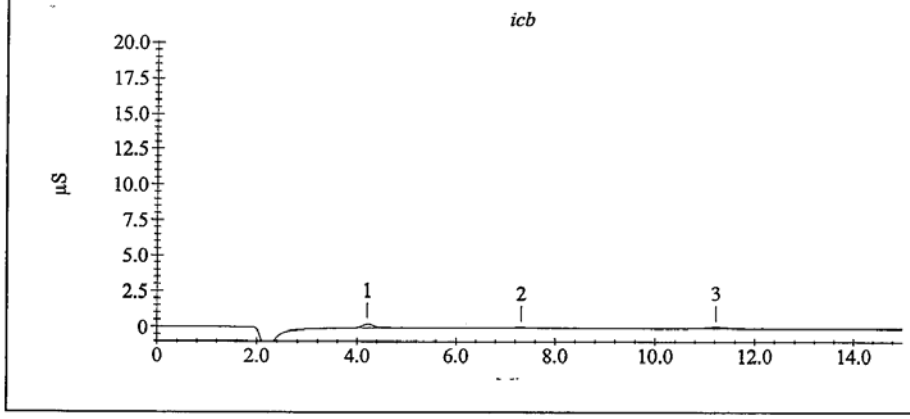
Sample Analysis Report

Sample Name : icb
Data File Name : C:\PeakNet\data\Sept 2003\09-17-03-1_003.DXD

Method File Name : c:\peaknet\method\inst#1 fl,cl,br,no3,so4.met
Date Time Collected : 9/17/03 3:48:30 PM
System Operator : GH

Peak Information : All Peaks

Peak #	Component Name	Retention Time	Amount (mG/L)	Peak Area	Peak Height
1	chloride	4.20	0.26	39716	2653
2	nitrate	7.32	0.09	11825	556
3	sulfate	11.22	0.19	35098	1063



: PeakNet 5.1 Page 1 of 1 Current Date : 9/17/03
Current Time : 16:06:05

SOP No.	Revision No.	Date	Page
AWC-IC-05	10	July 31, 2006	20 of 22

TITLE: Ion Chromatography: Methods 300.0 / 9056 / SM 4110B

SUPERCEDES: Revision 9

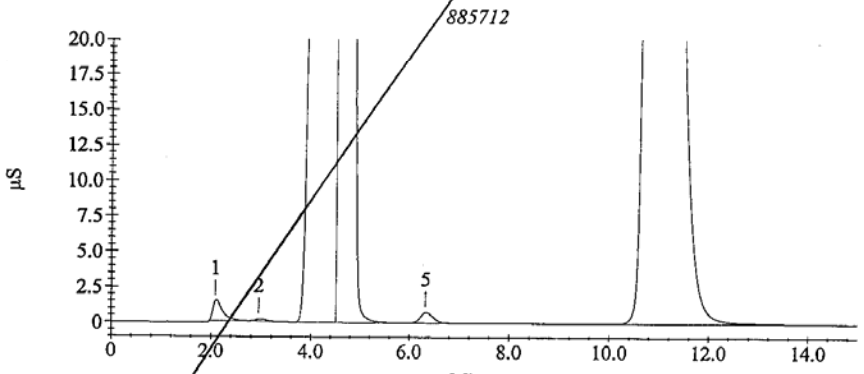
Sample Analysis Report

Sample Name : 885712
Data File Name : C:\PeakNet\data\Sept 2003\09-17-03-1_004.DXD

Method File Name : c:\peaknet\method\inst#1 fl,cl,br,no3,so4.met
Date Time Collected : 9/17/03 4:06:08 PM
System Operator : GH

Peak Information : All Peaks

Peak #	Component Name	Retention Time	Amount (mG/L)	Peak Area	Peak Height
1		2.08	0.00	205209	13955
2	fluoride	2.95	0.08	24614	1572
3	**chloride**	**4.29**	**176.21**	**76343372**	**2886731**
4		4.68	0.00	44459987	2914252
5	bromide	6.33	1.04	135717	7497
6	sulfate	10.92	172.32	48957673	1430799



: PeakNet 5.1

Page 1 of 1

Current Date : 9/17/03
Current Time : 16:23:43

SOP No.	Revision No.	Date	Page
AWC-IC-05	10	July 31, 2006	21 of 22

TITLE: Ion Chromatography: Methods 300.0 / 9056 / SM 4110B

SUPERCEDES: Revision 9

Sample Analysis Report

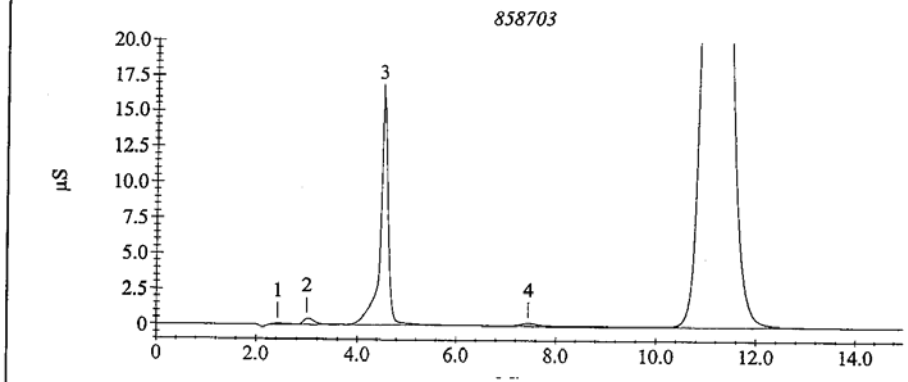
Sample Name : 858703
Data File Name : C:\PeakNet\data\Sept 2003\09-17-03-1_005.DXD

Method File Name : c:\peaknet\method\inst#1 fl,cl,br,no3,so4.met
Date Time Collected : 9/17/03 4:23:45 PM
System Operator : GH

Peak Information : All Peaks

Peak #	Component Name	Retention Time	Amount (mG/L)	Peak Area	Peak Height
1		2.43	0.00	17949	914
2	fluoride	2.99	0.19	75806	4539
3	chloride	4.51	4.73	1843230	154117
4	nitrate	7.45	0.28	41747	1934
5	sulfate	11.09	66.32	21197849	669560

confirmed



: PeakNet 5.1

Page 1 of 1

Current Date : 9/17/03
Current Time : 16:41:22

22.4 Wet Chemistry Batch Summary & Data Review Summary

SOP No.	Revision No.	Date	Page
AWC-IC-05	10	July 31, 2006	22 of 22

TITLE: Ion Chromatography: Methods 300.0 / 9056 / SM 4110B

SUPERCEDES: Revision 9

WET CHEMISTRY BATCH SUMMARY

Parameter _____ Method _____ Batch #_____

Comment #	Comment
1	NA
2	Sample(s) was diluted for matrix interference.
3	Sample(s) was diluted for excessive foaming/
4	Sample(s) was diluted for turbidity.
5	NA
6	NA
7	NA
8	Sample(s) was diluted for high concentration of target analyte
9	Sample(s) was diluted for turbidity.
10	Sample(s) was diluted for color.
11	There was insufficient volume for a lower dilution.
12	Sample(s) was diluted for viscosity.
13	Sample(s) was diluted for other reason (detail required)
14	Sample(s) required re-run to verify result.
15	Sample(s) requires re-run to verify deviation from historical result.
16	Sample(s) requires re-run for CCB failure.
17	Sample(s) affected by elevated CCB are greater than 10x detection limit.
18	Sample was colored.
19	Sample(s) was received outside of Holding Times.
20	Sample(s) contained a high amount of settleable material.
21	Sample(s) contained a high amount of suspended material.
22	Sample(s) were centrifuged for turbidity.
23	There was insufficient volume for analysis of sample at method required volume.
24	There was insufficient volume for re-analysis of the sample(s).
25	There was insufficient volume for dilution of the sample(s).
26	There was insufficient volume for Dup/Spk.
27	Sample(s) was cloudy
28	See accompanying Job Exception Report.

Comments and Corrective actions

#_____ Sample(s)_____

#_____ Sample(s)_____

#_____ Sample(s)_____

#_____ Sample(s)_____

CCV/CCB Compliant? NA_____ YES_____ NO_____(see reason below)

Other_____

_____

_____

Technician _____ Date_____

2nd Review _____ Date_____ **Number of Reanalysis for this batch:**_____

Review _____ Date_____ WC Summary Rev 1 7-2003

STL Buffalo
LABORATORY STANDARD OPERATING PROCEDURE

SOP No.	Revision No.	Effective Date	Page
AWC-IRON-56	5	June 13, 2006	1 of 11

TITLE: Ferrous Iron

SUPERCEDES: Revision 4

REVIEWED AND APPROVED BY:	SIGNATURE	DATE
Verl D. Preston, Quality Manager		
Christopher Spencer, Laboratory Director		
Peggy Gray-Erdmann, Supervisor		

1.0 IDENTIFICATION OF TEST METHODS

1.1 This method is taken from Standard Methods method 3500FE-D.

2.0 APPLICABLE MATRIX

2.1 This method is applicable to surface and saline waters and aqueous domestic and industrial wastes.

3.0 REPORTING LIMIT

3.1 The quantitation limit is 0.10mg/L.

4.0 SCOPE AND APPLICATION

4.1 This method is for determining total and ferrous iron in environmental water samples.

5.0 SUMMARY OF TEST METHOD

5.1 The solution is treated with acid and with 1,10- phenanthroline, this forms an orange red color, which obeys Beer's Law.

6.0 DEFINITIONS

6.1 Standard definitions are found in section 3 of the Laboratory Quality Manual.

TITLE: Ferrous Iron

SUPERCEDES: Revision 4

7.0 INTERFERENCES

7.1 Strong oxidizing agents, cyanide, nitrite, and phosphates (especially polyphosphates), chromium, zinc in concentrations exceeding 10 times that of iron; cobalt, and copper in excess of 5mg/L and nickel in excess of 2mg/L.

7.2 Bismuth, cadmium, mercury, molybdate and silver precipitate phenanthroline.

7.3 If noticeable amounts of color or organic matter are present, the sample can be evaporated, gently ashed, and re-dissolved in acid.

8.0 SAFETY

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

8.1 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.

8.2 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.
1 – Always add acid to water to prevent violent reactions.			
2 – Exposure limit refers to the OSHA regulatory exposure limit.			

TITLE: Ferrous Iron

SUPERCEDES: Revision 4

9.0 EQUIPMENT AND SUPPLIES

9.1 25ml sample cells

9.2 Spectrophotometer

9.4 Eppendorfs for measurements of 0.025 to 5ml

10.0 REAGENTS AND STANDARDS

10.1 Ferrous Iron Reagent Powder pillows from HACH.

10.2 Stock iron solution: FAS (Ferrous Ammonium Sulfate) slowly add 20ml conc. H_2SO_4 to 50ml of water and dissolve 1.404 grams of Fe $(NH_4)_2(SO_4)_2$ $6H_2O$. Add 0.1N potassium permanganate ($KMNO_4$). Dropwise until a faint pink color persists. Dilute to 1000ml with water.

11.0 SAMPLE COLLECTION, PRESERVATION, SHIPMENT AND STORAGE

11.1 The value of the determination depends greatly on the care taken to obtain a representative sample. Iron in well water or tap samples may vary greatly in both concentration and form, depending on the amount of flushing before and during sampling.

11.2 Ferrous iron samples must be preserved by placing samples at $4^{\circ}C$ until analysis is to be done. The rapid change in the Ferrous- Iron to Ferric Iron ratio after sampling requires that analysis be done as soon as possible. Optimally, the Ferrous-Iron should be determined at the time of sampling.

12.0 QUALITY CONTROL

12.1 Initial Calibration Curve (ICAL): A calibration curve must be analyzed every three months at a minimum. Acceptance criteria is a correlation coefficient (R value) of ≥ 0.995. An acceptable calibration verification sample and calibration blank must bracket each group of sample analyses. All quality control data should be maintained and available for easy reference or inspection.

12.2 Initial Calibration Verification and Laboratory Control Sample (ICV/LCS): (2.0ppm) Prepare by adding .25ml of the 200ppm stock standard into 25ml of distilled water. An ICV is made from a second source and must be analyzed once after every curve. LCSs must be analyzed after every 10 samples and at the beginning and end of the analytical batch.

12.3 Initial Calibration Blank and Method Blank (ICB/MBLK): To determine freedom from contamination, prepare one method blank (MBLK) at the beginning and end of the analytical procedure and another (CCB) after every ten samples. An initial Calibration Blank must be run after every curve. The blank consists of 25ml of distilled water that gets the same treatment as the samples and standards. The ICB and MBLK must exhibit values less than the reporting limit.

12.3.1 All blanks associated with DOD QSM and AFCEE samples must be less than 1/2 the STL Buffalo quantitation limit for Ferrous Iron.

12.4 Matrix Duplicate (MD): A sample duplicate must be run for every group of twenty or fewer samples.

12.5 Matrix Spike (MS): A sample spike must be analyzed for every group of twenty or fewer samples. A spike is 0.125ml of the 200ppm stock solution (1ppm).

13.0 CALIBRATION AND STANDARDIZATION

13.1 A calibration curve must be analyzed at a minimum of once every three months. Acceptance criteria is a correlation coefficient (R value) of $\geq$ 0.995. The curve will consist of a total of 5 points: 0, 0.1, 0.5, 1.0, 3.0 ppm.

13.1.2 Prepare calibration by diluting the stock iron solution (FAS) according to the following table.

Volume Fe Standard	Concentration Fe
0ml diluted to 25ml	Blank
0.0125ml	0.1ppm
0.0625ml	0.5ppm
0.125ml	1.0ppm
0.375ml	3.0ppm

14.0 PROCEDURE

14.1 Ferrous Iron Procedure:

14.2.1 Mix sample thoroughly and fill a sample cell with 25 ml of sample. Place the prepared sample into the cell holder. Measure the color intensity on the spectrophotometer at 510nm. This sample is not spiked nor do you add the Ferrous pillow packet. This is used for the blank correction. Enter this value under the blank absorbance column on the excel spreadsheet.

14.2.2 Take this sample and add the contents of one Ferrous Iron Reagent Powder Pillow tot the sample cell. Swirl to mix. Wait three minutes for the reaction period. Place the prepared sample into the cell holder. Measure the color intensity on spectrophotometer at 510nm. Enter this value under the sample absorbance column on the excel spreadsheet.

15.0 CALCULATIONS

15.1 Calculate sample results from the calibration curve by using the linear regression curve.
NOTE: In the case of any dilutions, correct the result by the dilution factor.

15.2 Measured Concentration by Linear Regression:

$$x = \frac{a - b}{m}$$

where:

a = area counts for analyte to be measured
m = slope
x = concentration
b = intercept

and

$$m = \frac{\sum x_i a_i}{\sum x_i^2}$$

$$b = Y_{ave} - b x_{ave}$$

15.3 Percent Recovery for LCS:

$$\%\ \text{Recovery (LCS)} = 100\left(\frac{E}{C}\right)$$

where:

E = obtained (experimental) value
C = true value

15.4 Relative Percent Difference (RPD):

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

where:

x_1 = analytical % recovery
x_2 = replicate % recovery

15.5 To calculate Ferric Iron subtract the final result for ferrous iron from the final result of the total iron.

16.0 METHOD PERFORMANCE

16.1. Method Detection Limit: A valid method detection limit for each analyte of interest must be generated. The MDL must be below the reporting limit for each analyte. The procedure for determination of the method detection limit is given in 40 CFR Part 136, Appendix B. See STL SOP S-Q-003, "Method Detection Limit Studies," current revision, for further guidance. Current STL Buffalo MDLs are maintained the QA department and are easily viewed in the laboratory LIMs system.

16.1.1. A one–time initial demonstration of performance for each individual method for water must be generated.

16.1.2. This requires quadruplicate analysis of a mid–level check standard containing all of the standard analytes for the method using the same procedures used to analyze samples, including sample preparation.

16.1.3. Calculate the average recovery and standard deviation of the recovery for each analyte of interest.

16.1.4. Compare these results with the acceptance criteria given in the Method or to laboratory historical limits (if available).

16.1.5. Repeat the test for any analyte that does not meet the acceptance criteria. Repeated failure for any analyte indicates the need for the laboratory to evaluate the analytical procedure and take corrective action.

16.2. Training Qualifications

16.3. The supervisor has the responsibility to ensure that this procedure is performed by an analyst who has been properly trained in its use and has the required experience.

16.4. The following analyst validation information is maintained for this method in the laboratory QA files.

16.5. The analyst must complete the laboratory safety orientation training that includes, but is not limited to, chemicals, PPE requirements, and electrical safety.

16.6. The analyst must read and understand this SOP.

16.7. The analyst must read and understand the Method used as reference for this SOP.

16.8. The analyst must complete a DOC or successfully analyze PT samples annually.

16.9. The analyst must complete the STL Quality Assurance Training.

17.0 DATA ASSESSMENT AND ACCEPTANCE CRITERIA FOR QUALITY CONTROL MEASURES

17.1 ICAL: calibration factor >0.995

17.2 ICV/LCS values must be between 90-110%.

17.3 Acceptance limits for sample duplicates are required to be calculated yearly and are available in AIMS.

17.4 Acceptance limits for sample spike values are calculated yearly and available in AIMS.

17.5 Method Blank:

17.5.1 Detected concentrations < PQL or

17.5.2 Detected concentrations < 10X amount in associated samples

18.0 CORRECTIVE ACTIONS FOR OUT-OF-CONTROL DATA

18.1 ICAL: Analysis cannot begin without an acceptable calibration curve. Instrument maintenance may be required. Please refer to STL Corporate Policy for information on the proper selection of calibration points.

18.2 ICV: Reanalyze calibration curve if unacceptable ICV is obtained.

18.3 LCS: Reanalyze the LCS.

18.3.1 If 2nd analysis is acceptable, analytical sequence can continue, however the previous 10 samples must be reanalyzed.

18.3.1 If 2nd analysis is unacceptable, analyze a new ICAL.

18.3.2 If LCS is below limits: Re-analyze all samples associated with an unacceptable LCS

18.3.3 If LCS is above limits: Re-analysis is not required if samples are ND.

18.4 Method Blank: Re-analyze all samples associated with an unacceptable method blank with the following exceptions:

18.4.1 Detected concentrations < PQL or

18.4.2 Detected concentrations < 10X amount in associated samples

18.5 MS/MSD:

18.5.1 Matrix interference can be assumed and corrective action is not required if both of the following conditions are met:

18.5.1.1LCS recovery is acceptable

18.5.1.2Recoveries in both MS and MSD are consistent (%RSD<30)

18.5.1.3If LCS is unacceptable – re-analysis of batch is required.

18.5.1.4If recoveries in MS/MSD are different (e.g.: one high, one low) further evaluation should be made. Matrix interference cannot be assumed in

this case. Discussion with the department supervisor, operations manager or QA manager should be included in the final decision process prior to releasing data.

19.0 CONTINGENCIES FOR HANDELING OUT-OF-CONTROL OR UNACCEPTABLE DATA

19.1 A Job Exception Form must be completed and filed with the Project Manager and QA Manager for any of the following conditions:

19.1.1. Holding times exceeded

19.1.2. Insufficient sample volume for re-analysis

19.1.3. In the event of unknown positives or sample matrix which present the analyst with questionable data, the project manager shall be notified so the client may be contacted and involved in the decision process and course of action

20.0 WASTE MANAGEMENT/ 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."

20.1 Waste Streams Produced by the Method

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

Acidic waste generated by the analysis. All samples and expired reagents are to be disposed of as "A" waste.

21.0 REFERENCE

21.1 Standard Methods 19th Edition, method 3500-Fe.

22.0 TABLES, DIAGRAMS, FLOWCHARTS AND VALIDATION DATA

21.1. Analytical Sequence

21.2. Analytical Batch

21.3. Wet Chemistry Batch Summary

TITLE: Ferrous Iron

SUPERCEDES: Revision 4

23.0 CHANGES FROM PREVIOUS REVISION

23.1 Updated the procedure in section 14.0 to include the Ferrous Iron Powder Pillows.

23.2 Updated sections 10.0, 12.0 and 13.0 to reflect new procedure.

23.3 Updated Attachment 22.2 to show excel spreadsheet.

22.1 Analytical Sequence

ICAL
ICB
ICV

LCS
MBLK
SAMPLE
SAMPLE
SAMPLE
SAMPLE
SAMPLE
SAMPLE
SAMPLE
SAMPLE
SAMPLE DUP
SAMPLE SPK
LCS
MBLK

STL Buffalo
LABORATORY STANDARD OPERATING PROCEDURE

SOP No.	Revision No.	Effective Date	Page
AWC-IRON-56	5	June 13, 2006	10 of 11

TITLE: Ferrous Iron

SUPERCEDES: Revision 4

22.2 Analytical Batch

Laboratory Bench Sheet
FERROUS IRON
Revision 0 June-2006

STL Buffalo

Analyst:			**Calibration Curve Information**					**BATCH #**		
Start Date:				Conc.(mg/L)	ABS.			**Instrument Information**		
Start Time:			STD1	0.000	0.000			Instrument:	Odyssey	
End Time:			Std. 2	0.100	0.055			Wavelength:	510	
			Std. 3	0.500	0.216			Parameter:	Ferrous Iron	
DATE OF CURVE=		6/2/2006	Std. 4	1.000	0.518			**Corr. Coef:**	0.9989	
SOP Information			Std. 5	3.000	1.390			**Slope:**	0.46397	
Number:	AWC-IRON-56							**Intercept:**	0.00895	
			Reagents Used			**Solution ID#**				
			Ferrous Iron Reagent Powder Pillow							
EQL:	0.01	mg/L								
ICV INFORMATION			**LCS Information:**					**Matrix Spike Information:**		
Solution #			Solution #					Solution #		
Concentration (mg/L)		2.00	Concentration (mg/L):		2			Concentration (mg/L):		1
ICV	True value:	2.00	LCS	True value:	2.00			MS	True Value	1.00

Job #	Sample ID	Sample Volume (mL)	Sample ABS.	Blank ABS.	Corrected ABS.	D.F.	Curve Conc. (mg/L)	Final Conc. (mg/L)	% Rec.	Comments
LCS	lcs	25			0.000	1	ND	ND	#VALUE!	
MBLK	BLANK	25			0.000	1	ND	ND		
		50			0.000	1	ND	ND		
		25			0.000	1	ND	ND		
		25			0.000	1	ND	ND		
		25			0.000	1	ND	ND		
		25			0.000	1	ND	ND		
		25			0.000	1	ND	ND		
		25			0.000	1	ND	ND		
		25			0.000	1	ND	ND		
		25			0.000	1	ND	ND		
		25			0.000	1	ND	ND		
		25			0.000	1	ND	ND		
LCS	lcs	25			0.000	1	ND	ND		
mblk	BLANK	25			0.000	1	ND	ND		
		25			0.000	1	ND	ND		
		25			0.000	1	ND	ND		
		25			0.000	1	ND	ND		
		25			0.000	1	ND	ND		
		25			0.000	1	ND	ND		
		25			0.000	1	ND	ND		
		25			0.000	1	ND	ND		
		25			0.000	1	ND	ND		
		25			0.000	1	ND	ND		
		25			0.000	1	ND	ND		
		25			0.000	1	ND	ND		
LCS	lcs	25			0.000	1	ND	ND		
mblk	blank	25			0.000	1	ND	ND		

Page 1 of 2

SOP No.	Revision No.	Effective Date	Page
AWC-IRON-56	5	June 13, 2006	11 of 11

TITLE: Ferrous Iron

SUPERCEDES: Revision 4

22.3 Wet Chemistry Batch Summary Sheet

WET CHEMISTRY BATCH SUMMARY

PARAMETER_____ **METHOD**_____ **BATCH** _____

COMMENTS	JOB NUMBER
WC Reporting Limit < STL Quant Limit	
WC Historical confirms within Hold Time	
WC Historical NO confirm & RE outside of HT	
WC Hold Time Exceedance-Dilution required	
WC Hold Time Exceedance-Instrument Failure	
WC Holding Time Exceedance by Date	
WC Holding Time Exceedance by Hours	
WC LCS within ERA limits outside internal	
WC LCS high recovery, sample ND	
WC MBLK hit but samples > 10X blank value	
WC RPD Exceedance for MS / SD	
WC Spike Failure HIGH MS only	
WC Spike Failure LOW MS only	
WC Spike Failure MS and SD	
WC BOD HT met- Oxygen depleted-RE out HT	
WC Carbonate Alkalinity, LCS/MBLK	
WC Reactivity Qualification	
WC TDS/Conductivity ratio outside of range	
WC TOX Breakthrough- no volume for redo	
WC TOX samples were centrifuged	
Other	

DILUTION CODES	REASON
002	Sample matrix effects
003	Excessive foaming
004	High levels of non-target compounds
008	High concentration of target analytes
009	Sample turbidity
010	Sample color
011	Insufficient volume for lower dilution
012	Sample viscosity
013	other

ICAL Compliant? YES NO NA IF NO, Why? _____
LCS/CCV Compliant? YES NO NA IF NO, Why? _____
CCB Compliant? YES NO NA IF NO, Why? _____
RPD Compliant? YES NO NA IF NO, Why? _____
ERA Compliant? YES NO NA IF NO, Why? _____

NUMBER of REANALYSIS FOR THIS BATCH:_____

Analyst_____ Date_____

Time Critical Batch Review_____ Date_____

Secondary Review & Closure_____ Date_____ WC Summary Rev 4 / 5-2005

STL Buffalo
LABORATORY STANDARD OPERATING PROCEDURES

SOP No.	Revision No.	Date	Page
AWC-Reactivity-100	5	December 8, 2005	Page 1 of 15

TITLE: REACTIVITY - METHOD SECT. 7.3

Supersedes: Revision 4

REVIEWED & APPROVED BY:	Signature	Date
Verl D. Preston, Quality Manager		
Christopher Spencer, Laboratory Director		
Peggy Gray-Erdmann, Supervisor		

1.0 IDENTIFICATION OF TEST METHODS

1.1 This method is taken from EPA solid waste methods; Section 7.3, 9012, 9014 and 9034.

2.0 APPLICABLE MATRIX

2.1 This method is applicable to both aqueous and solid wastes.

3.0 REPORTING LIMIT

3.1 The reporting limit is 10.0 mg/kg.

4.0 SCOPE AND APPLICATION

4.1 This method is applicable to all wastes, with the condition that wastes that are combined with acids do not form explosive wastes.

4.2 This method provides a way to determine the specific rates of release of hydrocyanic acid and hydrogen sulfide upon contact with a weak aqueous acid.

4.3 This test measures only the hydrocyanic acid and hydrogen sulfide evolved using the specific test conditions. It is not intended to measure forms of cyanide or sulfide other than those evolved under the test conditions.

SOP No.	Revision No.	Date	Page
AWC-Reactivity-100	5	December 8, 2005	Page 2 of 15

TITLE: REACTIVITY - METHOD SECT. 7.3

Supersedes: Revision 4

5.0 SUMMARY OF TEST METHOD

5.1 An aliquot of acid is added to a fixed weight of waste in a closed system. The generated gas is swept into a scrubber. The concentrations of hydrocyanic acid and hydrogen sulfide are determined by titration.

6.0 DEFINITIONS

6.1 Standard definitions can be found in section 3.0 of the STL Buffalo Laboratory Quality Manual.

7.0 INTERFERENCES

7.1 Interferences are undetermined.

8.0 SAFETY

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

8.1 *SPECIFIC SAFETY CONCERNS OR REQUIREMENTS*

Potassium Cyanide will form Hydrogen Cyanide (HCN) gas when combined with strong acids. Breathing HCN gas may result in death.

Sodium Sulfide will form Hydrogen Sulfide (HS) gas if combined with strong acids. Inhalation of HS gas may be fatal.

8.2 *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.

TITLE: REACTIVITY - METHOD SECT. 7.3

Supersedes: Revision 4

Material (1)	Hazards	Exposure Limit (2)	Signs and symptoms of exposure
Acetone	Flammable	1000 ppm-TWA	Inhalation of vapors irritates the respiratory tract. May cause coughing, dizziness, dullness, and headache.
Potassium Cyanide	Poison Corrosive	5 Mg/M3 TWA as CN	**This material will form Hydrogen Cyanide (HCN) gas when combined with strong acids. Breathing HCN gas may result in death.** Corrosive to the respiratory tract. May cause headache, weakness, dizziness, labored breathing nausea and vomiting, which can be followed by weak and irregular heart beat, unconsciousness, convulsions, coma and death. Solutions are corrosive to the skin and eyes, and may cause deep ulcers, which heal slowly. May be absorbed through the skin, with symptoms similar to those noted for inhalation. Symptoms may include redness, pain, blurred vision, and eye damage.
Sulfuric Acid	Corrosive Oxidizer Dehydra-dator	1 mg/m^3	This material will cause burns if comes into contact with the skin or eyes. Inhalation of vapors will cause irritation of the nasal and respiratory system.
Sodium Hydroxide	Corrosive	2 Mg/M3-Ceiling	Severe irritant. Effects from inhalation of dust or mist vary from mild irritation to serious damage of the upper respiratory tract, depending on severity of exposure. Symptoms may include sneezing, sore throat or runny nose. Contact with skin can cause irritation or severe burns and scarring with greater exposures. Causes irritation of eyes, and with greater exposures it can cause burns that may result in permanent impairment of vision, even blindness.
Sodium Sulfide	Corrosive	10 ppm-TWA 15 ppm-STEL	Will **for**m **H**ydrogen Sulfide (HS) gas if combined with strong acids. Inhalation of HS gas may be fatal. Symptoms include painful conjunctivitis, headache, nausea, dizziness, coughing and, in extreme cases, pulmonary edema and possible death. Irritant. Contact with skin can produce serious caustic burns with painful inflammation and possible destruction of tissue. Inflammation, tearing and pain may be expected. Severe contact can cause destruction of tissue.
Sodium Hydroxide	Corrosive	2 Mg/M3-Ceiling	Severe irritant. Effects from inhalation of dust or mist vary from mild irritation to serious damage of the upper respiratory tract, depending on severity of exposure. Symptoms may include sneezing, sore throat or runny nose. Contact with skin can cause irritation or severe burns and scarring with greater exposures. Causes irritation of eyes, and with greater exposures it can cause burns that may result in permanent impairment of vision, even blindness.
1 – Always add acid to water to prevent violent reactions.			
2 – Exposure limit refers to the OSHA regulatory exposure limit.			

SOP No.	Revision No.	Date	Page
AWC-Reactivity-100	5	December 8, 2005	Page 4 of 15

TITLE: REACTIVITY - METHOD SECT. 7.3

Supersedes: Revision 4

9.0 EQUIPMENT AND SUPPLIES

9.1 3-neck round bottom flasks (500ml capacity)

9.2 Ring stands

9.3 Stir plates

9.4 500 ml capacity scrubbers

9.5 Stir bars (approx. 1" length)

9.6 Flexible Tygon tubing for connections

9.7 Nitrogen gas source with flow meter

9.8 Copper tubing

9.9 Addition funnels (250 ml capacity)

9.10 Rubber stoppers for 3-neck flask

9.11 500 ml capacity jars

9.12 Fume hood

9.13 Analytical Balance

9.14 Buret

10.0 REAGENTS AND STANDARDS

10.1 Sulfuric Acid (0.01N), H_2SO_4: Add 70 ul concentrated H_2SO_4 into the 250 ml funnels of reagent water.

10.2 Sodium Hydroxide Solution (0.25N), NaOH: Purchased premade from VWR.

10.3 Cyanide Reference Solution, (1000 mg/L): 1000mg/L Free Cyanide standard purchased premade. It should be noted that a Free Cyanide standard must be used, as no heat is used in the method.

10.4 Sulfide Reference Solution, (570 mg/L): Dissolve 4.02 g of Na2S $9H_2O$ in approximately 900 ml of reagent water and dilute to 1 liter.

10.5 Standard Iodine Solution, (0.0250N): Dissolve 3.16 g Iodine and 23.0 g KI in approximately 900 ml reagent water and dilute to 1 liter.

10.6 Starch Indicator: Dissolve 10.0 g of soluble starch in 1 liter hot reagent water. Solution should be boiled until all of the starch is dissolved.

10.7 Sodium Thiosulfate Titrant (0.025N): Dissolve 6.21 g sodium thiosulfate ($Na_2S_2O_3$ $5H_2O$) and 0.4 g sodium hydroxide (NaOH) into 1 liter of distilled water. This titrant must be standardized before use as described in 13.2 and again monthly.

10.8 1:1 HCL: 500 ml HCL into 1 liter of reagent water.

10.9 Sodium Phosphate Buffer: 138g Sodium Phosphate Monobasic – Monohydrate into 1L DiH2O.

10.10 Pryidine-Barbituric Acid: Place 15g of Barbituric acid into a 250 ml volumetric flask, add just enough reagent water to wash the sides of the flask and wet the barbituric acid. Add 75 mls of pyridine and mix. Add 15 mls of concentrate HCL, mix, and cool to room temp. Dilute to 250 mls with DiH2O and mix. Refrigerate and store in a dark bottle.

10.11 Chloramine-T solution: Dissolve 1.0g Cloramine-T into 100mls of DiH2O. This solution must be made daily.

11.0 SAMPLE COLLECTION, PRESERVATION, SHIPMENT AND STORAGE

11.1 Samples containing, or which are suspected of containing a combination of cyanide and sulfide wastes should be collected with a minimum of aeration.

11.2 The sample bottle should be filled completely, excluding all headspace, and stoppered.

11.3 Although there are no method specified holding times it is suggested that all samples be tested as quickly as possible.

11.4 If unable to analyze immediately, samples should be stored at 4+/-2°C to decrease volatilization and in the dark to decrease photo decomposition.

11.5 Although samples can be preserved as described below, STL Buffalo does not recommend chemical preservation since this will cause dilution of the sample, increase ionic strength, and possibly change other physical or chemical characteristics of the waste. This may also affect the rate of release of the hydrocyanic acid and hydrogen sulfide.

11.5.1 Adjusting the sample pH to >12 with 6N NaOH can preserve cyanide wastes.

11.5.2 Adjusting the sample pH to >9 with 6N NaOH and adding four drops of 2N zinc acetate per 100 mls sample can preserve sulfide wastes.

TITLE: REACTIVITY - METHOD SECT. 7.3

Supersedes: Revision 4

12.0 QUALITY CONTROL

12.1 An acceptable calibration verification sample and calibration blank must bracket each group of sample analyses. All quality control data should be maintained and available for easy reference or inspection.

12.2 Laboratory Control Standard (LCS): Prepare a calibration standard using 10.0 ml of the 1000 mg/L cyanide reference solution (10.3), and 10.0 ml of 570 ppm sulfide reference solution (10.4). This must be carried through the entire distillation procedure at one per twenty samples. Begin the analytical procedure by running an LCS and then run one LCS after each additional 10 samples. Obtained values must be 10-100% of the true value for H2S and 20-100% of the true value for HCN.

12.3 Method Blank (MBLK): To determine freedom from contamination, prepare one calibration blank (MBLK) at the beginning of the analytical procedure and another (MBLK) after every ten samples and at the end of the analytical procedure. The blank consists of 10.0 ml reagent water that gets the same treatment as the samples and standards. The calibration blank must exhibit values less than the reporting limit.

12.4 Sample Duplicate (MS): A sample duplicate must be analyzed in every group of twenty or fewer samples. The RPD between duplicate analyses should be less than 20%.

13.0 CALIBRATION AND STANDARDIZATION

13.1 Standardization of cyanide reference solution (10.3):

13.1.1 Place 10.0 ml of the cyanide reference solution (10.3) into a flask. Add a few drops of rhodanine indicator (10.8) and titrate with silver nitrate solution (10.4) to a brownish pink color. 1ml titrant=1mg cyanide.

13.2 Standardization of 0.025N sodium thiosulfate titrant (10.9):

13.2.1Dissolve approximately 2.0 g KI in an Erlenmeyer flask with approximately 125 ml distilled water. Add a few drops of concentrated H_2SO_4 and 20.0 ml of the potassium biiodate standard (10.10). Dilute to 200 ml and titrate with sodium thiosulfate titrant, adding starch indicator toward the end, when a pale straw color is reached. Calculate the actual normality of the sodium thiosulfate by the following calculation:

$$\text{Normality } Na_2S_2O_3 = \frac{(20)(0.025)}{\text{mls } Na_2S_2O_3}$$

SOP No.	Revision No.	Date	Page
AWC-Reactivity-100	5	December 8, 2005	Page 7 of 15

TITLE: REACTIVITY - METHOD SECT. 7.3

Supersedes: Revision 4

14.0 PROCEDURE

14.1 Add 500 ml 0.25N sodium hydroxide (10.2) to a calibrated scrubber.

14.2 Assemble the system and adjust the flow rate of nitrogen to 60 ml/min.

14.3 Add 10.0 grams of the waste to be tested to the round bottom 3-neck flask.

14.4 Add approximately 250 ml 0.01N sulfuric acid (10.1) to the dropping funnel.

14.5 With the nitrogen flowing, begin stirring with a magnetic stirrer and stir plate. Begin the dropwise addition of the sulfuric acid from the dropping funnel.
NOTE: The stirring speed should remain constant and should not be fast enough to create a vortex.

14.6 After 30 minutes, close off the nitrogen and disconnect the scrubber. Determine the amount of cyanide and sulfide in the scrubber as described below.

14.7 Cyanide determination: Done by Cyanide method 335.2

14.7.1 Into a disposable culture tube add 1.5 ml of sodium phosphate buffer solution, 5 mls of sample and 0.2 ml of chloramine-T solution. Vortex to mix, wait 1-2 minutes. As soon as the time is up, add 0.5 ml of pyridine-barbituric and 2.8 mls of DI water. Vortex to mix, wait 8 minutes before reading on the spectrophotometer at a wavelength of 578 nm, zeroing the instrument on the calibration blank.

14.7.2 All samples must be read after 8 minutes and before 15 minutes, prepare only 10-20 samples at a time to ensure this time frame

14.7.3 If the concentration of cyanide exceeds the calibration curve for any sample, a dilution is required. A bench dilution may be prepared by pipetting an appropriate volume of distilled sample into a flask and dilution with 0.25 N NaOH. Repeat 14.7.1 for the colorimetric determination. A positive result for cyanide should be confirmed by reanalysis.

14.8 Cyanide determination done by Cyanide method 335.4

14.8.1 The Cyanide portion of this analysis may be run by Method 335.4 on the Lachat instrumentation.

14.9 Sulfide determination:

14.9.1 Into a 500ml flask place 1 ml of standard iodine solution (10.6). Bring the volume up to approximately 20 ml with reagent water.

14.9.2 Add 10 ml 6N HCl to the flask.

14.9.3 Pipet 200 ml of the scrubber solution into the flask, keeping the tip of the pipet below the surface of the iodine solution.

14.9.4 If the iodine color remains, record the amount used up to this point. If the yellow color disappears, add more iodine solution in 1 ml increments until the color remains. Record the total amount of iodine solution used.

14.9.5 Add enough starch indicator (10.7) for the solution to turn dark blue and titrate with 0.025N sodium thiosulfate (10.9) until the blue disappears. Record the volume of titrant used.

14.9.6 Final results should be reported as Total Releasable H_2S (mg/kg). Calculate as shown in 15.2.

15.0 CALCULATIONS

15.1 Cyanide (mg/kg): $\frac{(\text{ml } AgNO_3 \text{ used for sample - ml } AgNO_3 \text{ used for blank})(\text{N } AgNO_3)(52.04)(2{,}000)}{\text{sample weight (grams)}}$

15.2 Sulfide (mg/kg): $\frac{[(\text{ml I2})(\text{N I2})] - [(\text{ml titrant})(\text{N titrant})]\ (16{,}030)}{200 \text{ mls}} * \frac{500\text{mls}}{\text{grams of sample}}$

15.3 Percent Recovery for LCS:

$$\%\ \text{Recovery (LCS)} = 100\left(\frac{E}{C}\right)$$

where:

E = obtained (experimental) value
C = true value

15.4 Percent Recovery for Spikes:

$$\%\ \text{Recovery} = \left[\frac{(SSR - SR)}{SA}\right] \text{ x } 100$$

where:

SSR= spiked sample result
SR = sample result
SA = spike added

TITLE: REACTIVITY - METHOD SECT. 7.3

Supersedes: Revision 4

15.5 Relative Percent Difference (RPD):

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

where:
x_1 = analytical % recovery
x_2 = replicate % recovery

16.0 METHOD PERFORMANCE

16.1. Method Detection Limit: A valid method detection limit for each analyte of interest must be generated. The MDL must be below the reporting limit for each analyte. The procedure for determination of the method detection limit is given in 40 CFR Part 136, Appendix B. See STL SOP S-Q-003, "Method Detection Limit Studies," current revision, for further guidance. Current STL Buffalo MDLs are maintained the QA department and are easily viewed in the laboratory LIMs system.

16.2. A one–time initial demonstration of performance for each individual method for both soils and water matrices must be generated.

16.2.1. This requires quadruplicate analysis of a mid–level check standard containing all of the standard analytes for the method using the same procedures used to analyze samples, including sample preparation.

16.2.2. Calculate the average recovery and standard deviation of the recovery for each analyte of interest.

16.2.3. Compare these results with the acceptance criteria given in the Method or to laboratory historical limits (if available).

16.2.4. Repeat the test for any analyte that does not meet the acceptance criteria. Repeated failure for any analyte indicates the need for the laboratory to evaluate the analytical procedure and take corrective action.

16.3. Training Qualifications

16.3.1. The supervisor has the responsibility to ensure that this procedure is performed by an analyst who has been properly trained in its use and has the required experience.

16.3.2. The following analyst validation information is maintained for this method in the laboratory QA files.

16.3.2.1. The analyst must complete the laboratory safety orientation training that includes, but is not limited to, chemicals, PPE requirements, and electrical safety.

16.3.2.2. The analyst must read and understand this SOP.

16.3.2.3. The analyst must read and understand the Method used as reference for this SOP.

16.3.2.4. The analyst must complete a DOC or successfully analyze PT samples annually.

16.3.2.5. The analyst must complete the STL Quality Assurance Training.

17.0 DATA ASSESSMENT AND ACCEPTANCE CRITERIA FOR QUALITY CONTROL MEASURES

17.1. Acceptance Criteria:

17.1.1. ICAL: Calibration factor >0.9995

17.1.2. LCS (second source): 10-100% recovery

17.1.3. Method Blank:

17.1.3.1. Detected concentrations < PQL

or

17.1.3.2. Detected concentrations < 10X amount in associated samples

18.0 CORRECTIVE ACTIONS FOR OUT-OF-CONTROL DATA

18.1 ICAL: Analysis cannot begin without an acceptable calibration curve. Instrument maintenance may be required.

18.2 ICV: Reanalyze calibration curve if unacceptable ICV is obtained.

18.3 LCS: Reanalyze the LCS.

18.3.1 If 2nd analysis is acceptable, analytical sequence can continue, however the previous 10 samples must be reanalyzed.

18.3.2 If 2nd analysis is unacceptable, analyze a new ICAL.

18.4 Method Blank: Re-extract all samples associated with an unacceptable method blank. Samples and data are acceptable if results are below detection limit.

18.4.1 Samples: If the LCS or MBLK fail high, all samples with results below the detection limit can be accepted. If the LCS fails low, all samples must be reanalyzed.

TITLE: REACTIVITY - METHOD SECT. 7.3

Supersedes: Revision 4

19.0 CONTINGENCIES FOR HANDLING OUT-OF-CONTROL OR UNACCEPTABLE DATA

19.1.1. A Job Exception Form must be completed and filed with the Project Manager and QA Manager for any of the following conditions:

19.1.2. Holding times exceeded

19.1.3. Insufficient sample volume for re-distillation

19.1.4. In the event of unknown positives or sample matrix which present the analyst with questionable data, the project manager shall be notified so the client may be contacted and involved in the decision process and course of action

20.0 WASTE MANAGEMENT/ 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."

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

- Acidic sample waste generated by sample digestion and disposed of in the "A" waste containers.
- Alkaline sample waste remaining in scrubbers is disposed of in the "D" waste containers.

21.0 REFERENCES

21.1 EPA Test Methods for Evaluation Solid Waste, Physical/Chemical Methods (SW-846), Third Edition, Update III, December 1996; Section 7.3, 9012, 9014 and 9034.

22.0 TABLES, DIAGRAMS, FLOWCHARTS AND VALIDATION DATA

22.1 Distillation Sequence

22.2 Analytical Sequence

22.3 Analytical Batch for Reactive CN

22.4 Analytical Batch for Reactive Sulfide

22.5 Wet Chemistry Batch Summary

TITLE: REACTIVITY - METHOD SECT. 7.3

Supersedes: Revision 4

23.0 CHANGES FROM PREVIOUS REVISION

23.1 Laboratory Director change, signature update

23.2 Updated Attachments 22.1, 22.2, 22.3, 22.4 and 22.5

23.3 Updated sections 15.0, 16.0, 17.0, 18.0, 19.0

22.1 Distillation sequence

LCS – 1000 ppm CN and 570 ppm H2S
MBLK
Sample
Sample
Sample
Sample
Sample
Sample
Sample
Sample
Sample
Sample
Sample
Sample
Sample
Sample
Sample
Sample
Sample
Sample
Sample
Sample duplicate
LCS - 1000 ppm CN and 570 ppm H2S
MBLK

22.2 Analytical Sequence

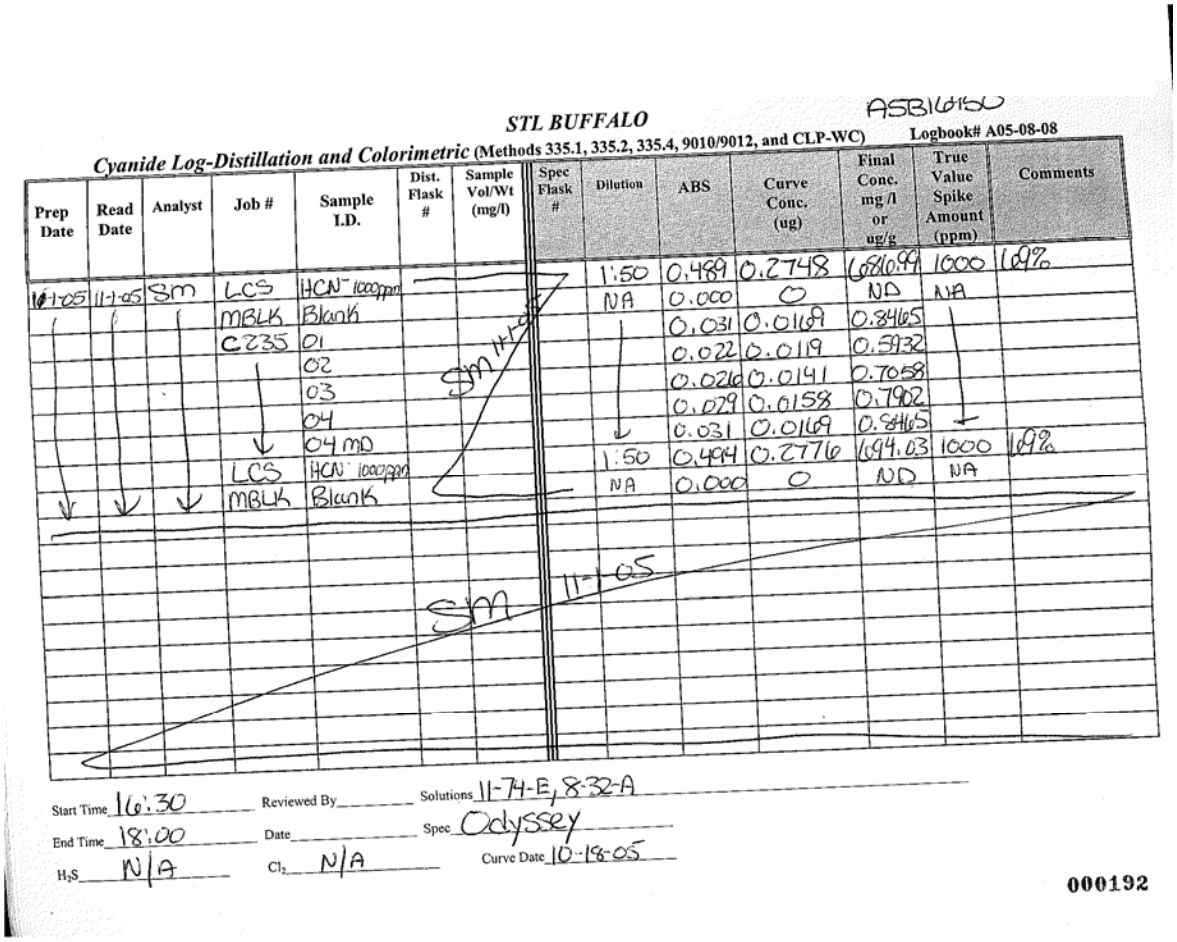
LCS – 1000 ppm CN and 570 ppm H2S
MBLK
Sample
Sample
Sample
Sample
Sample
Sample
Sample

TITLE: REACTIVITY - METHOD SECT. 7.3

Supersedes: Revision 4

Sample
Sample
Sample duplicate
LCS – 1000 ppm CN and 570 ppm H2S
MBLK

22.3 Analytical Batch – Reactive Cyanide



STL BUFFALO

ASB16150

Cyanide Log-Distillation and Colorimetric (Methods 335.1, 335.2, 335.4, 9010/9012, and CLP-WC) Logbook# A05-08-08

Prep Date	Read Date	Analyst	Job #	Sample I.D.	Dist. Flask #	Sample Vol/Wt (mg/l)	Spec Flask #	Dilution	ABS	Curve Conc. (ug)	Final Conc. mg/l or ug/g	True Value Spike Amount (ppm)	Comments
10-7-05	11-1-05	SM	LCS	HCN 1000ppm				1:50	0.489	0.2748	6860.99	1000	69%
			MBLK	Blank				NA	0.000	0	NA	NA	
			C235	01					0.031	0.0169	0.8465		
				02					0.022	0.019	0.5932		
				03					0.026	0.0141	0.7058		
				04					0.029	0.0158	0.7902		
				04 MD					0.031	0.0169	0.8465		
			LCS	HCN 1000ppm				1:50	0.494	0.2776	694.03	1000	69%
			MBLK	Blank				NA	0.000	0	ND	NA	

SM 11-1-05

Start Time 16:30 Reviewed By _____ Solutions 11-74-E, 8-32-A

End Time 18:00 Date _____ Spec Odyssey

H_2S N/A Cl_2 N/A Curve Date 10-18-05

000192

SOP No.	Revision No.	Date	Page
AWC-Reactivity-100	5	December 8, 2005	Page 14 of 15

TITLE: REACTIVITY - METHOD SECT. 7.3

Supersedes: Revision 4

22.4 Analytical Batch - Reactive Sulfide

STL BUFFALO
REACTIVITY LOG
Logbook # A04-8-6

Date	Anal.	Job #	Sample I.D.	N_2 Flow	Sample Wt. (g)	Scrubber Vol. (ml)	Time (Min)	Total Available Sulfide: I_2 Vol. (ml)	Total Available Sulfide: Sample Vol. (ml)	Total Available Sulfide: Titrant Vol. (ml)	Total Available Sulfide: Final conc. H_2S (mg/kg)	Comments
11-23-05	SM	LCS	HCN^-	100	10	500	30	N/A			1	HCN^- Only
		LCS	H_2S					2	200	1.8	201	35%
		MBLK	Blank					1	200	1.0	ND	
		D235	01					1	200	1.1	ND	
		↓	02					1	200	1.1	ND	
		D348	01					1	200	1.0	ND	
		D266	01					1	200	1.0	ND	
		↓	02					1	200	1.1	ND	
		D241	01					1	200	1.1	ND	
			02					1	200	1.0	ND	
		↓	03					1	200	1.0	ND	
		D309	01					1	200	1.0	ND	
		D320	01					1	200	1.0	ND	
		LCS	H_2S					2	200	1.8	201	35%
		MBLK	Blank					1	200	1.0	ND	
		D322	01					1	200	1.1	ND	
		D372	01					1	200	1.1	ND	
		↓	01 MD	N/A				1	200	1.1	ND	
↓	↓	LCS	H_2S	N/A				2	200	1.8	201	35%

Start/End Time 15:00/19:00 Solutions CHA-120-F, 8-20-C, CHA-75-E, CHA-94-B, 9-17-B, CHA-16-I

Reviewed By/ Date _____ N I_2= 0.025 N NaS_2O_3= 0.025

SOP No.	Revision No.	Date	Page
AWC-Reactivity-100	5	December 8, 2005	Page 15 of 15

TITLE: REACTIVITY - METHOD SECT. 7.3

Supersedes: Revision 4

22.5 Wet Chemistry Batch Summary

WET CHEMISTRY BATCH SUMMARY

PARAMETER_____ METHOD_____ BATCH _____

COMMENTS	JOB NUMBER
WC Reporting Limit < STL Quant Limit	
WC Historical confirms within Hold Time	
WC Historical NO confirm & RE outside of HT	
WC Hold Time Exceedance-Dilution required	
WC Hold Time Exceedance-Instrument Failure	
WC Holding Time Exceedance by Date	
WC Holding Time Exceedance by Hours	
WC LCS within ERA limits outside internal	
WC LCS high recovery, sample ND	
WC MBLK hit but samples > 10X blank value	
WC RPD Exceedance for MS / SD	
WC Spike Failure HIGH MS only	
WC Spike Failure LOW MS only	
WC Spike Failure MS and SD	
WC BOD HT met- Oxygen depleted-RE out HT	
WC Carbonate Alkalinity, LCS/MBLK	
WC Reactivity Qualification	
WC TDS/Conductivity ratio outside of range	
WC TOX Breakthrough- no volume for redo	
WC TOX samples were centrifuged	
Other	

DILUTION CODES	REASON
002	Sample matrix effects
003	Excessive foaming
004	High levels of non-target compounds
008	High concentration of target analytes
009	Sample turbidity
010	Sample color
011	Insufficient volume for lower dilution
012	Sample viscosity
013	other

ICAL Compliant?	YES	NO	NA	IF NO, Why? _____
LCS/CCV Compliant?	YES	NO	NA	IF NO, Why? _____
CCB Compliant?	YES	NO	NA	IF NO, Why? _____
RPD Compliant?	YES	NO	NA	IF NO, Why? _____
ERA Compliant?	YES	NO	NA	IF NO, Why? _____

NUMBER of REANALYSIS FOR THIS BATCH:_____

Analyst_____ Date_____

Time Critical Batch Review_____ Date_____

Secondary Review & Closure_____ Date_____ WC Summary Rev 4 / 5-2005

STL Buffalo
LABORATORY STANDARD OPERATING PROCEDURES

SOP No.	Revision No.	Effective Date	Page
AWC-TOC-15	10	December 8, 2005	1 of 17

TITLE: TOTAL ORGANIC CARBON: METHODS 415.1/9060

SUPERCEDES: Revision 9

REVIEWED & APPROVED BY:	SIGNATURE	DATE
Christopher Spencer, Laboratory Director		
Verl D. Preston, Quality Manager		
Peggy Gray-Erdmann, Supervisor		

1.0 IDENTIFICATION OF TEST METHOD

1.1. This SOP was derived from EPA Method 415.1 and SW846 Third Edition Method 9060.

2.0 APPLICABLE MATRIX

2.1. Applicable matrices are all aqueous samples or solid wastes.

3.0 REPORTING LIMIT

3.1. The standard reporting limit is 1.0 mg/l.

4.0 SCOPE AND APPLICATION

4.1. This method is applicable to all aqueous samples including ground, drinking, surface, and saline waters, as well as domestic and industrial wastes.

4.2. Solid waste may be analyzed for leachable Total Organic Carbon after first generating a leachate using the ASTM Shake Extraction Procedure.

TITLE: TOTAL ORGANIC CARBON: METHODS 415.1/9060

SUPERCEDES: Revision 9

5.0 SUMMARY OF THE TEST METHOD

5.1. Total Carbon (TC), Total Inorganic Carbon (TIC) and Total Organic Carbon (TOC) are all determined by wet oxidation. Each form of carbon is ultimately measured as carbon dioxide (CO_2) by a nondispersive infrared detector (NDIR) that has been calibrated to directly display the mass of CO_2 detected.

5.2. TIC is determined by measuring the carbon dioxide released by sample acidification. As the pH of the sample is lowered, carbonate and bicarbonate ions are converted to dissolved carbon dioxide. The dissolved carbon dioxide is carried into a NDIR calibrated to directly display the mass of carbon dioxide detected.

5.3. TOC is determined by measuring the carbon dioxide released by chemical oxidation of the organic carbon in the sample. After the sample has been acidified and purged of TIC, sodium persulfate, a strong oxidizer is added. This oxidant quickly reacts with organic carbon in the sample at 100°C to form carbon dioxide. When the oxidation reaction is complete, the carbon dioxide is purged from the solution and detected as described for TIC.

5.4. TC is determined by measuring the carbon dioxide released by complete oxidation of all carbon present in the sample (inorganic and organic). For this analysis, first add acid and persulfate to the sample and allow a specific reaction time to convert all carbon present to carbon dioxide. When the reaction is complete, the resulting carbon dioxide is purged from the solution and detected as described for TIC.

5.5. Glassware for this analysis must be HCl washed.

6.0 DEFINITIONS

6.1. All definitions are consistent with those described by STL's Corporate Quality Assurance Management Plan.

6.2. Organic carbon: carbon present in the form of organic carbon-based compounds.

6.3. Inorganic carbon: carbon present in the form of inorganic compounds; usually carbon dioxide.

7.0 INTERFERENCES

7.1. Inorganic halides in samples compete with the organics for persulfate. The Model 1010 is able to analyze samples with up to 30 mg of chlorine without any modification. When samples contain over 30 mg of chlorine, additional persulfate reagent, increased TOC react time, and a halide scrubber option are necessary.

TITLE: TOTAL ORGANIC CARBON: METHODS 415.1/9060

SUPERCEDES: Revision 9

8.0 SAFETY

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

8.2. SPECIFIC SAFETY CONCERNS OR REQUIREMENTS

8.2.1. The Sodium Persulfate is a strong oxidizer. Avoid contact with combustible materials, organic materials, strong reducing agents, and excess heat.

8.3. PRIMARY MATERIALS USED

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

Material (1)	Hazards	Exposure Limit (2)	Signs and symptoms of exposure
Sulfuric Acid	Corrosive Oxidizer Dehydra-dator	1 mg/m^3	This material will cause burns if comes into contact with the skin or eyes. Inhalation of vapors will cause irritation of the nasal and respiratory system.
Phosphoric Acid	Corrosive	1 Mg/M3 TWA	Inhalation is not an expected hazard unless misted or heated to high temperatures. May cause redness, pain, and severe skin burns. May cause redness, pain, blurred vision, eye burns, and permanent eye damage.
Sodium Persulfate	Oxidizer Corrosive	0.1 Mg/M3-TWA as Persulfates	Causes irritation to the respiratory tract. Symptoms may include sore throat, shortness of breath, inflammation of nasal passages, coughing, and wheezing. Causes severe irritation or burns to the skin and eyes. Symptoms include redness, itching, pain and burns. May cause allergic skin reactions. Can cause eye damage.
1 – Always add acid to water to prevent violent reactions.			
2 – Exposure limit refers to the OSHA regulatory exposure limit.			

TITLE: TOTAL ORGANIC CARBON: METHODS 415.1/9060

SUPERCEDES: Revision 9

9.0 EQUIPMENT AND SUPPLIES

9.1. O·I·Analytical Carbon Analyzer (Model 1010) with corresponding autosampler; O·I·Analytical (Model 1051).

9.2. Sample vials (40-mL vials with caps and septa).

9.3. 100 mL volumetric flasks

9.4. Class A pipettes.

9.5. Eppendorfs with a range of 100ul to 1000ul and 500ul to 5000ul.

10.0 REAGENTS AND STANDARDS

10.1. Carbon-free water used for initial calibration blanks (ICB) and method blanks (MBLK) to make up all standards.

10.2. 1000ppm Stock Standard KHP standard purchased from two separate vendors, (KHP #1 and KHP #2). ERA standard purchased from Environmental Resource Associates may be substituted for 60 PPM KHP standard.

10.3. 60.0-ppm initial calibration verification (ICV) and Laboratory Control Standard (LCS): dilute 60.0-ml stock 1000 ppm KHP #2 STD to 1000 ml in a volumetric flask.

10.4. Matrix Spike Solution (MS): 880 ul of 1000 mg/l KHP#2 STD is added to 40 ml of sample. The final expected concentration of spike in the sample is 20 mg/l.

10.5. Prepare calibration standards by diluting the following volumes of 1,000 mg/l KHP #1 STD with 100 ml carbon free water in a 100 ml volumetric flask. Invert several times before using. Measure standards as indicated in the following table.

Concentration	Volume
100.0 mg/l	10.0 ml 1,000mg/l KHP STD dilute up to 100 ml with carbon free DiH_2O.
50.0 mg/l	5.0 ml 1,000mg/l KHP STD dilute up to 100 ml with carbon free DiH_2O.
10.0 mg/l	1.0 ml 1,000mg/l KHP STD dilutes up to 100 ml with carbon free DiH_2O.
1.0 mg/l	0.1 ml 1,000mg/l KHP STD dilute up to 100 ml with carbon free DiH_2O.
0 mg/l	Carbon free DiH_2O.

10.6. (20%) Sodium Persulfate reagent. Add 400g of $Na_2S_2O_8$ to carbon free deionized water to create a total final reagent volume of 2 liter. Stirring may be necessary; do not heat. The shelf life for this solution is approximately three weeks.

10.7. (5%) Phosphoric Acid reagent. Prepare a (5%) phosphoric acid solution by adding 118.0 ml of reagent grade (85%) H_3PO_4 to carbon free deionized water to create a total final volume of 2 liter caution; exothermic).

10.8. UHP Nitrogen tanks (carrier gas).

16.0 SAMPLE COLLECTION, PRESERVATION, SHIPMENT AND STORAGE

16.1. Aqueous samples should be collected in 40-mL vials with caps and septa with zero headspace, preserved to pH<2 with Hydrochloric Acid, and stored at 4±2°C until time of analysis.

16.2. Soil samples should be collected in a 4oz glass wide jar and stored at 4±2°C until time of analysis.

16.3. Holding time for preserved samples is 28 days from sample collection.

17.0 QUALITY CONTROL

12.1 ICV: Initial Calibration Verification: must be analyzed immediately after the calibration curve using a second source standard (separate from the curve) to verify that the calibration curve is acceptable.

12.2 ICB: Calibration blanks must be analyzed after every calibration curve.

12.3 LCS: A LCS must be analyzed at the beginning of every sequence, and for every 10 or fewer samples analyzed.

12.4 MBLK: Method blanks must be analyzed at the beginning of every sequence, and for every 10 or fewer samples analyzed.

12.5 Matrix Duplicate (MD) and a sample Matrix Spike (MS) must be analyzed every 20 samples or fewer for method 415.1. A MD and MS must be analyzed every 10 samples of fewer for method 9060.

12.5.1 3rd Edition and 40 CFR protocols require MD and MS every 20 samples.

12.5.2 New York State ASP protocol requires MD and MS for every 10 samples.

18.0 CALIBRATION AND STANDARDIZATION

18.1. The TOC-1010 must be calibrated using a five-point curve at a minimum frequency of three months (section 10.4).

18.2. Prepare standard curve by plotting instrument response against concentration values. The curve is a linear curve. A calibration curve may be fitted to the calibration solution concentration/response data

using the computer. Acceptance or control limits should be established using the difference between the measured values of the calibration solution and the "true value" concentration. Acceptance criteria for the calibration curve is a correlation coefficient (R value) $\geq$0.995.

19.0 PROCEDURE

19.1. The autosampler carousel is capable of holding up to 53 sample vials. Place LCS's, Blanks, MS, MD and samples in appropriate slots in the carousel (Attachment 22.1). It is recommended that 5 reagent blanks be run at the beginning of each analytical run.

19.2. The autosampler carousel is secured to the autosampler deck and covered with the carousel cover. (Note: the carousel cover must be in place to operate the autosampler.)

19.3. Set up the analytical sequence by entering the number of samples and number of injections per sample (consult the instrument software manual for details).

19.3.1. For Method 415.1, a minimum of two injections is required.

19.3.2. Method 9060 requires four injections.

19.4. Activate "Auto Start" from the software menu (consult the instrument software manual for details). A printout of results will follow. (Consult O·I's Technical manuals for software specific functionality).

19.5. Dilute any samples that have an excessive amount of particles, as to not clog the needle. (Note dilution on run log).

14.6 Leachable TOC samples are prepared using ASTM Leaching procedure. The extract is analyzed on the instrument.

19.5.1. Dry weights must be performed on all soil samples to adjust the final reported concentration.

20.0 CALCULATIONS

15.1 If the result of any injection exceeds the linear range of the calibration, the sample must be re-analyzed using a dilution.

15.2 Leachable TOC samples and soil QC are calculated in AIMS:

$$\frac{\textit{TOC result (mg/l)}}{\text{Weight of the sample (g)}} \times \textit{final volume (soil and de ionized water) (ml)}$$

Then dry weight correct this calculated result. Result is divided by Dry Weight.

15.3 Percent Recovery for Analyses Involving Spikes:

$$\% \text{ Recovery } = \left[\frac{(SSR - SR)}{SA} \right] \text{ x } 100$$

where:

SSR = spiked sample result
SR = sample result
SA = spike added

15.4 Relative Percent Difference (RPD):

$$RPD = \frac{|x_1 - x_2|}{\left(\frac{x_1 + x_2}{2} \right)} \text{ x } 100$$

where:

x_1 = analytical % recovery
x_2 = replicate % recovery

15.5 Percent Recovery for LCS**:**

$$\% \text{ Recovery (LCS)} = 100 \left(\frac{E}{C} \right)$$

where:
E =obtained (experimental) value
C =true value

16.0 METHOD PERFORMANCE

16.1. Method Detection Limit: A valid method detection limit for each analyte of interest must be generated. The MDL must be below the reporting limit for each analyte. The procedure for determination of the method detection limit is given in 40 CFR Part 136, Appendix B. See STL SOP S-Q-003, "Method Detection Limit Studies," current revision, for further guidance. Current STL Buffalo MDLs are maintained the QA department and are easily viewed in the laboratory LIMs system.

16.2. A one–time initial demonstration of performance for each individual method for both soils and water matrices must be generated.

16.2.1. This requires quadruplicate analysis of a mid–level check standard containing all of the standard analytes for the method using the same procedures used to analyze samples, including sample preparation.

16.2.2. Calculate the average recovery and standard deviation of the recovery for each analyte of interest.

16.2.3. Compare these results with the acceptance criteria given in the Method or to laboratory historical limits (if available).

16.2.4. Repeat the test for any analyte that does not meet the acceptance criteria. Repeated failure for any analyte indicates the need for the laboratory to evaluate the analytical procedure and take corrective action.

16.3. Training Qualifications

16.3.1. The supervisor has the responsibility to ensure that this procedure is performed by an analyst who has been properly trained in its use and has the required experience.

16.3.2. The following analyst validation information is maintained for this method in the laboratory QA files.

16.3.2.1. The analyst must complete the laboratory safety orientation training that includes, but is not limited to, chemicals, PPE requirements, and electrical safety.

16.3.2.2. The analyst must read and understand this SOP.

16.3.2.3. The analyst must read and understand the Method used as reference for this SOP.

16.3.2.4. The analyst must complete a DOC or successfully analyze PT samples annually.

16.3.2.5. The analyst must complete the STL Quality Assurance Training.

17.0 DATA ASSESSMENT AND ACCEPTANCE CRITERIA FOR QUALITY CONTROL MEASURES

17.1 ICAL: calibration factor >0.995

17.2 ICV (second source): Within $\pm$10% of true value

17.3 LCS: Within $\pm$10% of true value

17.4 Method Blank:

17.4.1 Detected concentrations < PQL or

17.4.2 Detected concentrations < 10X amount in associated samples

17.5 MS/MSD: acceptance limits are calculated yearly based on historical results and available in the LIMs system.

17.6 MD: Duplicate RPD <= 20.0%

17.7 RPD between sample injection must be within 10% of each other. Reanalysis is required of any sample in which the injections RPD exceed 10%.

18.0 CORRECTIVE ACTIONS FOR OUT-OF-CONTROL DATA

18.1 ICAL: Analysis cannot begin without an acceptable calibration curve. Instrument maintenance may be required. Please refer to STL Corporate Policy for information on the proper selection of calibration points.

18.2 ICV: Reanalyze calibration curve if unacceptable ICV is obtained.

18.3 CCV: Reanalyze the CCV.

18.3.1 If 2nd analysis is acceptable, analytical sequence can continue, however the previous 10 samples must be reanalyzed.

18.3.2 If 2nd analysis is unacceptable, analyze a new ICAL.

18.4 Method Blank: Method Blank values must be less than the STL Quantitation limit. Re-analyze all samples associated with an unacceptable method blank with the following exceptions:

18.4.1 Detected concentrations < PQL or

18.4.2 Detected concentrations < 10X amount in associated samples

18.4.3 All blanks associated with DOD QSM and AFCEE samples must be less than half the Quantitation limit.

18.5 LCS:

18.5.1 If below limits: Re-analyze all samples associated with an unacceptable LCS

18.5.2 If above limits: Re-analysis is not required if samples are ND.

18.6 MS/MSD:

18.6.1 Matrix interference can be assumed and corrective action is not required if both of the following conditions are met:

18.6.1.1MSB recovery is acceptable

18.6.1.2Recoveries in both MS and MSD are consistent (%RSD<30)

18.6.2 If recoveries in MS/MSD are different (e.g.: one high, one low) further evaluation should be made. Matrix interference can not be assumed in this case. Discussion with the department supervisor, operations manager or QA manager should be included in the final decision process prior to releasing data.

18.7 If there is a noticeable difference between the injection replications, reset the sample. Each replicate injection should agree within ±10%.

19.0 CONTINGENCIES FOR HANDLING OUT-OF-CONTROL OR UNACCEPTABLE DATA

19.1 A Job Exception Form must be completed and filed with the Project Manager and QA Manager for any of the following conditions:

19.1.1. Holding times exceeded

19.1.2. Insufficient sample volume for re-analysis

19.1.3. In the event of unknown positives or sample matrix which present the analyst with questionable data, the project manager shall be notified so the client may be contacted and involved in the decision process and course of action

20.0 WASTE MANAGEMENT/ POLLUTION PREVENTION

20.1. 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."

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

20.2.1. Acidic waste from the auto-analyzer must be disposed of in the "A" waste container.

21.0 REFERENCE

21.1. Standard Methods for the Examination of Water and Wastewater, 18th Edition, American Public Health Association/ American Water Works/ Water Environment Federation, Washington, DC.

21.2. Method 415.1, "Methods for Chemical Analysis of Water and Wastes", U.S. Environmental Protection Agency, Environmental Monitoring and Support Laboratory, Revised March 1983

21.3. Method 9060, "Test Methods for Evaluating Solid Waste"; SW-846, Third Edition, 12/96.

22.0 TABLES, DIAGRAMS, FLOWCHARTS AND VALIDATION DATA

22.1. Analytical Run Sequence for Method 415.1

22.2 Analytical Run Sequence for Method 9060

22.3 Analytical Batch

22.4 Wet Chemistry Batch Summary and Data Review Checklist

23.0 CHANGES FROM PREVIOUS REVISION

23.1. Updated section 10.3 with the addition of an ERA standard.

23.2. Updated formulas for Sodium Persulfate and Phosphoric Acid so that the final volume is 2 liters.

23.3. Updated sections 15.0, 16.0, 17.0, 18.0 and 19.0

23.4. Updated section 13.1 to say that a curve is to be run every three months or sooner if needed.

23.5. Updated section 14.3 to include 5 reagent blanks to be run before each analytical sequence.

23.6. Updated attachments 221.1 and 22.3

23.7. Analytical Run Sequence for Method 415.1

Calibration Curve: analyzed at a minimum of once every three months
ICV
ICB

LCS
MBLK
Sample
Sample
Sample
Sample
Sample
Sample
Sample
Sample
Sample
Sample

LCS
MBLK
Sample
Sample

Sample
Sample
Sample
Sample
Sample
Sample
Sample Duplicate (MD)
Sample Spike (MS)
LCS
MBLK

23.8 Analytical Run Sequence for Method 9060

Calibration Curve: analyzed at a minimum of once every three months
ICV
ICB

LCS
MBLK
Sample
Sample
Sample
Sample
Sample
Sample
Sample
Sample
Sample Duplicate (MD)
Sample Spike (MS)
LCS
MBLK
Sample
Sample
Sample
Sample
Sample
Sample
Sample
Sample
Sample Duplicate (MD)
Sample Spike (MS)
LCS
MBLK

TITLE: TOTAL ORGANIC CARBON: METHODS 415.1/9060

SUPERCEDES: Revision 9

23.9 Analytical Batch

Page 1 of 2

SEQUENCE

090203-1 Tue Sep 02 11:13:08 2003

Pos/ Vial	Sample Name	Method	Run Type	# Rep	Vol (mL)	# Blk	Ovr Rng	Remarks
1	CLEANING	default	Sample	5	1.000	0	No	
2	ICV	default	Chk. 1	4	1.000	0	No	
3	ICB	default	Chk. 2	4	1.000	0	No	
4	835001	default	Sample	2	1.000	0	No	
5	831609	default	Sample	4	1.000	0	No	
6	831610	default	Sample	4	1.000	0	No	
7	831611	default	Sample	4	1.000	0	No	
8	831612	default	Sample	4	1.000	0	No	
9	831613	default	Sample	4	1.000	0	No	A3B09803
10	831614	default	Sample	4	1.000	0	No	
11	831615	default	Sample	4	1.000	0	No	
12	831616	default	Sample	4	1.000	0	No	1-176-A
13	832201	default	Sample	4	1.000	0	No	1-176-B
14	CCV	default	Chk. 1	4	1.000	0	No	1-176-C
15	CCB	default	Chk. 2	4	1.000	0	No	
16	832202	default	Sample	4	1.000	0	No	CHA-22-17
17	832203	default	Sample	4	1.000	0	No	
18	832204	default	Sample	4	1.000	0	No	
19	832205	default	Sample	4	1.000	0	No	
20	832602	default	Sample	4	1.000	0	No	
21	832603	default	Sample	4	1.000	0	No	
22	832604	default	Sample	4	1.000	0	No	
23	832604 DUP	default	Sample	4	1.000	0	No	
24	832605	default	Sample	4	1.000	0	No	
25	832605 SPK	default	Sample	4	1.000	0	No	
26	CCV	default	Chk. 1	4	1.000	0	No	
27	CCB	default	Chk. 2	4	1.000	0	No	
28	832606	default	Sample	4	1.000	0	No	
29	832607	default	Sample	4	1.000	0	No	
30	832608	default	Sample	4	1.000	0	No	
31	832609	default	Sample	4	1.000	0	No	
32	832610	default	Sample	4	1.000	0	No	
33	832611	default	Sample	4	1.000	0	No	
34	832612	default	Sample	4	1.000	0	No	
35	832613	default	Sample	4	1.000	0	No	
36	832614	default	Sample	4	1.000	0	No	
37	832615	default	Sample	4	1.000	0	No	
38	CCV	default	Chk. 1	4	1.000	0	No	
39	CCB	default	Chk. 2	4	1.000	0	No	
40	832616	default	Sample	4	1.000	0	No	
41	832617	default	Sample	4	1.000	0	No	
42	832619	default	Sample	4	1.000	0	No	
43	832620	default	Sample	4	1.000	0	No	
44	833001	default	Sample	4	1.000	0	No	

TITLE: TOTAL ORGANIC CARBON: METHODS 415.1/9060

SUPERCEDES: Revision 9

Page 2 of 2

SEQUENCE

090203-1 Tue Sep 02 11:13:08 2003

Pos/ Vial	Sample Name	Method	Run Type	# Rep	Vol (mL)	# Blk	Ovr Rng	Remarks
45	833002	default	Sample	4	1.000	0	No	
46	833003	default	Sample	4	1.000	0	No	
47	833001 DUP	default	Sample	4	1.000	0	No	
48	833005	default	Sample	4	1.000	0	No	
49	833005 SPK	default	Sample	4	1.000	0	No	
50	CCV	default	Chk. 1	4	1.000	0	No	
51	CCB	default	Chk. 2	4	1.000	0	No	

SOP No.	Revision No.	Effective Date	Page
AWC-TOC-15	10	December 8, 2005	15 of 17

TITLE: TOTAL ORGANIC CARBON: METHODS 415.1/9060

SUPERCEDES: Revision 9

```
**********************************************************************
**                          CONFIGURATION                           **
**********************************************************************

Analysis Mode:  TIC/TOC     Spl Intro:  Autosampler 53

Loop Size:  1 mL          Actual Volume  1mL   5mL   10mL   25mL
                          -------------  ----  ----  -----  -----
                           Loop A (uL):  1030  5080  10000  25000
                           Loop B (uL):  1030  5090  10000  25000

Tray Type:              53 Vial  Vial Option:          Septum Piercing
Needle Depth:           95 %     Preacid Volume (uL):              000
Wash Needle Depth:      95 %     Preacid Purge Time (min:sec):    0:00

             TIC    TOC    TC
Blank       ----   ----   ----           Linearization Coeff:  62000
 Average:     50   2732      0

                                Sample Transfer Times (sec)
          Initial Fill          |          Loop Fill          | Sample Inject
     Non-AS    AS  AS w/Sep | Non-AS    AS  AS w/Sep |     (all)
     ------  ----- -------- | ------  ----- -------- | ----------
1mL:    6.0   4.5      3.5 |    1.2   1.2      1.0 |        4.5
5mL:    8.1   7.2      6.8 |    5.1   5.1      4.2 |        9.3
10mL:  14.2  12.2     11.0 |   10.5  10.5     10.5 |       16.5
25mL:  35.0  35.0     32.0 |    n/a   n/a      n/a |       38.0
```

SOP No.	Revision No.	Effective Date	Page
AWC-TOC-15	10	December 8, 2005	16 of 17

TITLE: TOTAL ORGANIC CARBON: METHODS 415.1/9060

SUPERCEDES: Revision 9

Pos/ Vial	Run Type	Rep #	Run Date	Run Time	Data Filename	TIC Area (cts)	TIC Mass (ugC)	TIC Conc (ppm)	TOC Area (cts)	TOC Mass (ugC)	TOC Conc (ppm)	TC Area (cts)	TC Mass (ugC)	TC Conc (ppm)	
1	Spl	1	02Sep2003	11:24	555149	421	0.423	0.4109	651	0.000	0.0000	-	-	-	
1	Spl	2	02Sep2003	11:33	555150	267	0.248	0.2403	772	0.000	0.0000	-	-	-	
1	Spl	3	02Sep2003	11:43	555151	251	0.229	0.2226	704	0.000	0.0000	-	-	-	
1	Spl	4	02Sep2003	11:53	555152	282	0.265	0.2570	810	0.000	0.0000	-	-	-	
1	Spl	5	02Sep2003	12:02	555153	88	0.043	0.0421	899	0.000	0.0000	-	-	-	
1	Spl	Avg	-	-	-	261	0.242	0.2346	767	0.000	0.0000	-	-	-	CLEANING
1	Spl	SDev	-	-	-	118.43	-	-	95.82	-	-	-	-	-	
1	Spl	%RSD	-	-	-	45.24	-	-	12.49	-	-	-	-	-	
2	Chk1	1	02Sep2003	12:13	555154	-	-	-	58249	65.705	63.7917	-	-	-	
2	Chk1	2	02Sep2003	12:23	555155	-	-	-	58982	66.542	64.6035	-	-	-	
2	Chk1	3	02Sep2003	12:32	555156	-	-	-	59382	66.998	65.0466	-	-	-	
2	Chk1	4	02Sep2003	12:42	555157	-	-	-	58671	66.187	64.2591	-	-	-	ICV
2	Chk	Avg	-	-	-	-	-	-	58821	66.358	64.4252	-	-	-	101%
2	Chk	SDev	-	-	-	-	-	-	479.70	-	-	-	-	-	
2	Chk	%RSD	-	-	-	-	-	-	0.82	-	-	-	-	-	
3	Chk2	1	02Sep2003	12:52	555158	-	-	-	878	0.256	0.2488	-	-	-	
3	Chk2	2	02Sep2003	13:02	555159	-	-	-	740	0.099	0.0960	-	-	-	
3	Chk2	3	02Sep2003	13:12	555160	-	-	-	777	0.141	0.1369	-	-	-	
3	Chk2	4	02Sep2003	13:22	555161	-	-	-	794	0.160	0.1558	-	-	-	ICB
3	Chk	Avg	-	-	-	-	-	-	797	0.164	0.1594	-	-	-	
3	Chk	SDev	-	-	-	-	-	-	58.36	-	-	-	-	-	
3	Chk	%RSD	-	-	-	-	-	-	7.32	-	-	-	-	-	
4	Spl	1	02Sep2003	13:32	555162	115229	131.397	127.5698	11861	10.414	10.1111	-	-	-	
4	Spl	2	02Sep2003	13:42	555163	119234	135.966	132.0056	12097	10.684	10.3725	-	-	-	8350
4	Spl	Avg	-	-	-	117231	133.681	129.7877	11979	10.549	10.2418	-	-	-	01
4	Spl	%RSD	-	-	-	2831.96	-	-	166.88	-	-	-	-	-	
						2.42	-	-	1.39	-	-	-	-	-	
5	Spl	1	02Sep2003	13:52	555164	67432	76.870	74.6308	2277	0.000	0.0000	-	-	-	
5	Spl	2	02Sep2003	14:02	555165	68125	77.660	75.3984	2228	0.000	0.0000	-	-	-	
5	Spl	3	02Sep2003	14:11	555166	65729	74.927	72.7446	2165	0.000	0.0000	-	-	-	8316
5	Spl	4	02Sep2003	14:21	555167	67224	76.632	74.4005	2194	0.000	0.0000	-	-	-	
5	Spl	Avg	-	-	-	67127	76.522	74.2936	2216	0.000	0.0000	-	-	-	09
5	Spl	SDev	-	-	-	1008.77	-	-	48.13	-	-	-	-	-	
5	Spl	%RSD	-	-	-	1.50	-	-	2.17	-	-	-	-	-	
6	Spl	1	02Sep2003	14:32	555168	36466	41.544	40.3336	1154	0.000	0.0000	-	-	-	
6	Spl	2	02Sep2003	14:41	555169	36724	41.838	40.6193	1121	0.000	0.0000	-	-	-	
6	Spl	3	02Sep2003	14:51	555170	36518	41.603	40.3912	1036	0.000	0.0000	-	-	-	
6	Spl	4	02Sep2003	15:01	555171	34449	39.243	38.0996	1041	0.000	0.0000	-	-	-	10
6	Spl	Avg	-	-	-	36039	41.057	39.8609	1088	0.000	0.0000	-	-	-	
6	Spl	SDev	-	-	-	1066.00	-	-	58.76	-	-	-	-	-	
6	Spl	%RSD	-	-	-	2.96	-	-	5.40	-	-	-	-	-	
7	Spl	1	02Sep2003	15:11	555172	174101	198.558	192.7751	3772	1.186	1.1519	-	-	-	
7	Spl	2	02Sep2003	15:21	555173	176425	201.210	195.3491	3656	1.054	1.0234	-	-	-	
7	Spl	3	02Sep2003	15:31	555174	179408	204.613	198.6530	3771	1.185	1.1508	-	-	-	
7	Spl	4	02Sep2003	15:40	555175	179072	204.229	198.2809	3779	1.194	1.1596	-	-	-	11
7	Spl	Avg	-	-	-	177251	202.152	196.2645	3744	1.155	1.1214	-	-	-	
7	Spl	SDev	-	-	-	2488.20	-	-	59.11	-	-	-	-	-	
7	Spl	%RSD	-	-	-	1.40	-	-	1.58	-	-	-	-	-	
8	Spl	1	02Sep2003	15:51	555176	264169	301.308	292.5325	3873	1.302	1.2637	-	-	-	
8	Spl	2	02Sep2003	16:00	555177	255730	291.681	283.1856	3529	0.909	0.8827	-	-	-	
8	Spl	3	02Sep2003	16:10	555178	264052	301.175	292.4029	3840	1.264	1.2272	-	-	-	
8	Spl	4	02Sep2003	16:20	555179	255175	291.048	282.5709	3899	1.331	1.2925	-	-	-	12
8	Spl	Avg	-	-	-	259781	296.303	287.6730	3785	1.202	1.1666	-	-	-	
8	Spl	SDev	-	-	-	5004.06	-	-	172.53	-	-	-	-	-	

TITLE: TOTAL ORGANIC CARBON: METHODS 415.1/9060

SUPERCEDES: Revision 9

22.3 Wet Chemistry Batch Summary and Data Review Checklist

WET CHEMISTRY BATCH SUMMARY

PARAMETER_____ **METHOD**_____ **BATCH** _____

COMMENTS	JOB NUMBER
WC Reporting Limit < STL Quant Limit	
WC Historical confirms within Hold Time	
WC Historical NO confirm & RE outside of HT	
WC Hold Time Exceedance-Dilution required	
WC Hold Time Exceedance-Instrument Failure	
WC Holding Time Exceedance by Date	
WC Holding Time Exceedance by Hours	
WC LCS within ERA limits outside internal	
WC LCS high recovery, sample ND	
WC MBLK hit but samples > 10X blank value	
WC RPD Exceedance for MS / SD	
WC Spike Failure HIGH MS only	
WC Spike Failure LOW MS only	
WC Spike Failure MS and SD	
WC BOD HT met- Oxygen depleted-RE out HT	
WC Carbonate Alkalinity, LCS/MBLK	
WC Reactivity Qualification	
WC TDS/Conductivity ratio outside of range	
WC TOX Breakthrough- no volume for redo	
WC TOX samples were centrifuged	
Other	

DILUTION CODES	REASON
002	Sample matrix effects
003	Excessive foaming
004	High levels of non-target compounds
008	High concentration of target analytes
009	Sample turbidity
010	Sample color
011	Insufficient volume for lower dilution
012	Sample viscosity
013	other

ICAL Compliant? YES NO NA IF NO, Why? _____
LCS/CCV Compliant? YES NO NA IF NO, Why? _____
CCB Compliant? YES NO NA IF NO, Why? _____
RPD Compliant? YES NO NA IF NO, Why? _____
ERA Compliant? YES NO NA IF NO, Why? _____

NUMBER of REANALYSIS FOR THIS BATCH:_____

Analyst_____ Date_____

Time Critical Batch Review_____ Date_____

Secondary Review & Closure_____ Date_____ WC Summary Rev 4 / 5-2005

STANDARD OPERATING PROCEDURE
STL BURLINGTON

DISSOLVED GASES IN GROUNDWATER
RSK-175
Applicable Matrix: Groundwater

APPROVAL SIGNATURES

William S. Cicero
Laboratory Director
Date: June 6, 2007

Kirstin L. McCracken
Quality Assurance Manager
Date: June 6, 2007

Bryce E. Stearns
Technical Director
Date: June 8, 2007

Mark T. Phillips
Department Manager
Date: June 6, 2007

1.0 SCOPE AND APPLICATION

1.1 This SOP describes the laboratory procedure for the determination of dissolved gases (methane, ethane and ethene) and carbon dioxide in groundwater. This procedure determines the concentration of dissolved gas in headspace. This procedure does not provide total sample concentration (concentration in headspace + concentration in water).

1.2 The target compounds that can be determined by this procedure and their associated Reporting Limits (RL) are provided in Table 1, Section 18.0.

2.0 SUMMARY OF METHOD

2.1 Samples are collected without headspace in 44 mL VOA vials. Samples for methane, ethane and ethene are preserved with hydrochloric acid at the time of collection. Samples for carbon dioxide are not preserved. Prior to analysis, the sample is transferred to a 22 mL serum vial and headspace is created using nitrogen. Samples for methane, ethane, ethene are loaded onto a headspace autosampler and analyzed by GC/FID. Samples for carbon dioxide are manually injected and analyzed by GC/TCD.

3.0 DEFINITIONS

3.1 Definitions are included in Appendix B.

4.0 INTERFERENCES

4.1 Non-target compounds from the sample matrix can cause interference, which may result in positive identifications of non-target compounds with retention times similar to those of target compounds. The extent of these interferences will vary depending on the nature of the samples.

5.0 SAFETY

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

5.2 Specific Concerns or Requirements

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

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

5.3 Primary Materials Used

Table 2, Section 18.0 lists those materials used in this procedure that have a serious or significant hazard rating along with the exposure limits and primary hazards associated with that material as identified in the MSDS. The table does not include all materials used in the procedure. A complete list of materials used can be found in Section 7.0. 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. Any questions regarding the safe handling of these materials should be directed to the laboratory's Environmental Health and Safety Coordinator.

6.0 EQUIPMENT AND SUPPLIES

Computer Hardware/Software:

- GC Acquisition Platform- VAX 4505 (GVAX) Multichrom V2.11.
- Data Processing- Hewlett-Packard 9000-series computers, an HP9000 D250 (Chemsvr4) and an HP 9000 K200 (Chemsvr5)/HP-UX 10.20 and Target V3.5.

GC/FID/TCD: with Dual columns, headspace autosampler, or equivalent.

GC Columns:

- FID- Rt-UPLOT, (30m x 0.53 mmID)
- TCD- CTR 1, (6 feet inner with porous polymer and 6 feet outer with molecular sieve).

Syringes-10 uL to 5.0 mL gas tight syringes with Luer-Lok tip.

Serum Vials with crimp top, 22 mL

Supply of ultrahigh purity argon, helium, hydrogen, and nitrogen.

Acetylene, 10,000 ppmv, Matheson or equivalent.

Nitrogen with Acetylene (500 ppmv): Using a gas tight syringe transfer 900 mL of 100 ppmv acetylene standard into a 6 L Summa Canister. Pressurize the canister with nitrogen to 28.08092 psig, which corresponds to a final volume of 18.0 L.

7.0 REAGENTS AND STANDARDS

7.1 Reagents

- VOA Free Reagent Water

7.2 Standards

Primary Source Stock Standard (Methane, Ethane and Ethene): Matheson Micromat 14 Mix Gas Mix or equivalent. The Matheson Micromat 14 Gas Mix is comprised of 1% methane, ethane, ethane, hydrogen, carbon dioxide, acetylene and carbon monoxide in nitrogen. NOTE: 1% is equivalent to 10,000 ppmv.

Primary Source Stock Standard (Carbon Dioxide): Matheson Micromat 14 Bone Dry CO_2 or equivalent. This mix contains 99.8% carbon dioxide in nitrogen. NOTE: 99.8% is equivalent to 998,000 ppmv

Second Source Stock Standards: Purchase a different lot of the primary source standard from the manufacturer.

CO_2 Working Standard (5% / 50,000 ppmv): Using a gas tight syringe transfer 912.2 mL of the primary source stock CO_2 standard into a 6 L Summa Canister. Pressurize the canister with nitrogen to 29.99976 psig, which corresponds to a final volume of 18.2448L. Use the same formulation to prepare the second source CO_2 working standard.

Use the primary and secondary source standards to prepare the calibration standards and QC samples. The recommended formulations for the calibration standards are provided in Appendix A. The formulation to prepare the continuing calibration verification standard (CCV) and QC samples are provided in Sections 10.0 and 11.0.

Prepare all standards using the following technique: Add 18 mL of reagent water to a 22 mL serum vial and cap the vial. Create 4 mL of headspace by spiking UHP nitrogen or for methane, ethane and ethane method blank and samples only, nitrogen with acetylene through the septa; then add an appropriate amount of the gaseous standard through the septa.

8.0 SAMPLE HANDLING AND PRESERVATION

8.1 Samples for analysis of methane, ethane and ethane should be collected in 44 mL VOA vials preserved with 1:1 HCl to a pH of less than 2 at the time of collection. Samples for analysis of carbon dioxide should be collected in 44 mL VOA vials without preservative. Immediately following collection, samples should be cooled and stored at 4º C ± 2º C until the time of analysis.

8.2 The holding time is 14 days from time of collection.

8.3 Unless otherwise specified by client or regulatory program, after analysis, samples are retained for a minimum of 30 days after provision of the project report and then disposed of in accordance with applicable regulations.

9.0 QUALITY CONTROL

9.1 The minimum frequency requirements, acceptance criteria and recommended corrective action for QC samples are summarized in Table 3, Section 18.0. Below is a summary of each type of QC sample that is analyzed with the method.

9.2 A Method Blank (MB) and Laboratory Control Sample (LCS) are prepared with each analytical batch. These samples show that the laboratory is in control, independent of the sample matrix.

9.3 A Matrix Spike and Matrix Spike Duplicate (MS/MSD) should be analyzed with each analytical batch if sufficient sample volume is provided. Project specific MS/MSD and Sample Duplicates (SD) are performed per client request. These samples show the effect of the sample matrix on the accuracy and precision of the method.

9.4 Instrumental QC standards include a five-point calibration (ICAL), an Initial Calibration Verification (ICV) standard, also referred to as a second source standard and Continuing Calibration Verification (CCV) standards are analyzed every 24 hours and at the end of each analytical sequence.

10.0 CALIBRATION AND STANDARDIZATION

10.1 Instrument Operating Conditions

The recommended instrument operating conditions are as follows:

FID:
Temperature Program: 40º for 3.5 minutes
FID Temperature: 200ºC
Injection Port Temperature: 50ºC
Carrier gas: Helium, 30 mL/min
Hydrogen (FID): 30 mL/min
Air (FID): 300 mL/min

TCD:
Temperature Program: 75º for 3.5 minutes
TCD Temperature: 150ºC
Injection Port Temperature: 50ºC
Filament Temperature: 185ºC
Injection Port Temperature:
Carrier gas: Argon, 60 mL/min

10.2 Initial Calibration

Perform a multi-point calibration with a minimum of five levels for each analyte to demonstrate linearity.

Prepare the calibrations standards using the formulations provided in Appendix A. Prepare separate calibration standards each for (methane, ethane and ethane) and carbon dioxide.

Analyze the calibration standards following the procedure that begins in Section 11.2. The data processing system calculates the Calibration Factor (CF), mean CF and Percent Relative Standard Deviation (%RSD). The %RSD for all target analytes must be $\leq$ 30% for the calibration to be considered acceptable. If the %RSD is outside criteria for any target compound, investigate the cause of the problem and correct prior to the analysis of samples.

10.3 Initial Calibration Verification

Immediately following initial calibration, verify the calibration with a second source (ICV) standard.

To prepare the ICV for methane, ethane and ethene, add 200 uL of the second source gaseous standard into a 22 mL vial that contains 18 mL of VOA free water and 4 mL of headspace to yield an ICV concentration equivalent to CAL Level 3.

To prepare the ICV for carbon dioxide, inject 1 mL of the second source gaseous standard into a 22 mL vial that contains 18 mL of VOA free water and 4 mL of headspace to yield an ICV concentration equivalent to CAL Level 3.

Analyze the standard following the procedure that begins in Section 11.2. The percent recovery of the ICV must be 70-130%. If this criterion is not met, correct the problem and reanalyze the ICV. If the reanalysis fails, remake the calibration standards and recalibrate. The acceptance criteria must be met on both columns.

10.4. Continuing Calibration Verification (CCV)

Prepare and analyze a CCV at a concentration equivalent to CAL Level 3, every 24 hours and at the end of the analytical sequence. The percent difference of the CCV must be $\pm$30% as compared to the initial calibration. If the criteria are not met, reanalyze the CCV. If the reanalysis of the CCV fails, take corrective action (See Troubleshooting). After corrective action, the sequence may be continued only if two immediate, consecutive CCVs analyzed at different concentrations are within acceptance criteria. If these CCVs do not meet the criteria, recalibrate prior to further analysis. Samples must be bracketed by passing CCVs, and samples before and after CCV failure must be reanalyzed, unless the CCV is high and there are no detects in the associated samples.

10.5. Troubleshooting

The following items can be checked in case of calibration failures:

ICAL Failure: Perform instrument maintenance. In extreme cases, install new columns.

CCV Failure: Perform instrument maintenance.

Auto-sampler failure: Reset the auto-sampler.

Power Failure: Reset run in Multichrom and re-acquire or re-initiate run sequence.

11.0 PROCEDURE

11.1 Sample & QC Preparation

Remove the samples from refrigerated storage and allow them to warm to room temperature.

Transfer the sample into a 22 mL vial with a crimp cap. Insert a 22-gauge needle into the septum. Using a 5 mL gas-tight syringe, inject 4 mL of UHP nitrogen or nitrogen with acetylene (methane, ethane and ethane) into the vial to create headspace. Withdraw the needle and syringe from the vial and shake the vial vigorously for several seconds.

To prepare a MS/MSD for methane, ethane and ethane, prepare two additional aliquots of the parent sample and add 200 uL of Matheson Micromat 14 Gas Mix into the headspace to yield a spike concentration equivalent to the mid-level calibration standard.

To prepare the method blank for methane, ethane and ethane, transfer 22 mL of VOA free reagent water into a 22 mL vial and seal with a crimp cap. Insert a 22-gauge needle into the septum. Using a 5 mL gastight syringe, inject 4 mL of nitrogen with acetylene into the vial.

To prepare the LCS for methane, ethane and ethene, inject 200 uL of the second source gaseous standard into a 22 mL vial that contains 18 mL of VOA free water and 4 mL of headspace to yield an ICV concentration equivalent to CAL Level 3.

To prepare a MS/MSD for carbon dioxide prepare two additional aliquots of the parent sample and add 1 mL of 5% carbon dioxide working standard into the headspace to yield a spike concentration equivalent to the mid-level calibration standard.

To prepare the method blank for carbon dioxide free, transfer reagent 22 mL of VOA free reagent water into a 22 mL vial and seal with a crimp cap. Insert a 22-gauge needle into the septum. Using a 5 mL gastight syringe, inject 4 mL of UHP nitrogen into the vial.

To prepare the LCS for carbon dioxide, inject 1 mL of the second source gaseous standard into a 22 mL vial that contains 18 mL of VOA free water and 4 mL of headspace to yield an ICV concentration equivalent to CAL Level 3.

11.2 Analysis

Arrange the samples in a sequence that begins with the calibration standards (ICAL if necessary or CCV) followed by the analysis of QC samples, field samples and continuing calibration verification standards (CCVs).

Establish the instrument operating conditions and calibrate the instrument(s) in accordance with Section 10.0. If an acceptable initial calibration already exists, begin the sequence with analysis of the continuing calibration verification standard.

For GC/FID analysis (methane, ethane, ethane), place the standards, samples, and blanks onto the Tekmar headspace autosampler and initiate the analytical sequence. The autosampler equilibrates the sample's water and headspace phases at 40°C and injects 100 uL of sample headspace onto the GC column, where target analytes if present are detected by the FID.

For GC/TCD analysis (carbon dioxide), manually inject 1000 uL of the standards and samples directly onto the column.

The data system identifies the target analytes by comparing the retention time to the retention times of the mid-point of the initial calibration. The data system calculates the concentration for each target analyte from the calibration curve. If the data system does not properly integrate a peak, perform manual integration. All manual integration must be performed and documented in accordance with laboratory SOP LP-QA-006.

After analysis is complete, evaluate the results against the performance criteria given in Section 10.0 and Table 3, Section 18.0 and perform corrective action as necessary.

Dilute and reanalyze samples whose results exceed the calibration range. The diluted analysis should result in a determination within the upper half of the calibration curve.

NOTE: When multiple dilutions are performed, the laboratory routinely reports the result from the appropriate diluted run (i.e. no target analyte above calibration range and the result for the analyte for which the dilution was performed is in the upper half of the calibration range). Undiluted and lesser dilutions are not routinely provided unless specifically requested by the client. For DoD work, the DoD QSM requires that the undiluted analysis or most concentrated dilution be reported along with the appropriate dilution (i.e. report multiple dilutions).

12.0 CALCULATIONS

12.1 Percent Recovery (%R)

$$\%R = \frac{C_s}{C_n} \times 100$$

Where:
C_s = Concentration of the Spiked Field or QC Sample
C_n = Nominal Concentration of Spike Added

12.2 Percent Recovery for MS/MSD (%R)

$$\%R = \frac{C_s - C_u}{C_n} \times 100$$

Where:
C_s = Concentration of the Spiked Sample
C_u = Concentration of the Unspiked Sample
C_n = Nominal Concentration of Spike Added

12.3 Relative Percent Difference (RPD)

$$RPD = \frac{C_1 - C_2}{\left(\frac{C_1 + C_2}{2}\right)} \times 100$$

Where:
C_1 = Measured Concentration of First Sample
C_2 = Measured Concentration of Second Sample

12.4 Calibration Factor (CF)

$$CF_i = \frac{\text{Peak area or height}_{(x)}}{\text{Standard concentration}_{(ug/L)}}$$

12.5 Mean Calibration Factor

$$\overline{CF} = \frac{\sum_{i=1}^{n} CF_i}{n}$$

Where: n = number of calibration levels

12.6 Standard Deviation of the Calibration Factor

$$SD = \sqrt{\frac{\sum_{i=1}^{n} \left(CF_i - \overline{CF}\right)2}{n-1}}$$

Where: n = number of calibration levels

12.7 Relative Standard Deviation of the Calibration Factor (%RSD)

$$\%RSD = \frac{SD}{\overline{CF}} \times 100$$

12.8 Percent Difference (CCV)

$$\%D = \frac{CF_v - \overline{CF}}{\overline{CF}} \times 100$$

Where: CF_v = Calibration Factor from the Continuing Calibration Verification (CCV)

12.9 Sample Concentration

$$Concentration = \frac{Ax}{CFav} \otimes DF$$

Where:
Ax = Peak area of analyte
CFav = Mean calibration factor

13.0 DATA ASSESSMENT, CORRECTIVE ACTION & REPORTING

13.1 Data Review and Corrective Action

Review the samples, standards and QC samples against the acceptance criteria in Table 3. If the results do not fall within the established limits, perform the recommended corrective action. If corrective action is unsuccessful, document the situation with a nonconformance report and/or qualify the data using an appropriate data qualifier (see Appendix C for data qualifier definitions). For additional guidance regarding the laboratory's protocol and required elements for each level of data review refer to laboratory SOP LP-QA-019.

13.2 Data Reporting

The laboratory's RL for each target analyte is provided in Table 1. Report the data to the RL adjusted for sample dilution/concentration. The reporting limit is the threshold value below which results are reported as non-detected. Report sample results that have concentrations for a target analytes less than the RL with the "U" qualifier.

Further guidance on the application and use of the MDL, RL, and QL is provided in laboratory SOP LP-QA-005.

13.3 Data Management and Records

Retain, manage and archive electronic and hardcopy data as specified in laboratory

SOP LP-QA-014.

14.0 METHOD PERFORMANCE

14.1 A Method Detection Limit (MDL) Study is performed at initial method set-up and subsequently once per 12 month period. The procedure and acceptance criteria for MDL studies are given in laboratory SOP LP–QA-005 *Procedures for the Determination of the Limit of Detection (LOD), Limit of Quantitation (LOQ) and Reporting Limit (RL).*

14.2 Each analyst must complete an initial demonstration of proficiency (DOC) before independent analysis of client samples and demonstrate repeated proficiency annually thereafter. The procedures for employee training and demonstration of proficiency are further described in laboratory SOP LP-QA-011 *Employee Training.*

15.0 POLLUTION PREVENTION & WASTE MANAGEMENT

15.1 Where reasonably possible technology changes have been implemented to minimize the potential for pollution of the environment. Employees will abide by this SOP and the policies in Section 13 of the Corporate Safety Manual for “Waste Management and Pollution Prevention.”

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

- Acidic Sample Waste / Satellite Container: 5 Gallon Plastic Bucket

Transfer the waste stream to the satellite container(s) located in your work area. Notify authorized personnel when it is time to transfer the contents of the satellite containers to the hazardous waster storage room for future disposal in accordance with Federal, State and Local regulations, The procedures for waste management are further given in the laboratory SOP LP-LB-001 *Hazardous Waste.*

16.0 REVISION HISTORY

16.1 Title Page: Updated approval signatures.
16.2 All Sections: Updated to provide separate calibration procedure for carbon dioxide.

17.0 REFERENCES

17.1. Method RSK-175, Revision 0, August 1994.

18.0 TABLES, DIAGRAMS,FLOWCHARTS.

18.1 Table 1:Target analyte list, Reporting Limits
18.2 Table 2: Primary Materials Used.
18.3 Table 3: QC Summary, Frequency, Acceptance Criteria and Corrective Action
18.4 Appendix A: Standard Preparation Tables
18.5 Appendix B: Definitions

18.6 Appendix C: Equations

Table 1: Target Analyte List and Reporting Limit

Compound	CAS Number	Reporting Limit (ug/L)
Methane	000074-82-8	2
Ethane	000074-84-0	4
Ethene	000074-85-1	3
Carbon Dioxide	000124-38-9	1000

Table 2: Primary Materials Used

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.			

Table 3: QC Summary, Acceptance Criteria and Recommended Corrective Action

QC Item	Frequency	Acceptance Criteria	Recommended Corrective Action
ICAL	Before sample analysis, when CCVs indicate calibration is no longer valid, after major instrument maintenance.	%RSD $\leq$ 30	Correct problem, reanalyze, repeat calibration.
ICV	After each initial calibration	%R (70-130)	Correct problem and verify second source standard. If that fails, repeat initial calibration.
CCV	Every 24 hours and at the end of the sequence	%D ± 30%	Re-analyze once, if still outside criteria perform corrective action, sequence can be re-started if two successive CCVs pass, otherwise repeat ICAL and all associated samples since last successful CCV, unless CCV is high and bracketed samples are non-detects.
MB	Every 20 samples	< RL	Examine project DQO's and take appropriate corrective action, which may include re-analysis of MB, re-extraction of batch, and/or non-conformance report (NCR). Corrective action must be documented on NCR. If there are no detects in samples, or if all detects are > 10 X MB level, re-prep and reanalysis may not be required.
LCS	Every 20 samples	%R (70-130)	Examine project DQO's and take appropriate corrective action, which may include re-analysis of LCS, re-extraction of batch, and/or non-conformance report (NCR). Corrective action must be documented on NCR. Flag all reported values outside of control limits.
MS/MSD SD	Every 20 samples if sufficient sample volume is available. Project specific MS/MSD and SD per client request	%R (70-130) RPD < 30	Examine project DQO's and take appropriate corrective action, which may include re-analysis of LCS, re-extraction of batch, and/or non-conformance report (NCR). Corrective action must be documented on NCR. Flag all reported values outside of control limits.

Appendix A: Calibration Standard Preparation Tables

The standard formulations contained in this appendix are recommended and are subject to change. If the concentration or volume of any of the stock standard changes, the standard preparation instructions must be adjusted accordingly. See laboratory SOP LP-QA-002 *Standard Preparation* for further guidance.

Calibration Standards (Methane, Ethane, Ethene)

Primary Source Matheson Micromat 14 Gas Mix (10,000 PPMV)	Level 1	Level 2	Level 3	Level 4	Level 5
Volume Added (uL)	4.7	50	200	600	1000

Final Concentration (ug/L)

Analyte	Level 1	Level 2	Level 3	Level 4	Level 5
Methane	1.7	18	73	218	363
Ethane	3.0	34	136	409	681
Ethene	3.2	32	127	381	636

Calibration Standard (Carbon Dioxide)

Primary Source CO_2 Working Standard (50,000 PPMV)	Level 1	Level 2	Level 3	Level 4	Level 5
Volume Added (mL)	0.2	0.5	1.0	1.5	2.0

Final Concentration (ug/L)

Analyte	Level 1	Level 2	Level 3	Level 4	Level 5
Carbon Dioxide	1000	2500	5000	75000	10000

Where :

ug/L= PPMV of Parent Standard x (molecular weight (g) / 24.47) x (volume added (mL) /18 mL)

Compound	Molecular Weight (g)
Methane	16
Ethane	30
Ethene	28
Carbon Dioxide	44

Appendix B: Terms and Definitions

Acceptance Criteria: specified limits placed on characteristics of an item, process or service defined in requirement documents.

Accuracy: the degree of agreement between an observed value and an accepted reference value. Accuracy includes a combination of random error (precision) and systematic error (bias) components which are due to sampling and analytical operations; a data quality indicator.

Analyte: The specific chemicals or components for which a sample is analyzed. (EPA Risk Assessment Guide for Superfund, OSHA Glossary).

Batch: environmental samples that are prepared and/or analyzed together with the same process, using the same lot(s) of reagents. A preparation/digestion batch is composed of one to 20 environmental samples of similar matrix, meeting the above criteria. An analytical batch is composed of prepared environmental samples (extracts, digestates and concentrates), which are analyzed together as a group.

Calibration: a set of operations that establish, under specified conditions, the relationship between values of quantities indicated by a measuring instrument or measuring system, or values represented by a material measure or a reference material and the corresponding values realized by the standards.

Calibration Curve: the graphical relationship between the known values or a series of calibration standards and their instrument response.

Calibration Standard: A substance or reference used to calibrate an instrument.

Continuing Calibration Verification (CCV): a single or multi-parameter calibration standard used to verify the stability of the method over time. Usually from the same source as the calibration curve.

Corrective Action: the action taken to eliminate the cause of an existing nonconformity, defect or other undesirable occurrence in order to prevent recurrence.

Data Qualifier: a letter designation or symbol appended to an analytical result used to convey information to the data user. (Laboratory)

The qualifiers that are routinely used for this test method are:

U: Compound analyzed for but not detected at a concentration above the reporting limit.
B: Compound is found in the sample and the associated method blank.
E: Compound whose concentration exceeds the upper limit of the calibration range.
D: Concentration identified from a dilution analysis.

X,Y,Z: Laboratory defined flags that may be used alone or combined as needed. If used, provide a description of the flag in the project narrative.

Demonstration of Capability (DOC): procedure to establish the ability to generate acceptable accuracy and precision.

Holding Time: the maximum time that a sample may be held before preparation and/or analysis as promulgated by regulation or as specified in a test method.

Initial Calibration: Analysis of analytical standards for a series of different specified concentrations used to define the quantitative response, linearity and dynamic range of the instrument to target analytes.

Intermediate Standard: a solution made from one or more stock standards at a concentration between the stock and working standard. Intermediate standards may be certified stock standard solutions purchased from a vendor and are also known as secondary standards.

Laboratory Control Sample (LCS): a blank matrix spiked with a known amount of analyte(s) processed simultaneously with and under the same conditions as samples through all steps of the procedure.

Matrix Spike (MS): a field sample to which a known amount of target analyte(s) is added.

Matrix Spike Duplicate (MSD): a second replicate matrix spike

Method Blank (MB): a blank matrix processed simultaneously with and under the same conditions as samples through all steps of the procedure. Also known as the preparation blank (PB).

Method Detection Limit (MDL): the minimum amount of a substance that can be measured with a specified degree of confidence that the amount is greater than zero using a specific measurement system. The MDL is a statistical estimation at a specified confidence interval of the concentration at which relative uncertainty is ±100%. The MDL represents a range where qualitative detection occurs. Quantitative results are not produced in this range.

Non-conformance: an indication, judgment, or state of not having met the requirements of the relevant specification, contract or regulation.

Precision: the degree to which a set of observations or measurements of the same property, obtained under similar conditions, conform to themselves.

Preservation: refrigeration and/or reagents added at the time of sample collection to maintain the chemical, physical, and/or biological integrity of the sample.

Quality Control Sample (QC): a sample used to assess the performance of all or a portion of the measurement system.

TestAmerica BURLINGTON
STANDARD OPERATING PROCEDURE

TOTAL ORGANIC CARBON IN SOILS AND SEDIMENT
Lloyd-Kahn Method

Applicable Matrix: Soils, Sediments, and Other Solids

APPROVAL SIGNATURES

William S. Cicero
Laboratory Director
Date: August 9, 2007

Kirstin L. McCracken
Quality Assurance Manager
Date: August 9, 2007

Bryce E. Stearns
Technical Director
Date: August 9, 2007

Jessica A Holzschuh
Department Manager
Date: August 9, 2007

1.0 SCOPE AND APPLICATION

1.1 This SOP describes the laboratory procedure for the determination of total organic carbon (TOC) in soils, sediments and other solids. A procedure for the determination of TOC in marine sediment high in inorganic carbon is provided in Appendix B.

1.2 The routine reporting limit is 500 mg/kg. Additional sample may be used (up to 25 mg) to achieve as low a reporting limit as 100 mg/kg.

2.0 SUMMARY OF METHOD

2.1 A small aliquot of sample, routinely 10.0 mg, is transferred to a tin capsule and treated with phosphoric acid, then dried in an oven at 105°C for 30 minutes to one hour. This serves to separate the organic carbon from inorganic carbonates and bicarbonates. The sample is then transferred to an instrument where it is pyrolyzed in an inductive type furnace. The carbon is converted to carbon dioxide and measured by a differential thermal conductivity detector.

2.2 This procedure is based on the EPA Region II Document Determination of Total Organic Carbon in Sediment, July 27, 1998, authored by Lloyd Kahn, Quality Assurance Specialist.

2.3 Dixon, Wilfrid J., and Massey, Frank J. Jr.: Introduction to Statistical Analysis (fourth edition). Edited by Wilfrid J. Dixon. McGraw-Hill Book Company, New York, 1983. P377 and P548.

3.0 DEFINITIONS

3.1 Definitions are included in Appendix A.

4.0 INTERFERENCES

4.1 Volatile organics in the sediments may be lost in the decarbonation step resulting in a low bias. Maintaining the sample at 4°C, analyzing the sample within the specified holding time, and analyzing the wet sample, may minimize bacterial decomposition and volatilization of the organic compounds.

5.0 SAFETY

Employees must be trained on and adhere to the policies and procedures for safety in the Corporate Safety Manual and this document.

5.1 Safety Concerns or Requirements

None

5.2 Primary Materials Used

Table 1, Section 18.0 lists those materials used in this procedure that have a serious or significant hazard rating along with the exposure limits and primary hazards associated with that material as identified in the MSDS. The table does not include all materials used in the procedure. A complete list of materials used can be found in section 7.0. 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. Any questions regarding the safe handling of these materials should be directed to the laboratory's Environmental Health and Safety Coordinator.

6.0 EQUIPMENT AND SUPPLIES

Drying Oven: Capable of maintaining a temperature of 105°C.

Carlo Erba Elemental Analyzer Model EA1108 and Model NA 1500 or equivalent.

Costech Elemental Analyzer: Model 4010 or equivalent.

Analytical Balance: Capable of weighing to the nearest 0.001mg.

Aluminum Trays that hold sample capsules for use at 105°C

Tweezers

5mm X 9mm tin capsules

Quartz Columns: Costech Analytical or equivalent.

Quartz wool: for segregating and containing column materials

Copper Wire, Reduced: Costech Analytical or equivalent.

Tungsten on Alumina: Costech Analytical or equivalent.

High Temperature Gloves

Clear Plastic Sample Trays: Costech Analytical or equivalent.

7.0 REAGENTS AND STANDARDS

7.1 Reagents

Reagent water

Phosphoric Acid, Concentrated: Reagent Grade, J.T. Baker recommended.

Phosphoric Acid Solution (1:19): Add approximately 100 mL of reagent water to a 200 mL volumetric flask. Add 18.34 g of concentrated phosphoric acid to the volumetric flask then adjust to volume with reagent water. Mix the solution well then transfer the solution to a 250 mL polyethylene bottle. Assign an expiration date of six months from date made and store the solution at room temperature.

7.2 Standards

Acetanilide Crystals of known Carbon percentage: Purchased from Costech Analytical. Used to check instrument calibration.

Sulfanilamide Crystals (41.84% Carbon): Purchased from Costech Analytical. This material is used to calibrate the instruments.

Laboratory Control Samples (LCS) Material, Organic Material of known Carbon percentage: Purchased from LECO Corporation.

Matrix Spike Material, 1632B trace elements in coal (80.11% Carbon)

8.0 SAMPLE COLLECTION, PRESERVATION, SHIPMENT & STORAGE

8.1 Samples should be collected in amber glass jars. Immediately following collection, the samples should be cooled to 4°C (±2) and maintained at that temperature until time of analysis.

8.2 The holding time is 14 days from date of collection, unless otherwise specified.

8.3 Unless otherwise specified by a federal, state or client-specific protocol, samples are disposed of after 30 days in a manner that complies with all applicable regulations.

9.0 QUALITY CONTROL

9.1 The following QC check samples are analyzed with each batch of 20 or less samples: Method Blank (MB) Laboratory Control Sample (LCS), Matrix Spike (MS) and a Sample Duplicate (DP). In addition to calibration (ICAL), instrument standardization is checked with acetanilide every 20 drops and at the end of the analytical sequence. The minimum frequency requirements, acceptance criteria and recommended corrective action for QC samples are summarized in Table 2, Section 18.0.

10.0 CALIBRATION AND STANDARDIZATION

10.1 Calibration Curve

Analyze a calibration curve each time the combustion column is changed (a combustion column is good for about 200 sample drops). The recommended formulations for each calibration level is provided in the following table:

Calibration Standard Sulfanimide	Weight[1] (mg)	% Carbon	Carbon (mg)
Calibration Level 1	0.100	41.84	0.0418
Calibration Level 2	0.500	41.84	0.2092
Calibration Level 3	1.00	41.84	0.4184
Calibration Level 4	1.75	41.84	0.7322
Calibration Level 5	2.50	41.84	1.046

[1]*These weights are approximate. Enter the actual weight used into the software program.*

Measure a single drop for each calibration point. The instrument software system plots peak area against mg of Carbon and calculates a correlation coefficient using standard linear regression. The correlation coefficient (r) must be $\geq$ 0.995 for the calibration to be considered acceptable. If it is not, repeat the calibration prior to further analysis.

10.2 Troubleshooting

- *Calibration passes at > 0.995 correlation, but LCS fails abnormally low*: Re-calibrate. Calibration usually needs to be > 0.999 correlation.

- *Carbon peak "maxes out" at instrument 1200mv (peak has flat top)*: Reanalyze sample at lower weight.

- *No peaks on any chromatograms, no results*: Gases to instrument may be off. Turn on all gasses at valve manifold.

- *Autosampler will not work at all*: Gasses to instrument may be off. Turn on all gasses at valve manifold.

- *Single chromatogram shows results at bottom of page, but no peak or baseline in chromatogram window*: Re-print single chromatogram.

- *Some or all chromatograms show carbon peak at same retention time as Acetanilide, but peak is not identified as carbon, or is identified as another element*: Retention time shifted. Adjust retention time in calibration window, and reprint chromatograms.

- *Upon recalibration, peaks are not being identified as carbon*: In calibration window, general tab, adjust retention time to match peaks. Starting at level 1, "Open Standard", open level1 curve pt. in calibration directory, click "Add Peak" button, click on peak itself. Increase level #, opening standard for each curve pt and add each peak. Carbon Tab should have all five calibration points on curve, if done correctly.

- *Peaks in chromatograms identified as carbon, but all results in summary table below chromatogram are zero*: Current calibration not associated with run when started. Open current calibration, copy first two columns for all points (5 rows) in small table in general tab. Then, open calibration that was associated with run (should be

empty) and paste into table in calibration tab. Reprint all chromatograms on run.

- *Software crashes during analysis*: Boot up software normally. Chromatograms already printed/analyzed are ok, but, sample that was analyzing during shutdown is lost. Restart table at next sample by un-checking "run" box for samples already run and sample that was lost.

- *Autosampler error causes few samples to remain in autosampler tray after run has finished*: Identify samples that got stuck. Create a new run and analyze stuck samples (with initial weights) with bracketing QC. No PBS/LCS needed.

- *Autosampler error causes many sequential samples to remain in autosampler tray after run has finished (usually end of run)*: Add rows onto existing table. Identify samples that did not get analyzed and repeat Ids and weights into added rows. Restart table. All analyzed samples' status should be blue(analyzed), added rows should be green (not analyzed yet).

- *Various result issues or odd peak shapes or baseline issues*: Column may be leaking or cracked. Change column, recalibrate.

11.0 PROCEDURE

11.1 Sample Preparation

Using tweezers, and working directly from the box, place a tin capsule on the analytical balance and tare. Using the small sample scoop, add approximately 10 mg (or more, if client requested) of sample to the capsule. Record the sample weight on the benchsheet. Remove the capsule from the balance and place into one of the aluminum holding trays. Weigh two separate aliquots into two separate tin capsules for each field sample. Record all weight measurements on the sample preparation log. For the method blank, set two empty tin capsules into an aluminum holding tray. For the LCS, weigh ~9 mg of LECO LCS material into two separate tin capsules and set them in sequence in an aluminum holding tray.

For the matrix spike, weigh out an additional sample aliquot and record its weight. Add 0.3 – 0.7 mg of matrix spike material and record this weight. For the sample duplicate, weigh out an additional sample aliquot. Prepare two aliquots for both the matrix spike and the sample duplicate.

Add two drops of 1:19 phosphoric acid to each tin capsule. Place the aluminum trays into a drying oven set to a temperature of 105ºC for 30-60 minutes or until all samples appear dry.

Using tweezers, pinch the top of each tin capsule closed and compress the capsule around the material inside. Work carefully so as not to tear the capsule, but crush it down to the smallest size. Set the prepared samples in line in a clear plastic sample tray for storage, or place directly into an autosampler tray for analysis. For the latter, leave positions open for the acetanilide check standards and associated calibration blanks.

Prepare the acetanilide standard and blanks as follows:

For each acetanilide spike, weigh ~0.5 mg of acetanilide material into a tin capsule. Fold the capsule up and compress down to the smallest size possible. Prepare enough acetanilde to ensure a frequency of every 20 drops and the end of the analytical sequence. For each associated calibration blank, leave an empty position in the autosampler tray.

11.2 Software Set-up and Analysis

If the column has been changed, generate a new calibration curve. If not, use the existing calibration curve for analysis. Each column will analyze approximately 200 individual sample drops. When the counter on the instrument approaches 200, watch the instrument data for signs that the column is deteriorating; poor peak resolution, trailing baselines, extraneous peaks. If a column change is necessary, refer to Appendix C for the procedure. After changing the column, generate a new calibration curve.

Select the appropriate channel: Channel 1 is the NA 1500, Channel 2 is the EA 1108, and Channel 3 is the Costech instrument, which has its own PC. At the main screen select the sample table icon. The last sample table that was run will be shown on the screen.

Open a new sample table, and select the appropriate number of sample positions for the analysis, then name the table with the date and a unique alpha designator (i.e. 061505a). In front of the %3r in the file name column of the sample table, add the sample table name to ensure that each individual chromatogram generated from this sample table has a unique filename associated with it.

If the combustion column has been changed and instrument needs to be calibrated, follow the procedure below:

Prepare a “bypass” drop to determine the retention time for carbon with the new column. The bypass is an aliquot of acetanilide. The weight is not needed. Drop the bypass into the instrument and initiate a singular analysis. Set the retention time for carbon in the software to match that of the bypass drop.

Identify the first five sample lines with the names Std1 through Std 5. Enter their respective weights in the weight column, assign them a level # in the level column (Std1 is level 1, Std2 is level 2, etc.) to alert the software the order in which to place the calibration standards. In the sample type column, use the drop down and select “standard” for each. Finally, use the drop down in the Standard name column and select “sulfanilamide” for each. Add the standards to the autosampler tray and hit “start” to run the calibration.

Sample Analysis:

Open a new sample tray and create a unique file name. When the instrument was last

calibrated, the software creates a calibration file with the same name as the sample table in which it was run. Open this file and save it with the same name as the sample table about to be run to ensure that the analysis is calculated from the most recent calibration. To do this, click on the calibration icon (looks like a little calibration curve) and use the file option to open the calibration file last performed. Save this file with the same name as your sample table. Click on the sample table icon (looks like a little sample table) to get back to your sample table.

Enter each sample ID and their respective weights and save the sample table. Enter a weight of 10 mg for the Method Blank (PBS) and instrument blanks.

An example analytical sequence follows:

Initial Calibration (calibration blank and 5 calibration standards)

Acetanilide	(1 drop)
Blank	(1 drop)
PBS	(2 individual drops)
LCS	(2 individual drops)
Sample	(2 individual drops)
Sample	(2 individual drops)
Sample	(2 individual drops)
Sample	(2 individual drops)
Sample	(2 individual drops)
Sample	(2 individual drops)
Sample	(2 individual drops)
Sample	(2 individual drops)
Sample	(2 individual drops)
Acetanilide	(1 drop)
Blank	(1 drop)

Add the samples and acetanilides to the autosampler tray and set the tray into the autosampler carriage. Turn the autosampler tray until the number 1 position is behind the post, in front of the autosampler. The tray is now set to run.

Click the “start” icon to begin the analysis

After analysis review the analytical results against the acceptance criteria given in Table 2, Section 18.0, and perform corrective action as necessary. Enter the results for all instrument blanks (including PBS) and any client sample exhibiting an area response at or lower than the lowest calibration standard into the low level Excel spreadsheet set up for this purpose. This spreadsheet calculates these low level results with a two point linear regression using the origin and the lowest calibration point. A more precise result for low level samples is determined this way. Report results in mg/kg Carbon and corrected for % solids

12.0 CALCULATIONS

12.1 Percent Carbon to mg/kg Carbon Conversion

% Carbon × 10,000 = mg/kg Carbon

12.2 LCS Percent Recovery (%R)

$$\%R = \frac{\text{LCS Result}}{\text{LCS True Value}} \times 100$$

12.3 MS Percent Recovery (%R)

$$\text{mg/Kg wet SA} = \frac{\text{Spike TV} \times \text{weight of MS added}}{\text{sample weight}} \times 1\,\text{million}$$

$$\text{mg/Kg dry SA} = \frac{\text{mg/Kg wet SA}}{\%\ \text{solid}} \times 100$$

$$\text{mg/Kg dry Carbon} = \frac{\text{mg/Kg wet Carbon (from instrument)}}{\%\ \text{solid}} \times 100$$

$$\%R = \frac{A - B}{C} \times 100$$

Where:
A= Average of three drops of MS sample result: mg/Kg dry carbon
B= Average of three drops of parent sample: mg/Kg dry carbon
C= Average of three drops of mg/Kg dry SA
SA= spike added (mg/Kg)
Spike TV= 0.8011(mg/Kg)

12.4 Relative Percent Difference (RPD)

$$RPD = \frac{|D_1 - D_2|}{\frac{D_1 + D_2}{2}} \times 100$$

Where:

D_1 = First Sample Value

D_2 = Second Sample Value (duplicate)

12.5 Dixon Test (Use 3-7 results)

1. Sort all the results in ascending order (low values to high).
2. Calculate the tau statistic for the low and high values.

3. Compare the calculated tau statistics (low and high) to critical values listed below.
4. If either calculated tau is higher than the critical value, reject that value and repeat the test.

Tau statistic for lowest value = $\tau_L = (X_2 - X_1) / (X_k - X_1)$
Tau statistic for highest value = $\tau_H = (X_k - X_{k-1}) / (X_k - X_1)$

Where:
X_2 = Second lowest value in sorted list.
X_1 = Lowest value in sorted list.
X_k = Highest value in sorted list.
X_{k-1} = Second highest value in sorted list.

Number of observations, k	Critical Values
3	0.941
4	0.765
5	0.642
6	0.560
7	0.507

13.0 DATA ASSESSMENT, CRITERIA & CORRECTIVE ACTION

13.1 Review the samples, standards and QC samples against the performance criteria given in Table 2. If the results do not fall within the established limits or criteria perform corrective action. If corrective action is not taken or unsuccessful, the situation should be documented and reported in the project narrative. All data that does not meet established criteria must be flagged and noted in the project narrative.

14.0 METHOD PERFORMANCE

14.1 An Initial Demonstration of Capability is required for each analyst before unsupervised performance of this method.

14.2 An Initial Method Detection Limit (MDL) determination for each test method referenced in this SOP is performed following the procedure described in the reference method, 40CFR, Part 136, Appendix B and laboratory SOP LP-LB-009. The MDL is verified or repeated when a significant change to the method occurs. Significant changes include the use of alternate reagents or standard reference materials, new instrumentation or the use of alternate sample preparation procedures.

15.0 POLLUTION PREVENTION & WASTE MANAGEMENT

15.1 The laboratory optimizes technology to minimize pollution and reduce the production of hazardous waste whenever possible.

15.2 Waste Streams generated by this method;

- Spent combustion columns

→ *Satellite Waste Container: Five Gallon Metal Bucket labeled "Glass Disposal"*

Transfer the waste stream to the appropriate hazardous waste satellite container located in your work area. Notify authorized personnel when it is time to transfer the contents of the satellite container to the hazardous waste storage room for future disposal in accordance with Federal, State and Local regulations. The procedures for waste management are further given in laboratory SOP LP-LB-001 Hazardous Waste.

16.0 REVISION HISTROY

16.1 Cover Page: Changed to reflect current management team.
16.2 Section 10.2: This section was added
16.3 Section 11.0: Number of drops was changed from 4 to 2.
16.4 Section 12.5: The Dixon Test was added.
16.5 Section 18.2, Table 2: Sample precision criteria added.
16.6 Section 18.5, Appendix D: Determination of Black Carbon in Sediment Procedure added.

17.0 REFERENCES

17.1 EPA Region II Document Determination of Total Organic Carbon in Sediment, July 27, 1998, authored by Lloyd Kahn, Quality Assurance Specialist.

17.2 Dixon, Wilfrid J., and Massey, Frank J. Jr.: Introduction to Statistical Analysis (fourth edition). Edited by Wilfrid J. Dixon. McGraw-Hill Book Company, New York, 1983. P377 and P548

18.0 TABLES, DIAGRAMS, FLOWCHARTS

18.1 Table 1: Primary Materials Used
18.2 Table 2: QC Summary
18.3 Appendix A: Definitions
18.4 Appendix B: TOC Procedure for High Concentration Marine Sediments (CITHON)
18.5 Appendix C: Column change procedure
18.6 Appendix D: Determination of Black Carbon in Sediment Procedure

Table 1: Primary Materials used

Material (1)	Hazards	Exposure Limit (2)	Signs and symptoms of exposure
Phosphoric Acid	Corrosive	1 Mg/M3 TWA	Inhalation is not an expected hazard unless misted or heated to high temperatures. May cause redness, pain, and severe skin burns. May cause redness, pain, blurred vision, eye burns, and permanent eye damage.
1 – Always add acid to water to prevent violent reactions.			

Table 2: QC Summary and Recommended Corrective Action

QC Sample	Frequency	Acceptance Criteria	Corrective Action
ICAL	Following each column change	r $\geq$ 0.995	Standards check, re-calibration.
Method Blank (PBS)	Once per batch of 20 samples	< RL DoD: ½ RL	Re-prepare and reanalyze batch.
LCS	Once per batch of 20 samples	%R (75-125)	Re-prepare and reanalyze batch.
Acetanilide	Every 20 drops and at the end of the analytical run	%R (85-115)	Re-prepare and reanalyze samples not surrounded by passing Acetanilides
Blank (paired with Acetanilide)	Following each Acetanilide	< RL	Re-prepare and reanalyze batch.
Matrix Spike	One per batch of 20 or less samples	%R (75-125)	Discuss outlier in project narrative
Sample duplicate	One per batch of 20 or less samples	RPD < 20	Discuss outlier in project narrative
Sample precsion	Each sample is run in duplicate	%RPD<40%	Analyze 2 more replicates and perform Dixon test for high and low outliers. If no rejects, average all 4 replicates. If 1 replicate is rejected, perform Dixon test on remaining 3 reps. Report the average of the remaining 2 or 3 replicates.

Appendix A: Definitions

Accuracy: the degree of agreement between a measurement and the true or expected value, or between the average of a number of measurements and the true or expected value.

Batch: environmental samples, which are prepared and/or analyzed together with the same process, using the same lot(s) of reagents. A preparation batch is composed of one to 20 environmental samples of a similar matrix, meeting the above mentioned criteria.

Calibration: the establishment of an analytical curve based on the absorbance, emission intensity or other measured characteristic of known standard.

Calibration Blank (ICB/CCB): a volume of reagent water acidified with the same acid matrix as in the calibration standards.

Calibration Standards: a series of known standard solutions used to calibrate the instrument response with respect to analyte concentration. A standard containing the analyte in question (sulphanilimide) is prepared at varying weights and analyzed. This standard is a separate source from the LCS. The sulphanilimide is used to calibrate the instrument response with respect to analyte concentration.

Continuing Calibration Verification (CCV): a prepared standard solution used to verify the stability of the instrument calibration and instrument performance during the analysis of samples.

Corrective Action: action taken to eliminate the causes of an existing non-conformance, defect or other undesirable situation in order to prevent recurrence.

Demonstration of Capability (DOC): procedure to establish the ability to generate acceptable accuracy and precision.

Holding Time: the maximum time that a sample may be held before preparation and/or analysis as promulgated by regulation or as specified in a test method.

Initial Calibration: Analysis of analytical standards for a series of different specified concentrations used to define the quantitative response, linearity and dynamic range of the instrument to target analytes.

Initial Calibration Verification (ICV): A prepared standard solution from a source separate from that of the calibration standards used to verify the concentration of the calibration standards and the adequacy of instrument calibration.

Laboratory Control Sample (LCS): a blank matrix spiked with a known amount of analyte(s), processed simultaneously with, and under the same conditions as samples, through all steps of the analytical procedure.

Matrix: the substrate of a test sample.

Matrix Duplicate (DP): duplicate aliquot of a sample processed and analyzed independently; under the same laboratory conditions; also referred to as Sample Duplicate; Laboratory Duplicate.

Matrix Spike (MS): field sample to which a known amount of target analyte(s) is added.

Matrix Spike Duplicate (MSD): a replicate matrix spike.

Method Blank: a blank matrix processed simultaneously with, and under the same conditions as, samples through all steps of the analytical procedure.

Method Detection Limit (MDL): the minimum amount of a substance that can be measured with a specified degree of confidence that the amount is greater than zero using a specific measurement system. The MDL is a statistical estimation at a specified confidence interval of the concentration at which the relative uncertainty is +100%. The MDL represents a range where qualitative detection occurs using a specific method. Quantitative results are not produced in this range.

Percent Solids (%S): the proportion of solid in a soil sample.

Appendix B: Marine Sediments High in Inorganic Carbon

Sample Preparation

Transfer approximately 10 g of a thoroughly mixed sample to an aluminum weigh dish, and dry in the 105°C oven. Grind the sample with the pink mortar and pestle to a fine powder. Record the weight of a 250 mL Teflon beaker then transfer ~ 5 g of the ground sample to this beaker.

If the sample is to be spiked, weigh the beaker to the nearest 0.1mg and record the weight. Likewise determine and record the weight of the added sample. Add 0.1g of NIST 1632b Trace Elements in Coal (80.11% Carbon) to the sample. Record the weight added. Evenly distribute the spike over the sample and use a glass stir rod to mix the spike with the sample. Do not use that stir rod with any other sample.

Use Talc-free latex gloves from this point on to minimize the risk of acid burns. Add several drops of 1:1 HCL to each sample and stir each sample with its own glass stir rod. Samples with high concentrations of inorganic carbon may effervesce to the point of overflowing the beaker, so take care to add the acid in small aliquots and stir vigorously. If the sample "boils over" it must be re-prepared. Continue to add 1:1 HCL in small aliquots until there is no further reaction, taking sample to dryness after each addition of acid with the hot plate provided.

Carefully rinse the stir rod and beaker walls with DI water using a fine-tipped squirt bottle. Use only what is needed to bring the entire sample to the bottom of the beaker. ***When adding water to acid use necessary precautions to avoid splashing!***

Dry the treated samples on the hot plate in the hood, after each acid/water addition. Do not add more than a total of 200 mL of 1:1 HCL to any sample.

NOTE*: Samples are hydroscopic and will absorb water if they are exposed to air for too long.*

Weigh beaker with residue and record the residue weight measurement. After the sample is thoroughly dry, scrape the sample residue from the beaker and grind to a powder using the pink mortar and pestle. Transfer the ground sample to a clean, dry 40-mL vial reserved for this analysis.

NOTE: *Depending on the nature of the sample, it may be difficult to completely remove the dried residue from the beaker or to grind it to a homogenous powder. Where difficulties are encountered, make a note on the preparation worksheet.*

Analysis

Perform TOC analysis on processed sample material as outlined in section 10.0 of this SOP.

Appendix C: Column Change Procedure

Turn off the helium and oxygen supplies to the instrument.

Dial the left furnace temperature to a reading of 052 (this equates to 520˚C). Wait until the temperature drops below 600˚C to remove the column.

Remove the panel covering the furnace and unscrew the autosampler connection from the top of the column.

Unscrew the fitting at the bottom of the column and remove.

Lift the column up and out of the furnace using high temperature gloves.

CAUTION: The column will still be 500-600˚C. Do not touch the center portion of the column. Place the spent column in the metal can designated for this purpose.

Lay a new quartz column on the bench top, measure and mark off for the following:

- One inch up from the bottom and add a ½ inch plug of quartz wool. Note: pack the quartz wool tightly enough for it to stay in place.
- Pour in 2 ½ inches of copper wire
- Pack another ½ inch quartz wool plug on top of the copper
- Pour in 3 inches of tungsten
- Pack a final ½ inch quartz wool plug on top of the tungsten

Place the new column into the furnace and reconnect the top and bottom fittings. Snug these up, but don't over tighten.

Replace the panel covering the furnace, dial the furnace temperature back to 102 (this equates to 1020˚C), and turn the helium and oxygen supplies back on.

When the instrument comes up to operating temperature, it is ready to calibrate.

Appendix D: Determination of Black Carbon in Sediment Procedure

1. Obtain a representative subsample of the sediment. Weight 10 grams of sample into a clean pre-tared aluminum drying pan or equivalent.

2. Dry the sample at 105^{o}C for at least 12 hours.

3. Grind the sample using a mortar and pestle.

4. Sieve the sample using a number 35 sieve (500 um).

5. Treat the sample with phosphoric acid. Add acid drop wise until effervescence is no longer observed.

6. Dry the sample at 105^{o}C for 1 hour.

7. Set aside an aliquot of the sample at this stage for direct TOC analysis, reported without correction for the IN623 percent solids. Continue with the sample for Black Carbon.

8. Place the dried sample into a clean crucible and cover the sample.

9. Bake the samples at 375^{o}C in a muffle for 24 hours.

10. Allow the samples to cool and transfer approximately 5.0 mg into each of three tin capsules.

11. Transfer the sample (in the tin capsules) to the TOC analyzer for analysis by the Lloyd Kahn Method.

12. The sample is pyrolyzed in an inductive type furnace, where the carbon is converted to carbon dioxide, which is measured using a differential thermal conductivity detector.

13. The results will be reported as mg/Kg Black Carbon.

References:

Orjan Gustafsson, Thomas D. Bucherli, Zofia Kukulska, Mette Andersson, Claude Largeau, Jean-Noel Rouzaud, Christopher M. Reddy and Timothy I. Eglinton (December 2001) Evaluation of a Protocol for the Quantification of Black Carbon in Sediments, Global Biogeochemical Cycles, Volume 15, pages 881-890.

Orjan Gustafsson, Farnaz Haghseta, Charmaine Chan, John MacFarlane & Philip M. Gschwend (1997) Quantification of the Dilute Sedimentary Soot Phase: Implications for PAH Speciation and Bioavailability, Environmental Science & Technology, Volume 31, pages 203-209.

ARCADIS BBL

Attachment 2

Test American Quality Manual

LABORATORY QUALITY MANUAL

STL BUFFALO
10 Hazelwood Drive
Amherst, New York 14228
(716) 691-2600

Approved by: (Signature / Date)

Christopher A. Spencer
Laboratory Director — 11/23/05

Verl D. Preston
Quality Manager — 11/23/2005

John R. Schove
Operations Manager — 11/23/05

Kenneth E. Kasperek
Technical Director / EH&S — 11/28/2005

CONTROLLED DISTRIBUTION

COPY # :

ISSUED TO :

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

TABLE OF CONTENTS

Tables

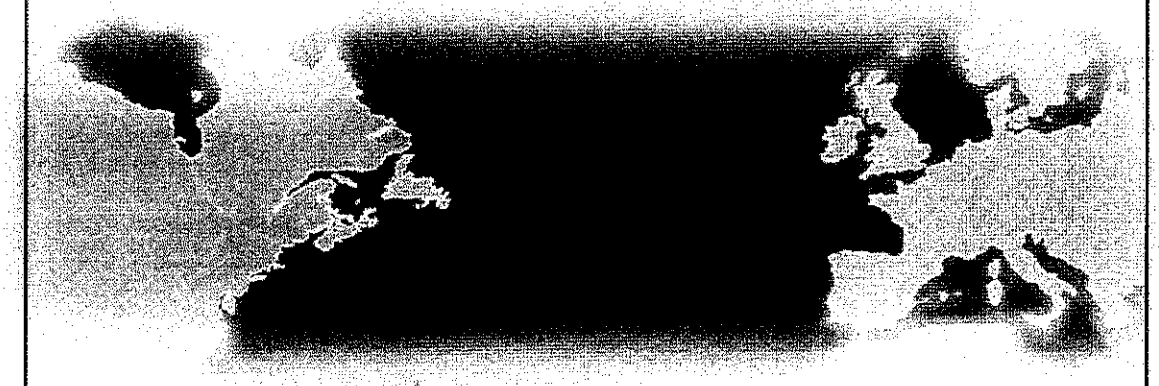
Figures

Appendix

SEVERN TRENT **STL**

Vision

STL will be the recognized industry leader for environmental analysis.



Mission

Through the innovation and dedication of our people, together with the quality of our systems, we will deliver levels of performance that delight our clients, retain the confidence of our stakeholders and enable the profitable growth of our business.

Severn Trent Laboratories

1.0 Introduction, Purpose, and Scope

1.1 STL Overview

STL Buffalo (STL) is a part of Severn Trent Laboratories, a major group of U.S. based companies. STL is a full-service environmental laboratory that provides quality comprehensive and integrated professional analytical services effectively and efficiently. A broad range of environmental testing services are offered that span a variety of matrices including aqueous, saline, solid, tissue and drinking water.

Associated with this activity are services to assure client requirements are known, communicated and satisfactorily addressed, and a deliverables package presenting the analytical results. The laboratory provides expert personnel for supervision, technical consultation, and project review for effective planning and implementation of analytical assignments.

STL operates under the regulations and guidelines of the following federal programs:

- Clean Water Act (CWA)
- National Pollution, Discharge, and Elimination System (NPDES)
- Occupational Safety and Health Administration (OSHA)
- Resource Conservation and Recovery Act (RCRA)
- Safe Drinking Water Act (SDWA)
- Toxic Substances Control Act (TSCA)

STL also provides services under various state and local municipal guidelines. A current table of analytical services and general service listing is presented on STL's website under the MySTL webpage or available from the laboratory. A current listing of STL Buffalo certifications (STLBuffCertList) is maintained by the laboratory on the company network directory. Copies of the actual certificates are available on the STL Buffalo intra-net site (BufNet).

1.2 Quality Assurance Policy

It is STL's policy to:

- Provide high quality, consistent, and objective environmental testing services that meet all federal, state, and municipal regulatory requirements.
- Generate data that are scientifically sound, legally defensible, meet project objectives, and are appropriate for their intended use.
- Promote employee adherence to quality documentation and implementation of Corporate Policies and Procedures
- Provide STL clients with the highest level of professionalism and the best service practices in the industry.
- Build continuous improvement mechanisms into all laboratory, administrative, and managerial activities.
- Maintain a working environment that fosters open communication with both clients and staff and ensures data integrity.

1.3 Management Commitment to Quality Assurance

STL management is committed to providing the highest quality data and the best service in the environmental testing industry. To ensure that the data produced and reported by STL meet the requirements of its clients and comply with the letter and spirit of municipal, state and federal regulations, STL maintains a quality system that is clear, effective, well communicated, and supported at all levels in the company.

Line organizations verify that specifications are achieved; QA organizations assist and provide oversight and verification of processes through planning, reviews, audits, and surveillances. The quality objectives are derived from this Laboratory Quality Manual (LQM), Standard Operating Procedures (SOPs) and Work Instructions.

1.4 Purpose

The purpose of the LQM is to describe STL's Quality System and to outline how that system enables all employees to meet the Quality Assurance (QA) policy. This LQM also describes specific QA activities and requirements and prescribes their frequencies. Roles and responsibilities of management and laboratory staff in support of the Quality System are also defined in this LQM.

1.5 Scope

This LQM is specific to STL Buffalo's quality systems and laboratory operations. All other STL locations have LQMs under the Corporate Quality Management Plan (QMP) or the Corporate QMP itself.

The laboratory is committed to ensuring that resources are available and deployed to meet client expectations. This includes gathering project information prior to sample receipt to ensure client expectations will be met with respect to:

- Sampling containers;
- Analytical methods employed;
- Accuracy and precision;
- Reporting limits;
- Personnel qualifications, training, and experience;
- Calibration and quality control measures employed;
- Regulatory requirements;
- Report contents;
- Supporting documentation, records and evidence; and
- Review of data

1.6 Servicing

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

The laboratory has established procedures for performing and verifying that client servicing meets requirements. Typical services provided are:

- Sample Containers/Supplies – *Container Management: Process Operation/Bottle Order Set-Up (APM-BottleOrder-03)*
- Project QAP preparation – Project Planning Process: *Project Information Requirements (APM-ProjInfo-20)*
- Regulatory advisory functions – Project Planning Process: *Project Information Requirements (APM-ProjInfo-20)*
- Consulting – Project Planning Process: *Project Information Requirements (APM-ProjInfo-20)*

Regulatory and advisory functions are addressed under the same procedures used for project planning.

2.0 References

The following references were used in preparation of this document and as the basis of the STL Quality System:

EPA Guidance for Preparing Standard Operating Procedures (SOPs), EPA QA/G-6, US EPA, Office of Environmental Information, EPA/240/B-01/004, March 2001.

EPA Requirements for Quality Management Plans, EPA QA/R-2, US EPA, Office of Environmental Information, EPA/240,B-01/002 March 2001.

EPA Requirements for Quality Assurance Project Plans, EPA QA/R-5, US EPA, Office of Environmental Information, EPA/240/B-01/003, March 2001.

EPA Quality Manual for Environmental Programs, 5360 A1, US EPA Office of Environmental Information – Quality Staff, May 2000.

General Requirements for the Competence of Testing and Calibration Laboratories, ISO/IEC 17025, December 1999.

Good Automated Laboratory Practices, Principles and Guidance to Regulations for Ensuring Data Integrity in Automated Laboratory Operations with Implementation Guidance, EPA 2185, US EPA Office of Information Resources Management, August 1995.

National Environmental Laboratory Accreditation Conference, Constitution, Bylaws, and Standards, EPA 600/R-04/003, US EPA Office of Research and Development, June 2003.

Quality Systems Manual for Environmental Laboratories, Department of Defense, Version 3.0, March 2005

Shell for Analytical Chemistry Requirements, US Army Corps of Engineers, December 1998.

Quality Systems for Analytical Services, U.S. Department of Energy, Rev. 1, April 2004.

This LQM was written to comply with the National Environmental Laboratory Accreditation Conference (NELAC) standards. Refer to Table 1 for a cross-section comparison of this LQM to the NELAC standards.

Table 1.

Correlation of QAPP Sections with NELAC 5.4.2.3 Quality Manual Requirements

NELAC Chapter 5.4.2.3 Quality Manual	Laboratory Quality Manual Section
a. Quality policy statement, including objectives and commitments	1.2 Quality Assurance Policy 4.2.1 Objectives of the Quality System
b. Organization and management structure	4.1 Organization and Management
c. Relationship between management, technical operations, support services and the quality systems	4.1.2 Roles and Requirements 4.2 Quality System
d. Records retention procedures; document control procedures	4.3 Document Control 4.12.2 Record Retention
e. Job descriptions of key staff and references to job descriptions of other staff	4.1.2 Roles and Requirements
f. Identification of laboratory approved signatories	4.1 Organization and Management
g. Procedures for achieving traceability of measurements	5.5 Measurement Traceability
h. List of all test methods under which the laboratory performs its accredited testing	5.3.1 Method Selection
i. Mechanisms for assuring the laboratory reviews all new work to ensure that it has the appropriate facilities and resources before commencing such work	4.4.2 Project-Specific Quality Planning
j. Reference to the calibration and/or verification test procedures used	5.4.3 Equipment Verification and Calibration 5.3.6.2 Data Review
k. Procedures for handling submitted samples	4.7.1 Sample Acceptance Policy 5.7 Sample Handling, Transport and Storage
l. Reference to the major equipment and reference measurement standards used as well as the facilities and services used in conducting tests	1.6 Servicing 4.1.1 Laboratory Facilities 5.4.2 Equipment Maintenance 5.4.3 Equipment Verification and Calibration
m. Reference to procedures for calibration, verification and maintenance of equipment	5.4.2 Equipment Maintenance 5.4.3 Equipment Verification and Calibration
n. Reference to verification practices including inter-laboratory comparisons, proficiency testing programs, use of reference materials and internal QC schemes	5.8.1 Proficiency Testing 5.8.2 Control Samples
o. Procedures for feedback and corrective action whenever testing discrepancies are detected, or departures from documented policies and procedures occur	4.9 Control of Non-Conformances 4.10 Corrective Action 4.11 Preventive Action 5.8.6 Permitting Departures from Documented Procedures
p. Laboratory management arrangements for exceptionally permitting departures from documented policies and procedures or from standard specifications	4.4.2 Project-Specific Quality Planning 5.8.6 Permitting Departures from Documented Procedures
q. Procedures for dealing with complaints	4.8 Complaints

Table 1.

Correlation of QAPP Sections with NELAC 5.4.2.3 Quality Manual Requirements

NELAC Chapter 5.4.2.3 Quality Manual	Laboratory Quality Manual Section
r. Procedures for protecting confidentiality and proprietary rights (including national security concerns)	4.7.2 Client Confidentiality and Proprietary Rights
s. Procedures for audits and data review	4.13 Internal Audits 4.14 External Audits 5.3.6 Data Reduction and Review
t. Process/procedures for establishing that personnel are adequately experienced in duties they are expected to carry out and are receiving any needed training	5.1.2 Training
u. Ethics policy statement developed by the laboratory and training personnel in their ethical & legal responsibilities	5.1.3 Ethics Policy
v. Reference to procedures for reporting analytical results	5.3.6 Data Reduction and Review 5.9 Project Reports
w. Table of contents, listing reference, glossaries and appendices	TOC Table of Contents Appendix List of Cited SOPs and Work Instructions

3.0 Terms and Definitions

Accuracy: The degree of agreement between a measurement and true or expected value, or between the average of a number of measurements and the true or expected value.

Audit: A systematic evaluation to determine the conformance to specifications of an operational function or activity.

Batch: Environmental samples, which are prepared and/or analyzed together with the same process, using the same lot(s) of reagents. A preparation batch is composed of 1 to 20 environmental samples of a similar matrix, meeting the above mentioned criteria. Where no preparation method exists (e.g., volatile organics, water), the batch is defined as environmental samples that are analyzed together with the same process and personnel, using the same lots of reagents, not to exceed 20 environmental samples. An analytical batch is composed of prepared environmental samples, extracts, digestates or concentrates that are analyzed together as a group. An analytical batch can include prepared samples originating from various environmental matrices and can exceed 20 samples.

Chain of Custody (COC): A system of documentation demonstrating the physical possession and traceability of samples.

Comprehensive Environmental Response, Compensation and Liability Act (CERCLA/Superfund): Legislation (42 U.S.C. 9601-9675 et seq., as amended by the Superfund Amendments and reauthorization Act of 1986 (SARA), 42 U.S.C. 9601et seq.

Compromised Sample: A sample received in a condition that jeopardizes the integrity of the results. See Section 4.7.1 of this LQM for a description of these conditions.

Confidential Business Information (CBI): Information that an organization designates as having the potential of providing a competitor with inappropriate insight into its management, operation or products.

Confirmation: Verification of the presence of a component using an additional analytical technique. These may include second column confirmation, alternate wavelength, derivatization, mass spectral interpretation, alternative detectors, or additional cleanup procedures.

Corrective Action: Action taken to eliminate the causes of an existing non-conformance, defect or other undesirable situation in order to prevent recurrence.

Data Audit: A qualitative and quantitative evaluation of the documentation and procedures associated with environmental measurements to verify that the resulting data are of acceptable quality.

Demonstration of Capability (DOC): Procedure to establish the ability to generate acceptable accuracy and precision.

Document Control: The act of ensuring that documents (electronic or hardcopy and revisions thereto) are proposed, reviewed for accuracy, approved for release by authorized personnel, distributed properly and controlled to ensure use of the correct version at the location where the prescribed activity is performed

ERA Sample: A control sample obtained from an independent source, used to monitor a specific element in the sampling and/or testing process.

Equipment Blank (EB): A portion of the final rinse water used after decontamination of field equipment; also referred to as Rinsate Blank and Equipment Rinsate.

Federal Insecticide, Fungicide and Rodenticide Act (FIFRA): Legislation under 7 U.S.C. 135 et seq., as amended.

Federal Water Pollution Control Act (Clean Water Act, CWA): Legislation under 33 U.S.C. 1251 et seq., Public Law 92-50086 Stat. 816.

Field Blank (FB): A blank matrix brought to the field and exposed to field environmental conditions.

Field Duplicate (FD): Duplicate field-collected sample most commonly used to assess the accuracy of the field collection process.

Field of Testing (FOT): A field of proficiency testing is based on NELAC's categorization of accreditation based on program, matrix and analyte.

Good Laboratory Practices (GLP): Formal regulations for performing basic laboratory operations outlined in 40 CFR Part 160 and 40 CFR Part 729 and required for activities performed under FIFRA and TSCA.

Holding Time: The maximum time that a sample may be held before preparation and/or analysis as promulgated by regulation or as specified in a test method.

Instrument Blank: A blank matrix that is the same as the processed sample matrix (e.g. extract, digestate, condensate) and introduced onto the instrument for analysis.

Internal Chain of Custody (COC): An unbroken trail of accountability that ensures the physical security of samples, data and records. Internal COC refers to additional documentation procedures implemented within the laboratory that includes special sample storage requirements, and documentation of all signatures and/or initials, dates, and times of personnel handling specific samples or sample aliquots.

Instrument Detection Limit (IDL): The minimum amount of a substance that can be measured with a specified degree of confidence that the amount is greater than zero using a specific instrument. The IDL is associated with the instrumental portion of a specific method only, and sample preparation steps are not considered in its derivation. The IDL is a statistical estimation at a specified confidence interval of the concentration at which the relative uncertainty is ±100%. The IDL represents a range where qualitative detection occurs on a specific instrument. Quantitative results are not produced in this range.

Laboratory Control Sample (LCS): A blank matrix spiked with a known amount of analyte(s), processed simultaneously with, and under the same conditions as, samples through all steps of the analytical procedure; also referred to as Matrix Spike Blank (MSB); Environmental Resource Associate Sample (ERA).

Laboratory Quality Manual (LQM): A document stating the quality policy, quality system and quality practices of the laboratory. The LQM may include by reference other documentation relating to the laboratory's quality system.

Limit of Detection (LOD): The minimum amount of a substance that an analytical process can reliably detect. Also referred to as the Method Detection Limit (MDL)

Matrix: The substrate of a test sample. Common matrix descriptions are defined in Table 2.

Table 2. Matrix Descriptions

Matrix	Description
Aqueous	Aqueous sample excluded from the definition of Drinking Water or Saline/Estuarine source. Includes surface water, groundwater, effluents, leachates and wastewaters.
Drinking Water	Aqueous sample that has been designated a potable water source.
Saline	Aqueous sample from an ocean or estuary, or other salt-water source such as the Great Salt Lake.
Liquid	Liquid with <15% settleable solids.
Solid	Soil, sediment, sludge, ash, paint chips, filters, wipes or other matrices with ≥15% settleable solids.
Waste	A product or by-product of an industrial process that results in a matrix not previously defined (i.e., drum liquid or oils).
Tissue	Sample of a biological origin such as fish tissue, shellfish, or plant material. Such samples shall be grouped according to origin.

Matrix Duplicate (MD): Duplicate aliquot of a sample processed and analyzed independently; under the same laboratory conditions; also referred to as Sample Duplicate; Laboratory Duplicate.

Matrix Spike (MS): Field sample to which a known amount of target analyte(s) is added.

Matrix Spike Blank (MSB): A blank matrix spiked with a known amount of analyte(s), processed simultaneously with, and under the same conditions as, samples through all steps of the analytical procedure; also referred to as Laboratory Control Sample (LCS).

Matrix Spike Duplicate (MSD): A replicate matrix spike.

Method Blank (MB): A blank matrix processed simultaneously with, and under the same conditions as, samples through all steps of the analytical procedure.

Method Detection Limit (MDL): The minimum amount of a substance that can be measured with a specified degree of confidence that the amount is greater than zero using a specific measurement system. The MDL is a statistical estimation at a specified confidence interval of the concentration at which the relative uncertainty is ±100%. The MDL represents a range where qualitative detection occurs using a specific method. Quantitative results are not produced in this range.

Non-conformance: An indication, judgment, or state of not having met the requirements of the relevant specifications, contract, or regulation.

Precision: An estimate of variability. It is an estimate of agreement among individual measurements of the same physical or chemical property, under prescribed similar conditions.

Preservation: Refrigeration and/or reagents added at the time of sample collection to maintain the chemical, physical and/or biological integrity of the sample.

Proficiency Testing: Determination of the laboratory calibration or testing performance by means of inter-laboratory comparisons.

Proficiency Test (PT) Sample: A sample, the composition of which is unknown to the analyst, that is provided to test whether the analyst/laboratory can produce analytical results within specified performance limits. Also referred to as Performance Evaluation (PE) Sample.

Proprietary: Belonging to a private person or company.

Quality Assurance (QA): An integrated system of activities involving planning, quality control, quality assessment, reporting and quality improvement to ensure that a product or service meets defined standards of quality with a stated level of confidence.

Quality Assurance (Project) Plan (QAPP): A formal document describing the detailed quality control procedures by which the quality requirements defined for the data and decisions pertaining to a specific project are to be achieved.

Quality Control (QC): The overall system of technical activities, the purpose of which is to measure and control the quality of a product or service.

Quality Control (QC) Sample: A control sample, generated at the laboratory or in the field, or obtained from an independent source, used to monitor a specific element in the sampling and/or testing process.

Quality Management Plan (QMP): A formal document describing the management policies, objectives, principles, organizational authority, responsibilities, accountability, and implementation plan of an agency, organization or laboratory to ensure the quality of its product and the utility of the product to its users.

Quality System: A structured and documented management system describing the policies, objectives, principles, organizational authority, responsibilities, accountability, and implementation plan of an organization for ensuring quality in its work processes, products (items), and services. The quality system provides the framework for planning, implementing, and assessing work performed by the organization and for carrying out required QA/QC.

Quantitation Limit (QL): The minimum amount of a substance that can be quantitatively measured with a specified degree of confidence and within the accuracy and precision guidelines of a specific measurement system. The QL can be based on the MDL, and is generally calculated as 3-5 times the MDL, however, there are analytical techniques and methods where this relationship is not applicable. Also referred to as Practical Quantitation Level (PQL), Estimated Quantitation Level (EQL), Limit of Quantitation (LOQ).

Raw Data: Any original information from a measurement activity or study recorded in laboratory notebooks, worksheets, records, memoranda, notes, or exact copies thereof and that are necessary for the reconstruction and evaluation of the report of the activity or study. Raw data may include photography, microfilm or microfiche copies, computer printouts, magnetic/optical media, including dictated observations, and recorded data from automated instruments. Reports specifying inclusion of "raw data" do not need all of the above included, but sufficient information to create the reported data.

Record Retention: The systematic collection, indexing and storing of documented information under secure conditions.

Reference Standard: A standard, generally of the highest metrological quality available at a given location, from which measurements made at that location are derived.

Reporting Limit (RL): The level to which data is reported for a specific test method and/or sample. The RL is generally related to the QL. The RL must be minimally at or above the MDL.

Resource Conservation and Recovery Act (RCRA): Legislation under 42 U.S.C. 321 et seq. (1976).

Safe Drinking Water Act (SDWA): Legislation under 42 U.S.C. 300f et seq. (1974), Public Law 93-523.

Sampling and Analysis Plan (SAP): A formal document describing the detailed sampling and analysis procedures for a specific project.
Selectivity: The capability of a measurement system to respond to a target substance or constituent.

Sensitivity: The difference in the amount or concentration of a substance that corresponds to the smallest difference in a response in a measurement system using a certain probability level.

Spike: A known amount of an analyte added to a blank, sample or sub-sample.

Standard Operating Procedure (SOP): A written document which details the method of an operation, analysis or action whose techniques and procedures are thoroughly prescribed and which is accepted as the method for performing certain routine or repetitive tasks.

Storage Blank: A blank matrix stored for 1 to 2 weeks with field samples of a similar matrix (volatiles only) that measures storage contribution to any source of contamination.

Systems Audit: A thorough, systematic, on-site, qualitative review of the facilities, equipment, personnel, training, procedures, record keeping, data validation, data management, and reporting aspects of a total measurement system.

Test Method: Defined technical procedure for performing a test.

Toxic Substances Control Act (TSCA): Legislation under 15 U.S.C. 2601 et seq., (1976).

Traceability: The property of a result of a measurement that can be related to appropriate international or national standards through an unbroken chain of comparisons.

Trip Blank (TB): A blank matrix placed in a sealed container at the laboratory that is shipped, held unopened in the field, and returned to the laboratory in the shipping container with the field samples.

Verification: Confirmation by examination and provision of evidence against specified requirements.

4.0 Management Requirements

The organizational chart of STL is presented in Figure 1. Corporate employees are located at various STL facilities as outlined in the organizational structure. The organizational chart of STL Buffalo is presented in Figure 2.

4.1 Organization and Management

The Laboratory Director and Quality Assurance Manager are responsible and have the signature authority for approving and implementing this plan. The Laboratory Director and/or his designee also have signatory authority for approval of work and release of reports. The following listing defines those employees that may act as report signatory designees for the Laboratory Director.

Technical Director
Operations Manager
Quality Manager
Customer Service Manager
Project Manager
Project Manager Assistant

Figure 1. STL Organization Chart

Figure 1. STL Organizational Chart

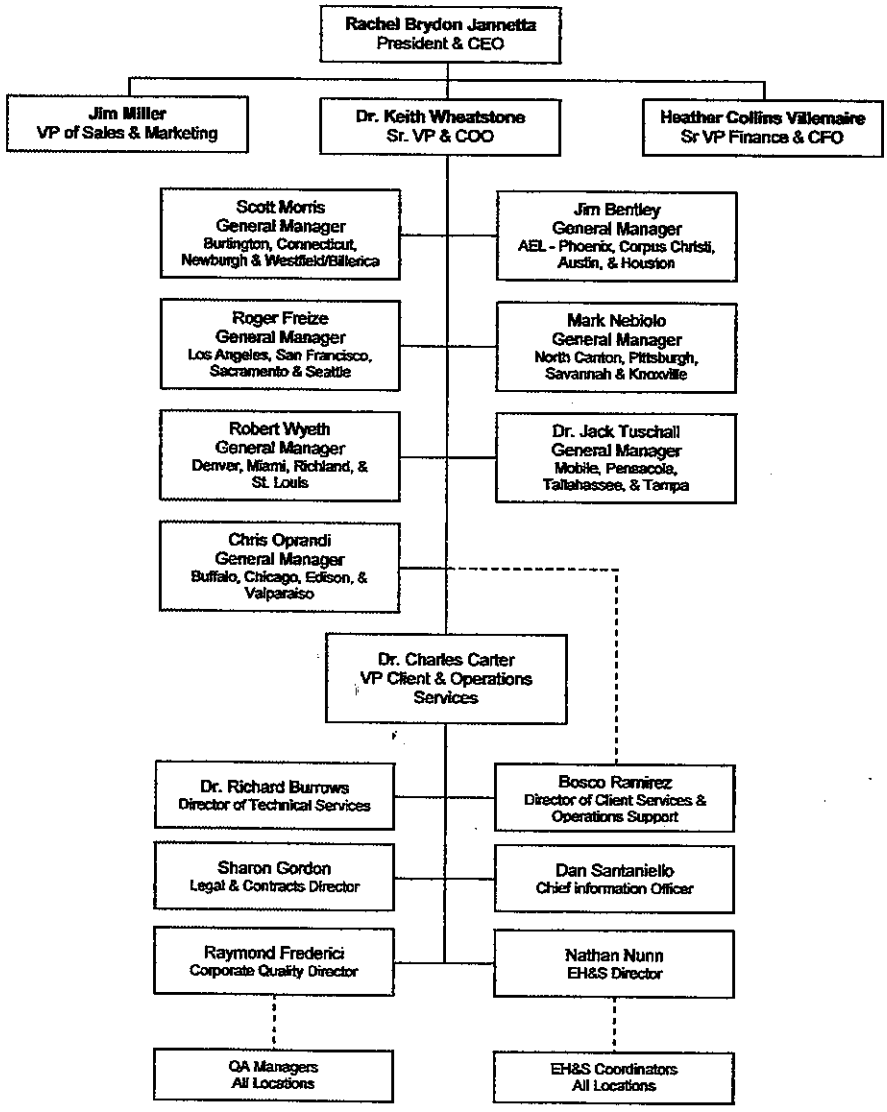
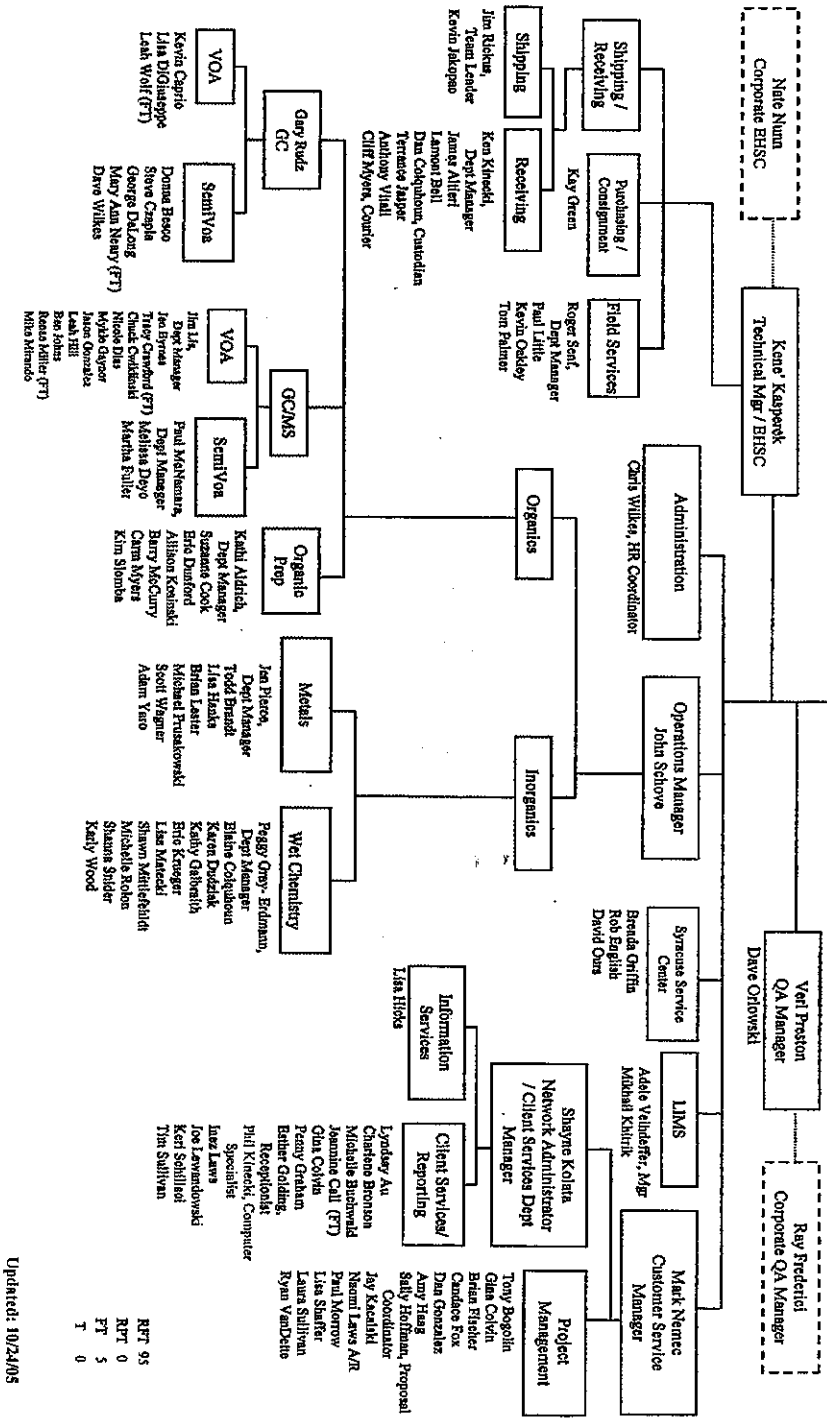


Figure 2. STL Buffalo Organizational Chart, November 2005



4.1.1 Laboratory Facilities

The laboratory is located in Amherst, New York, which is approximately 10 miles from Buffalo, New York, and is staffed by 100 professionals. The laboratory is comprised of 28,000 square feet of state-of-the-art commercial laboratory and office space and houses both inorganic and organic operations. The facility is divided into separate work areas to facilitate sample throughput. These areas include the following:

- Sample receipt and refrigerated storage
- Bottle kit preparation
- Organic and TCLP sample preparation
- Metals digestion
- Wet chemistry laboratory
- Organic instrumentation laboratories
- Metals instrumentation laboratory

The main instrumentation laboratories are equipped with state-of-the-art instrumentation and sufficient duplicate equipment to provide back-up service for most major systems. A listing of laboratory equipment and instrumentation is referenced as STL Buffalo Capital Equipment List, (STLBuffEquipList). Table 3 is a summary of the major laboratory instruments.

Table 3. Major Equipment List

GC	GC/MS	ICP	ICP/MS	CVAA	HPLC	Auto Analyzer	IC	TOC	TOX
20	12	2	1	2	1	4	3	2	1

Each of the laboratory areas has separate heating, ventilation, and air conditioning systems. Non-destructive gas chromatographic detectors, and GC/MS rotary pumps are vented out of the instrumentation through charcoal filters.

4.1.2 Roles and Responsibilities

The specific duties and responsibilities of the Laboratory Director, Technical Director/Environmental Health & Safety Officer, Quality Assurance Manager, Operations Manager, Customer Service Manager/Project Managers, Laboratory Supervisors, Sample Management Coordination, Information Technology Manager, and Chemists/Technicians and Data Packaging Specialists are as follows.

In the absence of any one individual, the staff or assistant within each department is professionally skilled in the ability to administer the function of the administrator or support personnel. This will allow for the continuance of the day-to-day operations of the laboratory.

4.1.2.1 Laboratory Director

The ultimate responsibility for the generation of reliable laboratory data rests with the Laboratory Director, who is accountable to his General Manager and oversees the daily operations of the laboratory. The Laboratory Director's responsibilities include allocation of personnel and resources, setting goals and objectives for both the business and employees, achieving the financial, business and quality objectives of STL. Furthermore, to see that all tasks performed in the laboratory are

conducted according to the requirements of this LQM, the Project Specifications and/or the appropriate QAPP; and to assure that the quality of service provided complies with the project's requirements.

The Laboratory Director has the authority to affect those policies and procedures to ensure that only data of the highest level of excellence are produced. As such, the Laboratory Director is responsible for maintaining a working environment which encourages open, constructive problem solving and continuous improvement.

4.1.2.2 Quality Assurance Manager

The Quality Assurance Manager (QAM) has the full-time responsibility to evaluate the adherence to policies and to assure that systems are in place to produce the level of quality defined in this LQM. The QAM is responsible for:

- Ensures that the laboratory's quality system and LQM meet the requirements of the Corporate QMP.
- Ensures IDL/MDL studies are completed and documented
- Ensures method validation studies are completed and documented
- Periodically performs data package inspections
- Performs data authenticity audits on 100% of analysts and instruments
- Assist in the preparation, compilation, and submittal of quality assurance project plans
- Reviews program plans for consistency with organizational and contractual requirements and advises appropriate personnel of deficiencies
- Maintains QA records
- Maintains certifications and accreditations
- Initiates and oversees both internal and external audits; documents root cause investigations for all noted deficiencies; and ensures timely audit closure
- Maintains a corrective action process for internally identified issues and ensures timely closure
- Manages the laboratory's PT Program and performs/documents root cause investigations for all failures
- Monitors to ensure the documentation of training and method demonstration is current
- Facilitates SOP development and document control
- Submits monthly QA reports to management

The QAM shall have the final authority to accept or reject data, and to stop work in progress in the event that procedures or practices compromise the validity and integrity of analytical data. The QAM is available to any employee at the facility to resolve data quality or ethical issues. The QAM shall be independent of laboratory operations and has an indirect reporting relationship to the STL Corporate QA Director.

4.1.2.3 Technical Director

The Technical Director is responsible for assessing the construction and management of the facility design, maintaining environmental conditions, technical and financial evaluation of capital equipment and capital budgeting and asset valuation.

In addition, the Technical Director solves day to day technical issues, provides technical training and guidance to staff, project managers and clients, investigates technical issues identified by operations personnel or QA, and directs evaluation of new methods.

4.1.2.4 Operations Manager

The Operations Manager reports to the Laboratory Director and oversees the daily operations of the analytical laboratory, maintaining a working environment that encourages open, constructive problem solving and continuous improvement.

The Operations Manager is responsible for supervision of laboratory staff, setting goals and objectives for the laboratory, ensuring compliance with project/client requirements and ensuring on-time performance, supervises maintenance of equipment and scheduling of repairs. Responsibilities also include implementation of the quality system in the laboratory and ensuring timely compliance with audit and QA corrective actions.

In addition, the Operations Manager works with the Technical Director in evaluating technical equipment and assessing capital budget needs.

4.1.2.5 Customer Service Manager/Project Managers

The laboratory recognizes the importance of efficient project management. The laboratory Project Managers (PM) are responsible for preparing the LIMs project technical specifications which summarize QA/QC requirements for the project, maintaining the laboratory schedule, communicating technical requirements to the laboratory, and advising the Operations Manager, QA and Laboratory Supervisors of all variances. The laboratory Project Manager will provide technical guidance and the necessary laboratory-related information to the preparer of project-specific QAPPs and provide peer review of the final document to ensure accuracy of the laboratory information.

4.1.2.6 Laboratory Supervisors

The Laboratory Supervisors are as follows:

- Sample Management Supervisor
- Organic Preparation Supervisor
- GC Analysis Supervisor
- GCMS Volatiles Supervisor
- GCMS Semivolatiles Supervisor
- Metals Supervisor
- Wet Chemistry Supervisor

The Laboratory Supervisors serve as the technical experts on assigned projects, provide technical liaison, assist in resolving any technical issues within the area of their expertise; and implement established policies and procedures to assist the Operations Manager in achieving section goals. The Laboratory Supervisors are responsible for ensuring that their personnel are adequately trained to perform analyses; that equipment and instrumentation under their control is calibrated and functioning properly; that system and performance audits are performed on an as-needed basis; provide input and review in the development and implementation of project-specific QA/QC requirements; and for providing the critical review of proposal and project work for programs as

directed by the Operations Manager and Laboratory Director. The Laboratory Supervisors coordinate these activities with the project management and quality assurance sections.

4.1.2.7 Sample Management Coordination

The Sample Custodian is designated as the Sample Management Coordinator for any work performed internally and responsible for the receipt and login of client samples. The sample custodian confirms the samples received against the Chain of Custody, transports the samples to the proper storage unit within the facility and tracks the disposal of client samples after the required holding time has expired.

4.1.2.8 Subcontract Sample Management Coordination

The Project Manager is designated as the Sample Management Coordinator for any work subcontracted under their management. The Project Manager verifies each subcontracting request to ensure that special client restrictions are not jeopardized (e.g., samples must be analyzed by the receiving affiliated or network laboratory and must maintain specific certification(s)). The Project Manager is also responsible for verifying the credentials; establishing the service agreement; ensuring data review; and invoicing of all laboratory subcontractors. The Project Manager discusses any deficiencies or anomalies with the subcontractor prior to reporting any data to the client.

4.1.2.9 Environmental Health and Safety Coordinator / Waste Management

The Health and Safety Coordinator is responsible for the safety and well-being of all employees while at the laboratory. This includes, but is not limited to, administering the Corporate Safety Manual that complies with federal regulations, MSDS training and review, conducting laboratory safety orientation and tours for all new employees, providing instructions on safety equipment, cleaning up laboratory spills, and instructing personnel of laboratory procedures for emergency situations. The Health and Safety Coordinator is on-call 24-hours a day, 7-days a week for all laboratory situations.

The Health and Safety Coordinator responsibilities additionally include waste management of laboratory generated hazardous waste in accordance with appropriate regulations. This includes maintenance of required documentation, such as waste manifests, segregation of waste in accordance with requirements, and training of personnel in proper segregation of waste and preparation of Safety related SOPs. The EHSC maintains overall EH&S program oversight, but may delegate specific day-to-day activities as necessary.

4.1.2.10 Information Technology Manager

The overall role of the Information Technology (IT) Manager is to enhance laboratory productivity through improved information access, flow, and security. For information to be of greatest value, it must be readily accessible and reliable. It is the responsibility of the IT Manager to provide software tools that allow quick and user friendly access to that information, while at the same time controlling access to that information to those that have the need and proper authority.

Information flow can be enhanced through automation. Automation is the minimization of human intervention in a process. Reduction in human intervention can result in significant error reductions and time savings. The IT Manager assists the laboratory in automation by providing hardware and software solutions to help minimize human intervention in data collection, processing, and storage.

The IT Manager is responsible for providing data security by controlling access, as mentioned above, and for providing for disaster recovery. Data stored on the central Laboratory Information Management System (LIMS) is the direct responsibility of the IT Manager. No fewer than two copies of all data should exist at any time so that lost or destroyed data can always be retrieved from an alternate source. These copies may consist of data within the system and on electronic storage media. Data stored electronically in other departments is the direct responsibility of those departments. However, the IT Manager is responsible for providing procedures and training to all laboratory operations, as appropriate, to assist in making backup copies of local data.

STL has established procedures for IT management:

- *Computer Systems Account and Naming Policy* – P-I-003
- *Computer Systems Password Policy* – P-I-004
- *Software Licensing Policy* – P-I-005
- *Virus Protection Policy* – P-I-006

4.1.2.11 Chemists / Technicians

Any effective laboratory quality assurance/quality control program depends on the entire organization, including management and every individual on the laboratory staff. Analysts and technicians must read and be familiar with the LQM, method SOPs and other essential standard operating procedures. They must know where SOPs are located and agree to adhere to them explicitly unless an error in the SOP is evident and they brought this to the attention of their supervisor or QA manager. They must receive ethics and data integrity training and sign an ethics agreement annually.

Analysts and technicians must ensure that their training records are up to date prior to performing a method without direct supervision. This includes maintaining their training file, filing demonstration of capability evidence and receiving supervisor approval.

The initial review for acceptability of analytical results rests with the analysts conducting the various tests. Observations made during the performance of an analytical method may indicate that the analytical system is not in control. Analysts must use quality control indicators to assure that the method is within acceptance limits, corrective action is taken or a non-conformance (job exception) report is documented/approved before reporting results.

4.1.2.12 Data Packaging Specialist

The Data Packaging Specialist is responsible for coordinating receipt of all data from the various service groups within the laboratory, reviewing data for compliance to laboratory QC criteria and/or criteria in the LIMs Project Profile Specification, and ensuring that data are reported in a timely manner and in the proper format.

4.2 Quality System

The quality system and quality objectives are driven by this LQM, SOPs and Work Instructions. Within these documents, the Laboratory Director and Quality Manager ensure that the quality policy is understood, implemented, and maintained at all levels of the organization. The development and implementation of appropriate accountabilities, duties, and authority by organizational positions are clearly delineated. Line organizations achieve and verify that specifications are achieved; the QAM provides oversight and verification of processes through planning, reviews, audits, and surveillances.

The Laboratory Director's leadership, support and direction ensure that the policies and procedures are implemented throughout the organization.

4.2.1 Objectives of the Quality System

The goal of the quality system is to ensure that business operations are conducted with the highest standards of professionalism and data integrity in the industry.

To achieve this goal, it is necessary to provide our clients with scientifically sound, well documented, regulatory compliant data, and to ensure that we provide the highest quality service available in the industry with uncompromising data integrity. A well-structured, organized and communicated quality system is essential in meeting this goal. The laboratory's quality system is designed to minimize systematic error, encourage constructive, documented problem solving, and provide a framework for continuous improvement.

This LQM, Work Instructions and the SOPs are the basis and outline for our quality and data integrity system and contain requirements and general guidelines under which the laboratory conducts operations. In addition, other documents may be used by the laboratory to clarify compliance with quality system or other client requirements. Within the LQM, SOP or Work Instruction numbers are noted in parenthetic text. These numbers refer to the laboratory procedure(s) associated with the subject item. A table listing these quality system policies and procedures is appended to this document.

The QA Manager is responsible for implementing and monitoring the Quality System. The QA Manager reports to the Laboratory Director on the performance of the quality system for review and continuous improvement. The QA Manager has sufficient authority, access to work areas, and organizational freedom (including sufficient independence from cost and schedule considerations) to:

- Initiate action to prevent the occurrence of any nonconformities related to product, process and quality system,
- Identify and record any problems affecting the product, process and quality system,
- Initiate, recommend, or provide solutions to problems through designated channels,
- Verify implementation of solutions, and
- Assure that further work is stopped or controlled until proper resolution of a non-conformance, deficiency, or unsatisfactory condition has occurred and the deficiency or unsatisfactory condition has been corrected.

The QA Manager identifies opportunities for continual improvement. When a situation arises where acceptable resolution of identified issues cannot be agreed upon at the laboratory, direct access to STL's Corporate QA Director is available. This provides laboratory QA personnel independence, where needed, to ensure that QA policies and procedures are enforced.

The QA Manager conducts annual training for all laboratory and administrative personnel to ensure their familiarity with the quality documentation and the implementation of the policies and procedures in their work.

4.3 Document Control

The laboratory maintains procedures to control documents and analytical data. Since an extensive quantity of data is generated and this is our primary product, document control is inherently segregated from data control, as described further in Sections 4.3.1 and 4.3.2.

4.3.1 Document Control Procedure

Organization, security and control of documents are necessary to ensure that confidential information is not distributed and that all current copies of a given document are from the latest applicable revision. Unambiguous identification of a controlled document is maintained by information in the document header: Document Number, Revision Number, Effective Date, and Number of Pages. Document control may be achieved by either electronic or hardcopy distribution. Documents may be controlled for a specific time period after issuance. In this case the document will be marked with the date issue and expiration date.

Controlled documents are authorized and records of their distribution and archiving are maintained by the QA Department. Controlled status is defined as the continuous distribution of document updates where document marked as either "Controlled" or "Uncontrolled". Uncontrolled status is defined as the single distribution of the current SOP. Document updates are not distributed to people holding documents marked "uncontrolled". For tracking purposes, a control copy number is assigned to documents distributed with a controlled status. All copy numbers are written or typed in red to easily identify the SOP as a controlled copy.

4.3.1.1 Document Revision

Changes to documents occur when a procedural change warrants a revision of the document. After document revisions are authorized, all outdated versions are removed from use and disposed or segregated from the active/current document versions. A single copy of the archived document is retained for historical purposes. This archived version is clearly identified as an "Archived Copy".

SOPs are reviewed and/or updated on a 12 month basis, which is tracked by an established review schedule *(SOP Master Index)*. These reviews are conducted by the analyst, QA Manager, Department Supervisor, Laboratory Director or the Health and Safety Coordinator, all of whom may provide the approval signature for each SOP.

4.3.2 Data Control

All raw data, such as bound logbooks, instrument printouts, magnetic tapes, electronic data, as well as final reports, are retained for a minimum period of 5 years, unless otherwise specified by client or regulatory requirements. Such data may be maintained longer, as defined by client and project requirements. Specifics on the procedure of archiving records and client or project specific requirements are contained in the SOP, Record Storage and Retention,*(AGP-RecordStorage-56).*

Raw data and reports are documented and stored in a manner which are easily retrievable. The procedure for maintaining raw data records is briefly described below:

- Instrument print-outs for conventional inorganic parameters are filed by parameter and month. Inorganic Metals are filed by Instrument and Filename. Generally, current year documents are kept on file in the laboratory sections.
- All raw data, for example, instrument print-outs and logbooks, are maintained in a secured storage area or records are scanned and retained on electronic media.

- The computer information is backed up on tape daily, and stored in a secured and temperature/humidity controlled environment to maintain the integrity of the electronic information in the event of system failure. Copies of all back-up tapes are maintained in secured off-site locations.
- All copies of client final reports are maintained in hard copy format or electronically (e.g., Adobe Acrobat).

4.4 Request, Tender, and Contract Review

4.4.1 Contract Review

For many environmental sampling and analysis programs, testing design is site or program specific and does not necessarily "fit" into a standard laboratory service or product. It is STL's intent to provide both standard and customized environmental laboratory services to our clients. To ensure project success, technical staff members perform a thorough review of technical and QC requirements contained in contracts. Contracts are reviewed for adequately defined requirements and STL's capability to meet those requirements.

All contracts entered into by the laboratory are reviewed for the client's requirements in terms of compound lists, test methodology requested, sensitivity, accuracy, and precision requirements. The reviewer ensures that the laboratory's test methods are suitable to achieve these regulatory and client requirements and that the laboratory holds the appropriate certifications and approvals to perform the work. The review also includes the laboratory's capabilities in terms of turnaround time, capacity, and resources to provide the services requested, as well as the ability to provide the documentation, whether hardcopy or electronic. If the laboratory cannot provide all services but intends to subcontract such services, whether to another STL facility or to an outside firm, this will be documented and discussed with the client prior to contract approval.

Any contract requirement or amendment to a contract communicated to STL verbally is documented and confirmed with the client in writing (e.g., letter, contract, e-mail, etc.). Any discrepancy between the client's requirements and STL's capability to meet those requirements is resolved in writing before acceptance of the contract. Contract amendments, initiated by the client and/or STL, are documented in writing for the benefit of both the client and STL. All contracts, QAPPs, Sampling and Analysis Plans (SAPs), contract amendments, and documented communications become part of the permanent project record.

4.4.2 Project-Specific Quality Planning

Communication of contract specific technical and QC criteria is an essential activity in ensuring the success of site specific testing programs. To achieve this goal, STL assigns a Project Manager (PM) to each client. The PM is the first point of contact for the client. It is the PM's responsibility to ensure that project specific technical and QC requirements are effectively evaluated and communicated to the laboratory personnel before and during the project (Project Information Requirements, APM-ProjInfo-20). QA department involvement may be needed to assist in the evaluation of custom QC requirements.

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

resources are sufficient to perform work for the client's project. Project management is positioned between the client and laboratory resources.

Prior to work on a new project, the dissemination of project information and/or project opening meetings may occur to discuss schedules and unique aspects of the project. Items to be discussed may include the project technical profile, turnaround times, holding times, methods, analyte lists, reporting limits, deliverables, sample hazards, or other special requirements. The PM introduces new projects to the laboratory staff through project kick-off meetings *(APM-ProjInfo-20)* or to the supervisory staff during production meetings. These meetings provide direction to the laboratory staff in order to maximize production and client satisfaction, while maintaining quality. In addition, project notes may be associated with each sample batch (e.g., Job) as a reminder upon sample receipt and analytical processing.

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

Such changes are also communicated to the laboratory through the management Production Meetings which are conducted two times per week. Such changes are updated to the project notes and are introduced to the managers at these meetings. The laboratory staff is then introduced to the modified requirements via the Project Manager or the individual laboratory section manager.

STL strongly encourages client visits to the laboratory and for formal/informal information sharing sessions with employees in order to effectively communicate ongoing client needs as well as project specific details for customized testing programs.

4.4.3 Data Quality Objectives

Data quality objectives (DQO) are qualitative and quantitative statements used to ensure the generation of the type, quantity, and quality of environmental data that will be appropriate for the intended application. Typically, DQOs are identified before project initiation and during the development of QAPPs and SAPs. The analytical DQOs addressed in this section are precision, accuracy, representativeness, completeness, and comparability.

The components of analytical variability (uncertainty) can be estimated when QC samples of the right types and at the appropriate frequency are incorporated into the measurement process of the laboratory. STL incorporates numerous QC samples to obtain data for comparison with the analytical DQOs and to ensure that the measurement system is functioning properly. The control samples and their applications, described in Section 5.8.2, are selected based on analytical method or client-specific requirements. Analytical QC samples for inorganic and organic analyses may include calibration blanks, instrument blanks, method blanks, laboratory control standards, calibration standards, matrix spikes, matrix duplicates and surrogate spikes.

The DQOs discussed below ensure that data are gathered and presented in accordance with procedures appropriate for its intended use, that the data is of known and documented quality, and are able to withstand scientific and legal scrutiny.

4.4.3.1 Precision

Precision is an estimate of variability. It is an estimate of agreement among individual measurements of the same physical or chemical property, under prescribed similar conditions. Precision is expressed either as Relative Standard Deviation (RSD) for greater than two measurements or as Relative Percent Difference (RPD) for two measurements. Precision is determined, in part, by analyzing data from LCSs, MS, MSD, and MD.

Precision also refers to the measurement of the variability associated with the entire process, from sampling to analysis. Total precision of the process can be determined by analysis of duplicate or replicate field samples and measures variability introduced by both the laboratory and field operations.

4.4.3.2 Accuracy

Accuracy is the degree of agreement between a measurement and the true or expected value, or between the average of a number of measurements and the true or expected value. It reflects the total error associated with a measurement.

Both random and systematic errors can affect accuracy. For chemical properties, accuracy is expressed either as a percent recovery (R) or as a percent bias (R - 100). Accuracy is determined, in part, by analyzing data from LCSs, MS and MSD.

Accuracy and Precision objectives employed by the laboratory are as defined in the CERCLA's Inorganic and Organic Statements of Work (SOW); statistically-derived control limits; or default limits as listed in each respective method SOP.

4.4.3.3 Representativeness

Representativeness is the degree to which data accurately and precisely represent a characteristic of a population, a variation in a physical or chemical property at a sampling point, or an environmental condition. Data representativeness is primarily a function of sampling strategy; therefore, the sampling scheme must be designed to maximize representativeness. Representativeness also relates to ensuring that, through sample homogeneity, the sample analysis result is representative of the constituent concentration in the sample matrix. STL makes every effort to analyze an aliquot that is representative of the original sample, and to ensure the homogeneity of the sample before sub-sampling.

4.4.3.4 Completeness

Completeness is defined as the percentage of measurements that are judged valid or useable. Factors negatively affecting completeness include the following: sample leakage or breakage in transit or during handling, loss of sample during laboratory analysis through accident or improper handling, improper documentation such that traceability is compromised, or sample result is rejected due to failure to conform to QC specifications. A completeness objective of greater than 90% of the data specified by the statement of work is the goal established for most projects.

4.4.3.5 Comparability

Comparability is a measure of the confidence with which one data set can be compared to another. To ensure comparability, all laboratory analysts are required to use uniform procedures (e.g., SOPs) and a uniform set of units and calculations for analyzing and reporting environmental data.

A measure of inter-laboratory comparability is obtained through the laboratory's participation in proficiency testing (PT) programs established with Water Supply (WS), Water Pollution (WP), Solid Waste (SW), and Underground Storage Tank (UST) programs. In addition, the laboratory employs the use of NIST or EPA traceable standards, when available, to provide an additional measure of assurance of the comparability of data.

Project representativeness and comparability are dependent upon the sampling plan on a project specific basis, and are therefore not covered in this LQM. Assessment of site and collection representativeness and comparability is performed by client or field engineer.

4.4.3.6 Additional DQOs

Method Detection Limits

The method detection limit (MDL) is the lowest concentration that can be detected for a given analytical method and sample matrix with 99% confidence that the analyte is present. The MDL is determined according to Appendix B of 40 CFR 136, "Guidelines Establishing Test Procedures for the Analysis of Pollutants". MDLs reflect a calculated (statistical) value determined under ideal laboratory conditions in a clean matrix, and may not be achievable in all environmental matrices. The laboratory maintains MDL studies for analyses performed; these are verified at least annually.

For the performance of non-routine methods, e.g., client/contract requirement, MDLs or Method Validation Studies will be completed on an as needed basis. The turnaround time for such studies will be as determined by the client and Project Manager. Such studies will be reviewed and approved by the client and/or regulatory agency prior to project implementation.

Instrument Detection Limits

There are a number of ways to determine Instrument Detection Limit (IDL) sensitivity (e.g., signal-to-noise ratio, precision of the low-level standard, lowest calibration curve point or the IDL study defined within CLP). The method and means in which IDLs are determined are documented and maintained in the QA department for each individual instrument.

IDLs are periodically generated for each element by the metals laboratory based on project or program requirements (i.e., CLP is quarterly for each instrument). These limits are used to gauge instrument sensitivity without the introduction of preparation method variance.

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 can report with acceptable quantitative error or client requirements, values specified by the EPA methods or other project and client requirements. The laboratory reporting limits are further related and verified by the lowest point on a calibration curve. Because of the high level of quantitative error associated with determinations at the level of the MDL, the laboratory endeavors to keep reporting limits higher than the MDL. Wherever possible, reporting is limited to values approximately 2-5x the respective MDL to ensure confidence in the value reported.

Client specific requests for reporting below the routine laboratory reporting limit or approaching the IDL or MDL are special circumstances not to be confused with the previous statement. Data evaluated down to the MDL/IDL is qualified as estimated with a 'J' for organic analyses, a 'B' for inorganic analyses or with a comment in the report case narrative.

MDL studies are performed annually, and reporting limits are assessed. If the MDL does not meet the routine laboratory reporting limit or the method specified limit, it is repeated or the laboratory reporting

limit is reassessed. If the laboratory continually demonstrates that the method reporting limits are not achieved, equipment, technique, and the method are reviewed to assure optimal performance or appropriate action is taken.

4.5 Subcontracting

Subcontracting is arranged with the consent of the client. Consent shall be requested in a timely manner and the client response shall be suitably prompt to ensure that it shall not be unreasonably refused. All QC guidelines specific to the client's analytical program are transmitted to the subcontractor and agreed upon before sending the samples to the subcontract facility. Proof of required certifications from the subcontract facility is maintained in the project records. Where applicable, specific QC guidelines, QAPPs, and/or SAPs are transmitted to the subcontract laboratory. Samples are subcontracted under formal Chain of Custody (COC).

Subcontract laboratories may receive an on-site audit by a representative of STL's QA staff if it is deemed appropriate by the QA Manager. The audit involves a measure of compliance with the required test method, QC requirements, as well as any special client requirements (e.g., Technical Profile and LIMS Project Notes). STL may also perform a paper audit of the subcontractor, which would entail reviewing the LQM, the last two PT studies, and a copy of any recent regulatory audits with the laboratory's responses.

Intra-company subcontracting may also occur between STL facilities. Intra-company subcontracting within STL is arranged with the documented consent of the client or a QAPP specification. The originating laboratory is responsible for communicating all technical, quality, and deliverable requirements as well as other contract needs.

Project reports from both STL and external subcontractors are not altered and are included in their original form in the final project report provided by STL. This clearly identifies the data as being produced by a subcontractor facility. If subcontract data are incorporated into the laboratory's report (i.e., imported), the report must explicitly indicate which lab produced the data for which methods and samples , as required in Section 5.9.4.

4.6 Purchasing Services and Supplies

Evaluation and selection of suppliers and vendors is performed, in part, on the basis of the quality of their products, their ability to meet the demand for their products on a continuous and short term basis, the overall quality of their services, their past history, and competitive pricing. This is achieved through evaluation of objective evidence of quality furnished by the supplier, which can include certificates of analysis, recommendations, and proof of historical compliance with similar programs for other clients. To ensure that quality critical consumables and equipment conform to specified requirements, all purchases from specific vendors are approved by a member of the supervisory or management staff.

Chemical reagents, solvents, glassware, and general supplies are ordered as needed to maintain sufficient quantities on hand. Purchasing guidelines for equipment and reagents meet with the requirements of the specific method and testing procedures for which they are being purchased. The measurements for evaluation and selection of suppliers; the acceptance of supplies and services; and certificates of conformance are described in the procurement SOP *(Procurement of Laboratory Supplies and Services, APH-Supply-08).*

4.6.1 Solvent and Acid Lot Verification

Pre-purchase approval is performed for solvents and acids purchased in large quantities unless a certificate of conformance has been furnished. These may include acetone, ethyl ether, hexane, methylene chloride, nitric acid, hydrochloric acid, sulfuric acid, and hydrogen peroxide. Each lot of incoming supplies requiring pre-approval is checked against the previously approved lot number. If the lot number is not approved, the lot is refused. If the lot number is an approved lot number, it is accepted and documented. Solvents and acids are pre-tested in accordance with STLs Corporate *Testing Solvents and Acids* procedure (S-T-001) for all of the STL laboratories.

4.7 Service to the Client

4.7.1 Sample Acceptance Policy

Samples are considered "compromised" if the following conditions are observed upon sample receipt:

- Cooler and/or samples are received outside of temperature specification.
- Samples are received broken or leaking.
- Samples are received beyond holding time.
- Samples are received without appropriate preservation.
- Samples are received in inappropriate containers.
- COC does not match samples received.
- COC is not properly completed or not received.
- Breakage of any Custody Seal.
- Apparent tampering with cooler and/or samples.
- Headspace in volatiles samples >6mm.
- Seepage of extraneous water or materials into samples.
- Inadequate sample volume.
- Illegible, impermanent, or non-unique sample labeling.

When "compromised" samples are received, it is documented on the hardcopy COC, the LIMS Sample Receipt Checklist or on an Analytical Receipt Resolution Form (ARRF); and the client is contacted for instructions. If the client decides to proceed with the analysis, the project report will clearly indicate any of the above conditions and the resolution.

4.7.2 Client Confidentiality and Proprietary Rights

Data and sample materials provided by the client or at the client's request, and the results obtained by STL, shall be held in confidence (unless such information is generally available to the public or is in the public domain or client has failed to pay STL for all services rendered or is otherwise in breach of the terms and conditions set forth in the STL and client contract) subject to any disclosure required by law or legal process. Technical, business and proprietary information provided by a client and data/information generated by the laboratory are restricted for the use within the laboratory for purposes of accomplishing the project. Client information is not to be used on other projects or revealed except in conjunction with project work to anyone outside the laboratory without permission of the client.

STL's reports, and the data and information provided therein, are for the exclusive use and benefit of client, and are not released to a third party without written consent from the client (*Client Confidentiality Section 6.9; APM-ProjInfo-20).*

4.8 Complaints

STL believes that effective client complaint handling processes have important business and strategic value. Listening to and documenting client's concerns captures "client knowledge" that helps to continually improve processes and outpace the competition. Implementing a client complaint handling process also provides assurance to the data user that the laboratory will stand behind its data, service obligations and products.

Client inquiries, complaints or noted discrepancies are documented, communicated to management, and addressed promptly and thoroughly. The investigation of the cause, resolution and authorization of corrective action is documented *[Data Quality Request (DQR); SOP AQA-DQR-65 or Corrective Action Notice (CAN); SOP AQA-CA-65)].*

Client complaints are documented by the employee receiving the complaint. The documentation can take the form of a Data Quality Review request (DQR) or in a format specifically designed for that purpose (e.g., phone conversation record or e-mail). The Laboratory Director, CSM, Technical Director and/or QA Manager are informed of client complaints and assist in resolving the complaint.

The nature of the complaint is identified, documented and investigated, and an appropriate action is determined and taken. STL Buffalo uses an automated documentation and tracking mechanism for the DQR process which provides a system for trend analysis of repeat complaints. In cases where a client complaint indicates that an established policy or procedure was not followed, the QA department is required to conduct a special audit to assist in resolving the issue. A written confirmation, or letter to the client, outlining the issue and response taken is strongly recommended as part of the overall action taken.

The number and nature of client complaints is reported by the QA Manager to the Corporate QA Director in the QA Monthly report. Monitoring and addressing the overall level and nature of client complaints and the effectiveness of the solutions is part of the Quality Systems Management Review *(QMP, Section 4.15.2 and SOP AQA-Management Review-45).*

4.9 Control of Non-conformances

Non-conformances include any out of control occurrence. Non-conformances may relate to client specific requirements, procedural requirements, or equipment issues. All non-conformances in the laboratory are documented at the time of their occurrence on a Job Exception Report, also known as a non-conformance report *(AQA-CA-65)*

All non-conformances that affect a sample and/or sample data become part of the affected project's permanent record. When appropriate, reanalysis is performed where QC data falls outside of specifications, or where data appears anomalous. If the reanalysis comes back within established tolerances, the results are approved. If the reanalysis is still outside tolerances, further reanalysis or consultation with the Section Manager, Project Manager or QA Manager for direction may be required. All records of reanalysis are kept with the project files.

Where non-conformances specifically affect a client's sample and/or data, the client is informed and action must be taken. Action can take the form of reporting and flagging the data, and including a description of the non-conformance in the project narrative.

4.10 Corrective Action

To consistently achieve technical and regulatory requirements, the laboratory data must be supported by an effective corrective action system. The system must be capable of isolating and rectifying both random and systematic errors. Identification of systematic errors, or errors that are likely to occur repetitively due to a defect or weakness in a system, is particularly valuable in maintaining an environment of continuous improvement in laboratory operations.

Mechanisms used to ensure problem definition include SOPs; internal and external audits and surveillances; and regular laboratory management meetings. When evaluation of performance against established criteria for good laboratory practices shows a condition that could adversely affect the quality of services provided, corrective action is initiated.

Any employee in STL can initiate a corrective action. The initial source of corrective action can also be external to STL (i.e., corrective action due to client complaint, regulatory audit, or PT(s)). When a problem that requires corrective action is identified, the following items are identified by the initiator on the corrective action report: the nature of the problem, the name of the initiator, and the date. If the problem affects a specific client project, the PM is informed immediately.

All corrective actions, whether immediate or long-term, will comprise the following steps to ensure a closed-loop corrective action process:

- Define the problem.
- Assign responsibility for investigating the problem.
- Determine a corrective action to eliminate the problem.
- Assign, and obtain commitment to, responsibility for implementing the corrective action.
- Implement the correction.
- Assess the effectiveness of the corrective action and verify that the corrective action has eliminated the problem.

4.10.1 Immediate Corrective Action

Immediate corrective actions to correct or repair non-conforming equipment and systems are generally initiated in response to adverse conditions identified through QC procedures. The analyst has relatively quick feedback that a problem exists, e.g., calibration does not meet or QC check samples exceed allowable criteria, and can take immediate action to repair the system.

The initial responsibility to monitor the quality of a function or analytical system lies with the individual performing the task or procedure. DQOs are evaluated against laboratory-established or against method or client specified QA/QC requirements. If the assessment reveals that any of the QC acceptance criteria are not met, the analyst must immediately assess the analytical system to correct the problem. When the appropriate corrective action measures have been defined and the analytical system is determined to be "in-control" or the measures required to put the system "in-control" have been identified and scheduled, the problem and resolution or planned action is documented in the

appropriate logbook or Job Exception Report. Data generated by an analytical system that is determined to be out-of-control must never be released without approval of the Section Manager, QA Manager, Laboratory Director and client notification.

When an acceptable resolution cannot be met or data quality is negatively affected, the analyst will notify their Section Manager and initiate a Job Exception. If a Job Exception is required, it is routed for proper authorizations and direction. Proper authorization and direction is given by the Project Manager and/or QA Manager. Based upon the circumstances and judgment of the Project Manager, in conjunction with the QA Manager, the client will be notified of the situation.

Data generated concurrently with an out-of-control system will be evaluated for usability in light of the nature of the deficiency. If the deficiency does not impair the usability of the results, data will be reported and the deficiency will be noted in the case narrative. Where sample results may be impaired, the Project Manager is notified by a written Job Exception Report and appropriate corrective action (e.g., reanalysis) is taken and documented.

A Job Exception documents analytical problems at the bench level. This form allows for the documentation of the out-of-control situation, actions undertaken to correct the problem and a return-to-control status. All Job Exceptions are signed/dated by the respective laboratory section manager.

The QA Manager has the authority to stop the analysis, e.g., failure to meet method or project requirements, and to hold all analyses of samples affected by an out-of-control situation. The method cannot be restarted without appropriate documentation leading to the QA Manager's approval and sign-off.

4.10.2 Long-term Corrective Action

Long-term corrective action is generally initiated due to QA issues, which are most often identified during internal and external audits (Sections 4.13 & 4.14). Typically, a deeper investigation into the root cause of the nonconformance is warranted, and the problem may take much longer to identify and resolve. Staff training, method revision, replacement of equipment, and LIMS reprogramming are examples of long-term corrective action.

4.10.3 Responsibility and Closure

The Section Manager is responsible for correcting out-of-control situations, placing highest priority on this endeavor. Associated corrective actions, once verified for effectiveness, are incorporated into standard practices. Ineffective actions will be documented and re-evaluated until acceptable resolution is achieved. Section Managers are accountable to the Operations Manager to ensure final acceptable resolution is achieved and documented appropriately.

The QA Manager also may implement a special audit (Section 4.13). The purpose of inclusion of the corrective action process in both routine and special audits is to monitor the implementation of the corrective action and to determine whether the action taken has been effective in overcoming the issue identified.

Any out-of-control situations that are not addressed acceptably at the laboratory level may be reported to the Corporate Quality Director by the QA Manager, indicating the nature of the out-of-control situation and problems encountered in solving the situation. This provides laboratory QA personnel non-laboratory management support, if needed, to ensure QA policies and procedures are enforced.

4.11 Preventative Action

The laboratory's preventive action programs improve, or eliminate potential causes of nonconforming product and/or nonconformance to the quality system. This preventive action process is a proactive continuous process improvement activity which can be initiated by clients, employees, business providers, and affiliates. The QA section has the overall responsibility to ensure that the preventive action process is in place, and that relevant information on actions is submitted for management review.

Preventive action opportunities may be identified from information obtained through activities related to but not limited to the corrective action process, performance evaluation program, internal audits, management review, and/or market trends, industry trends and competitive comparisons.

Established standard practices for preventive action are included in the Corrective and Preventive Action SOP *(AQA-CA-35)*; the Data Quality Request SOP *(AQA-DQR-65)* and the Quality System Management Review SOP *(AQA-Management Review-45)*. These procedures describe the information sources used to detect, analyze, and eliminate potential causes of nonconformities and to ensure effective implementation of solutions.

4.12 Records

4.12.1 Record Types

Record types are described in Table 4.

4.12.2 Record Retention

Data reports are filed electronically as .pdf files by job number. Hardcopy COC files are maintained and are filed with the original Job File in job number order.

Laboratory data, project management files, QA records (e.g., PT scores/corrective actions; MDLs/IDLs, statistical analysis, QAPPs, etc..), Human Resources information, etc.., are compiled by date order. The same procedure is followed both in current and archived hardcopy storage.

Upon archiving, a record is made in the Archive Logbook and a number is assigned for each storage box of records. This logbook documents the contents (description and dates) of each storage box. Records are maintained for the periods defined in Tables 5 and 6. On an annual basis, the storage boxes are reviewed and those records subject to disposal are purged.

Table 5 outlines the laboratory's standard record retention time. For raw data and project records, record retention is calculated from the date the project report is issued. For other records, such as Controlled Documents, QC, or Administrative Records, the retention time is calculated from the date the record is formally retired. Records related to the programs listed in Table 6 have lengthier retention requirements and are subject to the requirements in Section 4.12.3.

Table 4. STL Record Types

Raw Data	Controlled Documents	QC Records	Project Records	Administrative Records

Raw Data	Controlled Documents	QC Records	Project Records	Administrative Records
See Section 3. Terms and Definitions	LQMs/ QAPPs	Audits/ Responses	COC Documentation	Accounting
	QMP (Corporate)	Certifications	Contracts and Amendments	Corporate Safety Manual, Permits, Disposal Records
	SOPs	Job Exceptions / DQRs	Correspondence	Employee Handbook
		Logbooks*	QAPP	Personnel files, Employee Signature & Initials, Training Records
		Method & Software Validation, Verification	SAP	
		Standards Certificates	Telephone Logbooks	Technical and Administrative Policies
	Work Instructions	MDL/IDL/IDC Studies	E-mails	
		PTs	Electronic Data Report	
		Statistical Evaluations		

*Examples of Logbook types: Maintenance, Instrument, Preparation (standard and samples), Standard and Reagent Receipt, Archiving, and Balance Calibration.

Table 5. STL Record Retention

Record Type		Archival Requirement *
Raw Data	All* (Electronic Data Reports (.pdf & EDD)	5 Years from completion
Controlled Documents	All*	5 Years from document retirement date
QC	All*	5 Years from archival
Project	All*	5 Years from project completion
Administrative	Personnel/Training	Indefinitely
	Accounting	10 years

* Exceptions listed in Table 6.

4.12.3 Programs with Longer Retention Requirements

Some regulatory programs and clients have longer record retention requirements than the laboratory's standard record retention time. These are detailed in Table 6 with their retention requirements and client-specific requirements are listed in the Record Retention and Storage SOP (*AQA-RecordStorage-56*). In these cases, the longer retention requirement is implemented and

noted in the archive. If special instructions exist such that client data cannot be destroyed prior to notification of the client, the container or box containing that data is marked as to who to contact for authorization prior to destroying the data.

Table 6. Special Record Retention Requirements

Program	Retention Requirement
NY Potable Water NYCRR Part 55-2	10 years
Commonwealth of MA – All environmental data 310 CMR 42.14	10 years
FIFRA – 40 CFR Part 160	Retain for life of research or marketing permit for pesticides regulated by EPA
Michigan Department of Environmental Quality – all environmental data	10 years
Minnesota – Drinking Water	10 years
Navy Facilities Engineering Service Center (NFESC)	10 years
OSHA - 40 CFR Part 1910	30 years
Pennsylvania – Drinking Water	10 years
TSCA – 40 CFR Part 792	10 years after publication of final test rule or negotiated test agreement
Louisiana – All environmental data	10 years

4.12.4 Archives and Record Transfer

Archives are indexed such that records are accessible on either a project or temporal basis. Archives are protected against fire, theft, loss, deterioration, and vermin. Electronic records are protected from deterioration caused by magnetic fields and/or electronic deterioration. Access to archives is controlled and documented.

STL ensures that all records are maintained as required by the regulatory guidelines and per this LQM upon facility location change or ownership transfer. Upon facility location change, all archives are retained by STL in accordance with this LQM. Upon ownership transfer, all final test reports generated by the laboratory will be submitted to the clients if not previously provided. Any further record retention requirements will be addressed in the ownership transfer agreement and the responsibility for maintaining archives is clearly established.

In the event that the laboratory is closed, all final test reports generated by the laboratory will be submitted to the clients if not previously provided. All records will then be transferred to STL's corporate record storage location. All boxes and contents will be appropriately labeled with the dates of destruction (Refer to Tables 5 and 6) and managed in accordance their policies.

4.13 Internal Audits

Quality assurance audits and surveillances are conducted to assess the performance of laboratory systems in meeting technical, regulatory and client requirements; and to evaluate the operational details of the QA program *(Systems Audits; S-Q-002)*. They provide a means for management to be apprised of, and to respond to, a potential problem before it actually impacts the laboratory operations. They also are a mechanism for ensuring closure of corrective actions resulting from external audits.

4.13.1 Audit Types and Frequency

A number of types of audits are performed at STL. These audit types and frequency are categorized in Table 7.

Table 7. Audit Types and Frequency

Audit Type	Performed by	Frequency
Systems	QA Department or Designee	Annual
Data	QA Department or Designee	**Data Report Review:** As necessary to ensure an effective secondary review process
		Analyst Data Audits: 100% of all analysts annually
		Electronic Data Audits: 100% of all organic instruments
Special	QA Department or Designee	As Needed

4.13.2 Systems Audits

Systems audits are technical in nature and are conducted on an ongoing basis by the QA Manager. Systems audits cover all departments of the facility, both operational and support. The review consists of laboratory systems, procedures, documentation and issues noted in external audits.

The audit report is issued by the QA Manager within 21 calendar days of the audit. The audit report is addressed to the Operations Manager and Department Supervisors and copied to the Corporate Quality Director and the Laboratory Director.

Written audit responses are required within 30 calendar days of the audit report issue. A maximum of one calendar month is given to address any recommended corrective actions. The audit response is directed to all individuals copied on the audit report. Where a corrective action may require longer than a calendar month to complete, the target date for the corrective action implementation is stated and evidence of the corrective action is submitted to the QA Department in the agreed upon time frame.

4.13.3 Data Audits

Data audits are focused to assess the level of customer service, SOP compliance, regulatory compliance, accuracy and completeness of test results and reports, documentation, and adherence to established QC criteria, laboratory SOPs, technical policy, and project specific QC criteria.

The QA Department provides feedback and/or corrections and revisions to project reports where necessary. Records of the data audits are kept, and the frequency of data audits is included in the monthly QA report. In performing data audits, it is essential that data be assessed in terms of

differentiating between systematic and isolated errors. Upon noting anomalous data or occurrences in the data audits, the QA Department is responsible for seeking clarification from the appropriate personnel, ascertaining whether the error is systematic or an isolated error, and overseeing correction and/or revision of the project report if necessary. Errors found in client project reports are revised and the revision sent to the client (Section 4.8). The QA Department is also responsible for assisting in the corrective action process where a data audit leads to identification of the need for permanent corrective action.

The frequency of data auditing may also be dependent upon specific clients and regulatory programs. All active laboratory logbooks and QC files are subject to periodic audits/ surveillances by the QA personnel.

4.13.3.1 Data Authenticity Audits

Data authenticity audits shall be performed on 100% of all analysts by the QA department or a designee independent from the operations. Performing data authenticity checks will typically include verifying raw data, evaluating calculation tools and independently reproducing the final results and comparing it to the hardcopy on randomly selected batches of data. The QA Manager will report the percentage of analysts reviewed (for the year) in the monthly QA report and should average about 8% per month.

4.13.3.2 Electronic Data Audits

Electronic data audits are performed on 100% of all organic instruments by the QA department or a designee independent from the operations. This may include Mint Miner® scanning of randomly selected batches of electronic data followed by a chromatography system review. The QA manager will report the percentage of instruments reviewed (for the year) in the monthly QA report and should average about 8% of instruments per month. Electronic data audits include spot-checking of manual integrations by QA personnel in order to determine that the manual integration is appropriate and documented according to Section 5.3.6.1.

4.13.4 Special Audits

Special audits are conducted on an as needed basis, generally as a follow up to specific issues such as client complaints, corrective actions, proficiency testing results, data audits, systems audits, validation comments, or regulatory audits. Special audits are focused on a specific issue, and report format, distribution, and timeframes are designed to address the nature of the issue.

4.14 External Audits

STL is routinely audited by clients and external regulatory authorities – both government and non-government. Whether the audit is scheduled or unannounced, full cooperation with the audit team is provided by the laboratory and administrative staff. STL recommends that the audits be scheduled with the QA Department so that all necessary personnel are available on the day of the audit.

4.15 Management Reviews

4.15.1 QA Reports to Management

A monthly QA report is prepared by the QA Manager and forwarded to the Laboratory Director and Corporate Quality Director. The reports include statistical results that are used to assess the effectiveness of the quality system. The required information for the monthly report is shown in Figure 3.

4.15.2 Quality Systems Management Review

A quality systems management review is performed at least annually by the Laboratory Director and QA Manager *(SOP AQA-Management Review-45)*. This review ensures that the laboratory's quality system is adequate to satisfy the laboratory's policies and practices, government requirements, certification, accreditation, approval requirements, and client expectations. Quality systems management reviews are accomplished through the evaluation and revision of this LQM, monthly quality assurance reporting and goal setting.

Management reviews of specific quality system elements may be performed through continuous improvement activities, monthly QA reports, process changes, SOP revisions, and/or audit reports/responses. Documentation of these reviews are not required unless it is inherent in the review mechanism (e.g., approval signatures on SOP revisions).

4.15.3 Monthly QA Report and Metrics

By the 3rd day of the month, the QA manager prepares a monthly QA report. The report is sent to the Laboratory Director, General Manager and Corporate Quality Director. The report contains a narrative summary and metrics spreadsheet. At a minimum, the report content contains the items listed below (Figure 3). During the course of the year, the Laboratory Director, General Manager or Corporate Quality Director may request that additional information be added to the report.

Figure 3. Monthly QA Report Format

1	Audits
	Internal System Audits External System Audits
2	Revised Reports / Client Feedback
	Revised Reports Client Complaints Client Compliments
3	Certification Changes
	Changes Losses / Revocations
4	Proficiency Testing
	Study participation and scores Combined PT scores Repeat failures
5	SOP Status
	Report the percentage of SOPs that have been revised or reviewed within the last 24 months.
6	Miscellaneous QA and Operational Issues
	Narrative outlining improvements, regulatory

	compliance issues and general concerns.
Appended	Metrics Spreadsheet
	Summarize metrics in template provided by the Corporate Quality Director

5.0 Technical Requirements

5.1 Personnel

5.1.1 General

STL management believes that its highly qualified and professional staff is the single most important aspect in assuring the highest level of data quality and service in the industry. The staff consists of professionals and support personnel that include the following positions:

- General Manager
- Laboratory Director
- Technical Director
- QA Manager
- Human Resource Manager
- Customer Service Manager
- Operations Manager
- QA Specialist
- Health & Safety Coordinator / Waste Management
- Project Manager
- Information Technology Manager
- Network Administrator
- Department Supervisor
- Analyst
- Sample Custodian
- Technician
- Data Reporting Specialist

In order to ensure that employees have sufficient education and experience to perform a particular task, job descriptions are developed for all personnel. Job Descriptions are located on the STL Intranet Site's Human Resources web-page:

http://stlnet.stl-inc.com/Corporate/HR/JobDescriptions/JobDescrip_index.htm.

5.1.2 Training

STL is committed to furthering the professional and technical development of employees at all levels. Selection of qualified candidates for laboratory employment begins with documentation of minimum education, training, and experience prerequisites needed to perform the prescribed task. Minimum education and training requirements for STL employees are outlined in Job Descriptions.

Orientation to the laboratory's policies and procedures, in-house method training, and employee attendance at outside training courses and conferences all contribute toward employee proficiency.

The QA section in conjunction with the Human Resources section are responsible for maintaining documentation of these activities.

Each laboratory section is required to maintain documentation associated with analytical training (e.g., training records, IDOCs, CDOCs, and controlled documents). The QA department maintains documentation of method proficiency (e.g., IDMPs, MDLs, MDLVs, PT Sample Tracking, QC Control Limits/Data). This information is available to managers and staff for planning and evaluation.

The following evidence items are maintained in the employees technical training file for each technical employee:

- An Ethics Agreement signed by each staff member (renewed each year).
- A Confidentiality Agreement signed by each staff member (renewed each year).
- Initial Demonstration of Capability (IDOC)
- The employee has read and understood the latest version of the laboratory's quality documentation.
- The employee has read and understood the latest, approved version of all test methods and/or SOPs for which the employee is responsible.
- Annual evidence of continued DOC that may include successful analysis of a blind sample on the specific test method; a similar test method; an annual DOC; or four successive and acceptable LCSs.
- Documentation of external training courses attended
- All training regarding QA policies and procedures

The Human Resource department maintains documentation and attestation forms on employment status & records; benefit programs; timekeeping/payroll; and employee conduct (e.g., ethics). This information is maintained in the employee's secured personnel file.

Table 8. STL Employee Minimum Training Requirements

Specialty	Experience
General Chemistry and Instrumentation	Six months
Gas Chromatography	One year
Atomic Absorption	One year
Mass Spectrometry	One year
Spectra Interpretation	Two years

Required Training	Time Frame [1]	Employee Type
Environmental Health & Safety	Month 1	All
Ethics	Month 1	All
Data Integrity	Month 1	Technical and PMs
Ethics Refresher	Annually	All
Quality Assurance	Quarter 1	All
Initial Demonstration of Capability	Prior to unsupervised method	Technical

(IDOC)	performance	

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

The quality assurance training includes an overview of regulatory programs and program goals, discussions about data integrity and data misrepresentation and an overview of laboratory quality control procedures and purposes.

When an analyst has not met these training requirements, they can perform a task under the supervision of a qualified analyst, peer reviewer or department supervisor, and are considered an analyst in training. The person supervising an analyst in training is accountable for the quality of the analytical data and must review and approve data and associated corrective actions.

Technical training is accomplished by the Operations Manager, Department Supervisor or a senior analyst to ensure method comprehension. All new personnel are required to demonstrate competency in performing a particular method by successfully completing an Initial Demonstration of Capability. IDOCs are performed by the analysis of four replicate QC samples. Results of successive LCS analyses can be used to fulfill the IDOC requirement. The accuracy and precision, measured as average recovery and standard deviation (using n-1 as the population), of the 4 replicate results are calculated and compared to those in the test method (where available). If the test method does not include accuracy and precision requirements, the results are compared to target criteria set by the laboratory. The laboratory sets the target criteria such that they reflect the DQOs of the specific test method or project. A IDOC Certification Statement is recorded and maintained in the employee's training file. Tabulated results summary and raw data are completed and signed by the analyst and section manager with the proper entries made onto the analysts training record. The data are submitted to the QA department for approval and entry into the master IDOC spreadsheet and filing. Figure 4 shows an example of a IDOC Certification Statement.

On an annual basis, each analyst's method capabilities must be evaluated. The requirement that a CDOC (Continued Demonstration of Capability) be completed for each method currently being analyzed must be presented for approval to QA in the same format as the IDOC discussed above.

Further details of the laboratory's training program are described in the SOP related to Laboratory Personnel Training *(AQA-TRAIN-10)*.

5.1.3 Ethics Policy

Establishing and maintaining a high ethical standard is an important element of a Quality System. In order to ensure that all personnel understand the importance the company places on maintaining high ethical standards at all times; STL has established an Ethics Policy (P-L-006) and an Ethics Agreement (Figure 5). Each employee signs the Ethics Agreement, signifying agreed compliance with its stated purpose. The ethics agreement is required to be re-signed on an annual basis.

Violations of this Ethics Policy will not be tolerated. Employees who violate this policy will be subject to disciplinary actions up to and including termination. Criminal violations may also be referred to the Government for prosecution. In addition, such actions could jeopardize the Company's ability to do work on Government contracts, and for that reason, the Company has a Zero Tolerance approach to such violations.

Ethics is also a major component of STL's quality and data integrity systems. Each employee is trained in ethics within two weeks of hire and quality training within three months of hire. Annually, ethics refresher training will be provided. Employees are trained as to the legal and environmental repercussions that result from data misrepresentation. A data integrity hotline is maintained by STL and administered by the Corporate Quality Director.

Figure 4. Demonstration of Capability Certification Statement

SEVERN TRENT STL

DOC Cert. Statement
Revision 6
October 12, 2005

SEVERN TRENT LABORATORIES - BUFFALO

TRAINING & DEMONSTRATION OF CAPABILITY CERTIFICATION STATEMENT

Employee: _____ Page _____ of _____

Method Number: _____ Date: _____

Parameters or Analytes: _____

Initial Demonstration of Capability: ☐

SOP Number: _____ Revision # _____ Date Read _____

Trained By: _____

Date training began: _____ Date training completed: _____

Continued Demonstration of Capability: ☐

SOP Number: _____ Revision # _____ Date Read _____

I CERTIFY that I have read and understand the SOP identified above. I have also submitted data associated with the demonstration of capability.

_____ _____
Employee Signature Date

We, the undersigned, CERTIFY that:

1. The analyst identified above, using the cited test method(s), which is in use at this facility for the analyses of samples under the National Environmental Laboratory Accreditation Program, have met the Demonstration of Capability.

2. The test method(s) was performed by the analyst(s) identified on this certification.

3. A copy of the test method(s) and the laboratory-specific Sops are available for all personnel on-site.

4. The data associated with the demonstration capability are true, accurate, complete and self-explanatory.

5. All raw data (including a copy of this certification form) necessary to reconstruct and validate these analyses have been retained at this facility, and that the associated information is well organized and available for review by authorized assessors.

John Schove _____ _____
Operations Manager Signature Date

Verl Preston _____ _____
Quality Assurance Manager Signature Date

Figure 5. STL Ethics Agreement

I understand that STL is committed to ensuring the highest standard of quality and integrity of the data and services provided to our clients. I have read the Ethics Policy of the Company.

With regard to the duties I perform and the data I report in connection with my employment at the Company, I agree that:

- I will not intentionally report data values that are not the actual values obtained;
- I will not intentionally report the dates, times, sample or QC identification, or method citations of data analyses that are not the actual dates, times, sample or QC identifications, or method citations;
- I will not intentionally misrepresent another individual's work;
- I will not intentionally report data values that do not meet established quality control criteria as set forth in the Method and/or Standard Operating Procedures, or as defined by Company Policy;
- I agree to inform my Supervisor of any accidental reporting of non-authentic data by me in a timely manner; and I agree to inform my Supervisor of any accidental or intentional reporting of non-authentic data by other employees; and
- If a supervisor or a member of STL management requests me to engage in or perform an activity that I feel is compromising data validity or quality, I will not comply with the request and report this action immediately to a member of senior management, up to and including the President of STL.

As a STL employee, I understand that I have the responsibility to conduct myself with integrity in accordance with the ethical standards described in the Ethics Policy. I will also report any information relating to possible kickbacks or violations of the Procurement Integrity Act, or other questionable conduct in the course of sales or purchasing activities. I will not knowingly participate in any such activity and will report any actual or suspected violation of this policy to management.

The Ethics Policy has been explained to me by my supervisor or at a training session, and I have had the opportunity to ask questions if I did not understand any part of it. I understand that any violation of this policy subjects me to disciplinary action, which can include termination. In addition, I understand that any violation of this policy which relates to work under a government contact or subcontract could also subject me to the potential for prosecution under federal law.

EMPLOYEE SIGNATURE:_____ Date: _____

Supervisor/Trainer:_____ Date:_____

5.2 Facilities

The laboratory is a secure facility with controlled and documented access. Access is controlled by keyless entry access cards, locked doors, and a staffed reception area. All visitors sign in and are escorted by STL personnel while at the facility. The laboratory is locked at all times.

The facility is designed for efficient, automated high-quality operations. The laboratory is equipped with Heating, Ventilation, and Air Conditioning (HVAC) systems appropriate to the needs of environmental testing laboratories. Environmental conditions in the facility, such as hood flow, are routinely monitored and documented.

The facility is equipped with structural safety features. Each employee is familiar with the location, use, and capabilities of general and specialized safety features associated with their workplace. STL also provides and requires the use of protective equipment including safety glasses, protective clothing, gloves, etc..

5.3 Test Methods

Routine analytical services are performed using standard EPA-approved methodologies. In some cases, modification of standard approved methods may be necessary to provide accurate analyses of particularly complex matrices.

5.3.1 Method Selection

Since numerous methods and analytical techniques are available, continued communication between the client and laboratory is imperative to assure the correct methods are utilized. Once client methodology requirements are established, this and other pertinent information is summarized by the Project Manager in the LIMs technical profile. These mechanisms ensure that the proper analytical methods are applied when the samples arrive for log-in. For non-routine analytical services (e.g., special matrices, non-routine compound lists, etc.), the method of choice is selected based on client needs and available technology.

Most of the test methods performed at STL originate from test methods published by a regulatory agency such as the US EPA and other state and federal regulatory agencies. These include, but are not limited to, the following published compendiums of test methods. A listing of methods in which the laboratory is capable of performing is listed in the laboratory's Master Methods Index and Preservation Table *(SOP ASR-Psrv-07).*

Guidelines Establishing Test Procedures for the Analysis of Pollutants Under the Clean Water Act, and Appendix A-C; 40 CFR Part 136, USEPA Office of Water.

Method 1664, Revision A: N-Hexane Extractable Material (HEM; Oil and Grease) and Silica Gel Treated N-Hexane Extractable Material (SGT-HEM) Non-polar Material) by Extraction and Gravimetry, EPA-821-R-98-003, February 1999.

Methods for Chemical Analysis of Water and Wastes, EPA 600 (4-79-020), 1983.

Methods for the Determination of Inorganic Substances in Environmental Samples, EPA-600/R-93/100, August 1993.

Methods for the Determination of Metals in Environmental Samples, EPA/600/4-91/010, June 1991. Supplement I: EPA-600/R-94/111, May 1994.

NIOSH Manual of Analytical Methods, 4th ed., August 1994.

Methods for the Determination of Organic Compounds in Drinking Water, EPA/600/4-88-039, December 1988, Revised July 1991, Supplement I, EPA-600-4-90-020, July 1990, Supplement II, EPA-600/R-92-129, August 1992.

Statement of Work for Inorganics Analysis, ILM04.2, ILM05.2 and ILM05.3 USEPA Contract Laboratory Program Multi-media, Multi-concentration.

Statement of Work for Organics Analysis, OLM04.2 (with OLM04.3 update) and OLC02.1, USEPA Contract Laboratory Program, Multi-media, Multi-concentration.

New York State Department of Environmental Conservation, Analytical Services Protocol, NYSDEC ASP.

Standard Methods for the Examination of Water and Wastewater, 18th/19th/20th edition; Eaton, A.D. Clesceri, L.S. Greenberg, A.E. Eds; American Water Works Association, Water Pollution Control Federation, American Public Health Association: Washington, D.C.

Test Methods for Evaluating Solid Waste Physical/Chemical Methods (SW-846), Third Edition, September 1986, Final Update I, July 1992, Final Update IIA, August 1993, Final Update II, September 1994; Final Update IIB, January 1995; Final Update III, December 1996.

Annual Book of ASTM Standards, American Society for Testing & Materials (ASTM), Philadelphia, PA.

The laboratory reviews updated versions to all the aforementioned references for adaptation based upon capabilities, instrumentation, etc., and establishes an implementation schedule. As such, the laboratory strives to perform only the latest versions of each approved method.

5.3.2 SOPs

STL maintains a Master Index of SOPs *(SOP Master Index)* for both Method and Process SOPs. Method SOPs are maintained to describe a specific test method. Process SOPs are maintained to describe function and processes not related to a analytical testing (e.g., administrative procedures).

Method SOPs contain the following information, but not necessarily in the order listed:

Title Page with Document Name, Document Number, Revision Number, Effective Date, Page Numbers and Total # of Pages, Authorized Signatures, Dates and Proprietary Information Statement (Figure 6).

1. Identification of Test Method
2. Applicable Matrix
3. Scope and Application, including test analytes
4. Summary of the Test Method
5. Reporting Limits
6. Definitions
7. Interferences
8. Safety
9. Equipment and Supplies
10. Reagents and Standards
11. Sample Collection, Preservation and Storage
12. Quality Control
13. Calibration and Standardization
14. Procedure
15. Calculations
16. Method Performance
17. Data Assessment and Acceptance Criteria for Quality Control Measures
18. Corrective Actions for Out-of-Control Data
19. Contingencies for Handling Out-of-Control or Unacceptable Data
20. Waste Management/Pollution Prevention
21. References
22. Tables, Diagrams, Flowcharts and Validation Data
23. Changes From Previous Revision

Process SOPs contain the following information, but not necessarily in the order listed.

Title Page with Document Name, Document Number, Revision Number, Effective Date, Page Numbers and Total # of Pages, Authorized Signatures, Dates and Proprietary Information Statement (Figure 6).

1. Scope
2. Summary
3. Definitions
4. Responsibilities
5. Procedure
6. References
7. Tables, Diagrams, and Flowcharts
8. Changes from Previous Revision

The QA Department is responsible for maintenance of SOPs, archival of SOP historical revisions, maintenance of an SOP Master Index, and records of controlled distribution. SOPs, at a minimum, undergo annual review (12 months). Where an SOP is based on a published method, the laboratory maintains a copy of the reference method.

Figure 6. Proprietary Information Statement

This documentation has been prepared by Severn Trent Laboratories, Inc. (STL) solely for STL's own use and the use of STL's customers in evaluating its qualifications and capabilities in connection with a particular project. The user of this document agrees by its acceptance to return it to STL upon request and not to reproduce, copy, lend, or otherwise disclose its contents, directly or indirectly, and not to use if for any other purpose other than that for which it was specifically provided. The user also agrees that where consultants or other outside parties are involved in the evaluation process, access to these documents shall not be given to said parties unless those parties also specifically agree to these conditions.

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

SOP Interim Change Form

The SOP Interim Change Form is used for implementation, documentation, and authorization of changes to SOPs *(Procedure for Writing, Reviewing and Revising SOPs, AQA-SOP-55)*. Immediate changes in SOPs may be necessary to accommodate improvements; to implement acceptable changes in practices; or to correct potential errors in the existing version. The reason for the change will be identified and a detailed description of the procedure change will be presented. Since this form will become part of the referenced SOP, until such time that the SOP is updated, it must be legible and comprehensible. The Interim Change Form must provide an exact description and identify the affected sections.

Once this form is completed and changes are authorized, it becomes an official part of the SOP for which it revises, and is subject to all document control and records management policies.

5.3.3 Method Validation

Laboratory developed methods are validated and documented according to the procedure described in Section 5.3.5.

5.3.4 Method Verification

Method verification is required when a validated standard test method or a method modification is implemented. The level of activity required for method verification is dependent on the type of method being implemented, or on the level of method modification and its affect on a method's robustness. Method modification often takes advantage of a method's robustness, or the ability to make minor changes in a method without affecting the method's outcome.

It is the responsibility of the Operations Manager to present to the QA Manager all applicable method validation studies for review and approval. The documented approval by the Operations Manager, Department Supervisor and QA Manager must be applied to all applicable validation records before the method is released for use. Method verification may require some, but not all, of the activities described in Section 5.3.5.

5.3.5 Method Validation and Verification Activities

Before analyzing samples by a particular method, method validation and/or method verification must occur. A complete validation of the method is required for laboratory developed methods. While method validation can take various courses, the following activities can be required as part of method validation. Method validation records are designated QC records and are archived accordingly.

Determination of Method Selectivity
Method selectivity is demonstrated for the analyte(s) in the specific matrix or matrices. In some cases, to achieve the required selectivity for an analyte, a confirmation analysis is required as part of the method.

Determination of Method Sensitivity
Sensitivity can be both estimated and demonstrated. Whether a study is required to estimate sensitivity depends on the level of method development required when applying a particular measurement system to a specific set of samples. Where estimations and/or demonstrations of sensitivity are required by regulation or client agreement, such as the procedure in 40 CFR Part 136 Appendix B, under the Clean Water Act, these shall be followed. The laboratory determines MDLs are described in Section 4.4.3.6 and the corporate procedure for *MDL Policy, (S-Q-003).*

Relationship of Limit of Detection (LOD) to the Quantitation Limit (QL)
An important characteristic of expression of sensitivity is the difference in the LOD and the QL. The LOD is the minimum level at which the presence of an analyte can be reliably concluded. The QL is the minimum level at which both the presence of an analyte and its concentration can be reliably determined. For most instrumental measurement systems, there is a region where semi-quantitative data is generated around the LOD (both above and below the estimated MDL or LOD) and below the QL. In this region, detection of an analyte may be confirmed but quantification of the analyte is unreliable within the accuracy and precision guidelines of the measurement system.

When an analyte is detected below the QL, and the presence of the analyte is confirmed by meeting the qualitative identification criteria for the analyte, the analyte can be reliably reported, but the amount of the analyte can only be estimated. If data are to be reported in this region, they must be done so with a qualification that denotes the semi-quantitative nature of the result.

Determination of Interferences
A determination that the method is free from interferences in a blank matrix is performed.

Determination of Range
Where appropriate, a determination of the applicable range of the method may be performed. In most cases, range is determined and demonstrated by comparison of the response of an analyte in a curve to established or targeted criteria. The curve is used to establish the range of quantitation and the lower and upper values of the curve represent upper and lower quantitation limits. Curves are not limited to linear relationships.

Demonstration of Capability
DOCs are performed prior to method performance.

Determination of Accuracy and Precision
Accuracy and precision studies are generally performed using replicate analyses, with a resulting percent recovery and measure of reproducibility (standard deviation, relative standard deviation) calculated and measured against a set of target criteria.

Documentation of Method
The method is formally documented in an SOP. If the method is a minor modification of a standard laboratory method that is already documented in an SOP, an SOP Appendix describing the specific differences in the new method is acceptable in place of a separate SOP.

Continued Demonstration of Method Performance
Continued demonstration of Method Performance is addressed in the SOP. Continued demonstration of method performance is generally accomplished by batch specific QC samples such as LCS and Method Blanks.

5.3.6 Data Reduction and Review

Analytical data are entered/downloaded directly into LIMS or recorded on pre-formatted bench sheets that are paginated and bound into laboratory logbooks. These logbooks are issued and controlled by the laboratory's QA Section. A unique document control code is assigned to each book to assure that chronological record keeping is maintained. Analytical data may also be electronically stored as a secure .pdf file.

Analytical data are referenced to a unique sample identification number for internal tracking and reporting. Both LIMS entries and logbook pages contain the following information, as applicable: analytical method, analyst, date, associated sample numbers, standard concentrations, instrument settings, and raw data. Entries are in chronological order and maintained so as to enable reconstruction of the analytical sequence.

The analyst is responsible for entering / recording all appropriate information, and for signing and dating all logbook entries daily. All entries and logbook pages are reviewed for completeness by a supervisor, peer reviewer or the analyst themselves. Data review checklists document the analytical review of the LIMS entries, logbook and associated QC indicators. Copies of instrument outputs

(chromatograms, mass spectra, etc.) are maintained on file or electronically with the analyst's signature/initials and date.

5.3.6.1 Data Reduction

The complexity of the data reduction depends on the analytical method and the number of discrete operations involved (e.g., extractions, dilutions, instrument readings and concentrations). The analyst calculates the final results from the raw data or uses appropriate computer programs to assist in the calculation of final reportable values.

For manual data entry, e.g., Wet Chemistry, the data are reduced by the analyst and updated to the LIMs. Both the data entry and raw data are then verified by the department supervisor or alternate analyst. The spreadsheets, or any other type of applicable documents, are signed by both the analyst and alternate reviewer to confirm the accuracy of the data and manual entry(s).

Manual integration of peaks will be documented and reviewed and the raw data will be flagged in accordance with the STL Corporate SOP entitled *Acceptable Manual Integration Practices (S-Q-004).*

Copies of all raw data and the calculations used to generate the final results, such as bound logbooks, are retained on file for a minimum of 5 years or as otherwise requested by the client/project.

Calculations and data reduction steps for various methods are summarized in the respective analytical SOPs or program requirements.

5.3.6.2 Data Review

All data, regardless of regulatory program or level of reporting, are subject to a thorough review process. The individual analyst continually reviews the quality of the data through calibration checks, quality control sample results and performance evaluation samples. Data review is initiated by the analyst during, immediately following, and after the completed analysis.

All levels of the review are documented on Data Review Checklists that are specific to each laboratory section *(Technical Data Review; AGP-DataReview-21).*

Primary Review

The primary review is often referred to as a "bench-level" review. In most cases, the analyst who generates the data (e.g., logs in, prepares and/or analyzes the samples) is the primary reviewer. In some cases, an analyst may be reducing data for samples run by an auto-sampler set up by a different analyst. In this case, the identity of both the analyst and the primary reviewer is identified in the raw data.

One of the most important aspects of primary review is to make sure that the test instructions are clear, and that all project specific requirements have been understood and followed.

Once an analysis is complete, the primary reviewer ensures, where applicable, that:

- Sample preparation information is complete, accurate, and documented.
- Calculations have been performed correctly.
- Quantitation has been performed accurately.
- Qualitative identifications are accurate.

- Manual integrations are appropriate.
- Data flags to indicate manual integrations are recorded.
- Manual integrations are authorized by a date and signature or initials of primary analyst.
- Client specific requirements have been followed.
- Method and process SOPs have been followed.
- Method QC criteria have been met.
- QC samples are within established limits.
- Dilution factors are correctly recorded and applied.
- Non-conformances and/or anomalous data have been properly documented and appropriately communicated.
- COC procedures have been followed.
- Primary review is documented by date and initials/signature of primary analyst.
- All unused portions of hardbound logbooks are 'Z'ed out; corrections are made with a single line drawn through the error and are dated and initialed

Any anomalous results and/or non-conformances noted during the Primary Review are documented on the Data Review Checklist and on a Job Exception ; and are communicated to the Supervisor and the Project Manager for resolution. Resolution can require sample reanalysis, or it may require that data be reported with a qualification. Non-conformances are documented per Section 4.9. Case narrative comments are generated by the primary reviewer for any unresolved anomalous results or non-conformances.

Secondary Review

The secondary review is also a complete technical review of a data and is performed by the Supervisor, peer analyst or data specialist. The secondary review is documented on the same Data Review Checklist as the primary review.

The following items are reviewed:

- Qualitative Identification
- Quantitative Accuracy
- Calibration
- QC Samples
- Method QC Criteria
- Adherence to method and process SOPs
- Accuracy of Final Client Reporting Forms
- Manual Integrations – Minimal requirement is to spot-check raw data files for manual integration, as verified by date and initials or signature (hardcopy or electronic) of secondary data reviewer. Some regulatory programs require 100% secondary review of manual integrations.
- Completeness
- Special Requirements/Instructions
- Review and approve case narrative comments

If problems are found during the secondary review, which are documented on the data review checklist, the reviewer must work with the appropriate personnel to resolve them. If changes are made to the data, such as alternate qualitative identifications, identifications of additional target analytes, re-quantitation, or re-integration, the secondary reviewer must contact the laboratory analyst and/or primary reviewer of the data so that the primary analyst and/or reviewer is aware of the appropriate reporting procedures.

Completeness Review
The completeness review includes the review of the case narrative which outlines anomalous data and non-compliances using project narrative notes, Job Exceptions and DQRs generated during the primary and secondary review. The completeness review addresses the following items:

- Is the project report complete?
- Does the data meet with the client's expectations?
- Were the data quality objectives of the project met?

Are QC outages and/or non-conformances approved and appropriately explained in the narrative notes?

The laboratory Department Supervisor, Data Reporting personnel and the Project Manager contribute to the completeness review.

5.3.7 Data Integrity and Security

This section details those procedures that are relevant to computer systems that collect, analyze, and process raw instrumental data, and those that manage and report data.

Security and Traceability
Access to the laboratory's LIMS system that collects, analyzes, and processes raw instrumental data, and those that manage and report data is both controlled and recorded. System users are granted access levels that are commensurate with their training and responsibilities.

Control of the system is accomplished through limitation of access to the system by users with the education, training and experience to perform the task knowledgeably and accurately. System users are granted privileges that are commensurate with their experience and responsibilities.

Computer access is tracked by using unique login names and passwords for all employees that have access to the computer system. Entries and changes are documented with the identity of the individual making the entry, and the time and date. Where a computer system is processing raw instrumental data, the instrument identification number as described in Section 5.4.1 is recorded. The system has the capability of maintaining audit trails to track entries and changes to the data. This function is activated on any computer system that has that capability (e.g., Enviroquant, Chemstation, TotalChrom).

Verification
All the LIMS software programs have been verified prior to use and prior to the implementation of any version upgrades. Verification involves assessing whether the computer system accurately performs its intended function. Verification generally is accomplished by comparing the output of the program with the output of the raw data manually processed, or processed by the software being replaced. The verification of LIMS software programs are conducted by the Information Technology Manager with the assistance of the QA Manager, Operations Manager and the Department Supervisors. The IT Manager documents the approval of the program verifications. All records of the verification are retained as QC records.

Validation

Software validation involves documentation of the verification of final calculated results. Software validation is performed by the QA manager on all in house programs. Records of validation include original specifications, identity of code, printout of code, software name, software version, name of individual writing the code, comparison of program output with specifications, and verification records as specified above. Records of validation are retained as QC records.

The QA manager must retain documentation of the validation process as defined above. The designated LIMS methods administrator at the laboratory has the responsibility to validate any LIMS methods, calculations or criteria codes prior to use for sample analysis.

Auditing
STLs LIMS System Managers continually review the control, security, and tracking of IT systems and software.

Version Control
The laboratory maintains copies of outdated versions of software and associated manuals for all software in use at the laboratory for a period of 5 years from its retirement date. The associated hardware, required to operate the software, is also retained for the same time period.

5.4 Equipment

5.4.1 Equipment Operation

STL is committed to routinely updating and automating instrumentation. The laboratory maintains state of the art instrumentation to perform the analyses within the QC specifications of the test methods. The laboratory maintains an Equipment List *(STLBuffEquipList)* that documents the following information:

- Identity
- Date In Service
- Manufacturer's Name, Model Number, Serial Number
- Current Location

All equipment is subject to rigorous checks upon its receipt, upgrade, or modification to establish that the equipment meets with the selectivity, accuracy, and precision required by the test method for which it is to be used. All manufacturer's operations and maintenance manuals are kept up to date and accessible for the use of the equipment operator. Documentation of equipment usage is maintained using analytical run and maintenance logbooks.

5.4.2 Equipment Maintenance

STL employs a system of preventative maintenance in order to ensure system up time, minimize corrective maintenance costs and ensure data validity. All routine maintenance is performed as recommended by the manufacturer and may be performed by an analyst, instrument specialist or outside technician. Maintenance logbooks are kept on all major pieces of equipment in which both routine and non-routine maintenance is recorded.

Any item of equipment or instrumentation that has been subjected to overloading or mishandling, provides suspected results, has been shown by verification or otherwise to be defective, is new or

not been used for an extended period of time, is taken out of service and tagged as "OUT-OF-SERVICE", *(AGP-OutofService-65)*

Any instrumentation that is brought back on-line must have MDLs and DOCs performed and have acceptance within prescribed criteria; or calibrated by a certified agency (e.g., balances or Class S weights) and tagged as being within calibration specifications; and proven to provide consistent measurements (e.g., refrigerators, eppendorf pipettes, ovens).

The return to analytical control following instrument repair is documented in the maintenance logbook. Notation of the date and maintenance activity is recorded each time service procedures are performed. Maintenance logbooks are retained as QA records.

Maintenance contracts are held on specific pieces of equipment where outside service is efficient, cost-effective, and necessary for effective operation of the laboratory. Table 9 lists STL's major equipment and the suggested maintenance procedures.

Table 9. Major Equipment Maintenance

Instrument	Procedure	Frequency
Leeman Mercury Analyzer	Check tubing for wear	Daily
	Fill rinse tank with 10% HCl	Daily
	Change dryer tube	As Needed
	Fill reductant bottle with 10% Stannous Chloride	Daily
ICP & ICP/MS	Check pump tubing	Daily
	Check liquid argon supply	Daily
	Check fluid level in waste container	Daily
	Check re-circulator levels	Monthly
	Clean or replace filters	As required
	Check torch	Daily
	Check sample spray chamber for debris	Monthly
	Clean and align nebulizer	Monthly
	Change pump oil	Monthly
	Change Cones	As required
	Change printer cartridge	As required
	Replace pump tubing	As required
UV-Vis Spectrophotometer	Clean ambient flow cell	As required
	Precision check/alignment of flow cell	As required
	Wavelength verification check	Annually
Auto Analyzers	Clean sampler	Daily
	Check all tubing	Daily
	Clean inside of colorimeter	Daily
	Clean pump well and pump rollers	Quarterly
	Clean wash fluid receptacle	Weekly
	Oil rollers/chains/side rails	Weekly
	Clean optics and cells	Quarterly

Table 9. Major Equipment Maintenance

Instrument	Procedure	Frequency
Agilent GC/MS	Pump oil-level check	Monthly
	Pump oil changing	Annually
	Analyzer bake-out	As required
	Analyzer cleaning	As required
	Resolution adjustment	As required
	COMPUTER SYSTEM AND PRINTER:	
	Air filter cleaning	As required
	Change data system air filter	As required
	Printer head carriage lubrication	As required
	Paper sprocket cleaning	As required
	Drive belt lubrication	As required
Gas Chromatograph	Compare standard response to previous day or since last initial calibration	Daily
	Check carrier gas flow rate in column	Daily via use of known compound retention
	Check temp. of detector, inlet, column oven	Daily
	Septum replacement	As required
	Glass wool replacement	As required
	Check system for gas leaks with SNOOP	W/cylinder change as required
	Check for loose/frayed power wires and insulation	As Required
	Bake injector/column	As Required
	Change/remove sections of guard column	As Required
	Replace connectors/liners	As Required
	Change/replace column(s)	As Required
Electron Capture Detector (ECD)	Detector wipe test (Ni-63)	Semi-annually
	Detector cleaning	As required
Flame Ionization Detector (FID)	Detector cleaning	As required
Photoionization Detector (PID)	Change O-rings	As required
	Clean lamp window	As required
HPLC	Change guard columns	As required
	Change lamps	As required
	Change pump seals	Semi-annually or as required
	Replace tubing	As required
	Change fuses in power supply	As required
	Filter all samples and solvents	Daily
	Change autosampler rotor/stator	As required
Balances	Class "S" traceable weight check	Daily, when used
	Clean pan and check if level	Daily
	Field service	At least Annually

Table 9. Major Equipment Maintenance

Instrument	Procedure	Frequency
Conductivity Meter	0.01 M KCl calibration Conductivity cell cleaning	Weekly As required
Turbidimeter	Check light bulb	Daily, when used
Deionized/Distilled Water	Check conductivity Check deionizer light Monitor for VOA's System cleaning Replace cartridge & large mixed bed resins	Daily Daily Daily As required As required
Drying Ovens	Temperature monitoring Temperature adjustments	Daily As required
Refrigerators/ Freezers	Temperature monitoring Temperature adjustment Defrosting/cleaning	Daily As required As required
Vacuum Pumps/ Air Compressor	Drained Belts checked Lubricated	Weekly Monthly Semi-annually
pH/Specific Ion Meter	Calibration/check slope Clean electrode	Weekly As required
BOD Incubator	Temperature monitoring Coil and incubator cleaning	Daily Monthly
Centrifuge	Check brushes and bearings	Every 6 months or as needed
Water baths	Temperature monitoring Water replaced	Daily Monthly or as needed

5.4.3 Equipment Verification and Calibration

All equipment is calibrated prior to use (Initial Calibration) to establish its ability to meet the QC guidelines contained in the test method for which the instrumentation is to be used. All sample measurements are made within the calibrated range of the instrument and in compliance with method requirements. The calibration data, which includes instrument conditions and standard concentrations, is documented in pre-formatted instrument injection logs or within LIMS itself. The preparation of all reference materials used for calibration is documented in standards preparation logbooks in accordance with *SOP AGP-STD-14 (Standards Traceability and Preparation Logbooks).*

Once an instrument is calibrated, ongoing instrument calibration is demonstrated (Continuing Calibration) at the appropriate frequency as defined in the test method. Refer to the STL Corporate Policy *Selection of Calibration Points (P-T-001),* for guidance on using calibration data. Any instrument that is deemed to be malfunctioning is clearly marked and taken out of service. When the instrument is brought back into control, acceptable performance is documented.

5.4.3.1 Instrument Calibration

Specific instrument calibration procedures for various instruments are summarized further in this section, and detailed in the respective analytical methods. Typically, more than one analytical method is available for an analysis. These various methods and other program requirements (e.g., U.S. EPA CLP, AFCEE, USACE, QAPPs, contracts, etc.) may specify different calibration requirements. Therefore, calibration details as specified in the respective laboratory SOPs, Technical Profiles, QAPP, program requirements, and contracts supersede the general instrument calibration procedures are described further in Table 10. Complete details are provided in each method SOP.

Table 10. Minimum Instrument Calibration Procedures

Technique	Activity	Minimum Requirements
Metals (ICAP)	Initial Calibration	Following a period of time sufficient to warm up the instrument, the ICP is calibrated prior to each analytical run or minimally every 24 hours. Calibration standards are prepared from reliable reference materials and contain all metals for which analyses are being conducted. Working calibration standards are prepared fresh daily. Prior to an analytical run, the instrument is calibrated using appropriate standards. An Initial Calibration Verification (ICV) standard is analyzed immediately after standardization, followed by an Initial Calibration Blank (ICB). The ICV is from a source other than that used for initial calibration and the ICB must be free of target analytes at and above the value to be reported or appropriate corrective action must be taken. ICP Interference Check Samples (ICSAB) are analyzed at the frequency described in each method SOP.
	Continuing Calibration	The initial calibration is verified during the analysis sequence by analysis of a Continuing Calibration Verification (CCV) standard and a Continuing Calibration Blank (CCB). The response of the CCV must be within the SOP-specified criteria (e.g., ± 10% recovery of the true value). The CCB must be free of target analytes at or above the value to be reported or appropriate corrective action must be taken. If any ICVs/CCVs or blanks exceed their acceptance criteria, appropriate corrective action must be taken.

Table 10. Minimum Instrument Calibration Procedures

Technique	Activity	Minimum Requirements
Inorganic Colorimetric Methods	Initial Calibration	A full initial standard calibration curve will be prepared for all colorimetric analyses. Working standards to define this curve will include a minimum of five (5) concentrations which cover the anticipated range of measurement, plus a calibration blank. At least one of the calibration standards will be at a concentration which will enable verification of instrument response near the reporting limit as defined in Section 8.6 or a level suitable for meeting specific program requirements. The requirement for an acceptable initial calibration is described in the analytical SOP. If the criteria are not met, appropriate corrective action must be taken. Calibration data, e.g., correlation coefficient, is entered into the laboratory notebook, or associated instrument printouts, and retained with the sample data. If the initial curve is not analyzed that day, a daily calibration verification must be analyzed. This daily calibration will at a minimum consist of a blank and a mid-range standard. Results must be within SOP-specified criteria. If not, reanalysis of the standards may be done once to verify the readings; otherwise, a new curve will be developed.
Inorganic Colorimetric Methods (cont'd.)		For procedures that require pretreatment steps, a minimum of one standard shall be prepared with the pretreatment. If the pre-treated standard is within SOP-specified criteria, the curve will be used. If the pre-treated sample is not within the criteria, the reason will be determined. If it is determined that the difference between the curves is inherent in the procedure, the curve will be based on the standards prepared and carried through the pretreatment. An ICV will be analyzed immediately after the standardization, followed by an ICB. The ICV must be from a source other than that used for initial calibration. The ICV must be within SOP-specified criteria and the ICB must be free of target analytes or appropriate corrective action must be taken.
	Continuing Calibration	The initial calibration is verified during the analysis sequence by analysis of a CCB and a CCV. If any ICVs/CCVs or blanks exceed their acceptance criteria, analysis is terminated, and the instrument is recalibrated. All samples since the last valid calibration verification are evaluated for acceptability or reanalyzed. (If the CCV is elevated and the samples are ND, the data are deemed acceptable.)
Ion Chromatography	Initial Calibration	The ion chromatograph will be calibrated every three months or sooner if calibration verification can not be achieved. Calibration standards will be prepared from appropriate reference materials and will include a blank and a minimum of three concentrations to cover the anticipated range of measurements. At least one of the calibration standards will be at a concentration which will enable verification of instrument response near the reporting limit. If SOP-specified calibration criteria cannot be achieved, appropriate corrective action must be taken. Calibration data, e.g., correlation coefficient, will be archived with sample raw data.

Table 10. Minimum Instrument Calibration Procedures

Technique	Activity	Minimum Requirements
	Continuing Calibration Verification	A calibration verification standard and blank will be analyzed each day prior to sample analysis, throughout the sequence at a frequency of 10% and at the end of the analysis shift. The response calculated as a percent recovery of the standard must meet SOP or program-specific criteria. The response of the blank must be less than the concentration to be reported for samples analyzed.
GC/MS	All GC/MS instrumentation is calibrated to set specifications prior to sample analysis. These specifications vary depending on the requirements of the analytical program and the designated analytical method.	
	Tuning and Mass Calibration	Mass spectrometers are calibrated with perfluorotributylamine (FC-43) or perfluorophenanthrene (FC- 5311) as required to ensure correct mass assignment. In addition, at the beginning of the daily work shift, the GC/MS system must be tuned with decafluorotriphenylphosphine (DFTPP) for semivolatiles analysis and 4-bromofluorobenzene (BFB) for volatiles analysis, and calibrated to target compounds. Autotunes are run with PFTBA(perfluorotributylamine),which is encased in a vial inside the mass spec. DFTPP and BFB are run daily (12 hours where appropriate) for SVOA and VOA respectively.
GC/MS (cont'd.)	Tuning and Mass Calibration	Laboratory work using SW-846 protocols, defines the work shift as a 12-hour period initiated by the injection of DFTPP or BFB. For drinking water programs (500 series methods), a 12-hour work shift is specified in the method for calibration frequency. For wastewater programs (600 series methods), the tune expires when the day's analytical sequence is complete; however, no time limit is given for the length of the daily GC/MS work shift; therefore a maximum of 24 hours for 624 and 625 is used. Ion abundances will be within the windows dictated by the specific program requirements.

Table 10. Minimum Instrument Calibration Procedures

Technique	Activity	Minimum Requirements
	Initial Calibration	After an instrument has been tuned, initial calibration curves (generally 3-5 points) are generated for the compounds of interest. The low level standard must be at a concentration which will enable verification of instrument response near the reporting limit or at a concentration acceptable to meet program requirements. The other standards must extend through the linear working range of the detector. The parameters requiring quantitation must meet SOP or program-specified criteria prior to initiation of sample analysis. Any sample extracts containing parameters of interest which exceed the concentration of the high level standard, must be diluted to bring the parameters within the range of the standards. Instrument response to these target compounds are evaluated against SOP-specified criteria. Linearity is verified by evaluating the response factors (RF) for the initial calibration standards against SOP-specified criteria. Once an acceptable calibration is obtained, samples may be analyzed up until the expiration of the tune. At that time, the instrument must be re-tuned prior to further analysis. After acceptable tuning, a continuing calibration standard may be analyzed in lieu of a full multi-point calibration if the SOP-specified criteria are met. The majority of compounds analyzed for GC/MS comprise EPA's Target Compound List (TCL) or Priority Pollutant List (PPL). For add-on compounds not on the current TCL or PPL, initial calibration may be performed using a single point calibration of the additional compound(s), unless prior arrangements are made for a full three-to-five point calibration. Calibration data, to include linearity verification, will be maintained in the laboratory's records of instrument calibrations. 3 to 5 point curves for all GCMS analytes, special list. The only case where single point standards are used is for quantitation of PCBs other than Arochlor 1016 or 1260.
	Continuing Calibration	During each operating shift, a single calibration standard may be analyzed to verify that the instrument responses are still within the initial calibration determinations, as defined in the specific SOPs. If criteria cannot be met, appropriate corrective action must be taken.
GC and HPLC	Gas chromatographs and high performance liquid chromatographs will be calibrated prior to use as described in analytical SOP or program requirements. Calibration standard mixtures will be prepared from appropriate reference materials and will contain analytes appropriate for the method of analysis or program requirements	
GC and HPLC (cont'd.)	Initial Calibration	Initial calibration will include three or more calibration standards covering the anticipated range of measurement. The low level standard must be at a concentration which will enable verification of instrument response near the reporting limit or at a concentration acceptable to meet program requirements. The other standards must extend through the linear working range of the detector. The parameters requiring quantitation must meet SOP or program-specified criteria prior to initiation of sample analysis. Any sample extracts containing parameters of interest which exceed the concentration of the high level standard, must be diluted to bring the parameters within the range of the standards.

Table 10. Minimum Instrument Calibration Procedures

Technique	Activity	Minimum Requirements
	Continuing Calibration	The response of the instrument will be verified for each analysis sequence by evaluation of a daily calibration verification standard at a mid-range concentration. In order to demonstrate that the initial calibration curve is still valid, the calibration check standard must be within SOP or program-specified acceptance criteria for the compounds of interest or the instrument must be recalibrated. For multi-analyte methods, this check standard may contain a representative number of target analytes rather than the full list of target compounds. Optionally, initial calibration (e.g., the full range of concentration levels) can be performed at the beginning of the analysis sequence. Within the analysis sequence, instrument drift will be monitored by analysis of a mid-range calibration standard every ten samples or 12 hour sequence (depending on the method protocol), including external QC. If the SOP or program-specified calibration criteria are not met for the compounds of interest, appropriate corrective action must be taken.

5.5 Measurement Traceability

5.5.1 General

Traceability of measurements is assured using a system of documentation, calibration, and analysis of reference standards. Laboratory equipment that are peripheral to analysis and whose calibration is not necessarily documented in a test method analysis or by analysis of a reference standard is subject to ongoing certifications of accuracy.

At a minimum, these include procedures for checking specifications for balances, thermometers, temperature, De-ionized (DI) and Reverse Osmosis (RO) water systems, automatic/eppendorf pipettes and other volumetric measuring devices. Wherever possible, subsidiary or peripheral equipment is checked against standard equipment or standards that are traceable to national or international standards [with the exception of class A glassware (including glass microliter syringes that have a certificate of accuracy)].

An external certified service engineer services laboratory balances on an annual basis. This service is documented on each balance with a signed and dated certification sticker. Balances are calibrated on each day of use. All thermometers and temperature monitoring devices are calibrated annually against a traceable reference thermometer. Temperature readings of ovens, refrigerators, and incubators are checked on each day of use

Laboratory DI and Elga water systems have documented preventative maintenance schedules and the conductivity of the water is recorded on each day of use

Procedures for maintenance and record keeping of support equipment are defined in SOP *Support Equipment: Maintenance, Record Keeping and Corrective Actions of Analytical Balances, Temperature Control Devices and Reagent Water (AGP-SupportEquip-02)*

5.5.2 Reference Standards

The receipt of all reference standards is documented in the departmental Chemical History Logbook. Standards are obtained from commercial vendors and sources may vary depending upon the availability of mixes and solutions from vendors. Each production unit is responsible to ensure, when available, that all standards are traceable to EPA, NIST or A2LA and are accompanied by a Certificate of Analysis that documents the standard purity. If a standard cannot be purchased from a vendor that supplies a Certificate of Analysis, the purity of the standard is documented by analysis.

The receipt of each dry chemical, purchased stock solution or reference material to be used as a standard is assigned a unique ID number. The chemical name, manufacturer, lot number, date received, expiration date, date opened and initials of the analyst who opened the chemical are documented. The expiration dates for ampulated solutions shall not exceed the manufacturer's expiration date. Expiration dates for laboratory-prepared stock and diluted standards shall be no later than the expiration date of the stock solution or material or the date calculated from the holding time allowed by the applicable analytical method, whichever comes first. Expiration dates for pure chemicals shall be established by the laboratory and be based on chemical stability, possibility of contamination, and environmental and storage conditions. Expired standard materials shall be either revalidated prior to use or discarded. Revalidation may be performed through assignment of a true value and error window statistically derived from replicate analyses of the material as compared to an unexpired standard. The laboratory labels all standard and QC materials with expiration dates.

The preparation of all daughter solutions, whether a single or multiple-component stock, intermediate, or working standard solution, is documented in a standard solution preparation logbook. This documentation references the Standard ID of the respective parent solution(s) used in its preparation, providing a solid trail back to the solution or chemical received from the vendor. These records include the standard name, final volume, matrix, final concentration, analyst initials, prep date and expiration date. A daughter solution should not have an expiration date which post-dates any of the parent solutions used in its preparation.

References standards are labeled with a unique Standard Identification Number, date received, and the expiration date. All documentation received with the reference standard or documentation of standard purity is retained as a QC record and references the Standard Identification Number. All efforts are made to purchase standards that are $\geq$ 97.0% purity. If this is not possible, the purity is used in performing standards calculations.

The accuracy of calibration standards is checked by comparison with a standard from a second source. In cases where a second standard manufacturer is not available, a different lot is acceptable for use as a second source. The appropriate QC criteria for specific standards are defined in laboratory SOPs. In most cases, the analysis of an ICV or LCS is used as the second source confirmation.

Storage conditions, such as shelf life, ambient or chilled, controlled or restricted access, wet or desiccated, etc., are in conformance with the specifications set in the associated method, the program requirements, or the manufacturer's recommendation, as appropriate.

5.5.3 Reagents

Reagents are, in general, required to be analytical reagent grade unless otherwise specified in method SOPs. Reagents must be, at a minimum, the purity required in the test method. The date

of reagent receipt and the expiration date as well as the date of reagent preparation (where applicable) are documented in the standards preparation logbooks.

5.6 Sampling

Sample representativeness and integrity are the foundations upon which meaningful analytical results rely. Where documented and approved SAPs and/or QAPPs are in place, they must be made available to the laboratory before sample receipt, and approved by laboratory management before sample receipt.

5.7 Sample Handling, Transport, and Storage

5.7.1 General

COC can be established either when bottles are sent to the field, or at the time of sampling. STL can provide all of the necessary coolers, reagent water, sample containers, preservatives, sample labels, custody seals, COC forms, ice, and packing materials required to properly preserve, pack, and ship samples to the laboratory. Complete details for sample container preparation are contained within *Sample Container Preparation and Shipment SOP (ASR-Bottle-03)*. A summary of sample receipt is as follows with complete details available within the *Receipt of Analytical Samples SOP (ASR-Receipt-05).*

Samples are received at the laboratory by the designated sample custodians and a unique LIMS job number is assigned. The following information is recorded for each sample shipment:

- Client/Project Name.
- Date and Time of Laboratory Receipt.
- Laboratory Job Number
- Signature or initials of the personnel receiving the cooler and making the entries.

Upon inspection of the cooler and custody seals, the sample custodian opens and inspects the contents of the cooler, and records the cooler temperature. If the cooler arrival temperature exceeds the required or method specified temperature range by $\pm 2^{\circ}C$ (for samples with a temperature requirement of $4^{\circ}C$, a cooler temperature of just above the water freezing temperature to $6^{\circ}C$ is acceptable); sample receipt is considered "compromised" and the procedure described in Section 4.7.1 is followed. All documents are immediately inspected to assure agreement between the test samples received and the COC.

Any non-conformance, irregularity, or compromised sample receipt as described in Section 4.7.1 is documented in an Analytical Receipt Resolution Form (ARRF) and brought to the immediate attention of the Project Manager for resolution with the client. The COC, shipping documents, documentation of any non-conformance, irregularity, or compromised sample receipt, record of client contact, and resulting instructions become part of the permanent project record.

Samples that are being tested at another STL facility or by an external subcontractor are repackaged, iced, and sent out under COC.

Following sample labeling as described in Section 5.7.2, the sample is placed in storage. Refrigerated storage coolers are maintained at $4 \pm 2^{\circ}C$ and the temperatures are monitored daily.

All samples are stored according to the requirements outlined in the test method, and in a manner such that they are not subject to cross contamination or contamination from their environment.

Access to the laboratory is restricted to laboratory personnel or escorted guests as described in Section 5.2. Therefore, once sample possession is relinquished to the laboratory, the sample is in a designated secure area (e.g., the laboratory facility) accessible only to authorized personnel. Locked storage coolers are available for protocol that require internal COC procedures.

5.7.2 Sample Identification and Traceability

The sample custodian organizes the sample containers, COCs, and all pertinent information associated with the samples. The sample identity is verified against all associated sample information. Any inconsistencies are documented via an ARRF and forwarded to the Project Manager for resolution with the client prior to identifying the sample(s) into LIMS.

Each sample container is assigned a unique Sample Identification Number that is cross-referenced to the client identification number such that traceability of test samples is unambiguous and documented. Each sample container is affixed with a durable sample identification label.

All unused portions of samples, including empty sample containers, are returned to the secure sample control area, unless it has been documented that the container was disposed.

5.7.3 Sub-Sampling

Taking a representative sub-sample from a container containing a soil or solid matrix is necessary to ensure that the analytical results are representative of the sample collected in the field. The size of the sample container, the quantity of sample fitted within the container, and the homogeneity of the sample need consideration when sub-sampling for sample preparation.

After thoroughly mixing the sample within the sample container or transfer to a suitable plastic bag, a sub-sample from various quadrants and depths of the sample are taken to acquire the required sample weight. Any non-homogenous looking material is avoided and noted as such within the sample preparation record.

5.7.4 Sample Preparation

Sample preparation procedures vary for each matrix and analytical method are as referenced in the laboratory SOPs.

5.7.5 Sample Disposal

Samples are retained in STL storage facilities for 30 days after the project report is sent unless prior written arrangements have been made with the client. Samples may be held longer or returned to the client per written request. Unused portions of samples are disposed of in accordance with federal, state and local regulations. The laboratory removes or defaces sample labels prior to disposal unless this is accomplished through the disposal method (e.g., samples are incinerated). Complete details on the disposal of samples, digestates, and extracts is available within the *Sample Disposal SOP (ASR-DISP-33) and Hazardous Waste Management SOP (AWM-HazMg-01).*

5.8 Assuring the Quality of Test Results

5.8.1 Proficiency Testing

The laboratory analyzes Proficiency Test (PT) samples as required for accreditation and as outlined in NELAC. The laboratory participates in the PT program semi-annually for each PT field of testing for which it is accredited, according to the NELAC PT field of testing published guidelines. This includes drinking water, wastewater and solid/soil matrices.

The laboratory also participates in various client PT programs, when submitted.

PT samples are handled and tested in the same manner (procedural, equipment, staff) as environmental samples. Results of PT samples are distributed to the laboratory line management for review and action, if required. Any required response to deficiencies are submitted to the QA department for review and are filed with the PT study records. PT test sample data are archived using the requirements for project and raw data record retention.

5.8.1.1 Double Blind Performance Evaluation

The laboratory participates in an annual double blind performance evaluation study. An external vendor is contracted to submit double blind samples to the laboratory. Both the level of customer service and the accuracy of the test results are assessed objectively by the external contractor, who provides a detailed report to the Corporate Quality Director and to the laboratory. This is administered as a double blind program in order to assess all facets of the laboratory's operations.

5.8.2 Control Samples

Control samples (e.g., QC indicators) are analyzed with each batch of samples to monitor laboratory performance in terms of accuracy, precision, sensitivity, selectivity, and interferences. Control samples must be uniquely identified and correlated to unique batches. Control samples further evaluate data based upon (1) Method Performance, which entails both the preparation and measurement steps; and (2) Matrix Effects, which evaluates field sampling accuracy, precision, representativeness, interferences, and the effect of the matrix on the method performed. Each regulatory program and each method within those programs specify the control samples that are prepared and/or analyzed with a specific batch.

Control sample types and typical frequency of their application are outlined Sections 5.8.2.1 through 5.8.2.5 and Tables 11 through 15. Note that frequency of control samples vary with specific regulatory, methodology and project specific criteria. Complete details on method and regulatory program control samples are as listed in Sections 7 and 8 typically of each method SOP.

5.8.2.1 Method Performance Control Samples: Preparation Batch

Sample preparation or pre-treatment is commonly required before analysis. Typical preparation steps include homogenization, grinding, solvent extraction, sonication, acid digestion, distillation, reflux, evaporation, drying and ashing. During these pre-treatment steps, samples are arranged into discreet manageable groups referred to as preparation (prep) batches. Prep batches provide a means to control variability in sample treatment.

Control samples are added to each prep batch to monitor method performance (Table 11) and are processed through the entire analytical procedure with investigative/field samples.

Table 11. Preparation Batch Control Samples

Control Sample Type	Details	
Method Blank (MB)	Use	Monitors for potential contamination introduced during the sample preparation and analytical processes.
	Typical Frequency [1]	1 per batch of ≤ 20 samples per matrix type per sample extraction or preparation method.
	Description	Organics: Laboratory pure water for water samples or a purified solid matrix for soil or solid samples (when available or when requested); solid matrices commonly include sodium sulfate, vendor or agency supplied soil or solid, or purchased sand; these solids may require purification at the laboratory prior to use. Inorganics: Laboratory pure water for both water and soil or sediment samples. Volume/weights are selected to approximately equal the typical sample volume/weight used in sample preparation; and final results in a soil/solid batch may be calculated as mg/kg or ug/kg, assuming 100% solids and a weight equivalent to the aliquot used for the corresponding field samples, to facilitate comparison to actual field samples.
Laboratory Control Sample (LCS)	Use	Measures the accuracy of the method in a blank matrix and assesses method performance independent of potential field sample matrix affects.
	Typical Frequency [1]	1 per batch of ≤ 20 samples per matrix type per sample extraction or preparation method. For multi-analyte methods, the LCS may consist of surrogates in the blank matrix, and or a representative selection of target analytes/internal standards.
	Description	Prepared from a reference source of known concentration and processed through the preparation and analysis steps concurrently with the field samples. Aqueous LCS's may be processed for solid matrices unless a solid LCS is requested; final results may be calculated as mg/kg or ug/kg, assuming 100% solids and a weight equivalent to the aliquot used for the corresponding field samples, to facilitate comparison with the actual field samples.
Known QC Sample	Use	Comply with regulatory requirements; check the accuracy of an analytical procedure; troubleshoot method performance problems; verify an analyst in training's ability to accurately perform a method; to verify the return-to-control after method performance problems; and may also be used as an LCS.
	Typical Frequency [1]	As defined by the client or QAPP.
Known QC Sample (cont'd.)	Description	Obtained from outside suppliers or agencies; generally require preparation from concentrated materials by dilution into a standard matrix; contain known analytes or compounds; acceptance limits are provided by the vendor.

[1] Denotes an STL required frequency.

Field blanks, equipment blank and trip blanks, when received, are analyzed in the same manner as other field samples. However, a field blank should not be selected for matrix QC, as it does not

provide information on the behavior of the target compounds in the field samples. Usually, the client sample ID will provide information to identify the field blanks with labels such as "FB", "EB", or "TB".

5.8.2.2 Method Performance Control Samples: Matrix

Matrix control samples include sample duplicates (MD), sample matrix spikes (MS), and sample surrogate spikes. These control samples help monitor for potential physical and chemical effects which may interfere with the precision and/or accuracy of the selected analytical method. Since interferences can enhance or mask the presence of target analytes, matrix control samples measure the degree of interference and are used to assist in the interpretation of the analytical results. The laboratory avoids performing matrix QC on known field blank samples, such as trip blanks and rinsates, since these samples are not indicative of the sample matrix.

Table 12. Matrix Control Samples

Control Sample Type	Details	
Matrix Duplicate (MD)	Use	Monitors the effect of site matrix on the precision of the method; and of the reproducibility of laboratory preparation and measurement techniques. **Note:** Precision may also be affected by the degree of homogeneity of the sample, particularly in the case of non-aqueous samples or aqueous samples with particulates. Sample homogeneity and matrix effect should be considered when field samples are used to assess reproducibility. **Note:** A field duplicate, when received, measures Representativeness of sampling and the effect of the site matrix upon precision.
Matrix Duplicate (MD) (cont'd.)	Typical Frequency [1]	1 per 20 samples per matrix or per SAP/QAPP [2].
	Description	Performed by analyzing two aliquots of the same field sample independently; analyzed for each associated sample matrix (e.g., when requested by the client or the analytical method).
Matrix Spike (MS)	Use	Measures the effect of site sample matrix on the accuracy of the method.
Matrix Spike (MS) (cont'd.)	Typical Frequency [1]	1 per 20 samples per matrix or per SAP/QAPP.
	Description	Aliquot of a field sample which is spiked with the analytes or compounds of interest; analyzed for each associated sample matrix (when requested by the client or analytical method). The determination of MS percent recovery (% R) requires an analysis of a fortified sample and a non-fortified sample under the same procedural conditions (e.g., sample volumes, dilutions, procedural conditions, etc.). The concentration determined in the non-fortified sample is subtracted from the fortified sample concentration before determining the %R. The degree of homogeneity of the sample, particularly in the case on non-aqueous samples or samples with particulates, may affect the ability to obtain representative recoveries.
Matrix	Use	Measures effect of site sample matrix on precision of method.

Table 12. Matrix Control Samples

Control Sample Type	Details	
Spike Duplicate (MSD)	Typical Frequency [1]	1 per 20 samples per matrix, when requested by the client or the analytical method, or per SAP/QAPP [2].
	Description	Alternative to sample duplicate. Generally, inorganic protocols specify an MD/MS and organic protocols specify an MS/MSD.
Surrogate Spike	Use	Measures method performance to sample matrix (organics only).
	Typical Frequency [1]	Every QC and analytical sample.
	Description	Compounds similar to the target analytes in structure, composition and chromatography, but not typically found in the environment, are added to each QC and analytical sample, prior to preparation (e.g., extraction). If the surrogates in an analytical batch do not all conform to established control limits, the pattern of conformance in investigative and control samples is examined to determine the presence of matrix interference or the need for corrective action.
Internal Standards	Use	Monitor the qualitative aspect of organic and inorganic analytical measurements.
	Typical Frequency [1]	All organic and ICP methods as required by the analytical method.
	Description	Used to correct for matrix effects and to help troubleshoot variability in analytical response and are assessed after data acquisition. Possible sources of poor internal standard response are sample matrix, poor analytical technique or instrument performance.

[1] Denotes an STL required frequency.
[2] Either an MSD or an MD is required per 20 samples per matrix or per SAP/QAPP.

5.8.2.3 Matrix QC Frequencies

The frequency of matrix QC indicators depends on regulatory program compliance, a project's data quality objectives, or a client's requirements. The following frequency will be applied to samples when the regulatory programs are known and it does not conflict with project or client requirements.

Table 13. EPA Program Requirements

Program	Description [1]
SDWA	MD performed at a 10% frequency or 1 per preparation batch of ≤10 samples, whichever is more frequent.
CWA	MS (GC methods) and MD is performed at a 10% frequency or 1 per preparation batch of ≤10 samples, whichever is more frequent. For GC/MS Methods, MS is performed at a 5% frequency or 1 per preparation batch of ≤20 samples, whichever is more frequent.
RCRA	MS/MSD or MS/MD is performed at a rate of 5% per client (independent of the preparation batch). For clients submitting less than 10 samples, the method matrix QC requirement may be satisfied by another client's sample within the same prep batch unless the paperwork indicates a client requirement for matrix QC.
U.S. EPA CLP	MS/MSD or MS/MD is performed at a rate of 5% or 1 set per Sample Delivery Group (SDG) per matrix, independent of the prep batch. Samples are processed in simultaneous or continuous batches.

[1] MS, MSD and MD may not be applicable to some analytical protocols because of the nature of the sample or protocol.

5.8.2.4 Method Performance Control Samples: Instrument Measurement

Control samples are used to ensure that optimum instrument performance is achieved. These samples help ensure that the proper identification and quantitation of target compounds or analytes are achieved. The instrument control samples appropriate to each analytical technique are described in laboratory SOPs for each respective method. A brief description of these checks is included in Table 14.

Table 14. Instrument Performance Control Samples

Control Sample Type		Description
		Inorganics
ICV	Use	Calibration standard of known concentration prepared from a source other than that used for the calibration standards.
	Sequence	Analyzed after the standard curve to confirm calibration.
ICB	Use	Blank water or solvent; confirms the calibration and assures that any potential contamination is less than the reporting limit.
	Sequence	Analyzed immediately after the ICV.
ICP Interference Check Samples (ICSA/ICSB)	Use	Verifies the absence of spectral interferences.
	Sequence	Analyzed consecutively at the beginning of each eight hour analytical sequence, after the ICV/ICB, and again at an eight hour frequency following a CCV/CCB. When CLP protocols are followed, the ICSA/B will be analyzed with the analytical sequence, before the final CCV/CCB.
Reporting Limit Verification	Use	Verifies linearity near the reporting limit for CLP metals analyses. (Note: CRI is at a level 2X the CRDL; CRA is near the CRDL).
Standard (CRA and CRI)	Sequence	Performed only when analyzing CLP Samples or as specified by the client or program. Analyzed after the ICB. The CRI is also analyzed at the end of the eight hour analytical sequence, prior to analysis of the final CCV/CCB.
CCV	Use	Confirm that the instrument performance has not significantly changed during the analytical sequence; to verify stable calibration throughout the sequence; and/or to demonstrate that instrument response did not drift over a period of non-use. May be made from a source other than that used for the standard curve, however if the ICV is 2nd source, the CCV may be same source.
	Sequence	Analyzed at 10% or every two hours, whichever is more frequent; also analyzed at the end of the analytical sequence.
CCB	Use	Water blank used to confirm that the baseline has not drifted and to monitor for contamination at the reporting limit.
	Sequence	Analyzed at a rate of 10% for inorganics and at a rate of 1 per 10 readings/injections or every two hours, whichever is more frequent, for CLP metals; also analyzed at the end of the analytical sequence.
ICP Metals Linear Range	Use	Verify linearity and document the upper limit of the calibration range for each element.

Table 14. Instrument Performance Control Samples

Control Sample Type	Description	
Analysis Standard (LRS)	Sequence	Performed quarterly with a blank and a minimum of five standard concentrations to cover the anticipated range of measurement; one of the calibration standards will be at or near the reporting limit. The calibration curve generated must have a correlation coefficient $\geq$ 0.995 in order to consider the responses linear over that range.
ICP Inter-Element Correction (IEC)	Use	Correction factors for spectral interference (particularly due to Al, Ca, Fe, and Mg).
	Sequence	Determined at least annually for all wavelengths used for each analyte reported by ICP; or any time the ICP is adjusted in any way that may affect the IECs.
Organics		
GC/MS Tuning & Performance	Use	Ensures correct mass assignment and is monitored through response to target compounds during initial and continuing calibration, with minimum response criteria for specified system performance check compounds (SPCCs), and linearity is verified by evaluating the response factors (RF) for calibration check compounds (CCCs).
	Sequence	Tuned at the beginning of the daily work shift. Throughout the analysis, blanks, internal standard areas, surrogates, chromatographic baseline, resolution of peaks, and overall quality of the chromatography are used collectively to monitor instrument performance.
GC & HPLC Instrument Performance	Use	Monitored through retention time shift evaluation, linearity checks, and degradation checks of selected target compounds (e.g., for Endrin or DDT as appropriate).
GC & HPLC Instrument Performance	Sequence	Continuing calibration verification (e.g., blanks, shifts in chromatographic baseline or retention times, resolution of peaks, and overall quality of the chromatography) throughout the analytical sequence is accomplished through analysis of calibration check standards.

5.8.2.5 Method Performance Control Samples: Analysis Batch

Matrix specific control samples are used to assess the precision and accuracy of the method as applied to the specific sample matrix. These indicators provide information on sample matrix effects that is independent of the efficiency of the preparatory technique. The method performance control samples appropriate to each analytical technique are identified in the respective method. A brief description of these checks is included in Table 15.

These control samples are performed to provide a tool for evaluating how well the method performed for the respective matrix. These values are used by the client to assess the validity of a reported result within the context of the project's data quality objectives. For matrix specific QC results falling outside laboratory control limits which are attributed to matrix affects, no systematic corrective action is taken.

Table 15. Analysis Batch Performance Control Samples

Control Sample Type	Description	
ICP Serial Dilution	Use	5X Dilution of a field sample (performed at the instrument) to check for possible physical and/or chemical interferences.
	Sequence	5% of field samples or 1 per ≤20 samples per batch.
Method of Standard Addition (MSA)	Use	When specified by the analytical protocol or by client request.
	Sequence	When specified by the analytical protocol or by client request.

5.8.3 Statistical Control Limits and Charts

Statistical control limits and control charts are used to establish method performance of a given analysis and to monitor trends of QC results graphically over time. Once a data base of the laboratory results for a method/matrix/QC analyte combination is established, the acceptability of a given analysis of that QC parameter (and of the analytical batch to which it belongs) can be evaluated in light of the laboratory's normal performance. This is intended to help identify problems before they might affect data. Often, patterns of response that are not at all evident in sets of numbers are very distinct when the same values are viewed as a chronological graph.

Establishment of Limits

The purpose of using statistical control limits is to define, for each analyte in a given method/matrix/QC type combination, a range of expected values. This range encompasses the random variation that occurs normally in the laboratory and allows one to evaluate control samples in that context, rather than according to an arbitrary or external set of values. Limits for accuracy and precision are defined below:

Accuracy

As recoveries of a QC analyte in a given matrix are tabulated over time, a mean value for recovery is established, as is the standard deviation (s) of those recoveries. If the analysis is in statistical control (e.g., if the set of QC recoveries over time show random variation about the mean) approximately 99.7% of all recoveries for that QC will fall within three standard deviations (3s) of the mean. Thus, assuming that the mean itself is an acceptable level of recovery, the values corresponding to 3s above and 3s below the mean are defined as the Control Limits. Any single recovery outside these values is assumed to have resulted from some circumstance other than normal variation and shall be investigated.

Roughly 95% of points should fall within 2s of the mean. The values +2s and -2s are the Warning Limits. Any normal result has approximately a 1/20 chance of being between 2s and 3s from the mean, so a result in this region doesn't necessarily warrant corrective action, but attention should be paid to such points.

Precision

Precision is used to indicate matrix variability so that appropriate decisions can be made by the client when repeated analyses vary significantly. The coefficient of variation, expressed as a percentage (e.g., the %RSD) for the data set used to calculate accuracy control limits defines the control limit for precision. Duplicate analyses of the QC samples, such as duplicates or MS/MSD, should have an RPD less than or equal to this established precision control limit to be considered free of matrix interferences.

The laboratory calculates statistical control limits on an annual basis, or more frequently if change have been made to the analytical process which affects the chemistry of the method. Such limits are available on a project or QAPP-specific basis.

5.8.4 Calibration

Calibration protocols are method-specific, are briefly described in Table 10 and are defined in the Sections 6 & 7 of the method SOPs.

5.8.5 Glassware Cleaning

All glassware is thoroughly cleaned prior to use to ensure that sample integrity is not affected from artifacts caused by contaminated glassware.

A summary of general cleaning procedures follows with details provided in the *Laboratory Glassware Cleaning* SOP *(AGP-Glass-04):*

General laboratory glassware is cleaned with a low- or non-phosphate detergent, followed by thorough rinsing with tap water and deionized water.

Volumetric flasks and pipettes used for inorganics (method dependent), test tubes and caps used for micro-COD procedures, phosphate glassware, and metals-related glassware include an acid-washing step.

BOD glassware, includes use of EPA approved disposable plastic bottles or cleaning with a nitric or sulfuric acid and/or a NOCHROMIX-washing step.

Organic glassware includes a solvent-wash.

5.8.6 Permitting Departures from Documented Procedure

Where a departure from a documented SOP, test method, or policy is determined to be necessary, or unavoidable, the departure is documented in a Job Exception and reported in the case narrative. In most cases, these departures can be made with the approval of the Department Supervisor, Project Manager and the client. Issues of serious concern, as determined by the Operations Manager, Department Supervisor or Project Manager, will be brought to the attention of the Laboratory Director and/or QA Manager. In some instances, it is appropriate to inform the client before permitting a departure. The Project Manager, in consultation with the QA Manager, will make the determination as to the degree of notification required by the client.

On rare occasions, special analytical techniques will be requested for research, project specific requirements, or client needs. In these instances, SOPs may not be available, however, the analyst will thoroughly record the analytical steps and observations within a bound preformatted logbook.

5.8.7 Development of QC Criteria, Non-Specified in Method/Regulation

Where a method or regulation does not specify acceptance and/or rejection criteria, the laboratory must examine the data user's needs and the demonstrated sensitivity, accuracy and precision of the available test methods in determining appropriate QC criteria.

Data users often need the laboratory's best possible sensitivity, accuracy, and precision using a routinely offered test method, or are unsure of their objectives for the data. For routine test methods that are offered as part of STL's standard services, the laboratory bases the QC criteria on statistical information such as determination of sensitivity, historical accuracy and precision data, and method verification data. The method SOP includes QC criteria for ongoing demonstration that the established criteria are met (e.g., acceptable LCS accuracy ranges, precision requirements, method blank requirements, initial and continuing calibration criteria, etc.).

In some cases, a routine test method may be far more stringent than a specific data user's needs for a project. The laboratory may either use the routinely offered test method, or may opt to develop an alternate test method based on the data user's objectives for sensitivity, accuracy, and precision. In this case, it can be appropriate to base the QC criteria on the data user's objectives, and demonstrate through method verification and ongoing QC samples that these objectives are met.

For example, a client may require that the laboratory to test for a single analyte with specific DQOs for sensitivity, accuracy, and precision as follows: Reporting Limit of 10 ppm, Accuracy $\pm$25%, and RSD of <30%. The laboratory may opt to develop a method that meets these criteria and document through the Method Blank results, MDL study, and LCS results that the method satisfies those objectives. In this case, both the method and the embedded QC criteria have been based on the client's DQOs.

In some cases, the data user needs more stringent sensitivity, accuracy, and/or precision than the laboratory can provide using a routine test method. In this case, it is appropriate that the laboratory provide documentation of the sensitivity, accuracy, and precision obtainable to the data user and let the data user determine whether to use the best available method offered by the laboratory, or determine whether method development or further research is required.

5.9 Project Reports

The SOP for data package assembly and reporting formats is *ARP-Report-125* and a summary of this procedure follows.

Analytical reports comprise final results (uncorrected for blanks and recoveries unless specified), methods of analysis, levels of reporting, surrogate recovery data, and method blank data. In addition, special analytical problems will be noted in the case narratives. The number of significant figures reported are consistent with the limits of uncertainty inherent in the analytical method. Consequently, most analytical results will be reported to no more than two (2) or three (3) significant figures. Data are normally reported in units commonly used for the analyses performed.

Concentrations in liquids are expressed in terms of weight per unit volume (e.g., milligrams per liter, mg/L). Concentrations in solid or semi-solid matrices are expressed in terms of weight per unit weight of sample (e.g., micrograms per kilograms, ug/kg). Reporting limits take into account all appropriate concentration, dilution, and/or extraction factors, unless otherwise specified by program requirements (e.g.,IRPMS reports).

A client report is generated with various steps of approval prior to printing of the final version. If any analytical anomalies were encountered during the analyses, e.g., an out-of-control matrix duplicate, it is documented in a case narrative. The case narrative is prepared by the respective operating unit, project manager, or other designated personnel and inserted in the final report.

The final report forms are printed, data packages are organized, a glossary of flags and acronyms is added, and reports are paginated.

5.9.1 General

The criteria described in Section 5.9.2 apply to all Project Reports that are generated under NELAC requirements. The criteria described in Section 5.9.3 and 5.9.4 apply to all Project Reports.

5.9.2 Project Report Content

- Title
- Laboratory name, address, telephone number, contact person
- Unique Laboratory Project Number
- Name and Address of Client
- Client Project Name (if applicable)
- Laboratory Sample Identification
- Client Sample Identification
- Matrix and/or Description of Sample
- Dates: Sample Receipt, Collection, Preparation and/or Analysis Date
- Definition of Data Qualifiers
- Reporting Units
- Test Methods
- Report Paginated

The following are required where applicable to the specific test method or matrix:

- Solid Samples: Indicate Dry or Wet Weight
- Whole Effluent Toxicity: Statistical package used
- If holding time $\leq$ 48 hours, Sample Collection, Preparation and/or Analysis Time
- Indication by flagging or narrative comment where results are reported below the quantitation limit.

5.9.3 Project Narrative

A Project Narrative and/or Cover Letter is included with each project report and, at a minimum, includes an explanation of any and all of the following occurrences:

- Listing of any subcontracted analyses and subcontractor location
- Non-conformances

- "Compromised" sample receipt (see Section 4.7.1)
- Method Deviations
- QC criteria failures
- Any authorized SOP deviations, non-conformances and QC failures must be covered in the case narrative, cover letter or within the report.

Project Release

The Project Manager or his designee authorizes the release of the project report with a signature.

Where amendments to project reports are required after issue, these are documented in the form of a DQR (refer to Section 4.8) and can be in the form of a separate document and/or electronic data deliverable resubmittal. The revised report is clearly identified as revised with the date of revision and the initials of the person making the revision. Specific pages of a project report may be revised using the above procedure with an accompanying cover letter indicating the page numbers of the project revised. The original version of the project report will be kept intact and the revisions and cover letter included in the project files.

5.9.4 Subcontractor Test Results

Subcontracted data are clearly identified as such, and the name, address, and telephone number for the laboratory performing the test is included in the project report. Subcontracted results from laboratories external to STL are not reported on STL report forms or STL letterhead. Test results from more than one STL facility are clearly identified with the name of the STL facility that performed the testing, address, and telephone number for that facility. Data from subcontractors' reports may be added to an STL electronic deliverable.

Data subcontracted within STL may be reported on the originating laboratory's report forms provided the following mandatory requirements are met:

- The name, address, and telephone number of the facility are provided.
- Analytical results produced by the STL intra-company subcontractor are clearly identified as being produced by the subcontractor facility.
- The intra-company subcontractor's original report, including the chain of custody is retained by the originating laboratory.
- Proof of certification is retained by the originating laboratory.
- All information as outlined in Section 5.9.2 is included in the final report where the report is required to be compliant with NELAC, for both the originating and subcontracting laboratory.

5.9.5 Electronic Data Deliverables

Electronic Data Deliverables (EDD) are routinely offered as part of STL's services. STL offers a variety of EDD formats. EDD specifications are submitted to the IT department by the PM for review and undergo the contract review process in Section 4.4.1. Once the laboratory has committed to providing diskettes in a specific format, the coding of the format may need to be performed. This coding is documented and validated. The validation of the code is retained as a QC record.

EDDs are subject to a review to ensure their accuracy and completeness. If EDD generation is automated, review may be reduced to periodic screening if the laboratory demonstrates that it can

routinely generate that EDD without errors. Any revisions to the EDD format are reviewed until it is demonstrated that it can routinely be generated without errors. If the EDD can be reproduced accurately and if all subsequent EDDs can be produced error-free, each EDD does not necessarily require a review.

5.9.6 Project Report Format

STL offers a wide range of project reporting formats, including EDDs, short report formats, and complete data deliverable packages modeled on the Contract Laboratory Protocol (CLP) guidelines. Regardless of the level of reporting, all projects undergo the levels of review as described in Section 5.3.6.

Appendix. List of Cited SOPs and Work Instructions

Cited Section No(s).	Status	Description	Document No.
1.1	Buff	Certification Listing	STLBuffCertList
1.6 5.7.1	Buff	Container Management: Process Operation/Bottle Order Set-Up	APM-Bottle Order-03
1.6 4.4.2	Buff	Project Management: Project Planning Process/Project Information Requirements	APM-ProjInfo-20
4.1.1 5.4.1	Buff	Capital Equipment Listing	STLBuffEquipList
4.1.2.9	Buff	Computer System Account and Naming Policy Computer System Password Policy Software Licensing Policy Virus Protection Policy	P-I-003 P-I-004 P-I-005 P-I-006
4.3.1.1 5.3.2	Buff	SOP Master Index	STLBuff_SOPIndex
4.3.2 4.12.3	Buff	Data Management: Record Storage and Retention	AGP-RecordStorage-56
4.4.2	Buff	Project Kick-Off Meetings	APM-ProjInfo-20
4.6	Buff	Procurement of Laboratory Supplies and Services	APH-Supply-08
4.6.1	STL	Testing Solvents and Acids	S-T-001
4.7.2	Buff	Client Confidentiality	APM-ProjInfo-20
4.8	Buff	Data Quality Request	AQA-DQR-65
4.8	Buff	Preventative or Corrective Action	AQA-CA-35
4.8 4.11	Buff	Job Exception Report (Non-conformance Report)	AQA-CA-35
4.11	Buff	Quality Systems Management Review	AQA-Management Review-45
4.11	Buff	Preventive Action Measures	AQA-CA-35
4.13	STL	Systems Audits	S-Q-002
5.1.2	Buff	Laboratory Personnel Training	AQA-Train-10
5.1.3	STL	Ethics Policy	P-L-006
5.3.1	Buff	Methods Capabilities & Index	ASR-Prsv-07
5.3.2	Buff	SOP Interim Change	AQA-SOP-55
5.3.5	STL	MDL Policy	S-Q-003
5.3.6.1	STL	Acceptable Manual Integration Practices	S-Q-004

Appendix. List of Cited SOPs and Work Instructions

Cited Section No(s).	Status	Description	Document No.
5.3.6.2	Buff	Data Review Checklists / Technical Data Review GC Extractables / HPLC GC Volatiles GC/MS: Volatiles and Semi-Volatiles Metals Wet Chemistry	AGP-DataReview-21
5.4.2	Buff	Instrument and Equipment Out-of-Service Tagging	AGP-OutofService-65
5.4.3	STL	Selection of Calibration Points	P-T-001
5.4.3	Buff	Standards Traceability and Preparation	AGP-STD-14
5.5.1	Buff	Balance Calibration, Care and Use	AGP-SuppEquip-02
5.5.1	Buff	Thermometer Calibrations	AGP-SuppEquip-02
5.5.1	Buff	Water Quality	AGP-SuppEquip-02
5.7.1	Buff	Sample Receipt: Handling and Processing	ASR-Receipt-05
5.7.1	Buff	Sample Container Preparation and Shipment	ASR-Bottle-03
5.7.5	Buff	Laboratory Waste Disposal Procedures	ASR-Disp-33 and AWM-Haz.Mg-01
5.8.5	Buff	Glassware Cleaning Procedures	AGP-Glass-04
5.9 5.9.6	Buff	Data Management: Reporting	ARP-Report-125

ARCADIS BBL

Attachment 3

Chain of Custody

ARCADIS
Infrastructure, environment, facilities

ID#:

CHAIN OF CUSTODY & LABORATORY ANALYSIS REQUEST FORM

Page ___ of ___

Lab Work Order #

Send Results to:

Contact & Company Name:	Telephone:
Address:	Fax:
City State Zip	E-mail Address:
Project Name/Location (City, State):	Project #:
Sampler's Printed Name:	Sampler's Signature:

Sample ID	Collection Date	Collection Time	Type (✓) Comp	Type (✓) Grab	Matrix

Preservative							
Filtered (✓)							
# of Containers							
Container Information							

PARAMETER ANALYSIS & METHOD

Keys

Preservation Key:
A. H_2SO_4
B. HCL
C. HNO_3
D. NaOH
E. None
F. Other: _____
G. Other: _____
H. Other: _____

Container Information Key:
1. 40 ml Vial
2. 1 L Amber
3. 250 ml Plastic
4. 500 ml Plastic
5. Encore
6. 2 oz. Glass
7. 4 oz. Glass
8. 8 oz. Glass
9. Other: _____
10. Other: _____

Matrix Key:
SO - Soil
W - Water
T - Tissue
SE - Sediment
SL - Sludge
A - Air
NL - NAPL/Oil
SW - Sample Wipe
Other: _____

REMARKS

Special Instructions/Comments:

☐ **Special QA/QC Instructions(✓):**

Laboratory Information and Receipt		Relinquished By	Received By	Relinquished By	Laboratory Received By
Lab Name:	Cooler Custody Seal (✓)	Printed Name:	Printed Name:	Printed Name:	Printed Name:
☐ Cooler packed with ice (✓)	☐ Intact ☐ Not Intact	Signature:	Signature:	Signature:	Signature:
Specify Turnaround Requirements:	Sample Receipt:	Firm:	Firm/Courier:	Firm/Courier:	Firm:
Shipping Tracking #:	Condition/Cooler Temp: _____	Date/Time:	Date/Time:	Date/Time:	Date/Time:

Distribution: **WHITE – Laboratory returns with results** **YELLOW – Lab copy** **PINK – Retained by BBL**