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Part I of III

REMEDIAL ACTION
SAMPLING AND ANALYSIS PLAN

VOLUME II: QUALITY ASSURANCE PROJECT PLAN

Defense Depot Memphis, Tennessee

Defense Logistics Agency

MACTEC

MACTEC Engineering and Consulting, Inc.

Air Force Center for Environmental Excellence
Contract No. F41624-03-D-8606
Task Order Nos. 0038 and 0080

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REMEDIAL ACTION SAMPLING AND ANALYSIS PLAN

VOLUME II: QUALITY ASSURANCE PROJECT PLAN

Defense Depot Memphis, Tennessee

Prepared for:
Air Force Center for Environmental Excellence
Contract No. F41624-03-D-8606
Task Order Nos. 0038 and 0080

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PREFACE

The Remedial Action (RA) Sampling and Analysis Plan (SAP) prescribes those procedures necessary to perform the field activities, laboratory activities, and other contract requirements related to RA support at the Defense Depot Memphis, Tennessee, program. The RA SAP consists of two documents – the Field Sampling Plan (FSP) and the Quality Assurance Project Plan (QAPP). This RA SAP was prepared in accordance with the requirements of the U.S. Environmental Protection Agency (USEPA) *Guidance for Quality Assurance Project Plans*, EPA 600/R-98/018 (USEPA, 1998); USEPA *Requirements for Quality Assurance Project Plans*, EPA 240/B-01/003 (USEPA, 2001b); and Air Force Center for Environmental Excellence (AFCEE) *Guidance for Contract Deliverables – Appendix B: Field Sampling Plans, and Appendix C: Quality Assurance Project Plans* (AFCEE, 2001).

The FSP describes field activities to be performed and defines the procedures and methods required to collect field measurements and samples. The QAPP consists of information used to define and measure data quality objectives (DQOs). Definition of the DQOs assists in determining the appropriate procedures for fieldwork and laboratory analysis. The QAPP describes the quality assurance and quality control procedures necessary to meet project DQOs.

Key MACTEC Engineering and Consulting, Inc., (MACTEC) personnel participating in this project include Mr. Thomas Holmes, project principal; Mr. Paul Brafford, senior chemist; Mr. John Quinn, senior geologist; and Mr. David Price, project manager. The RA SAP was prepared by MACTEC under Contract No. F41624-03-D-8606, Task Order Nos. 0038 and 0080, for AFCEE and the Defense Logistics Agency.

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LIST OF ACRONYMS

AFCEE	Air Force Center for Environmental Excellence
A2LA	American Association of Laboratory Accreditation
APHA	American Public Health Association
ASTM	American Society for Testing and Materials
C	Celsius
C-C	Chain of Custody
CFR	Code of Federal Regulations
COC	Chemical of Concern
CVAA	Cold-vapor Atomic Absorption
DDMT	Defense Depot Memphis, Tennessee
DO	Dissolved Oxygen
DQE	Data Quality Evaluation
DQI	Data Quality Indicator
DQO	Data Quality Objective
DRI	Design-related Investigation
ECD	Electron Capture Detector
EQuIS	Environmental Quality Information System
FR	Federal Register
FSP	Field Sampling Plan
FSR	Field Sampling Report
GC	Gas Chromatography
IC	Ion Chromatograph
ICP	Inductively Coupled Plasma
ICS	Interference Check Sample
IS	Internal Standard
LCS	Laboratory Control Sample
LCSD	Laboratory Control Sample Duplicate
LQM	Laboratory Quality Manual
MACTEC	MACTEC Engineering and Consulting, Inc.
MCAWW	Method for Chemical Analysis of Water and Wastes

LIST OF ACRONYMS
(Continued)

MDL	Method Detection Limit
µg/kg	Micrograms per Kilogram
mg/kg	Milligrams per Kilogram
mg/L	Milligrams per Liter
mL/min	Milliliters per Minute
MS	Matrix Spike
MSD	Matrix Spike Duplicate
NIST	National Institute of Standards and Technology
ORP	Oxidation-reduction Potential
PARCC	Precision, Accuracy, Representativeness, Completeness, and Comparability
PCB	Polychlorinated Biphenyl
QA	Quality Assurance
QAPP	Quality Assurance Project Plan
QC	Quality Control
%R	Percent Recovery
RA	Remedial Action
RL	Reporting Limit
RPD	Relative Percent Difference
RSD	Relative Standard Deviation
SAP	Sampling and Analysis Plan
SOP	Standard Operating Procedure
SQL	Sample Quantitation Limit
STL	Severn Trent Laboratories
SVOC	Semivolatile Organic Compound
TAT	Turnaround Time
TCLP	Toxicity Characteristic Leaching Procedure
TOC	Total Organic Carbon
USACE	U.S. Army Corps of Engineers
USEPA	U.S. Environmental Protection Agency
VFA	Volatile Fatty Acid
VOC	Volatile Organic Compound

1.0 QUALITY ASSURANCE PROJECT PLAN

The Quality Assurance Project Plan (QAPP) outlines the quality assurance (QA)/quality control (QC) procedures to be utilized during the analyses of samples and the management of data generated in support of remedial actions (RAs) at Defense Depot Memphis, Tennessee (DDMT). This QAPP is general and includes several analytical test methods that may be utilized at DDMT. Those methods not included in this QAPP will be presented in the site-specific Work Plans.

The elements discussed in the QAPP include:

- Laboratory organization and responsibilities
- Data quality objectives (DQOs)
- Sample handling
- Analytical procedures
- Laboratory QC procedures
- Data reduction and calculation of precision, accuracy, representativeness, completeness, and comparability (PARCC)
- Laboratory documentation
- Data assessment procedures

1.1 PROJECT SCOPE, BACKGROUND, AND OBJECTIVES

DQOs are developed for field and laboratory operations to clarify study objectives, define the appropriate type of data stipulated, and specify tolerable levels of potential decision errors to establish the quality and quantity of data needed to support decisions. DQOs determine the type, quantity, and quality of data needed to meet project objectives and reach defensible decisions. Project-specific DQOs are presented in each project Work Plan.

The DQO process leads to the specification of sample handling procedures; preparatory (extraction/digestion), cleanup, and determinative methods; target analytes; method detection or reporting limits (RLs); field and laboratory QC samples; measurement quality objectives (QC acceptance limits) for PARCC parameters; required corrective actions; and data assessment procedures necessary to meet the

intended use of the data. The general DQOs for the DDMT field investigations are presented in Section 2.0.

The goal of the investigations and field activities is to generate data of sufficient quality and quantity to meet the overall project objectives. Data required for each project may consist of screening and definitive data. The general use and definitions of the data are presented below. General use categories are as follows:

- **Design-related Investigations (DRIs)** – Data collected in DRIs are to be provided to the Remedial Design contractor for incorporation into the RD; the data will also be used in development of RA Work Plans and as baseline data to assess RA effectiveness.
- **Confirmation Sampling** – Data collected for this activity will be used to confirm that constituents are not present above established cleanup goals, and to confirm that the final remedy cleanup objectives have been achieved in support of National Priorities List deletion.
- **Performance/Long-term Monitoring** – Data collected for this activity will monitor the progress of constituent reduction for sites where a remedial alternative is already in place.
- **Health and Safety** – Data will be collected during field activities to establish the level of protection needed for the fieldwork party and other site personnel. These data will be gathered during intrusive activities through the use of organic vapor analyzers, Draeger® tubes, and an explosimeter.

In addition, data collection may be required to support site characterization, risk assessment, and/or engineering design alternative activities. The types of sample media that will be required to meet the data needs of the general DDMT program will mainly consist of soil, groundwater, and effluent water.

1.2 PROJECT LABORATORY ORGANIZATION AND RESPONSIBILITIES

The project laboratories are responsible for providing the sample shipping containers, chain of custody (C-C) forms, and chemical analysis and reporting as designated by project-specific DQOs. Specific laboratory organization and personnel descriptions are presented in the Laboratory Quality Manuals (LQMs) located in Appendix A. Figure 3-1 of the Field Sampling Plan (FSP) presents the MACTEC Engineering and Consulting, Inc., (MACTEC) project personnel and organization. Subsection 2.3 of the FSP includes a summary of the subcontractors.

Severn Trent Laboratories (STL), located in North Canton, Ohio, and ETC, located in Memphis, Tennessee, will provide the chemical analyses scoped for investigations at DDMT. To successfully perform work for U.S. Department of Defense clients, the laboratories are certified by the National Environmental Laboratory Accreditation Council to perform the tests scoped for the investigations at DDMT. Both laboratories have implemented QA/QC specific to the project DQOs in their laboratory operations and Standard Operating Procedures (SOPs). DDMT's STL project manager is Mr. Roger Toth, and the ETC project manager is Ms. Connie Bradberry. Future projects requiring analytical services may result in the selection of another vendor(s).

If selection of another analytical service vendor(s) is necessary, the following vendor information will be compared for compliance with project requirements and a memorandum generated to document the compliance review and stored in the project file:

- Quality manual
- Organization and responsibilities
- SOPs
- Certifications
- Method detection limits (MDLs)
- RLs
- Control limits for method precision and accuracy
- Performance evaluations
- Performance audit corrective action

If selection of an additional analytical method from an approved service vendor is necessary, the following method information will be reviewed and included in a memorandum to document project requirements and stored in the project file:

- Analytical method SOP
- Calibration procedures and frequency
- Comparison of MDLs/RLs to applicable standards
- Field and laboratory QC sample number and frequency
- Precision and accuracy limits for duplicate samples, laboratory control samples (LCSs), and matrix spike (MS)/matrix spike duplicate (MSD) samples
- Laboratory and field blank results

- Holding times
- Sample containers, volumes, types, and preservation
- Generation of Data Quality Evaluation (DQE) SOP

2.0 DATA QUALITY PROGRAM OBJECTIVES

This section presents the information and objectives included in the data quality program for DDMT. These include the data verification, review, and validation tasks performed for the applicable QC elements.

Data verification is the most basic assessment of data. The purpose of data verification is to ensure that the records associated with a specific dataset actually reflect all of the processes and procedures used to generate them, and to evaluate the completeness, correctness, consistency, and compliance of the dataset against a standard or contract. In this context, "completeness" means that all required hardcopy and electronic deliverables are present. Data verification will be performed by MACTEC's senior/project chemist, in-house chemists, and database manager.

Data review is performed by the laboratory as part of its standard procedures. Subsection 5.2 details the actions performed by the laboratory during the data review process.

Data evaluation/validation is performed to generate a DQE case narrative report (submitted as an appendix to the project report). MACTEC's senior chemist will determine whether the data meet project-specific data quality criteria and contract requirements. MACTEC's project chemist and other in-house chemists produce the DQE report. These acceptance criteria and requirements are found in the DQO summary from the Sampling and Analysis Plan (SAP), project-specific Work Plans, field oversight findings, laboratory audits, and any other available quality indicators.

2.1 DATA CATEGORIES

Anytime chemical data are generated, the quality of the data must be assessed before use. The type and degree of assessment required depend upon the acceptance and performance criteria of the project. Data assessment is the all-inclusive process used to measure the effectiveness of a particular data-gathering activity. Several levels of data assessment exist, including data verification, data review, and data evaluation/validation. Specific details of each data assessment level are presented in Section 5.0.

As previously stated, screening and definitive data may be required to meet the project-specific DQOs. Data generated during field activities are categorized according to QA/QC elements. Data quality is

classified according to the following data categories as defined in *Guidance for the Data Quality Objective Process* (U.S. Environmental Protection Agency [USEPA], 1994a):

- Screening Data – Screening data are generated by rapid, less precise methods of analysis with less rigorous sample preparation. Sample preparation steps may be restricted to simple procedures, such as dilution with a solvent, instead of elaborate extraction/digestion and cleanup. Screening data provide analyte identification and quantitation, although the quantitation may be relatively imprecise. At least 10 percent of the screening data are confirmed using analytical methods and QA/QC procedures and criteria associated with definitive data. Screening data without associated confirmation data are not considered to be data of known quality.

  Screening data may be generated at DDMT through the use of colorimetric and/or titrametric field tests (ferrous iron and carbon dioxide) and multiple field quality parameter instruments that measure the potential of hydrogen (pH), specific conductance, dissolved oxygen (DO), temperature, oxidation-reduction potential (ORP), and turbidity. The field tests for ferrous iron and carbon dioxide are presented in Appendix C of the FSP.

- Definitive Data – Definitive data are generated using rigorous QA/QC procedures and analytical methods, such as approved USEPA reference methods. Data are analyte-specific, with confirmation of analyte identity and concentration. Methods produce tangible raw data (e.g., chromatograms, spectra, and digital values) in the form of paper printouts or computer-generated electronic files. Data may be generated on-site or at an off-site location, as long as the QA/QC requirements are satisfied. For the data to be definitive, either analytical or total measurement error must be determined. Table 2-1 summarizes the QA/QC elements related to each data category. Definitive data will be generated for most of the samples collected at DDMT. The overall DQO for analytical completeness of definitive data generated during field investigations is 90 percent for soils and waters.

2.2 PRECISION, ACCURACY, REPRESENTATIVENESS, COMPLETENESS, AND COMPARABILITY

The PARCC criteria measure the usability of environmental data as it relates to project objectives. Evaluation of these criteria ultimately reveals the representativeness and bias, if any, present in the sampling and analytical processes. The field program will be accomplished through the collection and analysis of field duplicates, rinsate blanks, trip blanks, and MS/MSDs. The analytical program will be assessed through the internal laboratory QC performed, including method blanks, LCSs, surrogate standards, internal standards (ISs), and calibration standards. These are discussed in more detail in Subsection 2.4.

2.2.1 Precision

Precision refers to the reproducibility or degree of agreement among duplicate measurements of a single analyte. The purpose of duplicate measurements is to characterize the precision of the sampling procedure under specified conditions. To measure precision in environmental samples, field duplicate samples are collected concurrently with the parent sample under the same field conditions. Although spiked in the laboratory, MS/MSD samples also provide field precision data. Analytical precision for a single analyte is expressed as a percentage of the difference between results of duplicate samples for a given analyte. The relative percent difference (RPD) for each compound or element is calculated using the equation below. Field duplicates will be collected at a frequency of 10 percent (i.e., 1 duplicate/split for every 10 field samples). Because of the collection procedures associated with and the nature of volatile organic compounds (VOCs) in soil samples, a co-located sample will be collected in lieu of a duplicate sample, but treated and labeled as a duplicate sample.

The closer the numerical values of the measurement, the more precise the measurement. The purpose of duplicate measurements is to characterize the precision of an analytical method. Precision determination will be performed in the laboratory by the analysis of laboratory duplicate samples and MS/MSD samples. Precision is expressed either as relative standard deviation (RSD) for replicate measurements greater than 2 or as RPD for duplicate measurements. The RSD for each compound or element is calculated using the following equation:

$$s = \sqrt{\frac{\sum_{i=1}^{n} (x_i - x)^2}{n-1}}$$

Where:

- s = sample standard deviation
- x = the mean
- x_i = the i^{th} data value
- n = number of data values
- $\sum$ = sum of

The RPD for each compound or element is calculated using the following equation:

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

Where:

A = Replicate value 1
B = Replicate value 2
RPD = Relative percent difference

2.2.2 Accuracy

Accuracy is a measure of the closeness of an observed value to the "true" value; e.g., theoretical or reference value, or population mean. The percent recovery (%R) of the compounds spiked into a matrix (via both LCSs, MS/MSDs, or surrogates) is used to evaluate the accuracy of the environmental sampling process. The recovery of an analyte from the LCS, an MS/MSD, and/or surrogate spikes is indicative of the impact a specific matrix may have on the accuracy of a specific compound or element. The compounds to be spiked for DDMT samples include the full list of analytes to be reported for the individual analysis.

The %R is defined as the observed concentration minus the sample concentration, divided by the true concentration of the spike added and multiplied by 100 to express percent.

$$\%R = \frac{X - T}{K} \times 100$$

Where:

X = Analytical result from the spiked sample
T = Analytical result from the unspiked aliquot
K = Known value of the spike
%R = Percent recovery

The bias and accuracy of field protocols are difficult to assess quantitatively. Sampling accuracy can be maximized, however, by the adoption of and adherence to a strict field QA program. Specifically, procedures will be performed following standard protocols. Equipment and instrumentation will be properly calibrated and well maintained. Trip blanks and equipment rinsates will be included in each

sample batch to provide representative data to assess the potential for cross-contamination. Through regular review of field procedures, deficiencies will be documented and corrected in a timely manner.

2.2.3 Representativeness

Representativeness is defined by the degree to which the data accurately and precisely represent an environmental condition. If the results are reproducible, the data obtained can be said to represent the environmental condition. Representativeness is ensured by collecting sufficient samples of an environmental medium, properly chosen with respect to place and time. The precision of a representative set of samples reflects the degree of variability of the sampled medium as well as the effectiveness of the sampling techniques and laboratory analysis. Samples that are not properly preserved or analyzed beyond holding times may not be considered representative. Review of sampling procedures, laboratory preparation, analysis holding times, trip blank analysis, and field blank analysis are essential to this assessment.

2.2.4 Completeness

Completeness is expressed as the percentage of usable data obtained from a measurement system compared to the amount that was expected to be obtained under correct or normal conditions. For data to be considered usable, they must meet some or all of the acceptance criteria specified in the analytical method used and must not result in "rejected" data points. Completeness will be calculated on a per-analysis-method, per-matrix, and per-site basis. The percent complete is used to evaluate whether sufficient data were acquired from the sampling event.

Field sampling conditions are often unpredictable and non-uniform. However, the objective of the field sampling program is to obtain samples for each analysis required at each site, provide sufficient sample material to complete those analyses, and collect the QC samples necessary to fully implement the field and laboratory QA/QC program. The overall DQO for field sampling and analytical completeness during field investigations is 90 percent for soils and waters.

Samples for which critical data points fail the DQOs may be reanalyzed (providing adequate sample volume and holding times are met) or resampled (with approval of the project manager) to meet the completeness goal. Critical data points are those points that are needed to meet the established DQOs, and include the chemicals of concern (COCs) as well as the field activities necessary to achieve the

DQOs. The use of the data with regard to qualification or resampling is determined by the Air Force Center for Environmental Excellence (AFCEE).

2.2.5 Comparability

Comparability is defined by the confidence with which one dataset can be compared to another dataset. Field and laboratory procedures affect comparability. To optimize comparability, only USEPA-established methods and U.S. Army Corps of Engineers- (USACE-) approved protocols have been selected or specified as appropriate for these investigations. By using standard sampling and analytical procedures, datasets will be comparable among DDMT sites.

2.3 METHOD DETECTION LIMITS AND REPORTING LIMITS

In order to meet DDMT project-specific DQOs, screening (where confirmed by definitive data) and definitive data will be compared to DDMT risk-based screening levels. Definitive data will be generated using USEPA methods with MDLs and/or RLs at or below screening levels to allow for sufficient qualitative and quantitative results (where achievable and feasible). Unless a reduced COC list is specified in the project-specific Work Plan, samples will be analyzed by the methods that include the compound/analyte list in Table 2-2.

All RL values meet the general DQOs as long as the RL is below the DDMT risk-based screening level. COCs with an RL above the screening level will be evaluated by comparison to the MDL. If the MDL is below or equal to the screening level, the MDL will be considered to meet the general DQOs. For COCs with an MDL above the screening level, alternative analytical methods will be evaluated to identify applicable methods with either the RL or MDL below the screening level. Such an evaluation was performed for 1,1,2,2-Tetrachloroethane; however, an alternative analytical method was not identified.

Table 2-2 presents comparisons of each of the method MDLs/RLs for target analytes to the DDMT background values and risk-based screening levels. A discussion of the laboratories' work processes and operations concerning MDLs may be found in the LQMs located in Appendix A.

Laboratory-established MDLs and RLs are updated annually per analytical method and matrix. However, the RLs used for DDMT will remain unchanged for the duration of the project. Updated MDL information will be provided when available from the laboratory. The laboratory-established detection

limits and RLs are listed in Appendix A. Method MDLs, RLs, and sample quantitation limits (SQLs) are defined below.

2.3.1 Method Detection Limit

MDL studies are conducted using spiked reagent water for water matrices and interference-free solid matrices processed through the appropriate analytical procedure, using simulated solid matrices such as Ottowa sand or sodium sulfate to generate soil limits. For inorganic parameters, MDLs for soil are established by applying the appropriate factor to convert the water MDL results of the studies to soil units. The RL is derived from the MDL and is set at the project-requested RL to meet project-specific DQOs.

The MDL is the minimum concentration of a substance that can be measured and reported with 99 percent confidence that the value is above 0. The MDLs are established using the required USEPA procedure specified in 40 Code of Federal Regulations (CFR) 136, Appendix B (*Definition and Procedure for the Determination of the Method Detection Limit* [Federal Register (FR), 1992]). A data pool of at least seven spiked replicates analyzed at a concentration approximately three times the anticipated MDL is generated. The MDL is estimated by employing the "t" distribution with a 99 percent confidence interval using the following equation:

$$MDL = \sqrt{(t)(S)}$$

Where:

t = a factor for n-1 degrees of freedom at the 99 percent confidence factor
S = the standard deviation of the data pool

The laboratories perform MDL studies on each instrument. Ongoing MDL verification is performed via MDL checks and MDL studies.

2.3.2 Reporting Limit and Sample Quantitation Limit

The RL is the lowest level that can be reliably achieved within specified limits of precision and accuracy during routine laboratory operating conditions as defined by SW-846. Project-specific RLs were

reviewed in comparison to risk-based screening levels. The SQL is the RL adjusted by the sample weight/volume extracted and analyzed, moisture content (soils and sediments only), and/or dilution.

SQLs are determined for each analyte reported in a sample according to sample size, percent moisture for soils, and dilution factors. Each analytical concentration will be reported as a numeric value at or greater than the MDL for inorganic and organic analyses. Samples with no detections (below the MDL) are reported as less than the SQL as approved by AFCEE. Detections below the SQL but above the MDL for inorganic and organic methods will be reported as estimated values. Water results will be reported in micrograms per liter concentrations for organics and in milligrams per liter (mg/L) for inorganics. Soil values will be reported on a dry weight basis in micrograms per kilogram (µg/kg) for organics and in milligrams per kilogram (mg/kg) for inorganics.

2.4 ELEMENTS OF QUALITY CONTROL

The following subsections discuss the QC elements relevant to analysis of environmental samples that will be followed during all analytical activities for producing definitive data.

2.4.1 Laboratory Control Sample

The LCS is analyte-free water for aqueous analyses or a choice of Ottawa sand, sodium sulfate, or glass beads 1 millimeter or smaller in diameter for soil spiked with all analytes reported for the method analyzed. Each analyte will be spiked at a level less than or equal to the midpoint of the calibration curve for each analyte. The LCS will be carried through the complete sample preparation and analysis procedure.

One LCS will be included in every analytical batch. If more than one LCS is analyzed in a batch, results from all LCSs analyzed will be reported. A QC failure of an analyte in any of the LCSs will require appropriate corrective action, as presented in Appendix B.

2.4.2 Matrix Spike/Matrix Spike Duplicate

An MS/MSD is an aliquot of sample spiked with known concentrations of all analytes reported for the method analyzed. The spiking occurs before sample preparation and analysis. Each analyte will be

spiked at a level less than or equal to the midpoint of the calibration curve. Only project spiked samples will be evaluated. The MS/MSD will be designated on the C-C.

A site-specific MS/MSD should be specified for each medium sampled for each site during each sampling event. Project managers should designate the MS/MSD to verify that the selections meet project requirements. At least 1 MS and 1 MSD will be designated for every 20 samples of each matrix type collected. When an MS and/or MSD recovery or RPD is outside the acceptance limits, corrective actions will be performed, as indicated in Appendix B.

2.4.3 Surrogates

Surrogates are organic compounds that are similar to the target analyte(s) in chemical composition and behavior in the analytical process, but that are not normally found in environmental samples. Surrogates are used to evaluate accuracy, method performance, and extraction efficiency. Surrogates will be added to environmental samples, controls, and blanks in accordance with the method requirements. Whenever a surrogate recovery exceeds the acceptance criteria, corrective action must be taken, as indicated in Appendix B.

2.4.4 Internal Standards

ISs are measured amounts of certain compounds added after preparation or extraction of a sample. They are used in an IS calibration method to correct sample results affected by column injection losses, purging losses, or viscosity effects. ISs will be added to environmental samples, controls, and blanks in accordance with the method requirements. When the IS results are outside the acceptance limits, corrective actions will be performed, as indicated in Appendix B.

2.4.5 Retention Time Windows

Retention time windows are used in gas chromatography (GC) analysis for qualitative identification of analytes. They are calculated from replicate analyses of a standard performed on multiple days. The procedure and calculation method is provided in SW-846 Method 8000B. Whenever a retention time window exceeds the acceptance criteria, corrective action must be taken, as indicated in Appendix B.

2.4.6 Interference Check Sample

The interference check sample (ICS), used in inductively coupled plasma (ICP) analyses, contains interfering and analyte elements of known concentrations. The ICS is used to verify background and inter-element correction factors, and is run at the beginning and end of each run sequence.

2.4.7 Method Blank

A method blank is an analyte-free matrix to which all reagents are added in the same volumes or proportions as those used in sample processing. The method blank is carried through the complete sample preparation and analytical procedure. The method blank is used to document impacts resulting from the analytical process and will be included in each analytical batch. The presence of analytes in a method blank at concentrations equal to or greater than the RL indicates the need for corrective action (Appendix B).

2.4.8 Equipment Blank

An equipment blank is a sample of American Society for Testing and Materials (ASTM) Type II reagent-grade water poured into, poured over, or pumped through the sampling device; collected in a sample container; preserved; and transported to the laboratory for analysis. Equipment blanks are used to assess the effectiveness of equipment decontamination procedures, and will be collected as specified in the FSP.

2.4.9 Trip Blank

The trip blank consists of a VOC sample vial filled in the laboratory with ASTM Type II reagent-grade water, transported to the sampling site, handled like an environmental sample, and returned to the laboratory for analysis. Trip blanks are not opened in the field. Trip blanks are prepared only when VOC samples are collected and are analyzed only for VOCs.

Trip blanks are used to assess the potential introduction of constituents from sample containers or during the transportation and storage procedures. A trip blank is included in each cooler containing samples to be analyzed for VOCs.

2.4.10 Field Duplicate

A field duplicate sample is a second sample collected at the same location as that of the original sample. Duplicate samples are collected simultaneously or in immediate succession, using identical recovery techniques, and treated in an identical manner during storage, transportation, and analysis. The sample containers are assigned a unique identification number in the field to hide their identity from the laboratory. Duplicate samples are used to assess precision of the sample collection process, and will be collected as specified in the FSP.

2.5 QUALITY CONTROL PROCEDURES

2.5.1 Holding Time Compliance

It is the responsibility of each laboratory associate processing a sample to ensure that holding times are met. The laboratory is responsible for meeting all holding times for properly preserved samples received within 48 hours of collection or if less than half the holding time has passed. If these conditions are not met, the laboratory will attempt to expedite sample analysis as soon as possible. When holding times are exceeded, the laboratory will identify and document the root cause of the holding time violation. Sample holding times are listed in Table 2-3.

2.5.2 Confirmation

Qualitative confirmation of results at or above the RL for samples analyzed by GC will be required and will be completed within the method-required holding times. For GC methods, a second column is used for confirmation.

2.5.3 Control Charts

Control charts are used to track the performance of LCS recoveries over time. Control charts are prepared and maintained by the laboratory as discussed in the individual LQMs (Appendix A).

2.5.4 Standard Materials

Standard materials used in calibration and to prepare samples will be traceable to a National Institute of Standards and Technology (NIST), USEPA, American Association of Laboratory Accreditation (A2LA),

or other equivalent AFCEE-approved source. The standard materials will be current and will not exceed expiration dates. Laboratory procedures for documenting standard materials are described in the LQMs (Appendix A).

A second source standard is used to independently confirm initial calibration. A second source standard is a standard purchased from a vendor that is different from the vendor supplying the material used in the initial calibration standards. The second source material can be used for the continuing calibration standards or for the LCS (but will be used for only one of the two). Two different lot numbers from the same vendor do not constitute a second source.

2.5.5 Supplies and Consumables

The laboratory will inspect supplies and consumables before their use in analysis. The materials description in the methods of analysis will be used as a guideline for establishing the acceptance criteria for these materials. Purity of reagents will be monitored by analysis of LCSs. An inventory and storage system for these materials will ensure use before manufacturers' expiration dates and storage under safe and chemically compatible conditions. The laboratories' procedures may be reviewed using the LQMs in Appendix A.

3.0 SAMPLING PROCEDURES

This section presents the laboratory procedures for sample receipt condition verification, handling requirements/sample storage, intra-laboratory custody requirements, corrective actions for incoming samples, and analytical parameter holding times.

3.1 SAMPLE CONTAINERS, VOLUMES, TYPES, AND PRESERVATION

The sample containers, sample volume, method of preservation, and holding times for the laboratories are presented in Table 2-3. Sample receipt, handling, and custody procedures are presented in the LQMs in Appendix A.

3.2 SAMPLE HANDLING AND CUSTODY

Samples received by the laboratory will be logged by a designated sample custodian or other properly trained associate. The laboratories will assign a unique identification code to each sample container received. Sample receipt protocols and storage conditions include the following:

- Determine whether the temperature requirement has been maintained during shipment. If the temperature is not between 0° and 6° Celsius (C), notify the field crew, site manager, and senior chemist immediately. Document the shipping container temperature on the C-C (Figure 3-1).
- Compare samples received to those listed on the C-C.
- Verify that sample holding times have not been exceeded.
- Examine all shipping records for accuracy and completeness.
- Sign each C-C, record the date and time of sample receipt immediately (only after the shipment is accepted), and attach the waybill.
- Note any problems associated with the coolers and samples on the cooler receipt form. Check sample preservation (if no notation is recorded on the C-C of a field check); determine the sample pH, if required; and record on the cooler receipt form (Figure 3-2). If preservation is not within prescribed limits, notify the field crew and the site manager immediately.
- Log each sample into the master logbook and computer file.

- Record sample numbers (from the master logbook) on each sample container, attach durable (water-resistant) laboratory sample container labels with the unique laboratory identification number, and test.
- Place the samples in proper laboratory storage.

3.2.1 Sample Log-in

The samples are entered in the laboratory sample log-in book and/or the Laboratory Information Management System, which contain the following information at a minimum:

- Project name or identification number
- Unique sample numbers (client and internal laboratory)
- Type of samples
- Required tests
- Date and time of laboratory receipt of samples
- Field identification supplied by field personnel

Notify the project manager and appropriate group/team leaders of sample arrival, and place the completed C-Cs and waybills and any additional documentation in the project file.

3.2.2 Sample Custody

Sample custody in the field begins with labeling each sample container, collecting and preserving the samples, and packaging samples for shipment to the designated laboratory. Proper documentation of field samples includes completing the logbook, the Field Sampling Report (FSR) for each sample, and the C-C record for each sample shipment.

A Request for Analysis form (Figure 3-3) will accompany the samples during shipment to the laboratories. The field chemist or field leader will retain a copy of the C-C and contact the laboratories daily to verify that samples were received intact and properly preserved.

3.2.3 Laboratory Sample Custody Records

All incoming samples must be accompanied by a C-C record and a Request for Analysis form completed in the field. If these forms do not accompany the incoming samples, the laboratory sample custodian will inform the laboratory project manager, who will contact the MACTEC site manager and/or project chemist for corrective action. An example of a C-C form is presented as Figure 3-1.

The sample custodian will enter the laboratory and test setup information into the computer. The laboratory will generate an intra-laboratory C-C and a sample confirmation for all samples submitted. Upon preparation and/or analysis, the associate removing the sample from storage will sign the sample out or in (date and signature) and log the tests to be performed.

3.2.4 Sample Storage

Once samples have been logged in, sample control personnel are responsible for placing the samples in the proper storage environment. Procedures specific to the laboratories can be found in the LQMs (Appendix A). The laboratories will assign certain individuals the duty of notifying the group/team leaders or their designees when samples must be analyzed immediately because of holding time requirements. The primary considerations for sample storage are:

- Maintaining the sample at the method-required temperature, if necessary
- Maintaining sample integrity through adequate protection from constituents from outside sources or from cross-contamination between samples

3.2.5 Sample Security and Tracking

The laboratory will maintain the integrity of the samples received, their associated extracts, and the data generated. Limited and controlled access to all laboratory areas will be maintained.

To receive samples from the custody room, the analyst will complete the applicable portion of the intra-laboratory C-C. The analyst is responsible for maintaining custody of samples during analysis.

The analyst will return the samples to the custody room when analyses are completed. Samples will be maintained in the custody room during non-duty hours unless analyses are complete and the sample is to be discarded. When the samples are returned, the analyst will complete the sample custody log entry. The intra-laboratory C-C will be maintained in the project file.

Samples and the associated extracts will be stored for at least 30 days after receipt of the final data report for those samples.

3.2.6 Corrective Actions for Incoming Samples

This subsection describes the laboratories' corrective action procedures. The laboratories' specific corrective action procedures are summarized in the LQMs located in Appendix A.

A Sample Receiving Checklist or equivalent form/system is generated by the laboratory sample control section during the log-in process to document anomalies identified upon receipt of samples in the laboratory. These anomalies are outside laboratory control and do not require corrective actions to be taken within the laboratory. The laboratory sample custodian will inform the laboratory project manager, who will initiate a telephone call to the MACTEC site manager or project chemist for corrective action. The laboratory project manager is responsible for resolving all sample receipt issues (such as how to proceed with the analysis of compromised samples and documenting the decision to do so) with MACTEC's site manager or project chemist. Discrepancies recorded on the Sample Receiving Checklists must be resolved before sample preparation and analysis. The completed Sample Receiving Checklist is stored in the laboratory project file. The laboratory report narrative will include an explanation of any sample receipt anomalies and corresponding corrective actions.

4.0 SCREENING AND DEFINITIVE ANALYTICAL METHODS AND CALIBRATION

The following subsections identify and describe the analytical methods utilized for the DDMT program and the calibration procedures followed in each method. The analytical methods utilized for DDMT were selected to meet the overall DQOs and were obtained from the following sources:

- *Test Methods for Evaluating Solid Wastes*, USEPA SW-846, third edition and updates (USEPA, 1996)
- *Method for Chemical Analysis of Water and Wastes* (MCAWW; USEPA, 1983)
- *Annual Book of ASTM Standards*, Vol. 04.08, Section 4 (ASTM, 1986)
- 40 CFR 261, 1 July 1991 (FR, 1991)
- *Standard Methods for Examination of Water and Wastewater*, 16th, 17th, 18th, and 20th editions (American Public Health Association [APHA] *et al.*, 1980, 1989, 1992, 1999)
- *Sample Preparation and the Determination of Dissolved Gases in Water by Using GC Headspace Equilibrium Technique,* STL SOP No. COI-GC-005 (USEPA RSK SOP-175-Modified), 10 September 2001 (Appendix C; STL 2001)
- *Requirements for the Preparation of Sampling and Analysis Plans,* EM200-1-3 (USACE, 2001)

Table 4-1 lists the analytical methods that may be utilized for each environmental activity per matrix. The following subsections describe the methods and justify their selection based on the DDMT program DQOs.

4.1 VOLATILE ORGANIC COMPOUNDS BY GAS CHROMATOGRAPHY/MASS SPECTROMETRY

VOCs in soil, groundwater, effluent water, and associated QC samples will be analyzed by USEPA Method 8260B. Method SW-8260B was selected to provide lower RLs for the analytes, comparability to the screening levels, and consistency with source constituents previously identified.

VOCs in soil samples will be prepared for analysis by USEPA Method 5035A. The samples will be collected using 5- or 25-gram Encore™ samplers. In Method 5035A, a 5-gram aliquot of soil is placed into a pre-weighed VOC vial with sodium bisulfate (for low-level analysis) or methanol (for high-level analysis) and a stir bar upon receipt at the laboratory. In lieu of sodium bisulfate preservation, soil

samples can also be frozen upon receipt at the laboratory and placed into organic-free deionized water for low-level analysis. The preserved sample vial is placed, unopened, into a purge and trap carousel in which organic-free water, ISs, and surrogates are added. The vial containing the sample is heated to 40°C, and the volatiles are purged onto an appropriate trap using an inert gas combined with agitation of the sample.

VOCs in groundwater and effluent water samples will be prepared for analysis by USEPA Method 5030B. In Method 5030B, a 5- or 25-milliliter aliquot of an aqueous sample is withdrawn from the sample vial. ISs and surrogates are added, and the sample is placed into a purge vessel. The VOCs from the sample are purged and captured using a purge and trap apparatus. The VOCs are then desorbed from the trap into the GC, where they are qualitatively separated and quantitatively detected with a mass spectrometer. The laboratory SOPs for the VOC and preparation methods are included in Appendix C.

4.2 SEMIVOLATILE ORGANIC COMPOUNDS BY GAS CHROMATOGRAPHY/MASS SPECTROMETRY

Semivolatile organic compounds (SVOCs) include acid and base-neutral organic compounds and are analyzed using USEPA Method 8270C. Method SW-8270C was chosen to test selected effluent samples for compliance with the Interim RA discharge permit requirements and soil confirmation samples for compliance with remediation goals for the Dunn Field disposal sites.

The samples are extracted using USEPA Method 3520C (continuous liquid-liquid extraction) for waters. In Method 3520C, the water sample is placed into a continuous liquid-liquid extractor; the sample is adjusted, if necessary, to a specific pH; surrogates are added; and the sample is extracted with methylene chloride for 18 to 24 hours. In Method 3550B, sample preparation includes weighing a well-mixed 30-gram aliquot of soil with anhydrous sodium sulfate and then solvent extracting the soil three times using the ultrasonic technique. The soil extract is separated from the sample by vacuum filtration. The extract for liquids and soils is then dried with sodium sulfate and concentrated.

ISs are added to the sample extract, which is directly injected into a GC, in which the SVOCs are qualitatively separated and quantitatively detected with a mass spectrometer. The laboratory SOPs for the SVOC and preparation methods are presented in Appendix C.

4.3 ORGANOCHLORINE PESTICIDES AND POLYCHLORINATED BIPHENYLS BY GAS CHROMATOGRAPHY WITH SECOND COLUMN CONFIRMATION

Organochlorine pesticides and polychlorinated biphenyls (PCBs) will be analyzed by USEPA Methods 8081A (pesticides) and 8082 (PCBs). USEPA Methods 3520C for water and 3550B for soil will be the preparation methods.

USEPA Methods 8081A and 8082 are GC methods, in which the sample extract is injected into a GC using the solvent flush technique, and the compounds in the GC effluent are detected by an electron capture detector (ECD). Results detected at or above the RL are confirmed by second column identification. The laboratory SOPs for the pesticide, PCB, and preparation methods are included in Appendix C.

4.4 CHLORINATED HERBICIDES BY GAS CHROMATOGRAPHY WITH SECOND COLUMN CONFIRMATION

Analyses for herbicides will be performed using USEPA Method 8151A. USEPA Methods 3520C for water and 3550B for soil will be the preparation methods.

This method is performed by hydrolyzing the chlorinated esters with potassium hydroxide. The sample is then acidified and extracted with solvent. The acids are converted to methyl esters using diazomethane. The esters are determined by employing an ECD or equivalent, and the results are reported as acid equivalents. Results detected at or above the RL are confirmed by second column identification. The laboratory SOPs for the herbicide method are presented in Appendix C.

4.5 MERCURY BY COLD-VAPOR ATOMIC ABSORPTION

Mercury will be analyzed in the samples collected during the investigations using cold-vapor atomic absorption (CVAA) following SW-7470A and SW-7471A for water and soil samples, respectively. Mercury is a biotoxic metal, and its concentration may have effects on microbial degradation of constituents.

The method is performed by digesting a sample with potassium permanganate and potassium persulfate. The mercury is then reduced to an elemental state. The elemental mercury is aerated from the solution, and the mercury vapor content is measured as it passes through a cell positioned in the light path of an

atomic absorption spectrophotometer. The laboratory SOPs for the mercury analysis are included in Appendix C.

4.6 METALS BY INDUCTIVELY COUPLED PLASMA

Metals analyses for groundwater, soil, and effluent water samples performed by ICP will follow SW-6010B. The concentrations of metals in water, particularly iron, will aid in the estimation of mass of constituents lost to biodegradation through iron reduction (*Technical Protocol for Implementing Intrinsic Remediation with Long-term Monitoring for Natural Attenuation of Fuel Contamination Dissolved in Groundwater* [Wiedemeier *et al.*, 1995]). Metals analysis for soils is required for confirmation of excavation at disposal sites.

Water samples will be acid digested using Method 3005A. Soil and sediment samples will be digested by Method 3050B. ICP analysis uses a radio-frequency ICP to produce element-specific atomic-line emission spectra. The spectra are dispersed by a grating spectrometer, and the intensities of the lines are monitored by photomultiplier tubes. The laboratory SOPs for Methods SW-6010B, 3005A, and 3050B are included in Appendix C.

4.7 METHANE, ETHANE, ETHENE, AND CARBON DIOXIDE

The presence of methane, ethene, ethane, and carbon dioxide will be determined by STL-LA SOP No. COI-GC-005 (STL, 2001), based on the paper *Dissolved Oxygen and Methane in Water by a Gas Chromatography Headspace Equilibration Technique* (Kampbell *et al.*, 1989). Method AM20Gax/SOP COI-GC-005, which has also been called "USEPA RSK SOP-175, Modified," may be found in Appendix C. This method was selected to help characterize the conditions of the groundwater for constituent fate and transport evaluation. Methane in groundwater is indicative of strongly reducing conditions and microbial degradation. Reducing conditions are conducive to dechlorination of polychlorinated ethenes to monochloroethene (vinyl chloride). This method may be used to determine the concentration of certain gases (i.e., methane, ethene, ethane, and carbon dioxide) dissolved in aqueous samples.

The water sample is collected in a 40-milliliter VOC vial free of headspace. A headspace is generated in the laboratory by replacing 10 percent of the water sample with high-purity helium. The sample bottle is agitated for 5 minutes, and a sample of the headspace is collected and injected onto a GC system, where the gaseous COCs are separated and detected by a flame ionization detector or thermal conductivity

detector. The concentration of the dissolved gas in the original water sample is determined using Henry's Law, the headspace concentration of the gas, the bottle volume, and the temperature of the sample.

4.8 TOTAL ORGANIC CARBON

Total organic carbon (TOC) in the sample will be determined by USEPA Method 9060M. Method 9060M was selected to help characterize the conditions of the groundwater for constituent fate and transport evaluation. Knowledge of the TOC content of the aquifer is important in sorption and solute retardation calculations (Wiedemeier *et al.*, 1995). This method measures carbonaceous materials in liquid samples. TOC in soil samples will be determined by the Walkley Black method (ASTM 2974-8D) or equivalent method. The laboratory SOPs for the TOC analyses are included in Appendix C.

4.9 BROMIDE, CHLORIDE, NITRATE, NITRITE, AND SULFATE

Analyses of anions (bromide, chloride, nitrate, nitrite, and sulfate) will be performed by USEPA 300.0/SW-9056. Anions were selected to help characterize the conditions of the groundwater for constituent fate and transport evaluation. Chloride is measured to ensure that groundwater samples are representative of the water constituting the saturated zone in which the dissolved constituents are present (Wiedemeier *et al.*, 1995). Nitrate concentrations are used to estimate the mass of constituents that can be biodegraded by denitrification processes, and sulfate concentrations are used as an indicator of anaerobic degradation (Wiedemeier *et al.*, 1995).

Method 300.0/SW-9056 identifies and quantitates anions by ion chromatography (IC). A 2- to 3-milliliter aliquot of sample is injected into a stream of carbonate-bicarbonate eluent and pumped through three exchange columns, where the anions are converted to their corresponding acids, into a conductivity detector. The laboratory SOPs for the anion analyses are presented in Appendix C.

4.10 ALKALINITY

Alkalinity analysis will be performed by MCAWW Method 310.1. Alkalinity was selected to help characterize the conditions of the groundwater for constituent fate and transport evaluation.

Method 310.1 identifies and quantitates alkalinity by titration. The aliquot of sample is titrated with sodium carbonate and sulfuric acid to an endpoint pH of 4.5. The laboratory SOPs for the alkalinity analysis are presented in Appendix C.

4.11 SULFIDE

Sulfide analysis will be performed by MCAWW Method 376.1. Sulfide was selected to help characterize the conditions of the groundwater for constituent fate and transport evaluation.

Method 376.1 identifies and quantitates sulfide. Sulfide is precipitated from the sample by the addition of zinc acetate, then oxidized with iodine into sulfur. The laboratory SOPs for the sulfide analysis are presented in Appendix C.

4.12 VOLATILE FATTY ACID

Volatile fatty acid (VFA) analysis will be performed by Standard Method 5560. VFAs were selected to help characterize the conditions of the groundwater for sodium lactate distribution and for constituent fate and transport evaluation.

Method 5560 identifies and quantitates VFAs by IC. A 2- to 3-milliliter aliquot of sample is injected into a stream of carbonate-bicarbonate eluent and pumped through an ion exclusion column, where the anions are converted to their corresponding acids, into a conductivity detector. The STL SOP for the VFA analysis is presented in Appendix C.

4.13 TOXICITY CHARACTERISTIC LEACHING PROCEDURE EXTRACTION

Disposal samples will be extracted by toxicity characteristic leaching procedure (TCLP) using SW-846 Method 1311 before analysis. TCLP extraction involves the separation of any liquid present from the solid sample, and extraction of the solid sample with an amount of extraction fluid, consisting of glacial acetic acid and, in some cases, sodium hydroxide, equal to 20 times the weight of the solid sample. The extraction fluid is a function of the alkalinity of the solid sample. A special zero headspace extractor vessel is used for the VOC analytes. The liquid initially separated from the solid sample is then combined with the extract (if compatible) and analyzed for the fractions requested according to the appropriate SW-846 method (e.g., 8260B or 6010B).

The disposal samples occasionally may require characterization for reactivity, ignitability, and/or corrosivity. The laboratory SOPs for determining these characteristics are presented in Appendix C.

4.14 FIELD CHEMICAL TEST METHODS

Field chemical test parameters include pH, temperature, specific conductance, DO, ORP, ferrous iron, carbon dioxide, and turbidity. These parameters were selected to acquire knowledge of the groundwater's natural condition and its ability to degrade constituents.

The field measurements of pH, temperature, and specific conductance will be used to determine the presence and activity of microbial populations, the solubility of oxygen and other geochemical species, and the ability of the water to conduct electricity, respectively (Wiedemeier *et al.*, 1995). DO concentrations are used to estimate the mass of constituents that can be biodegraded by aerobic processes (Wiedemeier *et al.*, 1995). ORP is an indicator of the relative tendency of a solution to accept or transfer electrons. Ferrous iron is an indicator of anaerobic biodegradation, in which ferric iron acts as an electron acceptor. Carbon dioxide concentrations are used to estimate the mass of constituents that can be biodegraded by anaerobic condition processes. Turbidity is used to measure the amount of suspended particles within groundwater.

4.15 CALIBRATION PROCEDURES AND FREQUENCY

Calibration of instruments and support equipment is required to ensure that the analytical system is operating correctly and functioning with the proper precision, bias (accuracy), and sensitivity. The frequencies of calibration and calibration verification are presented in the LQMs (Appendix A), are based on the various analytical methods and industry standards, or may be based on project-specific DQOs. Calibration of equipment is performed with physical and chemical reference standards, as presented below.

4.15.1 Physical Reference Standards

Physical reference standards associated with periodic calibrations include weights for calibrating scales/balances and certified thermometers for calibrating working thermometers. Whenever possible, physical reference standards will be calibrated by a body that can provide traceability to nationally or internationally recognized standards.

4.15.2 Chemical Reference Standards and Reagents

This subsection describes chemical reference standards and reagents. Chemical reference standards are generally associated with operational calibration. These standards include reference materials traceable to recognized standard suppliers. These may include vendor-certified materials traceable to national or international standard reference materials (e.g., NIST or A2LA). Reagents, including critical solvents and acids used for sample preparation and/or analysis, are subjected to internal evaluation at the laboratory. The laboratory chemical reference standards and reagents program involves chemical testing on a lot-by-lot basis. This program is discussed in the LQMs located in Appendix A.

4.15.3 Standard Verification

Standards are verified by quantitative comparison to a second known standard before data are reported. The standard must meet specified QC criteria for the independent/second source initial calibration verification.

4.15.4 Operational and Continuing Calibration Procedures

The calibration procedures, preparation of calibration standards, and frequency of initial and continuing calibration checks for each laboratory are described for each analytical method in the following subsections, and are presented in Appendix B. At a minimum, each instrument and other equipment used by a laboratory must be calibrated and maintained at the recommended intervals prescribed by the analytical method employed. In those cases where it has been demonstrated that more frequent calibration or maintenance is required, the base method will be enhanced as necessary.

Initial Calibration Curve – An analytical instrument is calibrated when an instrument response can be related to the concentration of an analyte. This relationship may be depicted graphically and referred to as a "calibration curve." Initial calibration curves must be established based on the requisite number of standards identified within the method for each target analyte (and surrogates for organics). The RLs will be established by the laboratory at the low standard for each target analyte. All reported concentrations for target analytes will be within the high and low initial calibration standards. Data generated below the low standard will be reported as estimated (J-flagged) values. Data generated above the high standard will be diluted within the calibration range and reanalyzed. The frequency requirements for the initial calibration vary among the individual methods and are presented in the following subsections.

VOCs and SVOCs by GC/Mass Spectrometry – Calibration for GC/mass spectrometry will be performed according to SW-846 methodology and the instrumentation manufacturers' recommendations, as described in the laboratory SOPs. GC/mass spectrometry compounds will be calibrated using five standards that bracket the linear range of the detector, or six standards if quadratic fit is to be used. The GC/mass spectrometer will be tuned to meet ion abundance criteria provided in Table 4-2 for 4-bromofluorobenzene for Method SW-8260B, and Table 4-3 for decafluorotriphenylphosphine for Method SW-8270C.

If the percent RSD of any compound (other than calibration check compounds – RSD less than or equal to 30 percent) is greater than 15 percent, calibration curves of area ratio versus concentration may be constructed using first-order regression fit of the five calibration points. The correlation coefficient must be greater than or equal to 0.995. If second- or third-order regression is used, six data points for second order and seven data points for third order will be used for calibration only if individual correlation coefficients are ≥0.990.

ISs for GC/mass spectrometry volatiles will be added to each sample, standard, LCS, MS/MSD, and method blank. The VOC ISs are as follows:

- Fluorobenzene
- Chlorobenzene-d_5
- 1,4-Dichlorobenzene-d_4

Table 4-4 presents the volatile analytes quantitated by each IS.

ISs for GC/mass spectrometry semivolatiles will be added to each sample, standard, LCS, MS/MSD, and method blank. The SVOC ISs are as follows:

- 1,4-Dichlorobenzene-d_4
- Naphthalene-d_8
- Acenaphthene-d_{10}
- Phenanthrene-d_{10}
- Chrysene-d_{12}
- Perylene-d_{12}

Table 4-5 presents the semivolatile analytes quantitated by each IS.

Organics by GC with Second Column Confirmation – The GC calibration will follow SW-846 methodology and instrument manufacturers' recommendations, as described in the STL SOPs. Calibration consists of five standards that bracket the linear range of the detectors. The concentration of the lowest standard will be at or below the project RL. Second column confirmation is required for samples that exhibit a positive result at or above the RL, except for multi-component compounds (toxaphene, chlordane, and PCBs). The confirmation system must contain a dissimilar column and is calibrated and subject to the same QC as the primary GC system. Data from both analyses will be reported, and the most reliable of the two results will be identified. The two results will not be averaged.

Before calibration, retention time windows for each standard on each GC column are determined whenever a new GC column is installed. The following procedure is used to establish retention time windows. Three injections of each standard are made over a 72-hour period at approximately equal intervals. The standard deviation is calculated from the three absolute retention times. For multi-response analytes, one major peak is chosen from the chromatographic profile for the retention time study. Retention time windows for each analyte are updated at least daily and are determined using the continuing calibration analyte retention time plus or minus three times the standard deviation determined in the study. If the retention time window for an analyte is too restrictive or zero, a retention time window width of 0.03 minute will be used.

CVAA Mercury – Mercury analysis by CVAA will follow SW-7470A and SW-7471A calibration criteria with five standards and manufacturers' recommendations, as outlined in the laboratory SOP. This includes a daily multi-point calibration run before sample analysis and calibration checks analyzed after every 10 samples. Instrument setting and alignment will be optimized by maximizing the energy setting. The cell will be aligned by minimizing the absorbency reading.

ICP and ICP "Trace" Metals – Inorganic analyses for metals may be performed using ICP "trace" methods. For metals by the "trace" ICP method, the calibration is identical to standard ICP calibration and follows manufacturers' recommendations and SW-6010B methodology, as detailed in the laboratory SOP. The instrument must be calibrated daily with one standard and a blank, or once every 24 hours, and each time the instrument is set up. Calibration checks will be analyzed at a frequency of every 10 samples. The instrument operation is verified by checking the automatic gain setting and optical alignment.

The initial calibration for ICP "trace" metals analysis must be established by adhering to the following:

- Calibration – Perform the initial calibration with a high-level standard and a calibration blank. The concentration of the single standard establishes the linear calibration range and must fall below the upper linear dynamic range of the instrument. To ensure accuracy of concentrations at the RL, verification of a low-level standard is prepared from the primary source standard and results must be within ±20 percent of its expected value. If the 20 percent criterion cannot be consistently met, the concentration of the daily low-level continuing calibration verification standard (and associated RLs) should be increased until compliance is attained.
- Standard Preparation – The standard may be a "mixed" solution, meaning it contains all the metals of interest (as long as the metals are compatible), or a set of standard solutions, where each standard contains a subset of the compatible metals of interest.

Methane, Ethane, Ethene, and Carbon Dioxide – Calibration of methane, ethane, ethene, and carbon dioxide will follow the laboratory method criteria. The SOP is presented in its entirety in Appendix C. Dissolved gas compounds will be calibrated using five standards that bracket the linear range of the detector. Each initial calibration must have an RSD less than 25 percent for each compound.

TOC – TOC analysis will follow SW-9060/EPA415.1 calibration criteria and manufacturers' recommendations, as described in the laboratory SOP. Areas of at least three injections of a primary standard are read, averaged, and set to the true value of the standard before a multi-point calibration is run. An independent standard is analyzed to check the validity of the calibration, and calibration checks are analyzed after every 10 samples.

Bromide, Chloride, Nitrate, Nitrite, and Sulfate – Anion analysis will follow SW-9056/USEPA 300.0 method calibration and the IC calibration requirements, as outlined in the laboratory SOPs. The instrument must be calibrated daily with at least 3 standards, and calibration checks analyzed after every 10 samples, as described in Appendix B. The eluent flow rate on the IC is adjusted to 2 milliliters per minute (mL/min) to achieve ion separation. The detector offset is adjusted to zero based on effluent eluent conductivity, and the regeneration flow rate (usually 2.5 to 3 mL/min) is adjusted with the fiber or membrane suppresser to maintain stability. The anions are identified by retention time as compared to standards and quantitated by measurement of peak area or peak height.

Wet Chemical Test Methods – Analytical systems for wet chemistry techniques will be calibrated or standardized before sample analysis. The calibration consists of defining the working range by use of a

series of standard solutions. The calibration will be verified on an ongoing basis (every 10 to 20 samples minimum, and at the end of the analytical sequence) to ensure that the system remains within specifications. The titrimetric wet chemistry techniques will use a primary standard to verify the standardization of the titrant. Calibration and standardization for wet chemistry test methods are discussed in the paragraphs below.

Alkalinity – The alkalinity calibration will follow USEPA 310.1 methodology, as described in the laboratory SOP. The pH meter is calibrated with two standards, and the titrant is standardized. Calibration checks are analyzed after every 10 samples.

Sulfide – The sulfide calibration will follow USEPA 376.2 methodology, as detailed in the laboratory SOP. The sodium thiosulfate and iodine titrant standards are standardized.

VFAs – The VFA calibration will follow Standard Method 5560, as outlined in the laboratory SOP. Calibration checks are analyzed after every 10 samples.

4.16 LABORATORY QUALITY CONTROL PROCEDURES

Laboratory overall method performance will be monitored by the inclusion of various internal QC checks that allow an evaluation of method control (batch QC) and the effect of the sample matrix on the data being generated (matrix-specific QC). The overall quality objectives are to implement procedures for the laboratory analysis and reporting of data that are indicative of the degree of quality consistent with their intended use. Laboratory QC samples consist of method blanks, instrument blanks, LCSs, and calibration verification samples. In addition to laboratory performance QC, matrix-specific QC is utilized to determine the effect of the sample matrix on the data being generated. It generally includes MS/MSDs, sample duplicates, and the use of surrogate compounds. Laboratory QC samples, acceptance criteria, and corrective actions by reference methods for inorganic and organic methods are presented in the LQMs located in Appendix A. The following subsections identify the specific internal QC measures to be used by the laboratory when performing the analytical tests.

4.16.1 Analytical Sequence Quality Control

Tables I-1 through I-8 of USACE, 2001, (Appendix D) include summaries of the QC samples to be included with each analytical sequence for seven of the SW-846 methods.

4.16.2 Batch/Matrix-specific/Performance-based Quality Control

Laboratory QC samples are added to the normal sample stream to demonstrate that the laboratory is operating within prescribed requirements for accuracy and precision. The type and frequency of specific laboratory QC samples depend on the specified analytical method. In general, SW-846 recommends that blanks, blank spikes (LCSs), and sample spikes (MS/MSDs) be analyzed at a frequency of one per batch. A batch is defined as samples that are analyzed together with the same method sequence, the same lots of reagents, and the same manipulations common to each sample within the same time period or in continuous sequential time periods. The laboratory LQMs presented in Appendix A contain details of batch-specific QC.

5.0 DATA REDUCTION, REVIEW, VERIFICATION, REPORTING, VALIDATION, AND RECORD KEEPING

The following subsections describe how the data are reduced by the laboratory and the procedures for calculation of the data quality indicators (DQIs). These DQIs consist of precision, accuracy, limits of reporting (MDLs and RLs), and completeness.

5.1 DATA REDUCTION

Computerized data stations are present for each major analytical instrument. Most data reduction is performed at the data station associated with a particular piece of equipment. The analyst performs the analysis and enters the data on the parameter bench sheet and corresponding data station(s). Bench sheets contain all necessary information to establish sample identity, integrity, calibration evaluation, analytical observations, and results to process and validate the sample test data. A bench sheet key provided to the analyst specifies how information is to be recorded (e.g., notation and significant figures), the data reduction formula, and the QC samples required and their control criteria. Calculations are performed by the data station at each instrument and/or specialized software utilized by the Management Information Systems Department. The protocols used for rounding and significant digits for numerical data are in accordance with the USEPA 600/4-79/019 publication *Handbook of Analytical Laboratory Quality Control in Water and Wastewater Laboratories* (USEPA, 1979).

Sample quantitation will be performed based on the formulas listed in the laboratory method SOPs presented in Appendix C. Laboratory soil calculations have been modified to report results in µg/kg for organic analysis and mg/kg for inorganic analysis. Calibration factors will be determined from the initial calibration.

5.1.1 Organic Analysis

The formulas used for external and internal calculations are presented in the laboratories' method SOPs. Compounds with calibrations that do not meet the RSD criterion of less than or equal to 15 percent will be quantitated from a calibration curve.

External Standard Method – External standard method of quantitation is usually performed for GC methods such as SW-8081A, SW-8082, and SW-8151A.

Internal Standard Method – The internal standard method of quantitation is usually performed for GC/mass spectrometry methods such as SW-8260B and SW-8270C.

5.1.2 Inorganic Analysis

The formulas used for inorganic analyses, such as metals and wet chemical tests, are presented in the laboratories' method SOPs. Most inorganic compounds are quantitated from a calibration curve. The correlation coefficient must be greater than or equal to 0.995.

Ferric Iron – Ferric iron will be derived from the total iron result obtained from Method SW-6010B and the ferrous iron result obtained in the field or analyzed by the laboratory. The following calculation will be used (APHA *et al.*, 1992):

$$\text{Ferric Iron (mg/L)} = \text{Total Iron (mg/L)} - \text{Ferrous Iron (mg/L)}$$

Where:

Total Iron = Total iron result obtained from SW-6010B
Ferrous Iron = Ferrous iron result obtained from field test kit or laboratory analysis

5.2 DATA QUALITY CONTROL REVIEW

There are various levels of assessment required for screening data and definitive data. The following subsections discuss the requirements of each.

5.2.1 Assessment of Screening Data

Screening level data are typically characterized by less stringent QA/QC procedures. Assessment of screening level data consists of checking available QA/QC indicators and confirming the results with definitive analyses, usually at a 10 percent frequency.

5.2.2 Assessment of Definitive Data

Definitive level data require more stringent QA/QC procedures than screening level data. The following subsections discuss the review elements involved in the assessment of definitive data.

Data Evaluation – The MACTEC senior chemist uses the results of the data review to summarize findings in the DQE case narrative report to determine the usability of the data. The DQE case narrative report lists all potential effects of QA/QC failures on the data, and the senior chemist assesses the impact of QA/QC failures on the attainment of the DDMT DQOs and contract compliance.

Data Qualifiers – Data qualifiers will be applied by the project chemist as appropriate to alert the data user of deficiencies in the data. If any data points require qualification, they will receive data qualifiers as described in Table 5-1. The data associated with compounds/analytes that do not meet initial calibration, continuing calibration, surrogate, and/or LCS criteria will be considered either unusable (flagged "R") or quantitative estimates (flagged "J"), as outlined in the data evaluation SOPs included in Appendix E. If ISs fail criteria (after corrective action is taken), compounds associated with the individual IS or surrogate will be considered either unusable (flagged "R") or estimated (flagged "J"). If sample analysis exceeds holding times, the data will be flagged as estimated ("J") if less than or equal to two times the requirement, or as unusable ("R") if greater than two times the recommended holding time. If the method blank was impacted, the results will be qualified according to MACTEC's DQE SOP as estimated, possibly biased high, or false positive based on blank data ("B"). MS and MSD data will be reviewed and qualified based on all the data available. If several QC limits are exceeded, the associated data will be considered as unusable, flagged "R", and not be used. Estimated data are not necessarily unusable data. Project-specific data such as precision, accuracy, and completeness goals will be reviewed, and the data will be validated subject to these goals. If these goals are not met for C-Cs, resampling and analysis may be necessary.

Data Verification – Definitive data assessment begins at the laboratory. Each laboratory is responsible for ensuring the chemical data generated are of sufficient quality to meet intended uses for the DDMT project. Once the data have met the laboratory's standards, data verification is performed to determine whether the data package is correct and complete.

Data Review – Data review documents the possible effects on the data that result from various QC failures. It does not, however, determine the worth of the data, nor does it include the assignment of qualifier flags. The results of all of the following examinations are reported in the data review:

1. Initial inspection. The laboratory chemists screen the data for errors and inconsistencies, checking C-Cs, sample handling procedures, analyses requested, sample description, sample identification, and cooler receipt forms. The chemists

verify that the data were examined by a laboratory manager or QA officer. Sample holding times and preservation are likewise checked and noted.

2. Examination of actual data. Chemists examine the data from laboratory matrix duplicates, blind duplicates, trip blanks, equipment blanks, LCSs, LCS duplicates (LCSDs), MS/MSDs, surrogate recoveries, and field samples to determine whether the data are of acceptable quality. RPDs for LCSs/LCSDs and MS/MSDs must also meet the data precision requirements of the method. Surrogate recoveries must fall within acceptable method limits, or the data may be qualified as estimated or unusable.

3. Recoveries on LCSs, MSs, and MSDs must indicate that acceptable data have been generated. MS/MSDs should be analyzed at least once per every 20 samples or once per preparation batch, whichever is greater, per day.

Examples of the specific types of reviews performed by MACTEC chemists include the following:

1. Sample Analysis Completeness – Were all samples analyzed? Were samples analyzed for the parameters listed in the SAP and project-specific Work Plans?

2. Evaluation of Holding Times – Were samples analyzed within the specified holding and extraction times?

3. Evaluation of QC – Were standard curves within method control limits? Were preparation or method blanks impacted? Were continuing calibration standards in control? Were formulas and calculations used in analyte quantitation correct? Were the LCS recoveries within QC limits? Was an MS/MSD analysis performed? How did field duplicates compare? Were corrective actions taken where necessary?

4. Establishment of MDLs and RLs – Were RLs met? If not, why?

5.3 DATA VERIFICATION/VALIDATION

The procedures used by MACTEC for data evaluation and validation are described below. The primary DQE is performed by MACTEC's staff or project chemist following the SOPs developed for DDMT. These SOPs are presented in Appendix E. The DQE narrative and qualified (flagged) data tables are reviewed by a senior chemist. The data qualifier flags are described in Table 5-1. The data review process was developed based on reference to the following USEPA and USACE documents:

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

- *USEPA Contract Laboratory Program National Functional Guidelines for Inorganic Data Review*, EPA 540/R-94/013 (USEPA, 1994b)

- *USEPA Region III National Functional Guidelines for Organic/Inorganic Data Review*, EPA 600/R-96/055 (USEPA, 1994c)
- *USEPA Contract Laboratory Program National Functional Guidelines for Low Concentration Organic Data Review*, EPA 540/R-00/006 (USEPA, 2001a)
- *USEPA Contract Laboratory Program National Functional Guidelines for Inorganic Data Review*, EPA 540/R-01/008 (USEPA, 2002)
- USACE, 2001
- Any other method-specific criteria as presented in USEPA, 1996, Update III

The laboratory and field QC data and field notes provide the information to evaluate the analytical data for accuracy, precision, and completeness with respect to the project-specific DQOs. The data are first evaluated based on field notes taken during collection of the samples to assess sampling conditions and sampling procedures, or whether changes to the planned procedures were necessary. Secondly, each sample shipment sent to the laboratory is assessed for adherence to method-prescribed holding times, proper C-C documentation, correct usage of sample containers, and sample integrity upon receipt by the laboratory. Examples of elements reviewed are presented below:

- Field Record Completeness – Were all field analyses performed? Were all samples collected? Were any problems encountered, and how were they resolved? Were all field records complete (e.g., C-C forms, FSRs, and boring logs)?
- Sampling and Decontamination Procedures Review – Were all field duplicates collected? How did they compare? Was a rinsate collected for the sampling event? Did the rinsate show constituents? Were the trip blanks impacted? Did samples arrive intact, properly preserved, and following proper shipping protocol?
- Identification of Valid Samples – Were the samples collected representative of on-site conditions? Were the wells properly constructed, or were adequate amounts of sample available from creeks or streams? Were there sources of potential constituents during sampling?
- Correlation of Field Test Data and Identification of Anomalous Field Test Data – Did data from different methods of measurement correlate?
- Review of the Results of Field QC Samples, Such as Rinsates, Trip Blanks, and Duplicates, Can Help in Assessing Sample Integrity and Precision – The field data and laboratory data will be reviewed and evaluated compared to the DQOs established in this QAPP. Data validation will be performed on all DDMT chemical samples (100 percent).

The laboratory's internal QC procedures for calibration, method validation, and performance evaluation include appraisal of method-prescribed tune (for GC/mass spectrometry) and calibration criteria, method blank analyses, LCS analysis, MS/MSD analyses, and assessment of surrogate and IS recovery where applicable. MACTEC's evaluation of the laboratory data focuses on exceptions to the planned QC activities, problems encountered, and the effectiveness of the methodologies used within the laboratory. The data are then evaluated overall with respect to the project DQOs and evaluated for completeness. The following subsections present the evaluation procedures used for the analytical data with respect to the project-specific DQOs.

5.3.1 Evaluation of Field Data Quality

QC samples are collected to assess the quality and representativeness of the field sampling activities and the accuracy of analytical results from the laboratory. Field QC samples will be collected in accordance with the procedures and protocols in the FSP and in project-specific Work Plans.

The QC samples are collected concurrently with the field samples to assess the accuracy and precision of sampling and analysis. The field QC samples that will be collected will consist of field duplicates, MS/MSDs, trip blanks, field blanks, and rinsates (equipment blanks), as defined in USACE, 2001. The QC samples are collected in the same types of containers as those used for the field samples, and treated in the same manner. The QC samples will be analyzed by the laboratory concurrently with the field samples. QC samples are evaluated for reproducibility where applicable and the impact of blank impacts if present.

Field duplicates are collected to assess sampling precision. They consist of replicate grab samples collected concurrently with the associated field samples. Although not collected at separate field locations, they are considered separate field samples for analytical purposes. Duplicate samples submitted to the laboratory are identified with unique sample codes to hide their identity from the laboratory and are typically referred to as "blind duplicates". Cross-references to the sample's true identity are annotated in field logbooks and Daily Quality Control Reports maintained by field sampling personnel.

Field duplicate samples are collected to meet the frequency of approximately 10 percent established by USACE. Poor precision is represented if, during evaluation of laboratory data, RPDs exceed those as outlined per analysis classification.

Field duplicate RPDs are calculated in a manner similar to that described for MS/MSDs for analytical values that are greater than or equal to the RL.

Trip blank samples are collected to assess whether cross-contamination of water samples collected for analysis of VOCs occurred during sampling and shipment to the laboratories. The trip blanks are placed in the sample shipping container with the aqueous and solid field samples to be analyzed for VOCs.

5.3.2 Evaluation of Laboratory Data Quality

Laboratory data are evaluated to assess adherence to method-prescribed calibration and/or continuing calibration criteria, method blank analysis results, analyte recoveries from LCSs, MS/MSD recoveries and RPDs, surrogate recoveries, and ultimately completeness. Except for completeness, these criteria are used to evaluate the accuracy and precision of the data generated by the laboratory. Furthermore, the USACE-specified control limits for the major USEPA SW-846 methodologies are presented in USACE, 2001, and data are evaluated based on those limits.

In general, control limits not addressed by USACE, 2001, default to laboratory-generated limits. Laboratory-established control limits are based on the mean %R plus or minus 3 standard deviations of the mean using a minimum population of 20 recovery values.

The accuracy of the laboratory data is assessed by consideration of:

- Recovery of spikes from field samples spiked with known amounts (MS and MSD)
- Recovery of surrogate spikes for most analyses by GC
- Recovery of analytes from LCS

To determine precision, duplicates and MS/MSDs are analyzed. The values reported for a spiked sample (MS) and a spiked duplicate (MSD) are used to calculate an RPD. At times, the laboratory may also analyze LCSDs and determine RPD. The control limits are those established by USACE, 2001. Where USACE, 2001, does not address a specific analytical method, the laboratory-established control limits are used. The laboratories' internal control limits are based on a statistical population of at least 20 RPD

values. They are calculated by determining the mean RPD plus three times the standard deviation for the upper limit and zero RPD as the lower limit.

To evaluate completeness, the number of valid data points obtained from the measurement systems is compared to the number that was expected to be obtained under correct or normal conditions. Project objectives stipulate that 90 percent of the data are expected to be valid based on the evaluation of the QC data.

Representativeness in the laboratory can be determined by ensuring that all sub-samples collected from a given sample represent the sample as a whole by premixing and homogenizing. However, overall representativeness is assessed by a review of the precision obtained from field and laboratory duplicate samples.

5.3.3 Data Quality Objective Reconciliation

During the DQO reconciliation process, the final version of the evaluated (and, if necessary, qualified) data is compared to the project DQOs established in the associated Work Plans. Once the analytical completeness for the data has been calculated (number of usable results/total number of results times 100 percent), the completeness value is compared to the project DQOs. If an analyte fails to meet the completeness goal, an evaluation of the impact that the failure will have on the DQOs is made. If the failure is determined to negatively impact the DQOs, resampling will be required to obtain usable replacement data.

5.3.4 Project Completeness Assessment

As discussed in Subsection 5.3.3, completeness is assessed for comparison to the established project DQOs. The project completeness is made up of the analytical completeness (calculated as indicated above) and the field completeness. Field completeness is impacted by problems encountered during the field effort (e.g., dry wells or soil boring refusal). The project is assessed as complete once the completeness goals are met or every reasonable effort has been made to obtain the goal and the dataset cannot be improved with additional sampling and analyses.

5.4 LABORATORY SAMPLE MANAGEMENT RECORDS

These records are the documents that provide objective evidence of the performance of a process or observations of an item. Laboratory sample management records ensure that results produced by the laboratory are scientifically and legally defensible, and that project events can be reconstructed. Further discussion of the laboratory sample management records can be found in the LQMs located in Appendix A.

5.5 DATA REPORTING PROCEDURES

Data will be reported initially by the laboratory, reviewed for data quality, and submitted in technical reports to AFCEE. Requirements for data reporting are presented in the following subsections.

5.5.1 Data Package Format and Contents

Reporting of analytical results for DDMT projects will include environmental and QC sample analysis data in hardcopy format as well as a computer disk containing the data utilizing the MACTEC electronic data deliverable format. Analytical hardcopy reports will contain the following items:

- Case narrative
- C-C records and sample receipt information
- Laboratory name
- Client name
- Date of issue
- Project identification
- Field sample number
- Laboratory sample number
- Sample matrix description
- Analytical method description and reference citation
- Individual parameter results (including second column and primary results where appropriate)
- Date of analysis (extraction initiated and completed, first run, and subsequent runs)

- MDLs and RLs achieved
- Concentration units
- Any special conditions
- Dilution or concentration factors
- Corresponding QC report (see below)

QC data are recorded on the QC report forms for the appropriate tests and correlated to the analytical results by the laboratory lot control numbers. The QC results are used to prepare control charts for each test and matrix type. QC reports will contain the following items:

- Narrative describing any noncompliant samples
- Initial and continuing calibration results
- Tuning results
- Method blanks, preparation blanks, and initial and continuing calibration blanks
- Surrogate results
- MS/MSD results
- LCS results
- IS area counts
- Dilution test and recovery test/post-digestion spike results
- Preparation and analytical run logs

5.5.2 Technical Reports

Analytical results will be included in the technical reports. The data will be presented in tables and appendices within the reports. Tables within the reports may include the following information:

- Sample identification number
- Sampling date
- Sampling depth (if applicable)
- Positive results and nondetect results
- RLs
- Background concentration (naturally occurring or anthropogenic)
- Analytical method number
- Parameter name
- Units of measure
- Explanation of data qualifier flags

Data will also be presented in tables within the appendices and discussed within the DQE case narratives. The appendices will include the following information:

- Data summary tables
- Surrogate recovery results
- LCS results
- MS/MSD results
- Equipment blank, trip blank, and ambient blank results
- Method blank results

5.5.3 Electronic Deliverables

The data will be presented electronically from each laboratory in the format specified by MACTEC. The analytical data will be submitted in an American Standard Code for Information Interchange (ASCII) format compatible with EarthSoft's Environmental Quality Information System (EQuIS) database. In some cases, laboratories responsible for limited or mobile analyses will be asked to submit the data as a Microsoft Excel™ file.

MACTEC will submit the data to AFCEE as a print document format file of the hard copy, the DQE Report forms and narrative, and the final qualified and flagged data summary tables.

5.6 DATA MANAGEMENT PROCEDURES

This subsection describes the project data management process, tracing the path of the data from their generation to their final use and storage (e.g., the field, the office, and the laboratory). The laboratory standard record-keeping procedures are described in the LQMs (Appendix A). Records may be either hardcopy or electronic. The record-keeping system allows for the reconstruction of all laboratory activities that produced the analytical results. Details regarding control of electronic records are provided in the LQMs (Appendix A).

Sample management and data collection activities are closely documented. For example, as a sample enters a specific laboratory for analysis, it is documented by intra-laboratory C-C. The following data flow occurs at each laboratory:

- Extraction and preparation dates for samples, standards, duplicates, spikes, and blanks are entered into bound notebooks.
- Final analytical results and QC data are dated and initialed by the analyst.
- Data calculations, %R, and precision data are checked by the laboratory supervisor, who then initials and dates the output.

- Results are entered into the laboratory computer system by direct entry or electronic transfer.

5.6.1 Laboratory Turnaround Time

In general, the standard turnaround time (TAT) for analytical sample results is 21 days from the day of receipt at the laboratory and 30 days for all deliverables. The TAT may vary depending on project-specific objectives.

5.6.2 Data Archival/Retention Requirements

Following site activities, all project documentation becomes a part of the final evidence file. Records must be retained for at least five years from the date of the completion of all data deliverables.

6.0 PERFORMANCE AND SYSTEM AUDITS

This section describes the performance and system audits that will be performed on-site and at the laboratories. Annual laboratory audits must be conducted internally for each analytical area to verify the following at a minimum:

- Procedures are compliant with SOPs.
- Documentation practices are complete and traceable to a certified source.
- Data reviews are complete, well documented, and effective.
- Data reporting practices, including electronic or manual data transfer and client report generation, are accurate and complete.

All audit findings, any corrective actions, and root cause determinations will be fully documented in QA reports to laboratory management. All necessary corrective actions must be verified complete within a reasonable timeframe. Audits performed by external agencies or accrediting authorities may not substitute for internally conducted laboratory audits. See the "Internal Audits" section of the LQMs (Appendix A) for a description of the internal audit procedures. External audits are regularly performed by regulatory and private accreditation authorities. The available audit reports from the National Environmental Laboratory Accreditation program and USACE will be reviewed annually to ensure laboratory adequacy for continued analysis of project samples.

7.0 PREVENTIVE MAINTENANCE

The laboratory will administer a Preventive Maintenance Plan that will be implemented to minimize downtime of laboratory instruments. Equipment maintenance is the responsibility of the analyst and the department manager. Repairs and/or modifications are recorded on maintenance records. Daily equipment checks include visual and/or manual inspections of cooling fans, pumps, indicator readings, detectors, and gas supplies, and other method-specific inspections. Service schedules are established for performing routine preventive maintenance on all major equipment. The frequency of maintenance must consider manufacturers' recommendations and previous experience. Preventive maintenance occurs as often as every day to as infrequently as once per year, depending on the type and use of the equipment. The laboratories adhere to strict maintenance schedules that include sustaining optimum working order, regular inspection, and necessary cleaning.

The frequencies of preventive maintenance, along with the recommended preventive maintenance schedules, are presented in the LQMs (Appendix A) for analytical instrumentation and equipment. Schedules may also be defined in a laboratory's operation-specific routine maintenance SOPs.

8.0 NONCONFORMANCE/CORRECTIVE ACTIONS

This section addresses notification and corrective actions that should be followed by field and laboratory personnel if there are deviations from the SAP or problems with samples upon receipt at the laboratory. subsection 3.2.6 contains information regarding corrective actions associated with sample receipt, and subsection 3.2.3 contains information regarding corrective actions should a C-C or Request for Analysis form not accompany incoming samples. Significant changes to or deviations from the approved SAP may not be made without the written approval of AFCEE. Section 6.0 of the FSP provides additional information.

Exceedances of matrix-specific QC samples (MS/MSDs) may be problematic because of matrix effect (signal enhancement or suppression) on the analysis, but should not be viewed as an indicator of poor laboratory performance. Necessary corrective actions will vary depending on the type of interference and are subject to analyst professional judgment. When these departures indicate potential for false negatives, lack of sensitivity, or inability to accurately detect the target analyte(s), the analyst will inform the laboratory project manager, who will contact the MACTEC senior chemist for direction regarding finding possible alternatives. Other options, such as taking measures to decrease the matrix effect by such techniques as implementing cleanup procedures, diluting the samples, or processing a smaller amount of sample, may be considered. However, consequences to the data (e.g., higher detection limits or less representative sample aliquot) must be assessed comparatively with project objectives.

9.0 REFERENCES

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TABLES

TABLE 2-1

DATA CATEGORY QA/QC ELEMENTS
REMEDIAL ACTION SAMPLING AND ANALYSIS PLAN
Defense Depot Memphis, Tennessee

Screening Data QA/QC Elements

- Sample documentation (location, date and time collected, batch, etc.);
- Chain-of-custody (when appropriate);
- Sampling design approach (systematic, simple or stratified random, judgmental, etc.);
- Initial and continuing calibration;
- Determination and documentation of detection limits;
- Analyte(s) identification;
- Analyte(s) quantitation;
- Analytical error determination[1]: An appropriate number of replicate aliquots, as specified in the QAPP, are taken from at least one thoroughly homogenized sample. The replicate aliquots are analyzed, and standard laboratory QC parameters (such as variance, mean, and coefficient of variation) are calculated and compared to method-specific performance requirements specified in the QAPP;
- Definitive confirmation; At least 10 percent of the screening data must be confirmed with definitive data as described below. At a minimum, at least three screening samples reported above the action level (if any) and three screening samples reported below the action level (or as non-detects, ND) should be randomly selected from the appropriate group and confirmed.

Definitive Data QA/QC Elements

- Sample documentation (location, date and time collected, batch, etc.);
- Chain-of-custody (when appropriate);
- Sampling design approach (systematic, simple or stratified random, judgmental, etc.);
- Initial and continuing calibration;
- Determination and documentation of detection limits;
- Analyte identification;
- Analyte quantitation;
- QC blanks (trip blank, method blank, rinsate blank);
- Matrix spike recoveries,
- Performance Evaluation (PE) samples (when specified): The laboratory participates in a number of performance testing (PT) programs that submit performance evaluation (PE) samples to the laboratory for analysis at regular intervals. The National Environmental Laboratory Accreditation (NELAC) Program, USEPA Water Pollution, Water Supply and Hazardous Waste Programs submit PE samples to the laboratory semiannually. In addition, the USACE and USN submit PE samples to the laboratory every 18 months. Project-specific PE samples wil be submitted to the laboratory when specified by AFCEE.
- Analytical error determination (measures precision of analytical method): An appropriate number of replicate aliquots, as specified in the QAPP, are taken from at least one thoroughly homogenized sample. The replicate aliquots are analyzed, and standard laboratory QC parameters (such as variance, mean, and coefficient of variation) are calculated and compared to method-specific performance requirements specified in the QAPP;
- Total measurement error determination (measures overall precision of measurement system from acquisition through analysis): An appropriate number of co-located samples as determined by the QAPP are independently collected from the same location and analyzed following standard operating procedures. Based upon these analytical results, standard laboratory QC parameters such as variance, mean, and coefficient of variation are calculated and compared to established measurement error goals. This procedure may be required for each matrix under investigation, and may be repeated for a given matrix at more than one location at this site.

[1]The procedures identified measure the precision of the analytical method, and are required when total measurement error is not determined by definitive confirmation.

Source: Data Quality Objectives Process for Superfund, EPA 540-R-93-071 (USEPA, 1994).

TABLE 2-2

PROJECT DETECTION, REPORTING, AND SCREENING LIMITS
REMEDIAL ACTION SAMPLING AND ANALYSIS PLAN
Defense Depot Memphis, Tennessee

METHOD	COMPOUND	UNITS	REPORTING LIMIT	METHOD DETECTION LIMIT*	DISCHARGE LIMIT	SCREENING LEVEL**
			WATER			
8260B	1,1,1-Trichloroethane	µg/L	1.0	0.21	20	200
	1,1,2,2-Tetrachloroethane	µg/L	1.0	0.22 (1)	1000	0.055
	1,1,2-Trichloroethane	µg/L	1.0	0.22 (1)	100	0.19
	1,1-Dichloroethane	µg/L	1 0	0.21	NL	810
	1,1-Dichloroethene	µg/L	1.0	0.18	100	7 0
	1,2-Dichloroethane	µg/L	1.0	0.16 (1)	NL	0.12
	1,2-Dichloropropane	µg/L	1.0	0 15	NL	5.0
	2-Butanone (MEK)	µg/L	10	0.39	NL	1900
	2-Hexanone (MBK)	µg/L	10	0.35	NL	NL
	4-Methyl-2-pentanone (MIBK)	µg/L	10	0.32	NL	NL
	Acetone	µg/L	10	0.74	NL	610
	Benzene	µg/L	1.0	0.22	NL	0.34
	Bromodichloromethane	µg/L	1.0	0.14	NL	0.18
	Bromoform	µg/L	1 0	0.17	NL	8.5
	Bromomethane	µg/L	1 0	0.36	NL	NL
	Carbon disulfide	µg/L	1.0	0.28	NL	1000
	Carbon tetrachloride	µg/L	1.0	0 19 (1)	40	0.17
	Chlorobenzene	µg/L	1.0	0.2	NL	100
	Chloroethane	µg/L	1.0	0 24	NL	NL
	Chloroform	µg/L	1.0	0 16	200	6.2
	Chloromethane	µg/L	1.0	0.14	NL	NL
	cis-1,2-Dichloroethene	µg/L	1.0	0.21	100	61
	cis-1,3-Dichloropropene	µg/L	1.0	0.12	NL	NL
	Dibromochloromethane	µg/L	1.0	0.19 (1)	NL	0.13
	Ethylbenzene	µg/L	1.0	0.19	NL	2.9
	m,p-Xylenes	µg/L	2.0	0.31	NL	10000
	Methylene chloride	µg/L	1.0	0.19	20	4.3
	Methyl tert-butyl ether (MTBE)	µg/L	5.0	0.18	NL	NL
	o-Xylene	µg/L	1.0	0 14	NL	10000
	Styrene	µg/L	1.0	0.13	NL	100
	Tetrachloroethene	µg/L	1.0	0.19	120	0.66
	Toluene	µg/L	1.0	0.17	40	720
	trans-1,2-Dichloroethene	µg/L	1.0	0.16	100	100
	trans-1,3-Dichloropropene	µg/L	1.0	0.17	NL	NL
	Trichloroethene	µg/L	1.0	0.28	800	0.56
	Vinyl acetate	µg/L	2.0	0.14	NL	NL
	Vinyl chloride	µg/L	1.0	0.21	NL	2.0
8270C	2,4,5-Trichlorophenol	µg/L	10	0.13	NL	NL
	2,4,6-Trichlorophenol	µg/L	10	0.16	NL	NL
	2,4-Dichlorophenol	µg/L	10	0 24	NL	NL
	2,4-Dimethylphenol	µg/L	10	0.23	NL	NL
	2,4-Dinitrophenol	µg/L	50	1.3	NL	NL
	2,4-Dinitrotoluene	µg/L	10	0.16	NL	NL
	2,6-Dinitrotoluene	µg/L	10	0.17	NL	NL
	2-Chloronaphthalene	µg/L	10	0.29	NL	NL
	2-Chlorophenol	µg/L	10	0.14	NL	NL
	2-Methylnaphthalene	µg/L	10	0.028	NL	NL
	2-Methylphenol (o-cresol)	µg/L	10	0.15	NL	NL
	2-Nitroaniline	µg/L	50	0.18	NL	NL
	2-Nitrophenol	µg/L	10	0.14	NL	NL

TABLE 2-2

PROJECT DETECTION, REPORTING, AND SCREENING LIMITS
REMEDIAL ACTION SAMPLING AND ANALYSIS PLAN
Defense Depot Memphis, Tennessee

METHOD	COMPOUND	UNITS	REPORTING LIMIT	METHOD DETECTION LIMIT*	DISCHARGE LIMIT	SCREENING LEVEL**
8270C	3,3'-Dichlorobenzidine	µg/L	50	0.19	NL	NL
(cont'd)	3-Nitroaniline	µg/L	50	0.094	NL	NL
	4,6-Dinitro-2-methylphenol	µg/L	50	1.8	NL	NL
	4-Bromophenyl phenyl ether	µg/L	10	0.29	NL	NL
	4-Chloro-3-methylphenol	µg/L	10	0.18	NL	NL
	4-Chloroaniline	µg/L	10	0.31	NL	NL
	4-Chlorophenyl phenyl ether	µg/L	10	0.21	NL	NL
	4-Methylphenol (p-cresol)	µg/L	10	0.2	NL	NL
	4-Nitroaniline	µg/L	50	0.11	NL	NL
	4-Nitrophenol	µg/L	50	1	NL	NL
	Acenaphthylene	µg/L	10	0.03	NL	NL
	Acenaphthene	µg/L	10	0.028	NL	NL
	Anthracene	µg/L	10	0.03	NL	NL
	Benzo(a)anthracene	µg/L	10	0.028	NL	NL
	Benzo(a)pyrene	µg/L	10	0.022	NL	0.2
	Benzo(b)fluoranthene	µg/L	10	0 043	NL	NL
	Benzo(g,h,i)perylene	µg/L	10	0.044	NL	NL
	Benzo(k)fluoranthene	µg/L	10	0.071	NL	NL
	Benzoic acid	µg/L	50	0.81	NL	NL
	Benzyl alcohol	µg/L	10	1.1	NL	NL
	bis(2-Chloroethoxy)methane	µg/L	10	0.24	NL	NL
	bis(2-Chloroethyl)ether	µg/L	10	0.19	NL	NL
	bis(2-Chloroisopropyl)ether	µg/L	10	0.21	NL	NL
	bis(2-Ethylhexyl)phthalate	µg/L	10	0.36	20	6.0
	Butylbenzylphthalate	µg/L	10	0.14	NL	NL
	Chrysene	µg/L	10	0 035	NL	NL
	Dibenzo(a,h)anthracene	µg/L	10	0.054	NL	NL
	Dibenzofuran	µg/L	10	0.025	NL	NL
	Diethylphthate	µg/L	10	0 12	NL	NL
	Dimethylphthalate	µg/L	10	0 27	NL	NL
	Di-n-butylphthalate	µg/L	10	0.13	60	NL
	Di-n-octylphthalate	µg/L	10	0.16	NL	NL
	Fluoranthene	µg/L	10	0.024	NL	NL
	Fluorene	µg/L	10	0.035	NL	NL
	Hexachlorobenzene	µg/L	10	0.075	NL	1.0
	Hexachlorobutadiene	µg/L	10	0.11	NL	NL
	Hexachlorocyclopentadiene	µg/L	50	1.5	NL	50
	Hexachloroethane	µg/L	10	0.21	NL	NL
	Indeno(1,2,3-cd)pyrene	µg/L	10	0 081	NL	NL
	Isophorone	µg/L	10	0.16	NL	NL
	Naphthalene	µg/L	10	0.031	20	NL
	Nitrobenzene	µg/L	10	0.21	NL	NL
	N-Nitrosodi-n-propylamine	µg/L	10	0.21	NL	NL
	N-Nitrosodiphenylamine	µg/L	10	0.18	NL	NL
	Pentachlorophenol	µg/L	10	2 (1)	NL	1.0
	Phenanthrene	µg/L	10	0.044	NL	NL
	Phenol	µg/L	10	0.14	20	NL
	Pyrene	µg/L	10	0 051	NL	NL
8081A	4,4-DDD	µg/L	0.05	0 0085	NL	NL
	4,4-DDE	µg/L	0.05	0.0076	NL	NL
	4,4-DDT	µg/L	0.05	0.0086	NL	NL

TABLE 2-2

PROJECT DETECTION, REPORTING, AND SCREENING LIMITS
REMEDIAL ACTION SAMPLING AND ANALYSIS PLAN
Defense Depot Memphis, Tennessee

METHOD	COMPOUND	UNITS	REPORTING LIMIT	METHOD DETECTION LIMIT*	DISCHARGE LIMIT	SCREENING LEVEL**
8081A	Aldrin	µg/L	0.05	0.0061	NL	NL
(cont'd)	alpha-BHC	µg/L	0.05	0.0062	NL	NL
	alpha-Chlordane	µg/L	0.05	0.0073	NL	2.0
	beta-BHC	µg/L	0.05	0 0068	NL	NL
	delta-BHC	µg/L	0.05	0.0064	NL	NL
	Dieldrin	µg/L	0.05	0.0067	NL	NL
	Endosulfan I	µg/L	0.05	0.0072	NL	NL
	Endosulfan II	µg/L	0.05	0.0072	NL	NL
	Endosulfan Sulfate	µg/L	0.05	0.0083	NL	NL
	Endrin	µg/L	0.05	0.0074	NL	2.0
	Endrin Aldehyde	µg/L	0 05	0.0091	NL	2.0
	Endrin Ketone	µg/L	0.05	0.013	NL	NL
	gamma-BHC (Lindane)	µg/L	0.05	0.0062	NL	0.2
	gamma-Chlordane	µg/L	0.05	0.0065	NL	2.0
	Heptachlor	µg/L	0.05	0.0062	NL	0.4
	Heptachlor Epoxide	µg/L	0.05	0.0065	NL	0.2
	Methoxychlor	µg/L	0.1	0.01	NL	40
	Toxaphene	µg/L	2.0	0.5	NL	3.0
8082	Aroclor 1016	µg/L	1.0	0 46	NL	0.5
	Aroclor 1221	µg/L	1.0	0.21	NL	0.5
	Aroclor 1232	µg/L	1.0	0.085	NL	0.5
	Aroclor 1242	µg/L	1 0	0.23	NL	0 5
	Aroclor 1248	µg/L	1.0	0.18	NL	0.5
	Aroclor 1254	µg/L	1.0	0.18	NL	0.5
	Aroclor 1260	µg/L	1.0	0.085	NL	0.5
8151A	2,4-D	µg/L	4.0	1.6	NL	70
	2,4,5-T	µg/L	1.0	0.27	NL	NL
	2,4,5-TP (Silvex)	µg/L	1.0	0 28	NL	50
6010B	Aluminum	µg/L	200	23	2000	NL
	Antimony	µg/L	10	4.1	NL	6 0
	Arsenic	µg/L	10	2.6	100	10
	Barium	µg/L	200	0.75	NL	2000
	Beryllium	µg/L	5	0.43	NL	4.0
	Cadmium	µg/L	2	0 28	20	5.0
	Calcium	µg/L	5000	330	NL	NL
	Chromium	µg/L	5	1.9	400	100
	Cobalt	µg/L	7	0.96	NL	NL
	Copper	µg/L	25	2.3	400	1300
	Iron	µg/L	100	49	20000	NL
	Lead	µg/L	3	1.7	300	15
	Magnesium	µg/L	5	24	NL	NL
	Manganese	µg/L	15	1.2	NL	NL
	Nickel	µg/L	40	2.5	300	NL
	Potassium	µg/L	5000	63	NL	NL
	Selenium	µg/L	5	3.7	NL	50
	Silver	µg/L	5	0.74	NL	NL
	Sodium	µg/L	5000	540	NL	NL
	Thallium	µg/L	10	4.5	NL	2.0
	Vanadium	µg/L	7	0.71	NL	NL
	Zinc	µg/L	20	14	1000	NL
7470A	Mercury	µg/L	0.0002	0.029	2.0	2.0
300.0	Bromide	mg/L	0.5	0 075	NL	NL
	Chloride	mg/L	1.0	0 097	NL	NL

TABLE 2-2

PROJECT DETECTION, REPORTING, AND SCREENING LIMITS
REMEDIAL ACTION SAMPLING AND ANALYSIS PLAN
Defense Depot Memphis, Tennessee

METHOD	COMPOUND	UNITS	REPORTING LIMIT	METHOD DETECTION LIMIT*	DISCHARGE LIMIT	SCREENING LEVEL**
	Nitrate	mg/L	0.1	0.016	NL	10
	Nitrite	mg/L	0.1	0.017	NL	1.0
	Sulfate	mg/L	1.0	0.11	NL	NL
310.1	Alkalinity	mg/L	5.0	1.5	NL	NL
376.1	Sulfide	mg/L	1.0	0.4	NL	NL
9060	Total Organic Carbon	mg/L	1.0	0.081	NL	NL
	Dissolved Organic Carbon	mg/L	1.0	0.081	NL	NL
RSK-175	Carbon dioxide	mg/L	0.17	0.07	NL	NL
	Ethane	mg/L	0.002	0.0003	NL	NL
	Ethene	mg/L	0.001	0.0004	NL	NL
	Methane	mg/L	0.001	0.0006	NL	NL
SM 5560	Acetic acid	mg/L	1.0	0.15	NL	NL
	Butyric acid	mg/L	1.0	0.16	NL	NL
	Formic acid	mg/L	1.0	0.22	NL	NL
	Lactic acid	mg/L	1 0	0.3	NL	NL
	Propionic acid	mg/L	1.0	0.17	NL	NL
	Pyruvic acid	mg/L	1.0	0.25	NL	NL
		SOIL				
8260B	1,1,1-Trichloroethane	µg/kg	5.0	0 69	NA	2000
	1,1,2.2-Tetrachloroethane	µg/kg	5 0	0 83	NA	NL
	1,1,2-Trichloroethane	µg/kg	5 0	0.5	NA	NL
	1,1-Dichloroethane	µg/kg	5.0	0.67	NA	23000
	1,1-Dichloroethene	µg/kg	5.0	0.81	NA	NL
	1,2-Dichloroethane	µg/kg	5.0	0.6	NA	NL
	1,2-Dichloropropane	µg/kg	5.0	0 62	NA	NL
	2-Butanone (MEK)	µg/kg	20	2.7	NA	8550
	2-Hexanone (MBK)	µg/kg	20	1.5	NA	NL
	4-Methyl-2-pentanone (MIBK)	µg/kg	20	1 6	NA	NL
	Acetone	µg/kg	20	5.8	NA	16000
	Benzene	µg/kg	5 0	0 69	NA	NL
	Bromodichloromethane	µg/kg	5.0	0.7	NA	NL
	Bromoform	µg/kg	5.0	0.72	NA	NL
	Bromomethane	µg/kg	5.0	1 4	NA	200
	Carbon disulfide	µg/kg	5.0	1	NA	NL
	Carbon tetrachloride	µg/kg	5.0	0.64	NA	NL
	Chlorobenzene	µg/kg	5.0	1.1	NA	1000
	Chloroethane	µg/kg	5.0	1.4	NA	NL
	Chloroform	µg/kg	5.0	0 71	NA	NL
	Chloromethane	µg/kg	5 0	1.8	NA	82
	cis-1,2-Dichloroethene	µg/kg	5.0	0.91	NA	NL
	cis-1,3-Dichloropropene	µg/kg	5.0	0.63	NA	NL
	Dibromochloromethane	µg/kg	5.0	0 6	NA	NL
	Ethylbenzene	µg/kg	5 0	1.2	NA	13000
	m,p-Xylenes	µg/kg	10	2.6	NA	NL
	Methylene chloride	µg/kg	5.0	1.2	NA	NL
	Methyl tert-butyl ether (MTBE)	µg/kg	20.0	0.67	NA	NL
	o-Xylene	µg/kg	5 0	1.2	NA	NL
	Styrene	µg/kg	5.0	1.1	NA	4000
	Tetrachloroethene	µg/kg	5.0	1.2	NA	NL

TABLE 2-2

PROJECT DETECTION, REPORTING, AND SCREENING LIMITS
REMEDIAL ACTION SAMPLING AND ANALYSIS PLAN
Defense Depot Memphis, Tennessee

METHOD	COMPOUND	UNITS	METHOD REPORTING LIMIT	METHOD DETECTION LIMIT*	DISCHARGE LIMIT	SCREENING LEVEL**
8260B (cont'd)	Toluene	µg/kg	5.0	0.72	NA	12000
	trans-1,2-Dichloroethene	µg/kg	5.0	0.76	NA	NL
	trans-1,3-Dichloropropene	µg/kg	5.0	0.66	NA	NL
	Trichloroethene	µg/kg	5.0	0.8	NA	NL
	Vinyl acetate	µg/kg	10.0	2	NA	NL
	Vinyl chloride	µg/kg	5.0	1.2	NA	NL
8270C	2,4,5-Trichlorophenol	µg/kg	330	4.9	NA	270000
	2,4,6-Trichlorophenol	µg/kg	330	6.8	NA	200
	2,4-Dichlorophenol	µg/kg	330	5.3	NA	NL
	2,4-Dimethylphenol	µg/kg	330	6 8	NA	NL
	2,4-Dinitrophenol	µg/kg	1600	41	NA	1000
	2,4-Dinitrotoluene	µg/kg	330	5.7	NA	NL
	2,6-Dinitrotoluene	µg/kg	330	5 8	NA	NL
	2-Chloronaphthalene	µg/kg	330	6.3	NA	NL
	2-Chlorophenol	µg/kg	330	3.6	NA	NL
	2-Methylnaphthalene	µg/kg	330	0.99	NA	NL
	2-Methylphenol (o-cresol)	µg/kg	330	6.7	NA	NL
	2-Nitroaniline	µg/kg	1600	5 1	NA	NL
	2-Nitrophenol	µg/kg	330	3.4	NA	NL
	3,3'-Dichlorobenzidine	µg/kg	1600	4.9	NA	NL
	3-Nitroaniline	µg/kg	1600	3.2	NA	NL
	4,6-Dinitro-2-methylphenol	µg/kg	1600	47	NA	NL
	4-Bromophenyl phenyl ether	µg/kg	330	4.7	NA	NL
	4-Chloro-3-methylphenol	µg/kg	330	51	NA	NL
	4-Chloroaniline	µg/kg	330	5.2	NA	NL
	4-Chlorophenyl phenyl ether	µg/kg	330	3.6	NA	NL
	4-Methylphenol (p-cresol)	µg/kg	330	5.8	NA	NL
	4-Nitroaniline	µg/kg	1600	3.6	NA	NL
	4-Nitrophenol	µg/kg	1600	81	NA	NL
	Acenaphthylene	µg/kg	330	1.4	NA	NL
	Acenaphthene	µg/kg	330	0.92	NA	29219000
	Anthracene	µg/kg	330	2.3	NA	100000000
	Benzo(a)anthracene	µg/kg	330	1.4	NA	21100
	Benzo(a)pyrene	µg/kg	330	2.3	NA	2110
	Benzo(b)fluoranthene	µg/kg	330	2.3	NA	21100
	Benzo(g,h,i)perylene	µg/kg	330	1.8	NA	NL
	Benzo(k)fluoranthene	µg/kg	330	2.3	NA	211000
	Benzoic acid	µg/kg	1600	42	NA	NL
	Benzyl alcohol	µg/kg	330	140	NA	NL
	bis(2-Chloroethoxy)methane	µg/kg	330	16	NA	NL
	bis(2-Chloroethyl)ether	µg/kg	330	4.1	NA	NL
	bis(2-Chloroisopropyl)ether	µg/kg	330	5.2	NA	NL
	bis(2-Ethylhexyl)phthalate	µg/kg	330	16	NA	1231000
	Butylbenzylphthalate	µg/kg	330	3 7	NA	100000000
	Chrysene	µg/kg	330	0.99	NA	2110000
	Dibenzo(a,h)anthracene	µg/kg	330	1.5	NA	2110
	Dibenzofuran	µg/kg	330	0.83	NA	NL
	Diethylphthate	µg/kg	330	6.2	NA	1285000
	Dimethylphthalate	µg/kg	330	6.4	NA	3309000
	Di-n-butylphthalate	µg/kg	330	5	NA	61561000
	Di-n-octylphthalate	µg/kg	330	11	NA	24624000

TABLE 2-2

PROJECT DETECTION, REPORTING, AND SCREENING LIMITS
REMEDIAL ACTION SAMPLING AND ANALYSIS PLAN
Defense Depot Memphis, Tennessee

METHOD	COMPOUND	UNITS	REPORTING LIMIT	METHOD DETECTION LIMIT*	DISCHARGE LIMIT	SCREENING LEVEL**
8270C	Fluoranthene	µg/kg	330	0.93	NA	22000000
(cont'd)	Fluorene	µg/kg	330	1.3	NA	26281000
	Hexachlorobenzene	µg/kg	330	1.4	NA	10700
	Hexachlorobutadiene	µg/kg	330	2.5	NA	NL
	Hexachlorocyclopentadiene	µg/kg	1600	2.9	NA	NL
	Hexachloroethane	µg/kg	330	4.9	NA	NL
	Indeno(1,2,3-cd)pyrene	µg/kg	330	2	NA	21100
	Isophorone	µg/kg	330	3.5	NA	NL
	Naphthalene	µg/kg	330	0.89	NA	188000
	Nitrobenzene	µg/kg	330	6 4	NA	NL
	N-Nitrosodi-n-propylamine	µg/kg	330	7 6	NA	NL
	N-Nitrosodiphenylamine	µg/kg	330	4.1	NA	NL
	Pentachlorophenol	µg/kg	330	45	NA	27000
	Phenanthrene	µg/kg	330	1.1	NA	NL
	Phenol	µg/kg	330	5.7	NA	100000
	Pyrene	µg/kg	330	1	NA	29126000
8081A	4,4-DDD	µg/kg	1 7	0.5	NA	99500
	4,4-DDE	µg/kg	1.7	0 35	NA	70200
	4,4-DDT	µg/kg	1.7	0.4	NA	70200
	Aldrin	µg/kg	1.7	0.3	NA	NL
	alpha-BHC	µg/kg	1.7	0.3	NA	3590
	alpha-Chlordane	µg/kg	1.7	0.35	NA	64600
	beta-BHC	µg/kg	1.7	0.4	NA	12600
	delta-BHC	µg/kg	1.7	0.37	NA	NL
	Dieldrin	µg/kg	1.7	0 34	NA	1080
	Endosulfan I	µg/kg	1.7	0.33	NA	3694000
	Endosulfan II	µg/kg	1.7	0.42	NA	3694000
	Endosulfan Sulfate	µg/kg	1 7	0.38	NA	3694000
	Endrin	µg/kg	1 7	0 34	NA	185000
	Endrin Aldehyde	µg/kg	1.7	0.89	NA	185000
	Endrin Ketone	µg/kg	1.7	0.7	NA	185000
	gamma-BHC (Lindane)	µg/kg	1.7	0.34	NA	17400
	gamma-Chlordane	µg/kg	1.7	0.31	NA	64600
	Heptachlor	µg/kg	1.7	0.29	NA	3830
	Heptachlor Epoxide	µg/kg	1.7	0.42	NA	8000
	Methoxychlor	µg/kg	3.3	0.51	NA	3078000
	Toxaphene	µg/kg	67	10	NA	15700
8082	Aroclor 1016	µg/kg	33	6.7	NA	37200
	Aroclor 1221	µg/kg	33	9 9	NA	7440
	Aroclor 1232	µg/kg	33	5.2	NA	7440
	Aroclor 1242	µg/kg	33	10	NA	7440
	Aroclor 1248	µg/kg	33	4.8	NA	7440
	Aroclor 1254	µg/kg	33	4.3	NA	7440
	Aroclor 1260	µg/kg	33	8	NA	7440
8151A	2,4-D	µg/kg	80	8.6	NA	NL
	2,4,5-T	µg/kg	20	2.2	NA	NL
	2,4,5-TP (Silvex)	µg/kg	20	2.6	NA	NL
6010B	Aluminum	mg/kg	20	2.3	NA	100000
	Antimony	mg/kg	1	0 23	NA	7
	Arsenic	mg/kg	1	0.4	NA	29
	Barium	mg/kg	20	0.15	NA	1600

TABLE 2-2

PROJECT DETECTION, REPORTING, AND SCREENING LIMITS
REMEDIAL ACTION SAMPLING AND ANALYSIS PLAN
Defense Depot Memphis, Tennessee

METHOD	COMPOUND	UNITS	REPORTING LIMIT	METHOD DETECTION LIMIT*	DISCHARGE LIMIT	SCREENING LEVEL**
6010B	Beryllium	mg/kg	0.5	0.031	NA	19000
(cont'd)	Cadmium	mg/kg	0.2	0.023	NA	451
	Calcium	mg/kg	500	15	NA	NL
	Chromium	mg/kg	0 5	0.12	NA	4483
	Cobalt	mg/kg	5.0	0.1	NA	661
	Copper	mg/kg	2.5	0.16	NA	669
	Iron	mg/kg	10	4.3	NA	NL
	Lead	mg/kg	0.3	0.23	NA	1536
	Magnesium	mg/kg	500	2.1	NA	NL
	Manganese	mg/kg	1.5	0.31	NA	1540
	Nickel	mg/kg	4.0	0.17	NA	20439
	Potassium	mg/kg	500	3.6	NA	NL
	Selenium	mg/kg	0.5	0.27	NA	5
	Silver	mg/kg	0.5	0.11	NA	34
	Sodium	mg/kg	500	70	NA	NL
	Thallium	mg/kg	1	0.35	NA	67.5
	Vanadium	mg/kg	5.0	0.11	NA	7154
	Zinc	mg/kg	2	0 87	NA	100000
7471A	Mercury	mg/kg	0.10	0.0045	NA	307
Walkley Black	Total Organic Carbon	mg/kg	100	100	NA	NL

Notes:

* The MDLs presented were provided by STL. MDLs provided by other laboratories will be compared with project RLs prior to implementation.

** Screening Levels for water are the lowest of the USEPA Maximum Contaminant Levels (Winter 2004) or the USEPA Region IX Preliminary Remediation Goals for Tap Water (October 2002). Screening Levels for soil are the Soil Remediation Goals as found in the Dunn Field Record of Decision.

(1) The MDLs for these VOC and SVOC compounds are higher than their corresponding screening levels because current VOC and SVOC analytical method technology can not achieve MDLs lower than those listed.

NA Not applicable

NL Not listed

TABLE 2-3

CONTAINERS, PRESERVATIVES, AND HOLDING TIMES
MATRIX: GROUNDWATER AND SOIL
REMEDIAL ACTION SAMPLING AND ANALYSIS PLAN
Defense Depot Memphis, Tennessee

Parameter	Units	Method	Container	Minimum Recommended Quantity (mL)	Preservative	Holding Time
Groundwater						
Volatile Organics Compounds	µg/L	SW 5030B/8260B	VOA w/ Teflon®-lined septum	3 X 40 (no headspace)	4°C; HCl to pH<2	14 days/7 days if unpreserved
Dissolved Gases: Methane, Ethane, Ethene	mg/L	STL SOP COI-GC-005 (EPA RSK SOP-175M)	VOA w/ Teflon®-lined septum	3 X 40 (no headspace)	4°C: HCl to pH<2	14 days
Carbon Dioxide	mg/L	STL SOP COI-GC-005 (EPA RSK SOP-175M)	VOA w/ Teflon®-lined septum	2 X 40 (no headspace)	4°C	7 days
Semi-Volatile Organics	µg/L	SW 3520C/8270C	G-TLC (amber)	1000	4° C	7 days extraction/ 40 days analysis
Pesticides/PCBs	µg/L	SW 3520C/8081A/8082	G-TLC (amber)	1000	4° C	7 days extraction/ 40 days analysis
Herbicides	µg/L	SW 3520C/8151A	G-TLC (amber)	1000	4° C	7 days extraction/ 40 days analysis
Metals ICP/Mercury	µg/L	SW 3005A/6010B /7470A	P	1000	HNO_3 to pH<2 (dissolved – filter on site)	6 months for ICP/28 days for mercury
Anions: Bromide, Chloride, Nitrate, Nitrite, Sulfate	mg/L	EPA 300.0/SW 9056	P, G	250	4°C	28 days (Br, Cl, SO_4) 48 hours (NO_3, NO_2)
Alkalinity	mg/L	EPA 310.1	P	250 (no headspace)	4°C	48 hours
Sulfide	mg/L	EPA 376.1	P	500 (no headspace)	4°C; Zinc Acetate & NaOH to pH > 10	7 days
Total Organic Carbon	mg/L	SW 9060	P, G	2 X 40 (no headspace)	4°C; H_2SO_4 to pH<2	28 days

TABLE 2-3

CONTAINERS, PRESERVATIVES, AND HOLDING TIMES
MATRIX: GROUNDWATER AND SOIL
REMEDIAL ACTION SAMPLING AND ANALYSIS PLAN
Defense Depot Memphis, Tennessee

Parameter	Units	Method	Container	Minimum Recommended Quantity (mL)	Preservative	Holding Time
Dissolved Organic Carbon	mg/L	SW 9060	P, G	2 X 40 (no headspace) (dissolved – filter on site)	4°C; H_2SO_4 to pH<2	28 days
Volatile Fatty Acids	mg/L	ASTM D 1552	VOA w/ Teflon®-lined septum	1 X 40 (no headspace)	4°C	28 days
CONTAINER AND SAMPLE HANDLING GUIDE MATRIX: FIELD TESTS FOR GROUNDWATER						
Parameter	**Units**	**Method**	**Container**	**Minimum Recommended Quantity (mL)**	**Preservative**	**Holding Time**
pH	Units	EPA 150.1	P, G	50	N/A	ASAP
Specific Conductance	mS/cm	EPA 120.1	P, G	250	4°C	24 hrs
Temperature	°C	EPA 170.1	P, G	50	N/A	ASAP
Turbidity	NTUs	EPA 180.1	P, G	250	4°C	48 hrs
Redox Potential	mV	SM 2580	P, G	50	N/A	ASAP
Dissolved Oxygen	mg/L	MCAWW 360.1	P, G	50	N/A	ASAP
Ferrous Iron	mg/L	HANNA Kits 38039/38041	P, G	50	N/A	ASAP
Carbon Dioxide	mg/L	HANNA Kit 3818	G	50	N/A	ASAP
Soil						
Volatile Organic Compounds – Encores *	µg/kg	SW 5035/8260B	G-TLC/ Encores™	4 X 5 gram Encores ™	4°C	48 hrs for preservation/ 14 days analysis
Volatile Organic Compounds – field preservation	µg/kg	SW 5030B/8260B	G	4 X 4 oz.	4°C	14 days analysis

TABLE 2-3

CONTAINERS, PRESERVATIVES, AND HOLDING TIMES
MATRIX: GROUNDWATER AND SOIL
REMEDIAL ACTION SAMPLING AND ANALYSIS PLAN
Defense Depot Memphis, Tennessee

Parameter	Units	Method	Container	Minimum Recommended Quantity (mL)	Preservative	Holding Time
Semi-Volatile Organic Compounds	µg/kg	SW 3550B/8270C	G	8 oz.	4° C	14 days extraction/ 40 days analysis
Pesticides/PCBs	µg/kg	SW 3550B/8081A/8082	G	8 oz.	4° C	14 days extraction/ 40 days analysis
Herbicides	µg/kg	SW 3550B/8151A	G	8 oz.	4° C	14 days extraction/ 40 days analysis
Metals ICP/Mercury	mg/kg	SW 3050A/6010B /7471A	G	8 oz.	4° C	6 months for ICP/28 days for mercury
TOC	mg/kg	Walkley Black	G	8 oz.	4°C	28 days
TCLP	mg/L	SW 1311	G-TLS/ Encore™	Extractables, metals, ignitability, reactivity, corrosivity- 16 oz. VOCs-25g Encore™ or 4 oz.	4°C	VOCs 14 days extraction/14 days analysis; Extractables- 14 days extraction/7 days preparation/40 days analysis; Metals-6 months extraction/6 months analysis; Mercury-28 days extraction/28 days analysis; ignitability/corrosivity 28 days; reactivity-14 days

* If collecting for volatile organic compounds only, an additional aliquot of soil must be obtained in a one 4-oz wide mouth jar for moisture content determination

Acronym Definitions:

P = Polyethylene
G = Glass
G-TLS = Glass with Teflon®-lined septum
G-TLC = Glass with Teflon®-lined cap
PTFE = Fluoropolymer Resin/Teflon®

TABLE 4-1

ANALYTICAL TEST METHODS
REMEDIAL ACTION SAMPLING AND ANALYSIS PLAN
Defense Depot Memphis, Tennessee

MATRIX: GROUNDWATER/EFFLUENT WATER

Parameter	Method [a]
Volatile Organic Compounds	SW 5030B/8260B
Dissolved Gases: Methane, Ethane, Ethene, and Carbon Dioxide	STL-LA SOP COI-GC-005, Rev 1, (USEPA RSK SOP-175)
Semi-Volatile Organics	SW 3520C/8270C
ICP Metals	SW 3005A/6010B
Pesticides	SW 3520C/8080A
PCBs	SW 3520C/8082
Herbicides	8151A
Mercury	SW 7470A
Anions – Bromide, Chloride, Nitrate, Nitrite, Sulfate	MCAWW 300.0A/SW 9056
Alkalinity	MCAWW 310.1
Sulfide	MCAWW 376.1
Total Organic Carbon	SW 9060
Dissolved Organic Carbon	SW 9060
Metabolic Fatty Acids	SM 5560

MATRIX: SOIL

Parameter	Method [a]
Volatile Organics	SW 5035/8260B
Semi-Volatile Organics	SW 3550B/8270C
Pesticides	SW 3550B/8081A
Polychlorinated Biphenyls	SW 3550B/8082
Herbicides	SW 3550B/8151A
ICP Metals	SW 3050A/6010B
Mercury	SW 7471A
Total Organic Carbon	Walkley Black
TCLP – Volatile Organics	SW 1311/5030B/8260B
TCLP – Semi-Volatile Organics	SW 1311/3520C/8270C
TCLP – Pesticides	SW 1311/3520C/8081A
TCLP – Herbicides	SW 1311/3520C/8151A
TCLP – Metals	SW 1311/3005A/6010B/7470A
Reactivity (H_2S and HCN)	SW 846 Chap. 7.3.3 2/7.3.4.2
Ignitability	SW 1010
Corrosivity (pH)	SW 9045C

TABLE 4-1

ANALYTICAL TEST METHODS
REMEDIAL ACTION SAMPLING AND ANALYSIS PLAN
Defense Depot Memphis, Tennessee

MATRIX: FIELD TESTS

Parameter	Method [a]
pH (unit)	MCAWW 150.1
Specific Conductance (µmhos/cm)	MCAWW 120.1
Temperature (°C)	MCAWW 170.1
Turbidity (NTUs)	MCAWW 180.1
Redox Potential (mV)	SM 2580
Dissolved Oxygen (mg/L)	MCAWW 360.1
Ferrous Iron (mg/L)	HANNA Kits 38039/38041
Carbon Dioxide (mg/L)	HANNA Kit 3818

Notes:

[a] MCAWW "Methods for Chemical Analysis of Water and Wastes", EPA-600/4-79-020, March 1983 and subsequent revisions.

SW-846 "Test Methods for Evaluating Solid Waste, Physical/Chemical Methods", Third Edition, November 1986 and its updates.

SM "Standard Methods for Examination of Water and Wastewater," American Public Health Association, American Water Works Association, and Water Pollution Control Federation, 20th Ed., 1999.

STL Severn Trent Laboratory SOP No. COI-GC-005, "Sample Preparation and the Determination of Dissolved Gases in Water By Using Gas Chromatography (GC) Headspace Equilibrium Technique (USEPA RSK SOP-175-Modified)", September 10, 2001.

Not all test methods may be presented in the SAP. Equipment blanks are analyzed for the same parameters as associated samples with the exception of dissolved organic carbon and field tests.

TABLE 4-2

BFB KEY IONS AND ABUNDANCE CRITERIA[a]
REMEDIAL ACTION SAMPLING AND ANALYSIS PLAN
Defense Depot Memphis, Tennessee

Mass	Ion Abundance Criteria
50	15-40% of mass 95
75	30-60% of mass 95
95	base peak, 100% relative abundance
96	5-9% of mass 95
173	less than 2% of mass 174
174	greater than 50% of mass 95
175	5-9% of mass 174
176	greater than 95% but less than 101% of mass 174
177	5-9% of mass 176

Notes:
[a] USEPA Method SW8260B, SW-846, 3rd Edition, Update III, December 1996 (USEPA, 1996).
BFB - 4 - Bromofluorobenzene

TABLE 4-3

DFTPP KEY IONS AND ABUNDANCE CRITERIA[a]
REMEDIAL ACTION SAMPLING AND ANALYSIS PLAN
Defense Depot Memphis, Tennessee

Mass	Ion Abundance Criteria
51	30-60% of mass 198
68	<2% of mass 69
70	<2% of mass 69
127	40-60% of mass 198
197	<1% of mass 198
198	Base peak, 100% relative abundance
199	5-9% of mass 198
275	10-30% of mass 198
365	>1% of mass 198
441	Present, but less than mass 443
442	>40% of mass 198
443	17-23% of mass 442

Note:
[a] J W. Eichelberger, L.E. Harris, and W.L. Budde. "Reference Compound to Calibrate Ion Abundance Measurement in Gas Chromatography-Mass Spectrometry," Analytical Chemistry, 47, 995 (1975). EPA Method 8270C, 3rd Edition, Update III, December 1996 (USEPA, 1996).
DFTPP - Decafluorotriphenylphosphine

TABLE 4-4

VOLATILE INTERNAL STANDARDS WITH CORRESPONDING ANALYTES ASSIGNED FOR QUANTITATION
REMEDIAL ACTION SAMPLING AND ANALYSIS PLAN
Defense Depot Memphis, Tennessee

Fluorobenzene	**1,4-Difluorobenzene-d_4**	**Chlorobenzene-d_5**
Chloromethane	Trichloroethene	Chlorobenzene
Vinyl chloride	1,2-Dichloropropane	Ethylbenzene
Bromomethane	Bromodichloromethane	Styrene
Chloroethane	2-Chloroethyl vinyl ether	Bromoform
Acetone	cis-1,3-Dichloropropene	1,1,2,2-Tetrachloroethane
Carbon disulfide	trans-1,3-Dichloropropene	Toluene-d_8 *
1,1-Dichloroethene	Dibromochloromethane	Xylenes, total
Methylene chloride	Dibromofluorobenzene*	4-Bromofluorobenzene *
trans-1,2-Dichloroethene	1,1,2-Trichloroethane	
1,1-Dichloroethane	Tetrachloroethene	
Vinyl acetate	Toluene	
cis-1,2-Dichloroethene	4-Methyl-2-pentanone	
2-Butanone	2-Hexanone	
Chloroform		
1,1,1-Trichloroethane		
Carbon tetrachloride		
Benzene		
1,2-Dichloroethane		
1,2-Dichloroethane-d_4 *		

Note:
* Surrogate

TABLE 4-5

SEMI-VOLATILE INTERNAL STANDARDS
WITH CORRESPONDING ANALYTES ASSIGNED FOR QUANTITATION
REMEDIAL ACTION SAMPLING AND ANALYSIS PLAN
Defense Depot Memphis, Tennessee

1,4-Dichlorobenzene-d_4	Naphthalene-d_8	Acenaphthene-d_{10}	Phenanthrene-d_{10}	Chrysene-d_{12}	Perylene-d_{12}
Phenol	Nitrobenzene	Hexachlorocyclopentadiene	4,6-Dinitro-2-methylphenol	Pyrene	Di-n-octyl phthalate
bis(2-Chloroethyl)ether	Isophorone	2,4,6-Trichlorophenol	N-Nitrosodiphenylamine	Butyl benzyl phthalate	Benzo(b)fluoranthene
2-Chlorophenol	2-Nitrophenol	2,4,5-Trichlorophenol	4-Bromophenyl phenyl ether	3,3'-Dichlorobenzidine	Benzo(k)fluoranthene
1,3-Dichlorobenzene	2,4-Dimethylphenol	2-Chloronaphthalene	Hexachlorobenzene	Benz(a)anthracene	Benzo(a)pyrene
1,4-Dichlorobenzene	Benzoic acid	2-Nitroaniline	Pentachlorophenol	Chrysene	Indeno(1,2,3-cd)pyrene
Benzyl alcohol	bis(2-Chloroethoxy)methane	Dimethyl phthalate	Phenanthrene	bis(2-Ethylhexyl)phthalate	Dibenz(a,h)anthracene
1,2-Dichlorobenzene	2,4-Dichlorophenol	Acenaphthylene	Anthracene	Terphenyl-d_{14} *	Benzo(g,h,i)perylene
2-Methylphenol	1,2,4-Trichlorobenzene	2,4-Dinitrophenol	Di-n-butyl phthalate		
bis(2-Chloroisopropyl)ether	Naphthalene	3-Nitroaniline	Fluoranthene		
4-Methylphenol	4-Chloroaniline	Acenaphthene	2,4,6-Tribromophenol *		
N-Nitrosodi-n-propylamine	Hexachlorobutadiene	4-Nitrophenol			
Hexachloroethane	4-Chloro-3-methylphenol	Dibenzofuran			
2-Fluorophenol *	2-Methylnaphthalene	2,4-Dinitrotoluene			
Phenol-d_5 *	Nitrobenzene-d_5 *	2,6-Dinitrotoluene			
		Diethyl phthalate			
		4-Chlorophenyl phenyl ether			
		Fluorene			
		4-Nitroaniline			
		2-Fluorobiphenyl *			

Note:
* Surrogate compounds

TABLE 5-1

DATA QUALIFICATION FLAGS
REMEDIAL ACTION SAMPLING AND ANALYSIS PLAN
Defense Depot Memphis, Tennessee

Flag	Positive Results	Non-Detect Results
FLAGS FOR DATA WITHIN ACCEPTANCE LIMITS (Usable as Reported)		
(no flag)	{Use datum without qualification}	{Use datum without qualification}
FLAGS FOR DATA WITHIN ACTION LIMITS (Usable With Qualification)		
J	Estimated quantitation based upon QC data	Estimated quantitation based upon QC data
B	Estimated quantitation: possibly biased high or false positive based upon blank data	(Not applicable)
FLAGS FOR DATA OUTSIDE OF ACTION LIMITS (Unusable)		
R	Datum rejected based upon QC data: do not use	Datum rejected based upon QC data. do not use

Note that if the QC results suggest contradictory flags, the following hierarchy should be used to select the appropriate flag to assign:

R>B>J

FIGURES

Figure 3-1

Chain of Custody Record

SEVERN TRENT **STL**
Severn Trent Laboratories, Inc.

STL 4124 (0901)

Client	Project Manager	Date	Chain of Custody Number 150570
Address	Telephone Number (Area Code) Fax Number	Lab Number	Page _____ of _____
City / State / Zip Code	Site Contact	Lab Contact	Analysis (Attach list if more space is needed)
Project Name and Location (State)	Carrier/Waybill Number		Special Instructions/ Conditions of Receipt
Contract/Purchase Order/Quote No.	Matrix / Containers & Preservatives		

Sample I.D. No. and Description (Containers for each sample may be combined on one line)	Date	Time	Air	Aq	Sed	Soil	Other	Unpres	H_2SO_4	HNO_3	HCl	NaOH	ZnAc/NaOH

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

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

Turn Around Time Required
☐ 24 Hours ☐ 48 Hours ☐ 7 Days ☐ 14 Days ☐ 21 Days ☐ Other _____

QC Requirements (Specify)

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

Comments

DISTRIBUTION WHITE - Returned to Client with Report CANARY - Stays with the Sample PINK - Field Copy

FIGURE 3-2

COOLER RECEIPT FORM

Contractor Cooler_____

LIMS#_____

QA Lab Cooler #_____

Number of Coolers_____

PROJECT:_____ Date received:_____

USE BOTTOM OF PAGE 2 OF THIS FORM TO NOTE DETAILS CONCERNING CHECK-IN PROBLEMS.

A. PRELIMINARY EXAMINATION PHASE: Date cooler was opened: _____
by (print)_____ (sign)_____

1. Did cooler come with a shipping slip (air bill, etc.)? ..... YES NO
   If YES, enter carrier name & air bill number here: _____
2. Were custody seals on outside of cooler? ..... YES NO
   How many & where _____, seal date: _____ seal name:_____
3. Were custody seals unbroken and intact at the date and time of arrival? ..... YES NO
4. Did you screen samples for radioactivity using the Geiger counter? ..... YES NO
5. Were custody papers in a plastic bag & taped inside to the lid? ..... YES NO
6. Were custody papers filled out properly (ink, signed, etc.)? ..... YES NO
7. Did you sign custody papers in the appropriate place? ..... YES NO
8. Was the project identifiable from custody papers? If YES, enter project name at the top of this form ..... YES NO
9. Were temperature blanks used? ..... YES NO
   Cooler Temperature_____(°C) Thermometer ID No._____
10. Have designated person initial here to acknowledge receipt of cooler: _____ (date) _____

B. LOG-IN PHASE: Date samples were logged in: _____
by (print) _____ (sign) _____

11. Describe type of packing in cooler: _____
12. Were all bottles sealed in separate plastic bags? ..... YES NO
13. Did all bottles arrive unbroken with labels in good condition? ..... YES NO
14. Were all bottle labels complete (ID, date, time, signature, preservative, etc.)? .... YES NO
15. Did all bottle labels agree with custody papers? ..... YES NO
16. Were correct containers used for the tests indicated? ..... YES NO
17. Were samples preserved to correct pH, if applicable? ..... YES NO
18. Was a sufficient amount of sample sent for tests indicated? ..... YES NO
19. Were bubbles absent in volatile organic analysis (VOA) samples? If NO, list VOA samples below ..... YES NO
20. Was the project manager called and status discussed? If YES, give details on the bottom of this form ..... YES NO
20. Who was called? _____ By whom? _____ (date)_____

FIGURE 3-3

Mactec
3200 Town Point Dr, Suite 100
Kennesaw, GA 30144

REQUEST FOR ANALYSIS

Project Manager: Tom Holmes
Project Chemist: Jessica Vickers
Project: DDMT

Matrix: Groundwater
Sample ID: MW-47

Container	No.	Preservation	Parameter	Method	Prep
40 mL VOA w/septum	3	HCL to pH<2 Cool to 4 C	VOCs	SW8260B	SW5030B
500 mL Plastic	1	No Preservative Cool to 4 C	Anions/Sulfate/Bromide/Alk	E310.1/E300.0	
40 mL VOA w/septum	2	HCL to pH<2 Cool to 4 C	Total Organic Carbon	SW9060	
40 mL VOA w/septum	2	HNO3 to pH <2/Cool to 4C Field Filter	Dissolved Organic Carbon	E415.1	
500 mL Plastic	1	ZnAc & NaOH to pH>9 Cool to 4 C	Sulfide	E376.1	
1 L Poly	1	HNO3 to pH <2 Cool to 4 C	Total Metals (As, Mn, Se)	SW6010B	
40 mL VOA w/septum	2	HCL to pH<2 Cool to 4 C	Methane/Ethane/Ethene	RSK 175	
40 mL Amber VOA w/septum	3	No Preservative Cool to 4 C	Metabolic Fatty Acids		

Comments:_____
Prepared By:_____ Checked By:_____

APPENDIX A

SEVERN TRENT LABORATORIES – NORTH CANTON LQM
ENVIRONMENTAL TESTING & CONSULTING, INC. – MEMPHIS LQM

SEVERN TRENT LABORATORIES – NORTH CANTON LQM

877 86

SEVERN TRENT STL

Laboratory Quality Manual
for

STL North Canton

4101 Shuffel Drive, NW
North Canton, Ohio 44720
(330) 497-9396

Approved by:

Christopher R Oprandi 8/30/02
Laboratory Director — Date
Christopher R. Oprandi

Mark Bruce 8/30/02
Technical Director — Date
Mark Bruce, Ph.D.

Beth Lambert 8/26/02
Quality Assurance Manager — Date
Beth Lambert

Revision 3.0
August 19, 2002
Controlled Copy Number: _____

Purpose and Scope

The purpose of this Laboratory Quality Manual (LQM) is to describe the implementation the Severn Trent Laboratories (STL) Quality System at the STL North Canton laboratory. The LQM is written within the guidelines of the STL Quality Management Plan (QMP), which applies to all STL laboratories. The organization of this LQM is based on the "EPA Requirements for Quality Management Plans" (EPA QA/R-2, August 1994). This LQM outlines specific policies, organization, responsibilities, and activities required to assure high quality laboratory services. The LQM also fulfills the requirements of our clients, government agencies, and NELAC to document the laboratory Quality System.

This LQM contains references to other essential STL quality documents. The company-wide QMP, STL North Canton LQM, and referenced policies and SOPs are interrelated. Together they provide an integrated quality foundation that meets the objectives of the STL Quality Assurance Policy, as stated in Section 1.2.

The requirements set forth in this document are applicable to all employees at the STL North Canton laboratory. The policies and practices described here are presented as minimum guidelines only. Based on good scientific judgment, more rigorous requirements may be applied by laboratory employees. Specific requirements delineated in project plans may supersede general quality requirements described in this manual. One such project requirement, OhioVAP, is listed below:

- Quality Manual Sections 3.3, 3.4, 3.5, and Table 3.4-1: Per OhioVAP OAC rule 374-300-4(B) – STL North Canton will retain project records for all samples performed under the OhioVAP program for a period of ten years. After 10 years, STL North Canton must notify the director of intent to destroy records.

- Quality Manual Section 8.2.1: Per OhioVAP OAC rule 3745-300-04(1)(6) – STL North Canton will not update SOPs and Quality Manuals associated with OhioVAP projects unless they have been reviewed and approved by the agency.

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1.0 Management Commitment and Organization

1.1 STL Mission Statement

We enable our customers to create safe and environmentally favorable polices and practices by leading the market in scientific and consultancy services. We provide this support within a customer service framework that sets the standard to which others aspire. This is achieved by people whose professionalism and development is valued as the key to success and through continued investments in science and technology.

1.2 STL Quality Assurance Policy

It is STL's policy to:

- provide high quality, consistent, and objective environmental testing services that meet all relevant federal, state, and municipal regulatory requirements;
- generate data that are scientifically sound, legally defensible, meet project objectives, and are appropriate for their intended use;
- provide STL clients with the highest level of professionalism and the best service practices in the industry;
- build continuous improvement mechanisms into all laboratory administration, and managerial activities; and
- maintain a working environment that fosters open communication with both clients and staff.

1.3 STL Management Statement of 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.

1.4 Ethics, Waste, Fraud and Abuse

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 Agreement (see Figure 1.4-1). Ethics is also a major component of the STL QA training program (see Section 4 for details). A central tenant is that management must

consistently convey the message to analysts that financial pressures can never be allowed to compromise the quality of work. See the following policies for further details on specific policies related to this section:

LQM Section 6 – Computer Hardware and Software
QA-008 – Data Recording Requirements
QA-010 – Maintaining Time Integrity
QA-011 – Acceptable Manual Integration Practices
P-T-001 – Selection of Calibration Points

1.5 Organizational Structure and Relationships

STL North Canton is a local operating unit of Severn Trent Laboratories, Inc., a Delaware corporation. Date of incorporation was August 27, 1997.

The organizational structure for Severn Trent Laboratories, Inc. is presented in Figure-1.5-1. The responsibilities and authorities of the members of the STL corporate staff employees are described in the STL QMP

STL North Canton has day-to-day independent operational authority that is overseen by corporate officers (e.g., President, Commercial Director, Chief Operating Officer, Corporate Quality Assurance, etc.). The STL North Canton laboratory operational and support staff work under the direction of the Laboratory Manager. The organizational structure for STL North Canton is presented in Figure 1.5-2. A list of key STL North Canton personnel is provided in Figure 1.5-3. The lab maintains Job Descriptions which contain general job responsibilities for all laboratory employees. The following section outlines responsibilities and authorities for all employees of the STL North Canton laboratory, as they relate to quality management.

The STL North Canton QA Manager (QAM) is <u>independent</u> from day-to-day laboratory operations, has no direct analytical testing responsibilities, and is free from financial and other undue pressures which might adversely affect the quality of work. The QAM, a key member of the laboratory's management team, has direct access to the Corporate Quality Assurance Manager on all matters involving quality. The QAM is available to any lab employee to resolve quality or ethical issues. The QAM, if required, has the authority to cease operations adversely affecting the validity or integrity of the analytical data.

Figure 1.4-1 STL Ethics Agreement

It is the policy of STL to incorporate the highest standard of quality with all analytical programs by adhering to the following practices:

STL will only offer environmental analyses for which it can consistently demonstrate compliance with high quality, traceable and legally defensible performance standards.
All STL staff is committed to the practice of complete honesty in the production and reporting of data.
Staff who are aware of misrepresentation of facts or data manipulation to bypass established QA/QC requirements, are required to immediately inform their supervisor or any member of the upper management.

All employees are asked to sign a copy of the statement below upon their first day of employment.

I, _____(print name) understand that high standards of integrity are required of me with regard to the duties I perform and the data I report in connection with my employment at the Company. I agree that in the performance of my duties at the Company:
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 identifications, 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; 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 the upper management, up to and including the president of Severn Trent Laboratories Inc.
I will not intentionally report data values that do not meet established quality control criteria as set forth in the Method and/or Standard Operation 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. I agree to inform my Supervisor of any accidental or intentional reporting of non-authentic data by other employees. I have read this Ethics Agreement and understand that failure to comply with the conditions stated above will result in disciplinary action, up to and including termination from the Company.

Compliance with this policy of business ethics and conduct is the responsibility of every STL employee. Disregard or failing to comply with this standard of business ethics and conduct could lead to disciplinary action, up to and including possible termination of employment.

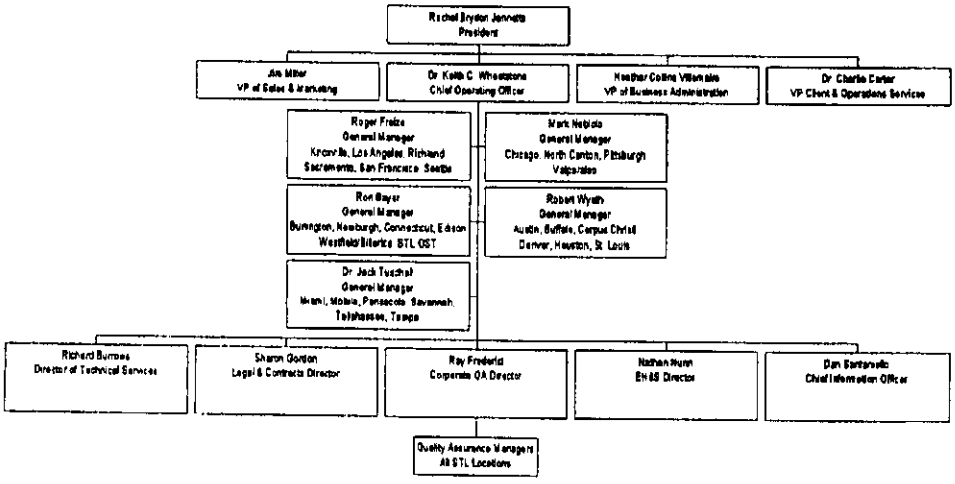
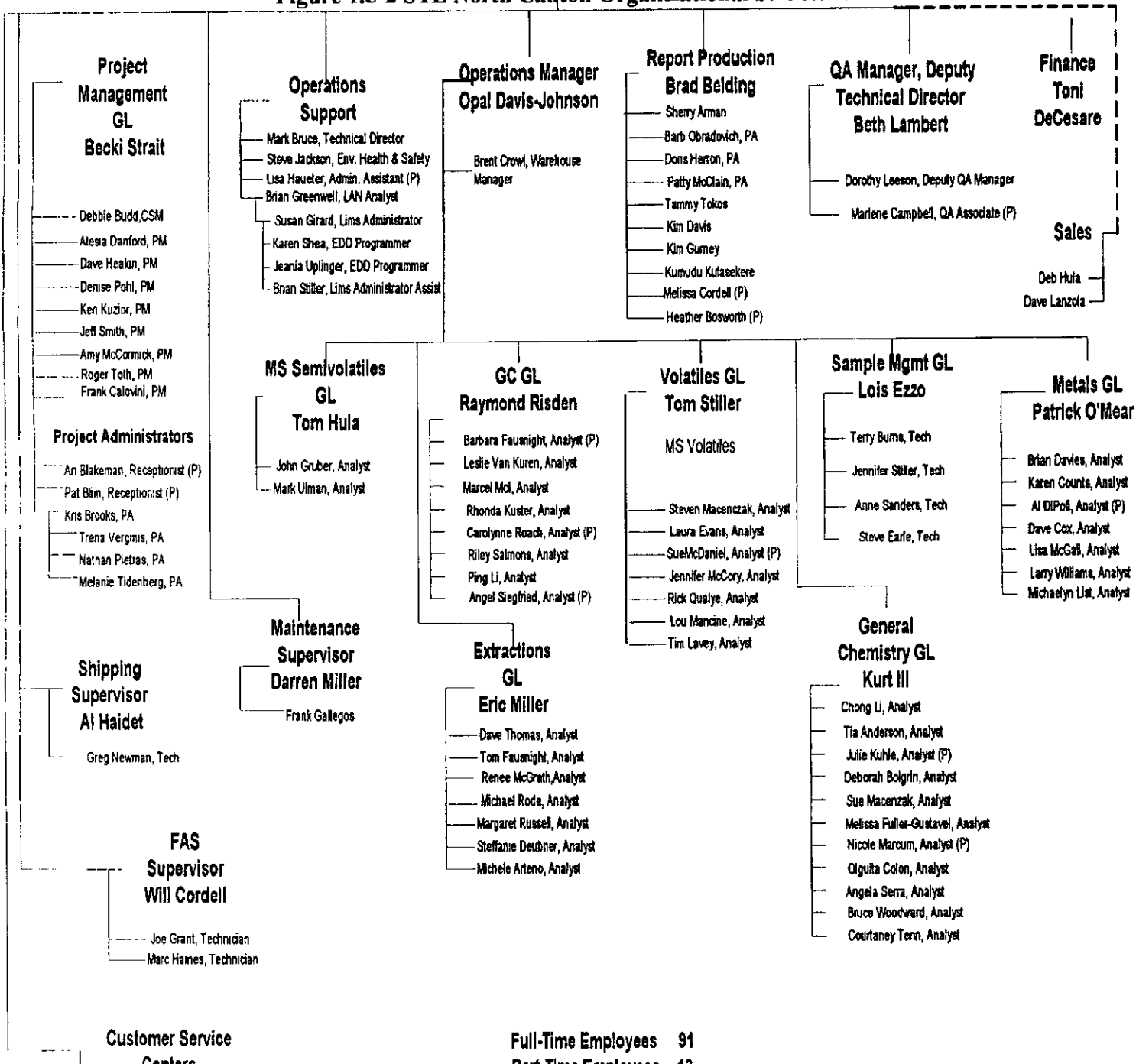


Figure 1.5-1 STL Organizational Structure

STL NORTH CANTON

Laboratory Director
Chris Oprandi

Figure 1.5-2 STL North Canton Organizational Structure



Full-Time Employees 91
Part-Time Employees 13
Total 104

*QA is included in North Canton headcount, but also reports to Corporate.
** Toni DeCesare and Sales are not included in NC head count
GL - Group Leader

Figure 1.5-3 STL North Canton Key Personnel

Name	Position	Education	Hire Date
Mark Nebiolo	General Manager, NE Reg	MS Biology	09/21/89
Christopher Oprandi	Laboratory Director	BS Chemistry	02/01/88
Opal Davis-Johnson	Operations Manager	BA Chemistry	06/30/86
Beth Lambert	Quality Assurance Manager	BA Chemistry, Physics	10/02/01
Rebecca Strait	Manager of Project Management	MBA Intl Bus	02/10/92
Stephen Jackson	Env. Health & Safety Coord	AS Environmental Science	09/30/91
Brad Belding	Report Production Supervisor	BS Chemistry	11/14/88
Mark Bruce	Technical Advisor	PhD Analytical Chemistry	12/07/87
Raymond Risden	GC & GCV Supervisor	BS Biology (Chem Minor)	05/04/92
Tom Hula	MS Semivoc. Supervisor	BA Chemistry	07/20/87
Tom Stiller	MS Volatiles Supervisor	BS Biology	02/01/88
Al Haidet	Shipping Supervisor	Work Experience	10/20/78
Lois Ezzo	Sample Control Supervisor	BA Biology	06/26/89
Darren Miller	Maintenance Supervisor	Work Experience	08/02/99
Will Cordell	Field Analytical Supervisor	BS Env Sci & Nat Res	08/03/92
Patrick O'Meara	Metals Supervisor	BA Biology	04/02/90
Kurt III	General Chemistry Supervisor	BS Chemistry	01/07/85
Eric Miller	Extractions Supervisor	Work Experience	11/21/94
Deborah Budd	Customer Service Manager	BA Biology	01/12/01
Alesia Danford	Project Management	Work Experience	01/04/84
David Heakin	Project Management	BS Chemistry	11/15/88
Ken Kuzior	Project Management	BS Chemistry	08/25/97
Amy McCormick	Project Management	Work Experience	04/09/90
Denise Pohl	Project Management	BA Biology	10/01/79
Jeff Smith	Project Management	AAS Fire Science	10/21/85
Roger Toth	Project Management	BS Environmental Sciences	12/11/89
Frank Calovini	Project Management	BS Microbiology	04/29/02

1.6 Quality Organization

All personnel are responsible for quality, which includes complying with all QA/QC requirements that pertain to their organizational/technical function.

1.6.1 Quality Assurance Manager

- Reports directly to the Laboratory Manager and, for all QA matters, to the Corporate QA Director to maintain independence of QA oversight
- Responsible for the implementing and communicating the QMP
- Maintains, approves, and implements the LQM

- Has joint signature authority, with the Laboratory Manager and Technical Manager for approval of quality documents, e.g., LQM, policies, and SOPs
- Directs controlled distribution of laboratory quality documents
- Provides Quality System training to all new personnel
- Reviews and approves documentation of analyst training records
- Serves as a focal point for QA and QC issues, reviews corrective actions and recommends resolution for recurring nonconformances within the laboratory
- Assists in maintaining regulatory analytical compliance, including maintaining certifications, and in this regard has signature authority for laboratory quality documents
- Monitors data quality measures via statistical methods to verify that the laboratory routinely meets stated quality goals
- Performs systems, data, contract compliance, and surveillance audits.
- Hosts external audits conducted by outside agencies
- Responsible for approving quality control reference data changes in the LIMS
- Oversees the selection, review, and approval of analytical subcontractors
- Prepares monthly QA Reports to management describing significant quality events
- Has 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

1.6.2 Laboratory Manager

- Reports directly to the Regional General Manager
- Responsible for implementation and adherence by lab staff to the STL QMP, STL North Canton LQM and all policies and procedures within the laboratory.
- Has signature authority for LQM, policies, SOPs, and contracts (as detailed in STL policy)
- Annually assesses the effectiveness of the QMP and LQM within the operation
- Maintains adequate trained staffing documented on organization charts
- Responsible for implementing internal/external audit findings corrective actions.

1.6.3 Operations Manager/Laboratory Supervisor

- Reports directly to the Laboratory Manager
- Supervises daily activities of the Operational Groups
- Schedules analytical operations
- Supervises QC activities performed as a part of routine analytical operations
- Implements data review procedures
- Supervises the preparation and maintenance of laboratory records
- Supervises maintenance of instruments and scheduling of repairs

- Works with the Project Managers and Group/Team Leaders to assure the requirements of projects are met in a timely manner
- Supervises daily activities of the Sample Control Group.

1.6.4 *Laboratory Technical Director*

- Reports directly to Laboratory Manager
- Responsible for the technical operation of the laboratory
- Responsible for coordinating the development and implementation of SOPs
- Has joint signature authority for LQM, policies, SOPs, and training records
- Performs technical training in area(s) of expertise
- Interfaces with management on technical needs and solving day-to-day technical issues
- Investigates technical issues related to projects as directed by QA
- Evaluates new methods, technical proposals, and statements of work
- Certifies technical laboratory personnel based on education and background to ensure that staff have demonstrated capability in the activities for which they are responsible by reviewing and signing analyst demonstrations.
- Performs other tasks as required by NELAC.

The Technical Director meets the requirements specified in the Section 4.1.1.1 of the NELAC standards.

1.6.5 *Project Manager Group Leader*

- Reports directly to the Laboratory Manager
- Supervises daily activities of the Project Management and Administrative Groups
- Works with the Operations Manager and/or Group/Team Leaders to ensure the requirements of projects are met in a timely manner

1.6.6 *Customer Service Managers (CSMs)*

- Reports directly to the Project Manager Group Leader
- Has signature authority for contracts for laboratory services, as detailed in STL policy, and for laboratory reports.
- Defines customer requirements through project definition
- Assesses and assures customer satisfaction
- Provides feedback to management on changing customer needs
- Brings together resources necessary to ensure customer satisfaction.

1.6.7 *Project Manager*

- Reports directly to the Project Manager Group Leader
- Monitors analytical and QA project requirements for a specified project
- Acts as a liaison between the client and the laboratory staff

- Prepares Quality Assurance Summary (QAS) or equivalent summary form and communicates project-specific requirements to all parties involved
- Assists the laboratory staff with interpretation of work plans, contracts, and QAPP requirements
- Oversees project data packages for completeness and compliance to client needs
- Has signature authority for final reports
- Keeps the laboratory and client informed of project status
- Together with the QA Manager, approves customer requested variances to methods and to standard laboratory protocols
- Monitors, reviews, and evaluates the progress and performance of projects
- Reports client inquiries involving data quality issues or data acceptability to the facility QA Manager and to the operations staff
- Conducts project reviews to assess the laboratory's performance in meeting customer requirements
- Prepares reissue requests for project data
- Responsible for meeting quality requirements.

1.6.8 *Group (Area) Leader, Team Leader or Supervisor*

- Reports directly to the Operations Manager
- Supervises daily activities of analyses within the group
- Supervises QC activities performed as a part of routine analytical operations
- Implements data review procedures
- Supervises the preparation and maintenance of laboratory records
- Evaluates instrument performance and supervises the calibration, preventive maintenance, and scheduling of repairs
- Oversees or performs review and approval of all analytical data
- Reports nonconformances to the appropriate managers
- Responsible for generation of SOPs for their section
- Responsible for meeting quality requirements.

1.6.9 *Analyst*

- Performs analytical methods and data recording in accordance with documented procedures
- Performs and documents calibration and preventive maintenance
- Performs data processing and data review procedures
- Reports nonconformances to the Supervisor/Manager and QA Manager
- Ensures sample and data integrity by adhering to internal chain-of-custody procedures
- Responsible for meeting quality requirements defined in this LQM and other supporting QA procedures.

1.6.10 Sample Custodian

- Ensures implementation of proper sample receipt procedures, including maintenance of chain-of-custody
- Reports nonconformances associated with condition-upon-receipt of samples
- Logs samples into the LIMS
- Ensures that all samples are stored in the proper environment
- Assists Environmental Health and Safety staff with sample disposal
- Responsible for meeting quality requirements.

1.6.11 Report Production Staff

- Accurately generates and compiles analytical reports and associated deliverables for delivery to the client
- Responsible for meeting quality requirements
- Produce as needed reports that meet the NELAC requirements.

2.0 Quality System and Description

2.1 Objectives of the STL Quality System

The Quality System is a set of management principles, objectives, policies, responsibilities, and implementation plans at the organizational and project-specific levels. The goal of the STL Quality system is to ensure that business operations are conducted with the highest level of professionalism in the industry. To achieve this goal, it is necessary to provide STL clients with not only scientifically sound, well documented, and regulatory compliant data, but also to ensure that STL provides the highest quality service available in the industry. A well-structured and well-communicated Quality System is essential in meeting this goal. STL's Quality System is designed to minimize systematic error, encourage constructive, documented problem solving, and provide a framework for continuous improvement within the organization.

2.2 Structure of the STL Quality System

At the highest level, the STL Quality Management Plan (QMP) is the basis for STL's Quality System. The QMP provides the guidance under which all STL facilities conduct their operations. This Laboratory Quality Manual (LQM) describes the implementation of the Quality System at the STL North Canton laboratory. This LQM and the series of associated quality documents described in Section 2.3 define the organization, project-specific principles, goals, controls, and tools of the Quality System as it is applied at this laboratory. The Quality System as described in this LQM demonstrates the commitment to accepted laboratory practices by STL North Canton.

2.3 Quality Assurance and Quality Controls

Quality Assurance (QA) is defined as the system of activities which ensures the quality of a process, product, or service. Quality controls (QC) are the tools used to monitor and regulate the the desired type and quality of product. The QA activities and QC controls employed in STL North Canton are defined in the following quality documents.

2.4 Quality Documents

The STL Quality System is developed from the reference documents shown in Table 2.4-1. The review and control of the STL North Canton documents described in the following subsections is described in Section 3 of this LQM. A cross-reference of the LQM to NELAC requirements quality manuals is presented in Table 2.4-2.

2.4.1 STL Quality Management Plan (QMP)

The requirements set forth in the QMP are applicable to all STL facilities. The policies and practices outlined in the QMP are minimum guidelines only. Requirements that are more rigorous may be applied for specific client or regulatory programs.

2.4.2 STL Company-Wide Policies

Severn Trent Laboratories has certain policies that apply company-wide. These policies are consistent with the QMP, and set forth requirements that all STL facilities are to follow.

2.4.3 Laboratory Quality Manual (LQM)

This STL North Canton LQM along with the associated policies and SOPs, provides the criteria and specifications for the generation of environmental analytical data. The LQM provides QC criteria for standard procedures, facility-specific instrumentation, and reporting.

2.4.4 STL North Canton Quality Policy Documents

Quality policies are referenced throughout the LQM that provide further detail to specific requirements of the QMP and LQM. These policies describe general quality objectives and guidelines in effect for this facility, rather than the details of specific practices. Refer to Table 2.4-3 for a list of the STL North Canton quality policies and required approvals. Table 2.4-4 lists the frequency of review for the documents.

2.4.5 Standard Operating Procedures

Standard Operating Procedures (SOPs) describe step-by-step instructions for performing a method or activity. In addition, there are SOPs which relate to other support services performed in the laboratory. Details of SOP format and document control are described in Policy QA-001, "Standard Operating Procedures" and Policy S-Q-001, "Official Document Control and Archive." SOPs that are actively used in this laboratory are listed in Table 8.2-2. SOPs are living documents and may supersede some requirements in this document until the LQM is updated annually.

2.4.6 Quality Assurance Project or Program Plans (QAPPs)

Regulations and contracts may contain QA requirements which are different from those described in this LQM. To address unique project requirements, Quality Assurance Project Plans (QAPjPs) may be prepared and implemented. The requirements documented in a

QAPjP, as agreed to by STL North Canton, take precedence over the LQM for that project. Typical specifications contained in a QAPjP or similar documentation include:

- New or modified testing methods
- Unique QC logic
- Special requirements for equipment use and maintenance
- Special handling due to safety considerations
- Project-specific detection and reporting limits
- Project-specific accuracy and precision limits or the statistical treatment of data
- Additional or unique documentation or records management requirements.

2.4.6.1 Quality Assurance Summary

Quality Assurance Summaries (QAS) or equivalent (e.g., Client Requirement Checklist in LIMS) are used to distill client-specific requirements typically documented in project QA plans onto a concise format, highlighting the requirements that are different than the laboratory standard practice. The summary describes for each project the required quality control samples, batching schemes, flagging conventions, deliverables, or other special client requests that may differ from routine laboratory operations. The QAS or equivalent is disseminated to laboratory operations by the Project Manager or Quality Assurance Manager to document client or program specific requirements. The QAS may be used alone or in conjunction with the project-specific QA plans.

2.4.7 Other Documents

Other documents which can affect the quality program may include the Chemical Hygiene Plan (CHP), memos, guidance documents, work instructions, and periodic management assessment reports. These documents may further define or guide the implementation of quality standards at STL but shall not conflict with the LQM or diminish the effectiveness of the Quality System

3.0 Document Control and Records Management

3.1 Objectives for Control of Quality Documents and Records

Quality Documents - The quality documents discussed in Section 2 of the LQM define the framework of the STL Quality System. Control and security of these documents are necessary to ensure that all staff have access to current policies and procedures at all times, to ensure that all changes to the policies and procedures are properly reviewed, to ensure that the history of use of documents can be reconstructed, and to ensure that confidential information is not improperly distributed. The system described in this section is designed to accomplish these objectives.

Vital Records – Vital records are the documents that provide objective evidence of the performance of a process or observations of an item. Records management ensures that results produced by the laboratory are scientifically and legally defensible, and ensures that project events can be reconstructed. Confidentially of the records and records retention requirements are discussed in this section.

3.2 Document Control Procedures

Unambiguous identification of a controlled document is maintained by identification of the following items in the document header:

- document title,
- unique document number,
- revision number,
- revision date,
- effective or implementation date, and number of pages

Controlled documents are marked as such, and the QA department keeps records of document distribution. Controlled distribution may be achieved by either electronic or hardcopy means. The effective date is the date when controlled copies are distributed. Controlled documents must be available in the immediate areas where the related work is performed. Details of the numbering system, required format, and restrictions for uncontrolled distribution of documents are in Policy # QA-001, "Standard Operating Procedures" and STL Corporate SOP # S-Q-001, "Official Document Control and Archive".

3.3 Document Review, Approval, and Revision

Controlled quality documents are authorized by the Laboratory Director, the Technical Manager, and the QA Manager. They indicate their authorization by signing the cover page of the document. STL North Canton quality documents, the individuals responsible for reviewing the documents, and the required frequency of review are listed in Table 3.3-1 and Table 3.3-2. In addition to periodic review and revision, quality documents must be

revised when a procedure or activity changes in a significant manner. Amendments to documents must be reviewed and approved by the same parties approving the original document, distributed in a controlled manner, and clearly indicated in the document. Obsolete versions of documents are removed from service when new revisions are issued. The QA Department maintains a record of history of use of all documents based on the effective date. For further details see Policy # QA-001, "Standard Operating Procedures"

3.4 Records Management

Records may be either hardcopy or electronic copies. It is not required to maintain both if they are properly secured and are complete and true copies. The record keeping system allows for reconstruction of all laboratory activities that produced the analytical results. The history of the sample is readily understood through the documentation. This includes

- chain-of-custody records, including intra-laboratory and inter-laboratory transfers of samples;
- records identifying the personnel involved in sampling, preparation, calibration, and testing;
- observations, calculations, and derived data;
- information relating to laboratory facilities, equipment, analytical test methods, and related laboratory activities (e.g., sample preparation, standards preparation, and data verification),
- original records clearly identifying all subcontracted test data, and
- a copy of the final test report

Requirements for data recording are described in Policy # QA-008, "Data Recording Requirements". Details concerning control of electronic records are given in Policy # QA-017, "Electronic Reporting". The types of vital records maintained are listed in Table 3.4-1

STL North Canton utilizes a controlled access public drive to post controlled electronic QA records such as MDLs, SOPs, Laboratory or QA forms, Certifications, etc. Only QA personnel have write access to the QA public drives labeled, "QAQC" and "QA". The records are maintained and backed-up according to the record retention policy. The processes for producing these electronic (or hardcopy) records are described in their specific QA Policy, SOP, and/or the STL QMP.

3.5 Document and Record Storage, Retention, and Disposal

It is the policy of STL North Canton that company records will be available to meet business needs and comply with all applicable legal records retention and disposition requirements. STL North Canton retains copies of records in a manner that allows prompt retrieval of documents and records for inspection purposes In accordance with NELAC, all quality

documents and records are stored for at least five years Other types of records have different retention requirement, refer to Table 3.4-1 for details.

STL North Canton retains copies of all vital records in a manner that allows prompt retrieval of documents and records for inspection purposes. In accordance with NELAC, all quality documents and records are stored for at least five years.

Specific projects and regulatory programs have longer record retention requirements than the standard STL record retention time. Refer to QMP Table 5 for a listing of examples of special program requirements. The inventory sheet accompanying the stored records must include disposal instructions, which take into account any special requirements, and who to contact for authorization prior to destroying the data

When records, as contained in files, are transferred to a records storage area or off-site storage area, they shall be placed in suitable containers and include an inventory sheet (hard copy or electronic) prepared by the person submitting the records. The contents of each container shall be compared to the inventory sheet and labeled If there are any discrepancies, the container and inventory sheet shall be returned to the person who prepared the box for correction. Archives are indexed such that records are accessible on a project or temporal basis. Archives are protected against fire, theft, loss, deterioration, and vermin. Backup copies of electronic media are stored in off-site archive facilities and are protected against deterioration caused by magnetic fields and/or electronic deterioration. Access to archives is controlled and documented. Further details of the laboratory's document and records archiving process are described in SOP # NC-QA-0019, "Records Information Management".

If the laboratory transfers ownership, vital records will be transferred to the new owner. If the laboratory goes out of business, vital records will be transferred to another operating STL laboratory or to our clients.

3.6 Data Confidentiality

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. 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.

In some cases the client may identify projects requiring confidentiality due to national security. Information concerning these projects will be limited only to those STL North Canton associates with a need to know.

The audit reports supplied by federal, state, and local regulatory agencies are public information and can be released without written consent of those agencies. However, specific client audits are confidential and must be approved by the client before releasing them to a third party.

4.0 Staff Qualification, Orientation and Training

All activities performed by STL North Canton shall be accomplished by qualified personnel. Each staff member must have the combination of experience and education needed to demonstrate the required knowledge for his or her position. Each must also have an appropriate general knowledge of laboratory operations, test methods, quality assurance and quality control procedures, and records management. Minimum training requirements are shown in Table 4.0-1. SOP # CORP-QA-0013 describes details of the training process and documentation. The Chemical Hygiene Plan (CHP) describes details for health and safety training.

4.1 Qualifications

STL North Canton maintains job descriptions for all positions. These job descriptions specify the minimum qualifications for education and experience, knowledge and skills, which are necessary to perform at a satisfactory level. Qualifications of professional staff are documented by resumes that include academic credentials, employment history, experience, and professional registrations. A copy of each person's resume is maintained in an electronic file, and is readily available for inspection

4.2 Orientation and Technical Training

Each new staff member shall receive orientation in quality and in health and safety Each new staff member shall be supervised in their assigned duties by their supervisor or a knowledgeable individual designated by the supervisor. The ability and authorization to perform independently shall be documented in the training files, as described below, with technical duties approved by the Technical Director or designee.

4.2.1 Quality Assurance (QA) Orientation

Each new staff member will receive a QA orientation. The QA Manager or designee will conduct this orientation within two weeks of the new employee's first day on the job. The orientation will, at a minimum, include the following topics:

- STL Quality System and hierarchy of quality documents (QMP, LQM, policies, and SOPs);
- key elements of the LQM and the Quality Control Policy (QA-003);
- introduction to the nonconformance memo (NCM) system and corrective action procedures;
- proper data recording practices;
- STL ethics agreement, including the potential consequences of unethical behavior; and
- the role of the QA department.

The QA orientation will be documented on a checklist, which is signed by the trainee. The documentation will be placed in the employee's training file.

4.2.2 Quality Training

Continued training in the mission and goals of the QMP and LQM shall be provided at least annually. These may be done in a single session or divided into separate sessions conducted at different times throughout the year. Formal training sessions are conducted and documented by the QA Manager or designee. In addition, each lab staff member shall read and document their awareness of the quality documents related to his or her position.

4.2.3 Health and Safety, Orientation and Training

Each new employee, contract worker, or working visitor is required to go through health and safety orientation and training as described in the CHP. The Health and Safety Coordinator must conduct the orientation as soon as possible after the individual reports to work and before chemicals are handled. More comprehensive health and training, both initial and on going, must be completed at the frequency given in the CHP

4.3 Training Files

Each active STL North Canton staff member has an individual training file maintained by the QA Manager or designee. This file can be documented on paper forms or in a database The following sections shall be included in the training files at a minimum:

- Resume - containing hardcopy or a reference to the electronic file
- Quality Assurance - containing documentation of QA/QC orientation and training completed
- Health and Safety - orientation and training documents
- Technical Proficiency - initial and on-going demonstrations of proficiency, one-on-one training, training courses or workshops on specific equipment or analytical methods is documented in this file. Note that documentation of awareness of technical SOPs is document on the demonstration of capability forms (see next section)

Other types of records to be included in the training file include work place regulatory compliance training, and professional development courses. The exact contents will vary depending upon a person's job function and tenure with the company. Details of requirements for training records and the approval process are given in SOP CORP-QA-0013.

4.4 Technical Proficiency Training

All new personnel are required to demonstrate competency in performing a particular method by successfully completing a Demonstration of Capability (DOC) before conducting analysis independently on client samples. On-going proficiency must be demonstrated annually.

DOCs are most commonly performed by analysis of four replicate QC check samples. Results of successive LCS analyses can be used to fulfill the DOC requirement. As required by the referenced method, the accuracy and precision, measured as average recovery and standard deviation (using n-1 population), of the four replicates are calculated and compared to the method limits or against current laboratory limits if multi-laboratory method acceptance limits are not specified. Single-blind proficiency samples and other NELAC acceptable proficiency samples are described in SOP # CORP-QA-0013. The DOC Certification documentation must be signed by the Technical Director and the Quality Assurance Manager and filed in the employee's training file (see example in CORP-QA-0013). The DOC Certification documentation must include a statement that the individual has read, understood, and agreed to perform the most recent version of the test procedure. In procedures such as %Solids, Color, Dissolved oxygen, Ignitability etc., where spiking is not an option and for which quality control samples are not readily available, the proficiency can be demonstrated by analyzing a duplicate sample provided the RPD≤10%.

Figure 4-1 Employee Minimum Training Requirements

Required Training	Time Frame	Employee Type
Environmental Health & Safety	Initial training before start of production work.	All
	Additional training as specified in the CHP	As required
Quality Assurance	Orientation within 2 weeks of hire date	All
	Annual QA program training	All
Technical Proficiency	Initial demonstration prior to unsupervised method performance	Technical staff
	Annual on-going demonstration	Technical staff

5.0 Procurement of Supplies and Services

Controlling the quality of supplies and services is necessary to ensure that STL North Canton provides high quality analytical services to our clients. The STL procurement program requires:

- assurance that purchased items and services meet requirements set by STL North Canton and perform as expected
- definitions and descriptions of the levels of documentation required for applicable technical and administrative procurement functions
- maintenance of records of all suppliers from whom we obtain services or supplies required for our analytical testing

Additional information is contained in QA Policies # QA-018, "Vendor Approval" and # QA-019, "Vendor Review and Oversight"

5.1 Selection of Vendors

Materials and supplies are purchased from approved vendors. Prospective vendors are selected based upon criteria appropriate to the materials or supplies provided. Policy # STL /PG-001 "Procurement and Contracts" details the process used. For national vendors and contracts, the vendor is selected by the STL Procurement Director through a competitive bidding process, strategic business alliance or negotiated vendor partnership. Potential vendors are required to complete a vendor acceptance application and are evaluated on the following criteria, as appropriate:

- the vendor's history of providing identical or similar products that perform satisfactorily in actual use
- the vendor's service record and ability to provide a complete product line and commensurate service
- the vendor's ability to administer inventory at the STL North Canton facility through an inventory management system that will ensure correct stocking levels as well as shelf-life tracking
- objective evaluation of the vendor's current quality records, supported by documentation
- results of audits by STL of the vendor's technical and quality capabilities

Vendors that provide measuring equipment, solvents, chemical standards, instrument service contracts, or subcontracted laboratory services shall be subject to more rigorous controls than vendors that provide off-the-shelf items.

5.2 Controlling Quality of Purchased Items

The quality of equipment, reagents, solvents, chemical standards, gases, and laboratory containers used in analyses must be of known quality so that their effect upon analytical results can be defined These quality specifications are derived from analytical method

requirements, project-specific requirements, and defined national standards for analytical testing. Quality specifications of materials are described in analytical SOPs. These quality specifications shall be included or referenced in the purchasing documents for the items being purchased. This includes specifications for the purity of standards, reagents, or chemicals, and technical specifications for accuracy and precision (e.g., Class A volumetric glassware). Reference to a catalogue number, model, lot number, or chemical grade is sufficient.

The Laboratory Director or designee has the responsibility for approving purchase orders. The section supervisors or designees are responsible for ensuring that the requested quality of materials ordered matches those received, for verifying that material storage is properly maintained and for removing materials from use when shelf life has expired

5.2.1 Evaluation of Off-the-Shelf Items

For items that are used regularly by STL North Canton where no unique requirements or specifications exist, the items may be purchased off-the-shelf. These items are ordered from the supplier on the basis of specifications set forth in the supplier's published product description. These include items such as glassware, filter paper, pipettes, and chromatography columns. The items are evaluated as a function of the standard analytical process.

5.2.2 Evaluation of Instruments

Evaluation of instruments purchased shall be conducted according to an acceptance-testing plan . The acceptance testing plan may be defined by the vendor or the method demonstration requirements specified in the laboratory analytical SOPs. Acceptance criteria may include instrument reliability, sensitivity, stability, selectivity, accuracy, precision, and ability to interface with existing computer systems

5.2.3 Evaluation of Critical Solvents and Acids

STL North Canton is part of a group of STL laboratories that conducts additional evaluations for certain solvents and chemical reagents where our criteria for purity are more stringent than the vendor's. These QRIs are subject to chemical analysis on a lot-by-lot basis before they are put into use. They are tested at one of the STL laboratories, and the chemical test results are evaluated by a designated quality representative. If the solvents or reagents meet the specifications given in SOP # CORP-QA-0001, "Quality Testing of Solvents, Acids, and Reagents (QRI Program)" an approval memorandum is issued to all participating laboratories. All laboratories then use the same lot, and reject any lots received at the facility that have not been tested.

5.2.4 Evaluation of Chemical Standards or Standard Reference Materials

Where available chemical standards will be traceable to the National Institute of Standards Technology (NIST) or an equivalent source. This is largely limited to physical and inorganic chemical standards. If NIST traceability is not commercially available, commercially certified materials shall be used, which are then tested for accuracy before reporting data. Details of the testing procedures and documentation are described in the laboratory SOP. Standards must be received with a certification report from the vendor with information such as purity/concentration, traceability, lot number, expiration date, preparation date, unique identification number, formula weight, density, radionucleide half-life, mass and/or volume of standards, and suggested storage requirements. Further details about labeling and handling of standards is described in Section 8 of this LQM.

5.2.5 Corrective Action for Failure to Meet Required Specifications

Corrective actions for failure of an item to meet required specifications are as follows:

- review of current supplies to eliminate the problem item
- notification to the STL Procurement Director to avoid additional problems at other STL labs
- return of the problem item to the vendor
- evaluate the impact on product or process

The QA Manager shall be notified of any significant or systematic quality problems The STL Procurement Director and the STL Quality Assurance Director shall be notified of any quality problems with national vendors.

5.3 Procurement of Subcontract Laboratory Services

Whether external to STL or not, all subcontracting from the STL North Canton laboratory to another laboratory is arranged with the documented consent of the client, in a timely response that 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. Documentation of required certifications from the subcontract facility are maintained in STL project records. Where applicable, specific QC guidelines, QAPjPs, and similar project documents 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 an assessment of compliance with the required test method, QC requirements, documentation, as well as any special client requirements.

Project reports received from external laboratories are not altered and are included in original form in the final report provided by STL. Intracompany subcontracting may also occur between STL facilities The originating laboratory is responsible for communicating QA/QC, reporting, and other project requirements.

The final report from STL North Canton clearly identifies what testing was performed by other laboratories, and, per NELAC, the certification status of the lab performing the work.

6.0 Computer Hardware and Software

The primary purpose of quality assurance systems for computer hardware and software is to protect the integrity of computer-resident data. Procedures are in place at STL-North Canton to assure that computer-resident data are accurate, traceable to a known source, protected against loss, and secure

STL's computer and hardware controls are based on the guidance in EPA's "Good Automated Laboratory Practices" (GALP), August, 1995. This includes both corporate level Information Technology (IT) functions and STL-North Canton IT functions. Some GALP requirements, such as management responsibilities and the training program, are addressed in other sections of the LQM. Some corporate level IT functions, such as the system change management procedures, is described in more detail in corporate IT documents. Table 6-1 provides a cross reference of practices outlined in Section 8 of the GALP manual to corresponding sections of STL's QA and IT documents. STL North Canton's listing of hardware and software are in Tables 6-2 and 6-3

6.1 Computer Hardware

Computer hardware used in the generation, measurement, or assessment of client data shall be of appropriate design and adequate capacity to function according to specifications Computer equipment must be installed in accordance with the manufacturer's recommendations, and undergo documented acceptance testing.

6.1.1 Wide-Area Systems

STL-North Canton's LIMs (QuantIMs) and the Office Network run on a wide-area network (WAN) serving multiple laboratories. The central node for the network is located at the Denver facility. The central processor is an IBM AS-400 with multiple servers and Cisco routers. Records for the system architecture, testing and maintenance, such as Initial Program Loads (IPLs), are documented in the AS-400 System Log, which is also in Denver The central System Administrator maintains records for installation of the network hardware.

6.1.2 Local Systems

The local systems consist of computer equipment for analytical instruments, data evaluation, and upload to the LIMS. A local-area network (LAN) supports the local office software. Testing, maintenance, and repair of the local computer hardware are the responsibility of the STL-North Canton LAN Analyst. The LAN Analyst maintains documentation for the local systems.

6.2 Facilities and Security

6.2.1 Central Computer Facilities

The environmental conditions of the facility housing the LIMS are controlled to protect against data loss Access to the central computer facility in Denver is restricted by keypad entry used by IT staff. The central computer room is temperature controlled, and has an Uninterrupted Power Supply (UPS) plus a power generator to ensure that the WAN functions are not disrupted by power failures. Backup media, such as tapes and disks, are maintained daily. In addition, full volume backup copies of the raw data are shipped offsite to a commercial facility specially designed to store electronic data

6.2.2 Local Computer Facilities

Facilities for housing local computer hardware must meet manufacturer's recommendations. Electronic data must be protected against environmental hazards such as fire, water damage, and strong electromagnetic fields. Data files will have backup copies made at regular intervals to protect against accidental loss through hardware or software failure.

6.2.3 Controlled Software Access

The integrity of data is also assured by maintaining limited access to administrative functions through a hierarchy of operating system shells controlled by passwords. Access is granted by the LAN Administrator depending on a persons experience, training, and assigned duties (see SOP# CORP-IT-0005 for more details).

Protection against unauthorized Internet access is provided by firewalls.

6.2.4 Virus Protection

Commercial virus protection programs are installed on all computers to detect and remove computer viruses. LAN Analysts are to be notified whenever a virus is detected so that they can isolate any portions of the systems that may be at risk.

6.3 LIMS Raw Data

QuantIMs raw data and instrument raw data from instrument data systems such as Target, IDB, and Chemstation are stored on the Office Automation servers (e.g., QARVCO01). The Systems Administrator and the LAN Analyst are responsible for maintaining the servers.

The individuals responsible for entering and recording raw data must be uniquely identified in the data, together with the date and time the data were entered. The instrument transmitting raw data must be uniquely identified, together with the date and time of the transmission. Further data recording requirements exist to document manual integrations (see Policy # QA-011 for details)

Procedures for verifying raw data are discussed in LQM Sections 8.8-8.8.3.

6.4 Software

If computer software is used to acquire, process, or report client data, that software is tested to ensure that it correctly performs its intended function. Software is validated or verified, depending upon its complexity, size, and whether it was purchased or developed by STL. The following definitions are used by STL:

- Validation - the process of establishing documented evidence, which provides a high degree of assurance that a specific process will consistently produce a product meeting, predetermined specifications and quality attributes. This process demonstrates and documents that the software performs correctly and meets all specified requirements

- Verification - the process of checking the accuracy of automatically (electronically) calculated information

6.4.1 Industry Standard Software

Industry standard software programs are defined as those, which are purchased and widely used without modification to the program itself. The program is initially verified for use by using test problems with known solutions to demonstrate that the program is operational for the desired application.

All purchased software must be used in accordance with the terms of its software license. Any use of software contrary to its license terms is expressly prohibited by STL.

6.4.2 Testing of STL-Developed Software

For programs used to process client data and developed within STL, and externally prepared programs, which are modified by STL, validation or verification must be performed. The process used is dependent upon the function of the software as follows:

- Large complex systems consisting of several programs operating in unison to produce an intended result must be validated.

- For smaller software which only performs numerical manipulation, sample sets of numbers for which results are known should be processed and the results verified In this case, known results are usually generated by performing hand calculations using the same equations and procedures as the software to verify that the software produces identical results

- Software that performs as part of instrument operation should be verified as previously described and by processing reference materials through the instrument system. Processed instrument response should be evaluated against expected instrument response and performance.

IT SOPs governing software development and testing include CORP-IT-0001, "Software and Hardware Change Management", CORP-IT-0007, "Software Testing, Validation and Verification", and CORP-IT-0013, "Software Quality Assurance".

6.4.3 Control of Software Changes

STL has a well established process for prioritizing and managing changes to LIMS and LIMS-related software (see CORP-IT-001 and CORP-IT-007). Proposals to modify software are written in a Software Enhancement Request, which includes a description of the task to be accomplished, the software to be modified, its functional requirements, and necessary algorithms. The Software Enhancement Request is submitted to the Change Management Committee for approval. The Committee includes representatives from each lab on the QuantIMs network. The Committee establishes a develop schedule and approves the resources needed. Documentation of changes, version control, and historical records of changes is the responsibility of the IT Manager of "Change Management and QA". Because these are modern networked systems, the documentation is kept on the network, rather than keeping redundant records at each facility as GALP suggests. All system software changes are developed in a test area and must pass the designed tests before it is installed in the working area.

The same principles of documenting software changes apply to spreadsheets, small databases, or other small programs that are used solely at the STL-North Canton lab The verification/validation records must explain the functional requirements, the algorithms and formulas used, the testing performed, and are maintained by the lab QA Manager or designee.

6.4.4 Software Maintenance

Software problems are presented to the local LIMS Administrator (LAS) in a Software Problem Report. The LAS presents the issue to a group of the network LASs. The problem is discussed to make sure it is understood, and then a solution is determined and prioritized. Changes to LIMS software for maintenance purposes are announced to each of the QuantIMs locations after revalidating the software

6.4.5 Software Revalidation

Whenever a program is changed, the change is evaluated to determine if it is significant enough to make revalidation necessary If features have been added, previous test

problems are rerun to demonstrate that their function has not been affected. New test problems are processed, as previously discussed, to verify added performance. If software revision changes the basic operation of the program, complete revalidation of the program may be required.

Spreadsheets and unprotected software used to acquire, process, or report client data must be documented and reverified when changes are made. The test problems used to provide initial verification is reprocessed and the results compared to demonstrate that performance of the software is unchanged

Laboratory operations are responsible for the generation of the validation and verification data for instrument level software. QA will maintain the necessary documentation. STL Information Technology is responsible for generation and maintenance of documentation relating to verification and validation of the STL QuantIMs system. This is described in Policy Attachment Number IT-013, Software Quality Assurance.

6.5 Comprehensive System Testing

Comprehensive system testing is performed periodically. Independent auditors, such as Price Waterhouse, include computer systems in their audits, which are commissioned by the laboratory executive management. Extensive testing of all software was performed for the lab's Y2K readiness exercises.

As described in LQM Section 9.2 2.1, the STL-North Canton QA Manager is responsible for ensuring an annual internal audit of all lab areas is performed, including the local IT functions

6.6 Records Retention

As required by NELAC, electronic raw data and computer documentation are stored for a minimum of five years See LQM Section 3.0 for further records retention details.

7.0 Contract Review and Project Planning

The generation of environmental analytical data is an intricate process Success is dependent upon the timely execution of interrelated steps. For many environmental sampling and analysis programs, testing design is site or project specific and is not necessarily the same as the laboratory's standard service. It is STL's intent to provide both standard and customized laboratory services to our clients, provided that any special requirements are documented in writing, and provided performing the work in this manner does not cause the laboratory to violate relevant regulatory requirements. STL North Canton has an organizational system in place to ensure that projects are properly planned prior to project initiation. This means that laboratory personnel understand project requirements, that the client clearly understands the lab's capabilities, that the laboratory has the facilities and resources needed to perform the required tests, that samples will be properly handled, that contingency plans are in place, and that analytical data will be reported in accordance with project needs.

7.1 Contract Review

The process of client request for proposal (RFP) and the laboratory's tender of a written response is a process of communication between both parties to understand project requirements and the laboratory's capabilities All contracts for new work entered into by STL North Canton are reviewed by the Customer Service Manager (CSM) or designee. Agreements for continuing work are the responsibility of laboratory Project Managers (PMs) or the CSM Depending on the size and scope of the proposed project, the Laboratory Director and other STL management staff can also be involved. Technical staff (Operations Manager, QA Manager, and IT staff) can be called upon to perform a review of the technical and QA/QC requirements. The CSM or PM, with this internal support, will work with clients to align project requirements with laboratory capabilities. Any contract requirement or contract modification communicated to STL verbally is documented and communicated to the client in writing Any discrepancy between client requirements and STL's capability to meet those requirements is resolved in writing before acceptance of the contract

All contracts, Quality Assurance Project Plans (QAPP), Sampling and Analysis Plans (SAPs), contract amendments and documented communications become part of the permanent project record as detailed in Section 3.5

7.2 Certifications and Approvals

A necessary part of the review and work acceptance procedure is the evaluation of project needs for laboratory certification. The persons reviewing the prospective project must determine if project work plans or regulatory permits are tied to specific laboratory certifications or approvals. Where such requirements exist, the laboratory must have the certifications or approvals in place before the work begins. QA personnel coordinate with

the state certification agencies to maintain or add additional parameters. Copies of current laboratory certifications are maintained by the QA office, and are available upon request.

7.3 Data Collection Process

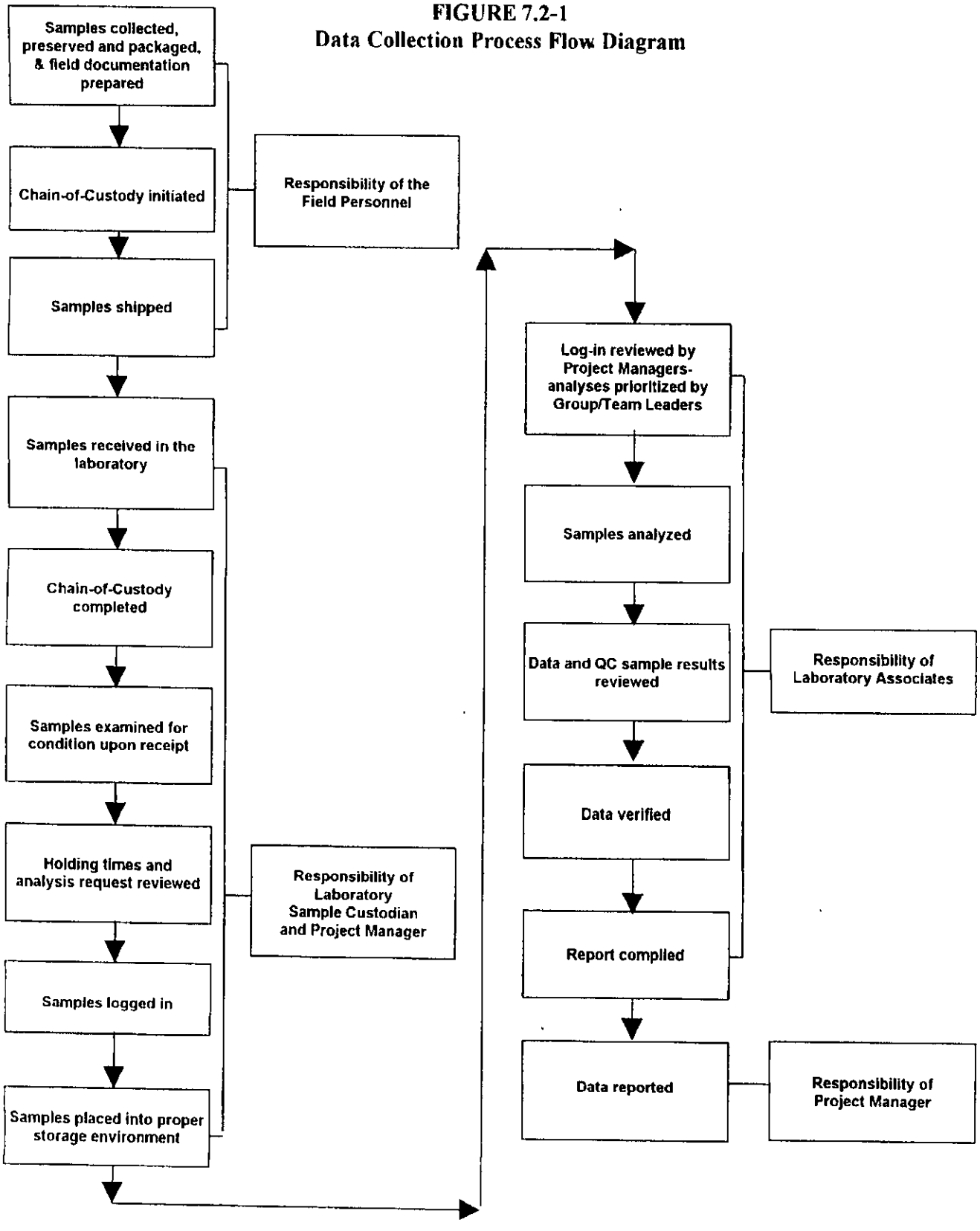
The sample collection and data generation processes are shown in Figure 7.2-1. These processes are designed to produce analytical data that accurately reflect the nature of the site or sampling point.

7.4 Project Organizational Responsibilities

Each laboratory client is assigned a single point of contact, usually a PM, to ensure that there is a strong line of communication between the client and STL North Canton. As a matter of policy, CSMs or designee, PMs, and Operations Managers work together to accomplish the following prior to receipt of samples at the laboratory:

- Samples are scheduled for arrival at the laboratory
- All unique project requirements have been identified and communicated to all appropriate personnel
- Standardized client, state, federal, or STL® programs are appropriately selected
- Fully-qualified and client approved subcontract laboratories have been selected if needed
- A review has been performed on all pre-project documents such as proposals, contracts, and/or QAPPs to identify the type of tests required and to ensure project requirements are within the scope of the laboratory being used
- All appropriate and required preparations have been made at the laboratory to accommodate or meet project requirements as described in proposals, contracts, and/or QAPPs
- It has been determined that the laboratory has the capability and the capacity to analyze the samples including equipment, staff, space and workload
- The laboratory is capable of meeting the required sample holding times and is able to report the resulting data within the time line specified by the client
- All known safety hazards associated with the samples have been communicated to all appropriate personnel.

FIGURE 7.2-1
Data Collection Process Flow Diagram



Approval and issuance of a quote, bid or contract document is documentation that this process has occurred. For particularly large or involved projects, STL North Canton encourages our clients to visit the laboratory and/or participate in kickoff meetings with the laboratory staff STL has found it very effective to invite the client into the laboratory's project preparations.

7.5 Communicating Project Requirements Internally

STL North Canton PMs shall document all project-specific requirements prior to receipt of samples. The LIMS system, QuantIMS, requires the PM to enter a "quote" before any samples can be logged in. In addition to price information, the "quote" is a detailed technical specification of the work to be performed. The quote includes identification of project personnel, numbers and types of samples, tests to be performed, reporting limits, QC to be performed, control limits, data qualifier flags to be used, significant figures to be used, and the types of deliverables required. This is the primary means of communicating routine project requirements to laboratory personnel.

Brief non-routine project requirements are entered into the comments section of the Client Requirements Checklist portion of the quote The Checklist is reviewed by analysts as analyses are being scheduled and before testing has started. If the special requirements are too lengthy for the Client Requirements Checklist, the PM must prepare a Quality Assurance Summary (QAS) or equivalent, which is a written document describing all requirements that are different than routine work. The QAS is referenced in the Client Requirements Checklist, and is distributed by the PM to each of the operational groups involved. For complex projects, project kickoff meetings are conducted by the PM with each of the operational groups involved.

7.6 Contingency Planning

An effective QA program must emphasize contingency planning, actions to prevent problems from reoccurring, and to ensure timely and effective completion of a measurement effort The following are considered relative to contingency planning.

7.6.1 Staffing

A primary objective is to ensure that qualified staff are available to perform the necessary analytical work, regardless of employee turnover, vacation, illness, or other absences STL North Canton is a relatively large laboratory with multiple staff capabilities for the majority of tests performed. However, other sources of trained personnel are potentially available to assist in the event of unforeseen absences. Given sufficient time for necessary orientation, temporary agency staff can be used. More significantly, STL is a large laboratory network and a large pool of qualified staff can be made available from other STL laboratories

7.6.2 Backup Instrumentation

Within STL North Canton, duplicate instrumentation is available for most methods to allow uninterrupted work flow if one piece of equipment fails. The laboratory may also choose to lease equipment. However, in circumstances where a catastrophic instrument failure occurs, alternative, but equivalent, methods may be recommended to the client for approval.

- Preventive Maintenance - STL's preventive maintenance program is designed to minimize analytical instrument malfunctions, permit simple adjustments, and to ensure fewer and shorter breakdowns of critical analytical equipment. (See Section 8.11, "Preventive Maintenance and Service".)

- STL Laboratories & Subcontractor Laboratories - To support the laboratory during peak periods or in the event of a critical instrument malfunction, STL has the capability to arrange for the use of other STL laboratories or other qualified analytical laboratories as subcontractors for short-term backup analytical support. **However, use of a subcontractor laboratory must be approved by the client in writing.** For projects requiring NELAC approval, the subcontractor must also be NELAC approved. See Section 5.3 for other procedures related to the control of subcontract laboratory services.

- Uninterruptable Power Supply - An Uninterruptable Power Supply (UPS) system which provides line conditioning and backup power to the LIMS computer system/server and laboratory instruments. This contingency allows sufficient time for the main computer system to be shut down and for data archival. All electronically generated data that are stored on the main or instrument computer systems and on individual personal computer (PC) hard drives are backed up at regular intervals. In the event that the main or instrument laboratory computer systems fail, the analytical data can be retrieved.

8.0 Work Processes and Operations

Many activities related to environmental projects activities are planned and designed externally to the laboratory or field operation, and are presented to the laboratory in the form of a contract, work plan, sampling and analysis plan (SAP) or QA Project Plan (QAPP). Laboratory and field activities are in turn planned, implemented, and assessed by STL to meet client requirements according to approved procedures and methodologies. The LQM provides the systems to document and implement these activities. The execution and assessment of the implemented operational systems are detailed in STL SOPs. The entire process is assessed on a regular basis for conformance to prescribed requirements.

Standard practices for STL North Canton operations are detailed in this section. Specific project or program requirements that differ from those described here can be met, but they must be explicitly stated in approved contracts, work plans, QAPPs or other project documents. Special project requirements can generally be accommodated provided that they are properly documented, communicated, and they do not cause the laboratory to violate relevant regulatory requirements.

Table 8 2-3 lists the test methods performed by STL North Canton Table 8.2-2 lists the SOPs associated with those methods. Table 8.0-1 provides a list of the major equipment in place at the laboratory, and Figure 8 11 (at the end of this Section) shows the laboratory floor plan.

8.1 Traceability of Measurements

STL documents all laboratory activities in sufficient detail to allow their reconstruction. To this end, documentation is generated to trace a sample from its point of origin, through receipt in the laboratory, analysis, reporting and disposal.

The required documentation includes, but is not limited, to:

- Chain of custody documenting movement and possession of samples
- Sample preparation
- Sample analysis
- Calibration and QC data associated with the samples
- Instrument maintenance
- Control of ancillary equipment and materials (e.g., DI water and glassware)
- Sample disposal
- Final reports

These topics are described in this section. Traceability of chemical standards is also discussed in Section 5 2.4

8.2 Analytical Methods

Whenever possible, STL operations use industry- and regulatory agency-recognized analytical methods from source documents published by agencies such as the Environmental Protection Agency (EPA), Department of Energy (DOE), and the American Society for Testing and Materials (ASTM) as described in STL's SOPs. Analytical methods performed

by STL Laboratories are given in Table 8.2-3 lists the methods routinely performed at the laboratory The methods pending or approved by a NELAC Accrediting Authority are indicated in the table.

Method performance data, as described in Section 8.2.2 below, are developed by the laboratory operations staff to demonstrate method proficiency. The operations staff and the QA staff evaluate and approve the performance data before a methodology is performed routinely. The method must also be described and documented in an SOP.

8.2.1 Standard Operating Procedures

SOPs are required for all repetitive analytical and administrative activities ranging from the receipt of samples in the laboratory through their analysis, reporting, and subsequent disposal Training, health and safety procedures, QC, method procedures, and instrument and equipment calibrations are included in SOPs. SOP requirements are discussed in the Policy # QA-001, "Standard Operating Procedures" The specifications in the policy meet NELAC requirements. Table 8.2-2 lists laboratory standard operating procedures

New SOPs and proposed SOP revisions are reviewed by technically qualified lab personnel. SOPs are controlled documents and are distributed and maintained as described in Policy QA-001. Requirements for SOP approval and frequency of review are listed in Tables 2.4-3 and 2 4-4. All significant modifications to the published method are described in a section of the SOP. All operations must be performed as described in these SOPs.

Planned changes in procedure which may occur due to expected sample matrix effects or project requirements are documented in the project files. These planned changes may be documented using nonconformance memos, NCMs (see discussion of NCMs in section 9.1), project-specific case narratives, or as modifications or additions to associated QAPPs. Every effort is made to obtain client written approval prior to implementing the change

Unplanned deviations in the SOPs, which may occur due to sample matrix or other events, are documented in NCMs and in the project-specific case narratives.

8.2.2 Method Validation and Verification

Before analyzing samples by a new method or method modification, the method must be verified or validated. After which, analyst capability must be demonstrated (see Section 4.4).

8.2.2.1 Method Verification

Method verification is required for methods developed by authoritative agencies, such as EPA or ASTM The level of verification can vary

depending on the type of method or level of modification, but generally should include:

- Determination of method sensitivity,
- Determination of working range,
- An initial demonstration of capability (as specified by NELAC), and
- A written SOP or project-specific written protocol.

Each of these are described in the next section.

8.2.2.2 Method Validation

A complete validation is required for methods developed by STL North Canton. While method validation can take a variety of courses, the following are the key concerns:

Determination of Method Selectivity

Method selectivity is the demonstrated ability to discriminate the analyte(s) of interest from other compounds in the specific matrix or matrices. 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

Method sensitivity is normally demonstrated using the 40CFR 136B method detection limit protocol (see MDLs, section 8.2 3, below), but can also be based on variance of blank results, and signal-to-noise ratios

Determination of Interferences

This is demonstrated by analyzing samples of the matrix of interest that is known to be free of the analyte(s) of interest.

Determination of Range

In most cases, analytical range is determined and demonstrated by comparison of the response of an analyte at different concentrations to targeted criteria. Often the targeted criteria are represented by the goodness of fit or linearity of the experimental data to a continuous mathematical function or curve. The curve is used to establish the range of quantitation, with the lower and upper values representing the upper and lower quantitation limits. Curves are not limited to linear relationships.

Determination of Accuracy and Precision

Accuracy and precision studies are generally performed using replicate analysis of samples of known concentration. The resulting percent recovery and relative standard deviation, or other precision measure, is calculated and compared to a set of target criteria

Documentation of Method

The method is formally documented in an SOP (see policy QA-001 for details). If a method modification is being performed for a specific short-

term project, the modification should be described in a written protocol that is approved by the lab's client, in addition to the in-house approvals required by QA-001.

Continued Demonstration of Method Performance
Continued ability of the lab to perform the method is addressed in the SOP. Generally this is accomplished with the specified calibration and batch QC requirements.

8.2.3 Method Detection Limits

It is STL North Canton's policy to follow the specification in the U.S. EPA 40 CFR Part 136 Appendix B in determining MDLs for chemical tests. The STL requirement for this procedure is further detailed in Policy QA-005 entitled "Determination of Method Detection Limits for Chemical Tests." This policy requires that the MDLs be determined for each analyte of interest representing the aqueous and solid matrices within the capability of the primary analytical methods. STL North Canton has performed MDLs per instrument. Ongoing MDL verification is performed via MDL checks or MDL studies. The laboratory's MDLs are given in Table 8.2-4 .

8.2.4 Instrument Detection Limits

Instrument Detection Limits (IDLs) are required to be performed quarterly for metals constituents and cyanide when analyses are performed in support of Comprehensive Environmental Response, Compensation and Liability Act (CERCLA) activities or when the USEPA CLP SOW protocol is required. IDLs are not required by the SW-846 methods, with the exception of method 6020.

When required, IDLs will be performed in accordance with the procedures defined in the applicable USEPA SOW, ILMO3.0 or subsequent versions, and Policy QA-014, "Determination of Instrument Detection Limits".

Prior to acceptance and use for reporting purposes, all data from detection limit studies and reporting limits must undergo technical review and approval by the laboratory management and QA staff.

8.2.5 Reporting Limits

Reporting limits are established and modified within STL according to the STL Policy QA-009, "Reporting Limits." Two reporting limit conventions are discussed in the policy: the standard Reporting Limit (RL) and the Project-Specific Reporting Limit (PSRL). The standard STL Reporting Limit (RL) is the lowest level at which measurements become quantitatively meaningful. The RL is always greater than the statistically determined MDLs. PSRLs are used when project data quality objectives (DQO) require a reporting limit other than the RL. PSRLs tailor STL's product to meet customer requirements. Higher PSRLs may be established based on maximum contaminant level (MCLs), applicable or relevant and appropriate requirements (ARARs), or project-specific data quality objectives

(DQOs). PSRLs below the lab's standard RL may be used, but they must be supported MDL and the instrument calibration. The STL RLs and PSRLs are maintained by the LIMS.

8.3 Data Quality Objectives

Data quality objectives (DQOs) 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 (EPA 1994)[1]. Typically, DQOs are identified during project scope and the development of sampling and analysis plans. In this LQM, however, we refer to only the analytical DQOs because laboratories generally do not have any authority over sample collection, shipment, or other field-related activities that may affect the data quality of the environmental sample before the sample is received in the laboratory. The EPA has established six primary analytical DQOs for environmental studies. These DQOs are precision, accuracy, representativeness, completeness, comparability, and detectability.

The components of analytical variability (uncertainty) can be estimated when QA and QC samples of the right types and quantities are incorporated into measurement procedures at the analytical laboratory. STL® incorporates numerous QA and QC samples to obtain data for comparison with the analytical DQOs and to ensure that the measurement system is functioning properly. The QA/QC samples and their applications, described in Section 8.4, are selected on the basis of method- or client-specific requirements. Field blanks, field duplicates, and performance evaluation (PE) samples are received from the client as unknown samples. Analytical laboratory QC samples for inorganic and organic analyses may include calibration or instrument blanks, method blanks, background, duplicates, replicates, laboratory control samples (LCSs), calibration standards, matrix spikes (MSs), matrix spike duplicates (MSDs), and surrogate spikes.

8.3.1 Precision And Accuracy

Precision is an estimate of variability, that is, it is an estimate of agreement among individual measurements of the same physical or chemical property, under prescribed similar conditions. The precision of a measurement system is affected by random errors. Precision is expressed either as relative standard deviation (RSD) for replicate measurements greater than two or as relative percent difference (RPD) for duplicate measurements. Table 8.6-1 illustrates the formulae used to calculate units of precision (i.e., RSD and RPD).

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. Systematic errors affect accuracy. For chemical properties, accuracy is expressed either as a percent recovery (R) or as a percent bias (R - 100).

[1] "Guidance for the Data Quality Objectives Process", EPA 600/R-96/005, September 1994.

The precision and accuracy measures that are to be used in evaluating inorganic, organic, and radionuclide constituents at STL are provided in Tables 8.4-5 through 8.4-7, in method-specific SOPs, and in the documentation for the analytical method of interest.

Precision and accuracy are determined, in part, by analyzing data from matrix spike and matrix spike duplicates, unspiked duplicates, LCSs, and single blind audit samples. For radiochemical determinations, counting statistics can also provide an estimate of uncertainty. A description of these QC samples is provided in Section 8.4.

8.3.2 Completeness

Completeness is a measure of the percentage of measurements that are judged to be valid measurements. At a minimum, the objective for completeness of data is 90% for each constituent analyzed.

8.3.3 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 (concentration) is representative of the constituent concentration in the sample matrix. At STL every effort must be made to analyze an aliquot that is representative of the original sample, and to ensure the homogeneity of the sample before subsampling.

8.3.4 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 (i.e., SOPs) and a uniform set of units and calculations for analyzing and reporting environmental data.

8.4 Quality Control Samples

Two types of Quality Control (QC) samples are field QC samples and laboratory QC samples. Field QC samples are collected during the sampling event and are useful in determining sampling precision and accuracy and monitoring for contamination that may occur during collection, transport or storage of environmental samples. Laboratory QC samples are routinely added at the laboratory to the normal sample stream. Successful analysis of these samples demonstrates that the laboratory is operating within prescribed requirements for accuracy and precision. In addition, utilizing matrix-specific laboratory QC samples, information regarding the effect of the matrix or field conditions on the

analytical results can be obtained. The following sections describe common field and laboratory QC samples.

8.4.1 Field QC Samples

When field QC sample collection and analysis are required for a project, it is the responsibility of the project sampling supervisor to ensure that this sampling is performed correctly and at the project-required frequencies. Field QC samples may or may not be identified as such to the laboratory and are considered by the laboratory as field samples for the purpose of QC batching, sample preparation and analysis. Field QC sample results are reported in the same manner as actual field samples, unless a specific deliverable is requested by the client. No correction of the analytical data is done in the laboratory based on the analysis of field QC samples. Field QC sample types, applicability to organic and inorganic analyses, precision and accuracy applications and by whom they are introduced are summarized in Table 8.4-1.

8.4.2 Laboratory QC Samples

Laboratory performance QC is required to ensure the laboratory systems (instrumentation, sample preparation, analysis, data reduction, etc.) are operating within acceptable QC guidelines during data generation as required to meet the client's objectives. Laboratory QC samples consist of method blanks (MB), instrument blanks, laboratory control samples (LCS) and calibration verification samples. In addition to laboratory performance QC, matrix-specific QC is utilized to determine the effect of the sample matrix on the data being generated. Typically, this includes matrix spikes (MS), matrix spike duplicates (MSD), sample duplicates, and the use of surrogate compounds.

Laboratory and matrix-spike QC sample types are summarized in Tables 8.4-2 through 8.4-4. In addition, Tables 8.4-5 through 8.4-7 list laboratory QC samples, acceptance criteria and corrective actions by reference method for inorganic methods, organic methods, and the USEPA CLP Statements of Work respectively. The following sections provide descriptions of laboratory QC samples and their frequency of use. Policy QA-003, "Quality Control Program", describes in detail the QC data evaluation process.

8.4.2.1 Quality Control (QC) Batch

The QC batch consists of a set of up to 20 field samples that behave similarly (i.e., same matrix) and are processed using the same procedures, reagents, and standards within the same time period. This definition of a QC batch is utilized by STL unless there is clear regulatory guidance, contract specifications, or differing client requirements that are explicitly documented. Environmental samples are associated to QC samples in the LIMs via the analytical batch, which is clearly indicated on each analytical

report as a cross-reference between the samples and associated QC. Further details and requirements for the application of the definition of QC batch are described in Policy QA-003.

8.4.2.2 Method Blank

The method blank (MB) is a QC sample that consists of all reagents specific to the method and is carried through every aspect of the procedure, including preparation, cleanup, and analysis. The method blank is used to identify any interferences or contamination of the analytical system that may lead to the reporting of elevated analyte concentrations or false positive data. Potential sources of contamination include solvent, reagents, glassware, other sample processing hardware, or the laboratory environment. In general, the method blank is a volume of deionized laboratory water for water samples, or a purified solid matrix for soil/sediment samples, that is processed as a sample. In the event that no appropriate solid matrix exists, deionized water may be used. The volume or weight of the method blank must be approximately equal to the sample volume or sample weight processed. A method blank shall be prepared with each group of samples processed.

8.4.2.3 Instrument/ Calibration Blank

The instrument blank is an unprocessed aliquot of reagent used to monitor the contamination of the analytical system at the instrument. System contamination may lead to the reporting of elevated analyte concentrations or false positive data. The instrument blank does not undergo the entire analytical process and generally consists of an aliquot of the same reagent(s) used for a sample dilution. Instrument blanks are also referred to as continuing calibration blanks (CCBs).

8.4.2.4 Laboratory Control Sample

A laboratory control sample (LCS) is a laboratory-prepared suitable clean matrix sample that is fortified with target analytes or a solid reference material purchased from an approved vendor. The LCS contains all target analytes specified in the method, and must contain the same analytes as the matrix spike and matrix spike duplicate. For certain regulatory or client programs, an LCS may contain a full list of analytes. However, in these cases, a subset of analytes, as defined by the program, is used to determine the acceptability of a batch of sample data. The LCS recovery data are used to monitor the analytical method performance in terms of analytical accuracy. On-going evaluation of the LCS recoveries demonstrates that the laboratory is performing the method within statistical control (i.e., accuracy and precision) in the absence of matrix interference. The LCS results, coupled with MS data, help determine

whether the laboratory performed the method correctly or the sample matrix affected the analytical results. When a laboratory control sample duplicate (LCSD) is required, a percent recovery for each target analyte is calculated, as well as a relative percent difference (RPD) between the LCS and the LCSD.

8.4.2.5 Matrix Spike

A matrix spike (MS) is an environmental sample to which known concentrations of target analytes have been added. MS samples are analyzed to evaluate the effect of the sample matrix on the analytical methodology. MS samples are generated by taking a separate aliquot of an actual field sample and spiking it with the selected target analyte(s) prior to sample extraction. The MS sample then undergoes the same extraction and analytical procedures as the unfortified client sample. Due to the potential variability of the matrix of each sample, these results may have immediate bearing only on the specific sample spiked and not on samples collected at other locations that are included in the QC batch.

8.4.2.6 Matrix Spike Duplicate

A matrix spike duplicate (MSD) is a second aliquot of a sample that is spiked with the selected target analyte(s) and analyzed with the associated sample and MS sample. The results of the MS and MSD are used together to determine the effect of a matrix on the accuracy and precision of the analytical process. Due to the potential variability of the matrix of each sample, the MS/MSD results may have immediate bearing only on the specific sample spiked and not all samples in the QC batch.

8.4.2.7 Sample Duplicate

A sample duplicate is a second aliquot of an environmental sample taken from the same sample container that is processed identically with the first aliquot of that sample. That is, sample duplicates are processed as independent samples within the same QC batch. The results are compared to determine the sample homogeneity and the precision of the analytical process.

8.4.2.8 Surrogates

Surrogates are organic compounds that are similar in chemical composition and behavior to the target analytes but that are not normally found in environmental samples. Surrogates are added to all appropriate samples and QC samples being tested for organic analytes to monitor the effect of the sample matrix and the procedure on the accuracy of the process.

8.4.2.9 Analytical Spike

An analytical spike is created by spiking target analytes into a prepared portion (i.e., post digestion) of a sample just prior to analysis. It provides information on matrix effects encountered during analysis such as suppression or enhancement of instrument signal levels. It is most often used in elemental analysis involving various forms of atomic emission or atomic absorption spectroscopy. A single analytical spike serves as a single point application of the "method of standard additions" or MSA.

8.4.2.10 Interference Check Sample

An interference check sample (ICS) is a solution containing known concentrations of both interfering and analyte elements. Analysis of this sample can be used to verify background and interelement correction factors.

8.4.2.11 Internal Standards

An internal standard (IS) is a compound or element with similar chemical characteristics and behavior in the analysis process to the target analytes, but is not normally found in environmental samples. The internal standard is usually added after sample preparation. The primary function of the internal standard is quantitation, however, it also provides a short-term indication of instrument performance.

8.5 Data Collection Operations

Laboratory analyses are designed to produce data that are representative of existing conditions present at the time the sample was obtained. The data collection design includes field sampling events, sample handling and custody, analytical operations, data recording procedures, data assessments, data verification, and data reporting requirements and techniques to assess limitations of data use. These operations are discussed in this section through section 8.10.

8.5.1 Field Collection and Shipment

In order to provide a sample that most accurately represents the test matrix, field sample collection personnel must abide by the sample collection guidelines and procedures established by involved regulatory agencies. A significant part of the efforts of regulatory agencies include the use of "approved" sample containers, chemical and physical preservation techniques, and observance of specified holding times. It is imperative that all samples be collected and preserved according to the appropriate analytical method specified in the QAPP (if one exists). Although the sampling may be performed by non-STL personnel, the importance of sampling and transportation of the sample to the laboratory is understood and must be considered

during data validation.

Sampling requirements must be communicated to the sampling team prior to field collection.

Field personnel are responsible for labeling each individual sample collected with the following information:

- Project name
- Unique client sample number
- Sample location (including as appropriate: borehole and depth or grid coordinates)
- Sampling date and time
- Sample preservation
- Analysis required.

An overriding consideration for the resulting analytical data is the ability to demonstrate that the samples have been obtained from the locations stated and that they have reached the laboratory without alteration. Evidence of collection, shipment, laboratory receipt, laboratory custody, and disposal must be documented to accomplish this. Figure 8.5-1 shows an example Chain-of-Custody (COC) form that is used by the STL laboratory to document this evidence. Field personnel are responsible for initiating the COC form.

The prompt shipment of samples to the laboratory is necessary to ensure that required holding times are met. Samples should be shipped by an overnight carrier, be hand-delivered, or transported in a manner that assures prompt delivery to the laboratory. Some sites require an extensive radioactive screening process before a sample may be shipped. In these cases, it is imperative for the Project Manager to maintain good communications with the client to assure proper staffing of the laboratory in response to a decreased holding time.

8.5.2 Sample Containers, Shipping Containers, Preservatives, and Holding Times

8.5.2.1 Sample Containers

A sample container is defined as the sealed enclosure, usually made of plastic or borosilicate glass that the sample is collected in and stored in until analysis. All sample containers provided by STL operations for environmental sampling are new, with the exception of some air sampling canisters, which must be recertified before reuse, and demonstrated to be clean for their appropriate use. All documentation certifying sample container cleanliness must be maintained by the laboratory or the vendor and can be provided to the client upon request.

FIGURE 8.5-1
Example STL Chain-of-Custody Form

Reference Document No. _____
Page 1 of _____

Project Name/No.: _____
Sample Team Members: _____
Profit Center No.: _____
Project Manager: _____
Purchase Order No.: _____
Required Report Date: _____

Sample Shipment Date: _____
Lab Destination: _____
Lab Contact: _____
Project Contact/Phone: _____
Carrier/Waybill No.: _____

Bill To: _____

Report To: _____

ONE CONTAINER PER LINE

Sample Number	Sample Description/Type	Date/Time Collected	Container Type	Sample Volume	Preservative	Requested Testing Program	Condition on Receipt	Disposal Record No.

Special Instructions:

Possible Hazard Identification:
Non-hazard ☐ Flammable ☐ Skin Irritant ☐ Poison B ☐ Unknown ☐

Sample Disposal:
Return to Client ☐ Disposal by Lab ☐ Archive _____ (mos.)

Turnaround Time Required:
Normal ☐ Rush ☐

QC Level:

1. Relinquished by: *(Signature/Affiliation)*	Date: _____ Time:	1. Received by: *(Signature/Affiliation)*	Date: _____ Time:
2. Relinquished by: *(Signature/Affiliation)*	Date: _____ Time:	2. Received by: *(Signature/Affiliation)*	Date: _____ Time:
3. Relinquished by: *(Signature/Affiliation)*	Date: _____ Time:	3. Received by: *(Signature/Affiliation)*	Date: _____ Time:

Comments:

The sample containers to be supplied are listed in Tables 8.5-1 through 8.5-5. Container volumes listed in these tables may be decreased with the approval of the laboratory QA Manager or Technical Director to accommodate reduced sample volumes required by the facility SOP or increased as applicable to accommodate duplicates and matrix spikes.

8.5.2.2 Shipping Containers

Shipping containers are defined as the sealed enclosure in which the sample containers are stored during shipment from the sample collection site to the analytical laboratory. Shipping containers must be of sufficient number and size to accommodate the samples in an upright condition. Shipping containers must also meet all requirements for the shipment of environmental samples.

Packaged samples must be shipped to the analytical laboratory in a safe manner that preserves the integrity of the samples. The most common method of sample shipment employs coolers or ice chests that are sealed with custody tape and shipping tape. These coolers must be durable and resistant to crushing during shipment. All coolers must be well maintained and cleaned to prevent cross-contamination of the samples. All coolers contain a plastic liner to limit contamination. It is the ultimate responsibility of the person collecting and packaging the sample for shipment to ensure that the shipping containers are clean and functional.

To help prevent sample breakage during shipment, additional consideration must be given to providing shock absorbency to all samples packaged inside the shipping container. Use of bubble-wrap around each sample container is the best way to provide this protection. Foam packing materials and vermiculite are also successfully used.

8.5.2.3 Sample Preservatives

Most analytes have a finite holding time in a given sample matrix. Sample preservation is the chemical or physical means by which samples are treated during and/or following sample collection to aid in the stability of the analytes of interest in that matrix. Sample holding times are also adversely affected when samples are improperly preserved, or shipped unpreserved. The preservation of samples at the time of sample collection will follow the requirements of the analytical methods used. This preservation includes the addition of reagents to deter chemical and biochemical degradation and the maintenance of refrigeration during transit and ultimate storage in the laboratory. The required preservatives for the analysis to be performed on each matrix are included in Tables 8.5-1 through 8.5-5.

8.5.2.4 Sample Holding Times

Holding time is defined as the maximum allowable time a sample can be stored after sample collection and preservation (or laboratory receipt for CLP) until appropriate processing occurs (preparation or analysis). The holding time may vary according to method or client requirements and is tracked and controlled via the LIMs. Tests designated with holding times as "analyze immediately or ASAP" are considered parameters that should be tested by field personnel or on-site. Each operation has a system in place to ensure that holding times are monitored by each group within the operating unit. It is the responsibility of each STL associate processing the sample to assure that holding times are met. STL is responsible for meeting all holding times for properly preserved samples received within 48 hours of collection or if less than half the holding time has passed. If these conditions are not met, STL will attempt to expedite sample analysis as soon as possible. When holding times are exceeded, the laboratory uses a Nonconformance Memo (NCM) to identify and document the root cause of the holding time violation.

Sample holding times are listed in Tables 8.5-1 through 8.5-5.

8.5.3 Sample Handling

STL North Canton's SOP, Sample Receiving - NC-SC-0005, describes the sample receipt and log-in process in detail. The following sections describe the general policies followed by STL.

8.5.3.1 Sample Receipt

Samples shall be received and logged in at STL by a designated sample custodian or other properly trained associate. Upon sample receipt, the sample custodian shall, as appropriate:

- Wear appropriate personal protective equipment. At a minimum, this consists of gloves, a lab coat, and safety glasses
- Examine the shipping containers to verify that the custody tape is intact
- Examine all sample containers for damage
- Open shipping containers in adequately ventilated areas to assure worker safety
- Determine if the temperature required by the requested testing program has been maintained during shipment. Document the shipping container temperature on the COC
- Compare samples received against those listed on the COC
- Verify that sample holding times have not been exceeded

- Examine all shipping records for accuracy and completeness
- Determine sample pH (if required for the scheduled analysis) (except VOA and TOX samples) and record on the cooler receipt form
- Sign and date the COC immediately (only after shipment is accepted) and attach the waybill
- Note any problems associated with the coolers and samples on the cooler receipt form and notify the PM who in turn notifies the client
- Attach durable (water-resistant) laboratory sample container labels with unique laboratory identification number and test
- Place the samples in proper laboratory storage.

A CUR or an equivalent form/system is generated by sample control during the sample log-in process to document anomalies identified upon the receipt of samples in the laboratory. These anomalies are outside of laboratory control and do not require corrective actions to be taken within the laboratory. The affected client shall be notified by the PM or designee of all CURs generated for their samples. The PM is responsible for resolving with the client how to proceed with the samples and documenting the decision to proceed with the analysis of compromised samples. CURs must be resolved prior to sample preparation and analysis. The completed CUR form shall be stored in the project file. An example CUR is shown in Figure 8.5-2. The report narrative will include an explanation of sample receiving related anomalies.

8.5.3.2 Exceptions or Discrepancies

STL reserves the right to reject samples for any of the following reasons:

- No custody seals as required by project
- No chain of custody documentation provided
- Preservation inappropriate for analysis requested
- Sample container inappropriate for analysis requested
- Sample received out of holding time for analysis requested
- Incomplete sample information provided
- Discrepancies between COC and sample labels
- Samples have high levels of polychlorinated dibenzo-p-dioxins/ dibenzo furans (PCDD/PCDFs)
- Samples have a high level gross alpha or beta radiation
- Samples are from a site known to contain chemical warfare agents (CWAs) and the samples have not been screened for them.

These or any other project exceptions or discrepancies are discussed with the client and agreed upon action taken.

Figure 8.5-2
Example STL Cooler Receipt Form/Narrative

Client:_____ Project:_____ Quote#:_____
Cooler Received on:_____ Opened on:_____ by:_____ (Signature)

Fedx ☐ Client Drop Off ☐ UPS ☐ Airborne ☐ Other:_____
Cooler ☐ Safe ☐ Foam Box ☐ Client Cooler ☐ Other:_____
STL Shipper No#:_____

1. Were custody seals on the outside of the cooler and intact? Yes ☐ No ☐
   If YES, Quantity_____ Location_____
   Were the custody seals signed and dated? Yes ☐ No ☐ NA ☐
2. Shipper's packing slip attached to this form? Yes ☐ No ☐
3. Were custody papers included inside the cooler and relinquished? Yes ☐ No ☐
4. Did you sign the custody papers in the appropriate place? Yes ☐ No ☐
5. Packing material used:

Peanuts ☐ Bubble Wrap ☐ Vermiculite ☐ Foam ☐ None ☐ Other :_____

6. Cooler temperature upon receipt _____ °C (see back of form for multiple coolers/temp)

METHOD: Temperature Vial ☐ Coolant ☐ Against Bottles ☐
COOLANT: Wet Ice ☐ Blue Ice ☐ Dry Ice ☐ Water ☐ None ☐

7. Were all the bottles sealed in separate plastic bags? Yes ☐ No ☐
8. Did all bottles arrive in good condition (Unbroken)? Yes ☐ No ☐
9. Did all bottle labels and tags agree with the custody papers? Yes ☐ No ☐
10. Were samples at the correct pH? Yes ☐ No ☐ NA ☐
11. Were correct bottles used for the tests indicated? Yes ☐ No ☐
12. Were air bubbles >6 mm in any VOA vials? Yes ☐ No ☐ NA ☐
13. Was a sufficient amount of sample sent in each bottle? Yes ☐ No ☐

Contacted PM _____ Date:_____ by:_____ via Voice Mail ☐ Verbal ☐ Other ☐
Concerning:

√	MACRO	MACRO
1. CHAIN OF CUSTODY		
	SR1A	Samples were received under proper custody procedures and without discrepancies.
	SR1B	The chain of custody and sample bottles did not agree. The following discrepancies occurred_____
2. SAMPLE CONDITION		
	SR2A	Sample(s)_____ were received or requested after the recommended holding time had expired.
	SR2B	Sample(s)_____ were received with insufficient volume
	SR2C	Sample(s)_____ were received in a broken container.
3. SAMPLE PRESERVATION		
	SR3A	Sample(s)_____ were further preserved in sample receiving to meet recommended pH level(s).
	SR3B	Sample(s)_____ were received with bubble > 6 mm in diameter (cc: PM)
4. NCM		
	SR4A	NCM has been generated. Refer to Clouseau for details
5. Other Anomalies (see below or back)		

Figure 8.5-2 - Continued
Example STL Cooler Receipt Form/Narrative

Client ID	pH	Date	Initials

Cooler	Temp	Method	Comments

Discrepancies Cont.

Macro Name:

Macro Name:

Macro Name:

Macro Name:

Other Anomalies:

8.5.3.3 Sample Log-in

Sample log-in activities at STL are fully documented in Sample Receiving SOP, NC-SC-0005. The following is a general description of the log-in process:

- Enter the samples in the laboratory sample log-in book, and/or the LIMS which contains the following information at a minimum:
- Project name or identification number
- Unique sample numbers (both client and internal laboratory)
- Type of samples
- Required tests
- Date and time of laboratory receipt of samples
- Field ID supplied by field personnel
- Notify the PM and appropriate Group/Team Leader(s) of sample arrival
- Place the completed COCs, waybills, and any additional documentation in the project file.

8.5.3.4 Sample Storage

The primary considerations for sample storage are:

- Maintenance at the method prescribed temperature, if required
- Maintenance of sample integrity through adequate protection from contamination from outside sources or from cross-contamination of samples. Low-level and high-level samples, when known, must be stored separately. Samples and standards must be stored in separate refrigerators or freezers. Storage areas for volatile organic test requests should be monitored weekly by the analysis of a holding (refrigerator) blank (an aliquot of contaminant-free water stored in a VOA vial)
- Security of samples within the laboratory.

The requirements listed in Tables 8.5-1 through 8.5-5 for temperatures and holding times shall be used. Placing of samples in the proper storage environment is the responsibility of sample control personnel. STL will assign individuals the responsibility of notifying the Group/Team Leaders or their designees if there are any samples which must be analyzed immediately because of holding time requirements.

8.5.3.5 Internal Sample Chain-of-Custody and Interlaboratory Transfers

Sample custody within STL laboratories is described in Sample Receiving SOP, NC-SC-0005. Internal COC may be required for programs defined

by state or federal agency. The sample custody documentation shall include the following minimum requirements:

- Name of associate taking custody of the sample from the sample storage area for preparation or analysis
- Dates sample removed from and returned to the sample storage area
- Identification of tests to be performed on the sample aliquot(s) selected by the associate
- Sample matrix
- Laboratory sample numbers
- Sample storage location.

Additional custody records can be provided by the laboratory at the specific request of the client. Access to STL is restricted to prevent any unauthorized contact with samples, extracts, or documentation.

Samples transferred to a different laboratory than the original receiving facility are transferred under chain-of-custody (COC). The COC is maintained whether the laboratory is another STL facility or a subcontracted laboratory. If the entire sample volume is transmitted, the original copy of the client's COC form will be used to document the relinquishing of the sample and will accompany the sample to its destination. A copy of the completed COC form shall be retained in the laboratory project file. In the case where an aliquot of a sample is shipped from the laboratory, a new COC will be generated by the laboratory and shipped with the sample aliquot. The original COC will be retained in the project file at the site holding the original sample container.

Samples are not transferred to other STL facilities or to subcontractor laboratories without prior approval of the client.

8.5.3.6 Subsampling

Sample preparation procedures are referenced and defined in the method SOPs.

8.5.3.7 Sample Disposal and Return Chain-of-Custody

After the requested analyses on the samples have been completed, any remaining portions of the samples will be maintained by the sample custodian until the samples are disposed of or returned to the client. The

disposal of each sample is recorded on the client's COC form, in LIMS, or referenced in the project file. Sample disposal procedures and documentation are described in operation-specific SOPs. STL's routine sample retention period is at least thirty days after the analytical report is issued to the client, unless otherwise specified by the client.

If samples are returned to the client rather than disposed of by the laboratory, the original COC or a new COC is used to document custody transfer back to the client from the laboratory. A copy of the completed COC is retained in the laboratory project file.

8.5.4 Calibration Procedures and Criteria

All equipment and instruments used at STL operations for quantitative measurements are controlled by a formal calibration program. Table 8.0-1 lists the lab's major analytical instrumentation , and Tables 8.5-6 through 8.5-9 outline calibration requirements. Calibrations may be periodic or operational. These are described in the lab's method SOPs. The Policy P-T-001, "Selection of Data Points Required for an Initial Calibration Curve," is applicable when the number of data points is not described in the method. At a minimum, these calibration procedures shall include:

- Instrument to be calibrated
- Reference standards used for calibration
- Calibration technique (e.g., linear, quadratic)
- Acceptable performance tolerances and corrective actions required if specifications are not met
- Frequency of calibration
- Calibration documentation requirements.

Whenever possible, recognized procedures such as those published by ASTM or the USEPA or procedures provided by manufacturers shall be adopted. If established procedures are not available, a procedure shall be developed considering the type of equipment, stability characteristics of the equipment, required accuracy, and the effect of operation error on the quantities measured.

8.5.4.1 Physical Reference Standards

Physical reference standards associated with periodic calibrations include weights for calibrating balances and certified thermometers for calibrating working thermometers. Whenever possible, physical reference standards shall be calibrated by a body that can provide traceability to nationally or internationally recognized standards. If these standards are not available, the basis for the reference standard shall be documented.

Physical reference standards shall be used only for calibration procedures and shall be stored separately from equipment used for analysis.

8.5.4.2 Chemical Reference Standards and Reagents

Chemical reference standards are generally associated with operational calibration. These standards include reference materials traceable to recognized standards suppliers. This may include vendor-certified materials traceable to national or international standard reference materials (e.g., NIST). This topic is also discussed in the Section on "Procurement of Supplies and Services" (see 5.2.4).

All chemical reference standards maintained in the laboratory for use in calibrations (or as QC spiking solutions) and reagents prepared in the laboratory shall be labeled or referenced to appropriate documentation (hard copy or electronic) with the following information at a minimum:

- A unique identification including concentration (solutions containing several analytes can be identified such that the solution constituents and concentrations can be referenced to a logbook)
- Medium prepared in
- Preparation date
- Expiration date
- Initials of preparer.

Vials containing standard solutions that are not large enough to accommodate labels listing the above information may be referenced to a laboratory logbook/ notebook entry or standards software. The expiration date of the working standard and reagent must not exceed the expiration date of the original material. These records should provide sufficient detail to allow one to reproduce the standard or reagent.

Records for all purchased standards and reagents shall include the date of receipt, the date opened, and, where applicable, the expiration date.

8.5.4.3 Standard Verification

When possible, reference standards are purchased from a STL preapproved vendor. Standards are verified by quantitation against a second known standard before reporting data. The standard for verification must meet the laboratory's criteria for the independent/second source ICV verification. Therefore, the verification of a new standard initial calibration with a second source ICV meets this verification requirement, some "bad acting" analytes may not meet these criteria and must be approved by the QAM before use. Standard spiking solutions and

surrogates shall be verified by analyzing an LCS with the new standards and verifying against historical criteria limits. Special standards that are obtained from another source must also be independently verified at the lab. Verification by the laboratory of a reference standard from neat materials is also necessary.

To extend the use of an expired standard, which may not be allowed by all programs, reverification is necessary provided that new analysis produces acceptable data. The verification of an expired standard is performed against a current, independent standard reference material by analyzing within a valid calibration and QC.

Stock and working standards and reagents are checked regularly for signs of deterioration, such as discoloration, formation of precipitates, or change in concentration. Care is exercised in the proper storage and handling of standard and reagent solutions. Standards and reagents are always stored separately from samples.

An independent or second source standard is used to verify initial calibrations. An independent/second source standard is defined as a standard composed of the same target constituents as, but from a different source than those used in the standards for the initial calibration. An independent standard may be a laboratory-prepared or a certified independent standard solution(s). Independence of reference material can be achieved by: (1) purchasing reference materials from two separate vendors, (2) using a different lot from the same vendor that is certified by the vendor as an independent standard or (3) having two separate individuals prepare the calibration and verification standard solutions if independent sources are not available.

8.5.4.4 Periodic Calibration

Periodic calibration is performed at prescribed intervals. In general, equipment that can be calibrated periodically is a distinct, singular purpose unit and is relatively stable in performance. These include balances, micropipettors, counters, thermometers, refrigerators, freezers, and ovens. Equipment employed at STL requiring periodic calibration are listed along with their respective calibration requirements in Tables 8.5-6. NELAC requires mechanical volumetric dispensing devices (except Class A glassware) to be checked for accuracy or at least a quarterly basis if in use. The laboratory unit has an SOP in place for the calibration of this equipment if in use at their location.

8.5.4.5 Operational and Continuing Calibration

Operational calibration is routinely performed as part of instrument usage, such as the development of a standard calibration curve (see Tables 8.5-7 to 8.5-9). The accuracy of initial calibrations are to be verified prior to sample analysis through the use of an independent standard in situations where the source method requires calibration verification.

Detailed requirements for operational and continuing calibration are contained in method-specific SOPs.

When an initial calibration is not performed on the day of analysis, the validity of the initial calibration verification must be verified prior to sample analyses by a continuing instrument calibration verification with each analytical batch.

- A continuing instrument calibration verification must be performed as outlined in Tables 8.5-7 (inorganics) and 8.5-8 (organics).

- Sufficient raw data records must be retained to permit reconstruction of the continuing instrument calibration verification, e.g., test method, instrument, analysis date, each analyte name, concentration and response, and calibration curve or response factor.

- When the acceptance criteria for the continuing calibration verification is exceeded high, i.e., high bias, and there are associated samples that are non-detects, then those non-detects may be reported. Otherwise the samples affected by the unacceptable calibration verification shall be reanalyzed after a new calibration curve has been established, evaluated and accepted.

- When the acceptance criteria for the continuing calibration verification is exceeded low, i.e., low bias, those sample results may be reported if they exceed a maximum regulatory limit. Otherwise the samples affected by the unacceptable verification shall be reanalyzed after a new calibration curve has been established, evaluated and accepted.

8.5.4.6 Calibration Failure

Equipment or instruments that fail calibration or become inoperable during use shall be tagged out (NCM created) to indicate they are out of calibration. Such instruments or equipment shall be repaired and successfully recalibrated before reuse. Following recalibration or verification, back to control will be documented in the injection/run

log and/or maintenance logbook through the routine identification of the required calibration runs specified by the standard operating procedure.

8.5.4.7 Calibration Records

Calibration shall be documented for each piece of equipment subject to calibration. All calibration records (periodic and operational) directly affect data and may not be limited to one project. These records shall be stored in either the quality records or the associated project files. Project files that include sample data shall either include the calibration records or include reference to them

8.6 Quality Assessment

The effectiveness of the QA practices is measured by the quality of data generated by the laboratory. Procedures are in place to detect, prevent, and correct quality problems and to ensure quality improvement. Items and processes that do not meet established requirements must be investigated to determine their cause. Improvements must be implemented in the operations that will prevent a recurrence of these quality problems and provide overall quality performance. All phases of laboratory work should be designed with the objective of preventing problems and improving quality on a continuous basis.

8.6.1 Data Quality Assessment

Data quality is judged in terms of precision, accuracy, representativeness, completeness and comparability. The areas of representativeness, comparability, and completeness for an overall project, inclusive of sampling issues, may be beyond the control of the laboratory. The elements over which the laboratory has direct control are precision, accuracy, and completeness relative to analytical testing results.

Precision and accuracy assessments are made as part of the evaluation of laboratory QC data generated during sample preparation and analysis. The QC samples employed at STL North Canton as part of routine sample analysis are summarized in Section 8.4 of this document. Table 8.6-1 shows the precision and accuracy measurements employed. Analytical method SOPs and STL Policy Number QA-003 include information on requirements for the type of QC samples, frequencies, and acceptance criteria. Additionally, the SOPs and Policy describe the appropriate actions to be taken when a QC sample result does not meet acceptance criteria.

8.6.2 Statistical Evaluation of Data

In-house limits for all QC data must be evaluated at least annually and compared to the limits published in the methods for applicable matrices. Method limits will

be employed until sufficient QC data are acquired. A minimum of 20 to 30 data points are recommended to establish the in-house QC limits. Calculated results of the QC (LCS) samples are evaluated by comparing against control limits (3-sigma).

Control charts are used to develop control limits, trouble-shoot analytical problems, and, in conjunction with the non-conformance system, to monitor for trends. Program-specific data analysis requirements for control charts are followed as required for data generated under those programs. These additional requirements shall be documented in a QAPP or QAS.

Precision and accuracy measurements employed by STL North Canton are shown in Table 8.4-3 through 8.4-7. Calculated results of these QC samples are evaluated using statistical tables or control charts.

8.7 Data Recording Procedures

To ensure data integrity, all documentation of data and records generated or used during the process of data generation must be performed in compliance with Policy Number QA-008, "Data Recording Requirements".

8.8 Data Reduction and Verification Procedures

Data review procedures comprise a set of computerized and manual checks applied at appropriate levels of the measurement process. Data review begins with the reduction or processing of data and continues through verification of the data and the reporting of analytical results. Calculations are checked from the raw data to the final value prior to reporting results for each group of samples. Data reduction can be performed by the analyst who obtained the data or by another analyst. Data verification starts with the analyst who performs a 100 percent review of the data to ensure the work was done correctly the first time. Data verification continues with review by a second reviewer who verifies that data reduction has been correctly performed and that the analytical results correspond to the data acquired and processed. This procedure is outlined in Figure 8.8-1.

8.8.1 Data Reduction and Initial Verification

Data reduction and initial verification may be performed by more than one analyst depending upon the analytical method employed. The preparation and analytical data may be reviewed independently by different analysts. In these instances, each item may not be applicable to the subset of the data verified or an item may be applicable in both instances. It is the responsibility of the analyst to ensure that the verification of data in his or her area is complete. The data reduction and initial verification process must ensure that:

- Sample preparation information is correct and complete including documentation of standard identification, solvent lot numbers, sample

amounts, etc.

- Analysis information is correct and complete including proper identification of analysis output (charts, chromatograms, mass spectra, etc.)
- Analytical results are correct and complete including calculation or verification of instrument calibration, QC results, and qualitative and quantitative sample results with appropriate qualifiers
- The appropriate SOPs have been followed and are identified in the project and/or laboratory records
- Proper documentation procedures have been followed
- All nonconformances have been documented
- Special sample preparation and analytical requirements have been met.
- The data generated have been reported with the appropriate number of significant figures as defined by the analytical method in the LIMS or otherwise specified by the client.

In general, data will be processed by an analyst in one of the following ways:

- Manual computation of results directly on the data sheet or on calculation pages attached to the data sheets
- Input of raw data for computer processing
- Direct acquisition and processing of raw data by a computer.

If data are manually processed by an analyst, all steps in the computation shall be provided including equations used and the source of input parameters such as response factors (RFs), dilution factors, and calibration constants. If calculations are not performed directly on the data sheet, they may be attached to the data sheets.

Manual integrations are sometimes necessary to correct misintegrations by an automatic data system software program, but must only be performed when necessary. Further discussion of manual integrations and the required documentation is given in Policy Number QA-011, "Acceptable Manual Integration Practices".

For data that are input by an analyst and processed using a computer, a copy of the input shall be kept and uniquely identified with the project number and other information as needed. The samples analyzed must be clearly identified.

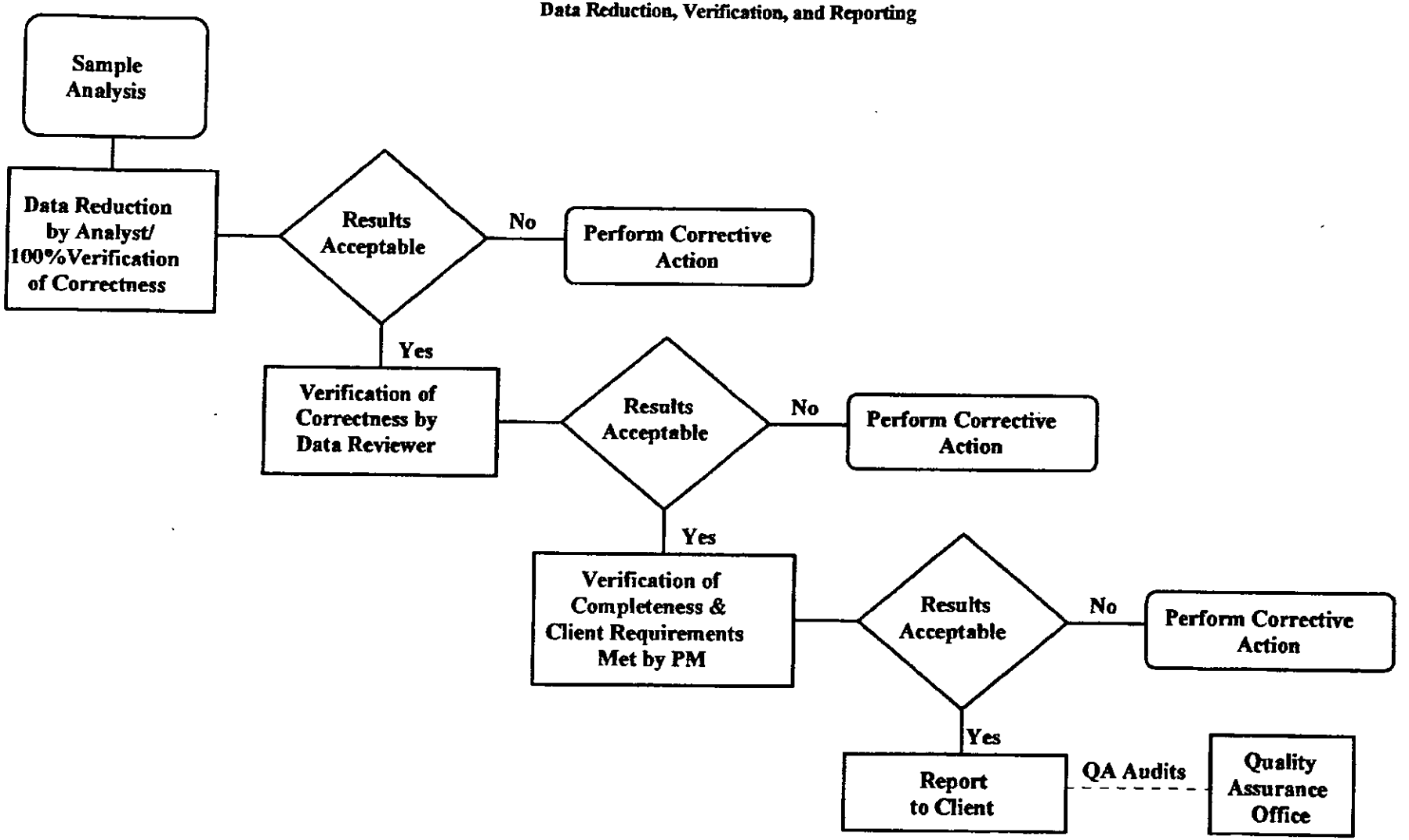
If data are directly acquired from instrumentation and processed, the analyst must verify that the following are correct:

- Project and sample numbers
- Calibration constants and RFs
- Units
- Numerical values used for reporting limits.

Analysis-specific calculations for methods are provided in SOPs. In cases where computers perform the calculations, software must be validated or verified, as described in Section 6.0 of this document, before it is used to process data.

The data reduction is documented, signed and dated by the analyst completing the process. Initial verification of the data reduction by the same analyst is documented on a data review checklist, signed and dated by the analyst. Data review requirements are described in Section 5.3.6 of the QMP.

FIGURE 8.8-1
Data Reduction, Verification, and Reporting



8.8.2 Data Verification

Following the completion of the initial verification by the analyst performing the data reduction, a systematic check of the data that has been fully reduced and checked through Level 1 review is performed by an experienced peer, supervisor, or designee. This check is performed to ensure that level 1 review has been completed correctly and thoroughly. The second level reviewer examines the data signed by the analyst. This review includes an evaluation of all items required in the raw data package. Any exceptions noted by the analyst must be reviewed. Included in this review is an assessment of the acceptability of the data with respect to:

- Adherence of the procedure used to the requested analytical method SOP
- Correct interpretation of chromatograms, mass spectra, etc.
- Correctness of numerical input when computer programs are used (checked randomly)
- Correct identification and quantitation of constituents with appropriate qualifiers
- Numerical correctness of calculations and formulas (checked randomly)
- Acceptability of QC data
- Documentation that instruments were operating according to method specifications (calibrations, performance checks, etc.)
- Documentation of dilution factors, standard concentrations, etc.
- Sample holding time assessment.

This review also serves as verification that the process the analyst has followed is correct in regard to the following:

- The analytical procedure follows the methods and specific instructions given on the project QAS or equivalent summary form
- Nonconforming events have been addressed by corrective action as defined on a nonconformance memo
- Valid interpretations have been made during the examination of the data and the review comments of the initial reviewer are correct
- The package contains all of the necessary documentation for data review and report production and results are reported in a manner consistent with the method used for preparation of data reports.

The specific items covered in the second stage of data verification may vary according to the analytical method, but this review of the data must be documented by signing the same checklist. Data review requirements are described in Section 5.3.6 of the QMP.

8.8.3 Completeness Verification

A third-level review is performed by the reporting and project management staff. This review is required before results are submitted to clients. This review serves to verify the completeness of the data report and to ensure that project requirements are met for the analyses performed. The items to be reviewed are:

- Analysis results are present for every sample in the analytical batch, reporting group, or sample delivery group (SDG)
- Every parameter or target compound requested is reported with either a value or reporting limit
- The correct units and correct number of significant figures are utilized
- All nonconformances, including holding time violations, and data evaluation statements that impact the data quality are accompanied by clearly expressed comments from the laboratory
- The final report is legible, contains all the supporting documentation required by the project, and is in either the standard STL format or in the client-required format.
- Implement checks to monitor the quality of laboratory results using correlation of results for different parameters of a sample (for example, does the TOC results justify the concentration of organic compounds found by GC/MS.)
- A narrative to accompany the final report will be finalized by the PM. This narrative will include relevant comments collected during the earlier reviews.

8.9 Data Reporting

8.9.1 Data Reports

STL North Canton is capable of developing a variety of data deliverable reports. Standard reports will contain:

- Cover Letter/Narrative - Information on sample types, tests performed, any problems encountered, and general comments are provided.
- Analytical Data - Data are reported by sample or by test with the appropriate significant figures and reporting limits, and have been adjusted for dilution, if appropriate. Pertinent information including dates sampled, received, prepared, extracted, and analyzed are provided.
- Laboratory Performance QC Information - The results of LCSs and method blanks analyzed with the project are listed. Any data or QC anomalies are discussed in the narrative.
- Matrix-Specific QC Information - Results of any sample duplicates and MS/MSDs analyzed with the samples as batch QC are reported. Other project-specific QC requested by the client are also reported. The results

include supporting information such as amount spiked, percent recovery, or percent difference/RPD.

- Methodology - Reference for analytical methodology used is cited.
- Other Deliverables - Other deliverables available include disk deliverables, CDROM, sample raw data packages, complete deliverable packages, and custom report formats. Requirements for electronic reporting are defined in Policy CORP-QA-017, "Electronic Reporting".

8.9.2 Final Report Details

STL North Canton will provide paginated reports or a uniquely defined, identifiable certificate/report (i.e. electronic file, CD). The report will include:

a) Report title, name, address and phone number of the laboratory.
b) Name and address of client/project name/client identification number.
c) Description (lab ID of sample).
d) Dates and Time of sample collections (if known), receipt, preparation and analysis.
e) If the required holding time is 48 or less, time of sample preparation and analysis.
f) Method identifiers traceable to all procedures used.
g) Reporting limit.
h) Test result with appropriate units and how reported (wet weight/dry weight). Also identify any results outside of quantitation limits. When required, a statement of the estimated uncertainty of the test result should be added.
i) If appropriate, description of any QC failures or deviations from SOPs.
j) Signature and title of the individual responsible for the report. Electronic signature is acceptable.
k) Date of issue.
l) All subcontract work must be clearly identified, and name and address of outside subcontractor noted.
m) Where relevant, a statement to the effect that the results relate only to the items tested or to the sample as received by the laboratory
n) Where relevant, a statement that the certificate or report shall not be reproduced except in full, without the written approval of the laboratory.

After final report any correction, addition, or deletion must clearly identify its purpose and meet the above reporting requirements as appropriate.

All applicable elements from above should be available for review if not issued in a formal report by an in-house or captive laboratory.

8.9.3 Verbal Results

STL North Canton, as a policy, discourages the release of data verbally or without full data review. If however, the client requests analytical results to be communicated verbally or by facsimile prior to final review, they must be clearly identified as "Preliminary" results. The client must understand that the data have not undergone the required levels of review and may potentially change.

8.9.4 Reporting Analytical Results

Sample results are reported according to analytical method SOPs or client specifications. Normally, the laboratory uses the STL North Canton Reporting Limit (RL) at which any analyte of interest detected at or above that level is reported as a positive value and any analyte of interest not detectable or detected below that level is reported as "not detected" at the RL. The laboratory will normally report results within the calibration, however, any reported results outside of the calibration range will be documented in the final report.

If a QC measurement is out of control and the data is to be reported, data qualifiers are reported with samples associated with failed QC measurements.

The laboratory must certify that the test results meet all NELAC requirements or provide reasons and/or justification if they do not.

In some cases a contract, QAPP, or documented client request may require the laboratory to report sample results in a specified manner. Some examples are given below:

- The laboratory may be requested to report all analytes of interest that are less than the laboratory's RL but are greater than the MDL. This data will be flagged with an appropriate qualifier or noted in the report case narrative. (See precautions in "Establishing Reporting Limits", Policy Number QA-009).
- The laboratory may be requested to report any tentatively identified compounds (TICs). These data will be flagged with an appropriate qualifier.
- The laboratory may be requested to report sample results using an RL that is higher than their normal level. In this case, only the analytes of interest found at or above that level would be reported as positive values. In this case, the laboratory will state the PSRL rather than the RL. All analytes of interest not detected or detectable below that level would be reported as "not detected" at the PSRL.

  In this situation, the laboratory must include documentation in the project file that supports the reporting procedure employed.

It is the responsibility of the laboratory to provide for a reporting system that assures that any problems associated with an analysis are properly documented on a nonconformance memo, communicated to the appropriate STL North Canton staff, and addressed appropriately in the data report.

8.9.5 Reissued Deliverables

If, after issuance of a report, STL North Canton observes any mistake that affects the results reported or the QC interpretation of those results, the client will be notified. After issuance of the report, the laboratory report remains unchanged. Any material amendments to a report after issue made only in the form of a further document, or data transfer must include the statement "Supplement to Test Report" or otherwise identified.

8.9.6 Client Confidentiality

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. STL's reports, and the data and information provided therein, are for the exclusive use and benefit of our clients, and are not released to a third party without written consent from the client. Data confidentiality is also discussed Section 3.6.

8.10 Data Validation

Data validation for STL refers to data reviews conducted in accordance with the USEPA CLP "Laboratory Data Validation Functional Guidelines for Evaluating Organic Analyses" and "Laboratory Data Validation Functional Guidelines for Evaluating Inorganic Analyses", or modifications thereof, for non-CLP type analyses.

This form of data validation provides an impartial evaluation of the laboratory's results. Data validation may be requested by the client for a percentage of data and is usually performed by a third party, one which was not involved with the sample analysis. Qualifiers are assigned to data, when required, according to the requirements of the data validation protocol being used.

8.11 Preventive Maintenance and Service

Facilities, instruments, equipment, and parts are subject to wear, deterioration, or change in operational characteristics. Within STL, preventive maintenance, coupled with vendor service agreements, is an organized program of actions taken to maintain facilities and equipment in control.

8.11.1 Analytical Instrumentation and Equipment

The primary purpose of the maintenance program is to prevent instrument and equipment failure and to minimize down time. A properly implemented maintenance program increases the reliability of a measurement system.

Each instrument or piece of equipment shall be uniquely identified. The laboratory maintains the following:

- Instrument/equipment inventory list
- Instrument/equipment major spare parts list or inventory
- External service agreement documents (if applicable)
- Instrument-specific preventive maintenance logbook or file for each functional unit.

The records of routine maintenance and non-routine maintenance shall include at a minimum:

- Name and serial number and/or unique ID of the item or equipment
- Details of maintenance performed
- Dates and results of recalibrations/ reverifications indicating return to control
- Analyst initials and the date maintenance was performed whether by the analyst or a contracted service representative.

Any item or equipment that does not perform to specifications or defective shall be taken out of service, and tagged as out of service until it has been repaired and shown by calibration/ verification to perform satisfactorily.

8.11.2 Frequency of Equipment Maintenance

The frequency of maintenance must consider manufacturer's recommendations and previous experience. Frequency of preventive maintenance along with the recommended preventive maintenance schedules are given in Tables 8.11-1 through 8.11-25 for analytical instrumentation and equipment or defined in operation specific routine maintenance SOPs. Frequency of maintenance for the facility systems is documented in the CHP.

8.11.3 Facilities

Another important aspect of the laboratory operation is the existence and maintenance of adequate, safe, and clean facilities including appropriate engineering controls such as proper ventilation, lighting, dust control, hoods, air flow, protection from extreme temperatures, waste disposal, and a source of stable power. The facility floor plan is provided in Figure 8.11.

The maintenance and use of these facilities and proper operations are described in the Chemical Hygiene Plan (CHP). The Laboratory Manager has

responsibility for ensuring a properly maintained facility. The Laboratory Director also has the responsibility for ensuring that facilities are available to store samples properly without contamination, work areas are equipped with adequate bench, hood and operational space, and that procedures are in place to ensure the areas are free from chemical contamination that may affect analytical results. The volatile laboratories are in a separate laboratory building from the semivolatile laboratories. The volatile laboratories utilize a separate air handling system with positive pressure to reduce possible contamination (refer to Floor Plan 8.11-1).

8.11.4 Facility Security

The laboratory building is a limited access, secure facility. To ensure that only authorized personnel are able to enter the building from an entrance that is not monitored, entry into each building is limited by use of electronic locks activated by magnetic keys which are issued only to authorized personnel.

During business hours, guest entry is possible only through the main entrance. This entrance is monitored at all times, by a receptionist. All guests are required to sign in by using a visitor logbook and wear a visitor's badge. Clients may drop off samples through a sample receiving door after calling for entry.

8.12 Requirements for Ancillary Equipment and Materials

8.12.1 Water

High purity water (e.g., ASTM reagent grade or equivalent water) will be used in all metals, wet chemistry, and organic analyses. Demonstration of contaminant-free water is shown through the analysis of method blanks consisting of the reagent water on a each working day for the analyte of interest. This water is obtained by the use of either a commercial ion-exchange deionizing, distillation, or reverse osmosis unit plus an appropriate polishing unit. The resulting water has a maximum conductivity of 1.0 umho-cm at 25°C or a minimum resistivity of 1.0 Mohm at 25°C. Conductivity or resistivity will be monitored and documented daily or on each day that water is dispensed for analytical use.

For volatile analyses the laboratory purchases bottled water that is further purified by purging with an inert gas before use to remove potential traces of organic solvents. This is described further in the Reagent Water SOP, NC-QA-0023.

8.12.2 Compressed Air and Gases

Ultra high-purity compressed gases from preapproved vendors or in-house gas generators will be used when required for instrumentation. These air and gases must meet the requirements and specifications of the analytical methods performed. In-line filters will be used when appropriate to minimize contamination and moisture from the gases.

8.12.3 Glassware Preparation

Glassware preparation procedures implemented at operating units are designed to ensure that contaminants are not introduced during sample analysis. Procedures describing glassware preparation are detailed in operation-specific Glassware Washing SOP, NC-QA-0014.

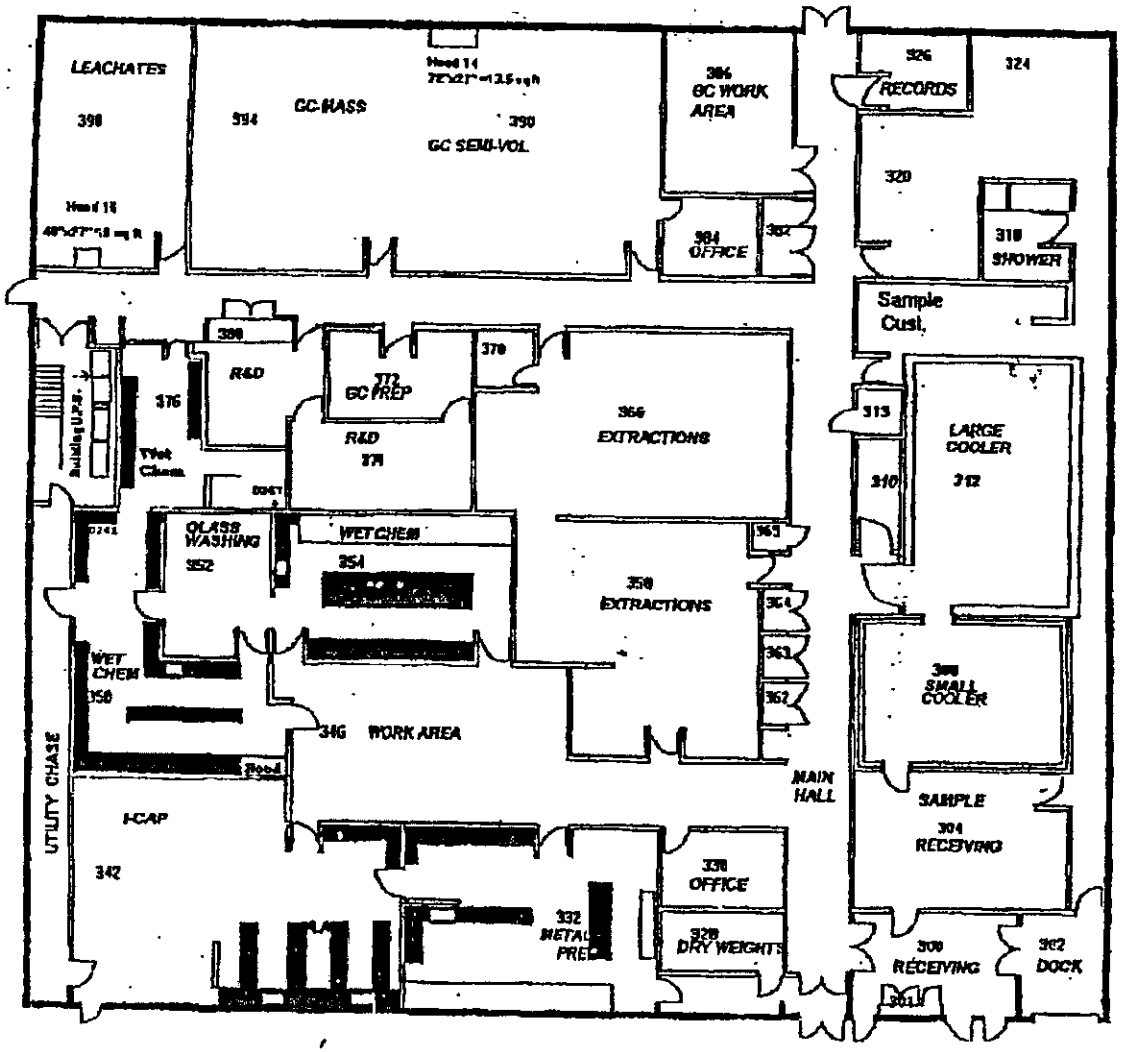
8.12.4 Chemical Storage

Storage of chemicals shall be conducted in a manner to minimize the potential for fire or release of hazardous material resulting from an unplanned chemical reaction. Refrigerators used for storing flammable liquids must have spark-free interior construction. Flammable solvents shall be stored in appropriate cabinets meeting all necessary codes. All chemicals are stored according to chemical compatibility. Further details regarding chemical storage are provided in the CHP.

8.12.5 Waste Management

The goal of STL's policy for waste management is to ensure that laboratory wastes are disposed of safely and in a manner consistent with applicable federal, state and local regulations. The waste disposal program is designed to assure that minimal harm to people and the environment shall result from the disposal of laboratory chemicals and samples. This goal is accomplished by requiring that the laboratory comply with the procedures presented in the CHP.

Figure 8.11 -1 Floor Plan



9.0 Quality Assessment and Response

9.1 Nonconformances

A nonconformance is an unplanned deviation from an established protocol or plan and in some cases may be exceptionally permitted departures from the documented policies and procedures or from standard specifications. The deviation may be the result of STL's actions as a systematic error, then termed a deficiency. A single isolated event or event beyond the control of STL is termed an anomaly.

Nonconformances can be identified on the basis of internal or external systems or performance audits, sample processing, routine calibration and monitoring of analytical and support equipment, or QC sample analyses. The Technical Director, Operations Manager, Project Manager, QA Manager, Group Leader, and Analyst may be involved in identifying the most appropriate corrective action. If previously reported data are affected, the issue is immediately brought to the attention of QA and Laboratory Management.

9.1.1 Nonconformance Memo (NCM)

All nonconformances, deficiencies and anomalies, are documented via an electronic process or on a paper form that meets NCM requirements as approved by QA. An allowed exception is log-in conformance problems, which are documented on a Condition Upon Receipt Form or equivalent (see Section 8.5). A detailed description of the procedure and responsibilities associated with nonconformance documentation, communication, and resolution is described in SOP # CORP-QA-0010, "Nonconformance and Corrective Action".

The Clouseau NCM program, available on the local-area network throughout the laboratory, is the main vehicle for documenting and communicating NCMs. The program allows anyone in the laboratory to document a nonconformance, explain the cause of the problem, and link to the LIMS system to identify the samples and clients involved. The program uses the local e-mail to automatically notify the person's supervisor, the Project Managers associated with the samples, and the QA department. The program is used to document approval and completion of the immediate corrective actions for the samples involved, and can be used to document long-term corrective actions. It provides a place to document resolution of problems with the clients and to query the associated database to examine trends and prepare management reports. A copy (paper or electronic) of the nonconformance memo will be kept in the project files along with the data it refers to. A copy, paper or electronic, shall also be kept in the quality files.

9.2 Client Complaints

Client inquiries and complaints are generally received through the PM or Customer Services Manager (CSM). Typically, the PM or CSM communicates with the client to determine the details of the inquiries, including technical data problems, deliverable issues, turn-around-time problems, etc. Technical and deliverable issues are coordinated by the PM and usually involve input from operations, QA, and management staff. A formal written response to the client is coordinated by the PM, but may on occasion be delivered by the CSM or the Account Manager. Details of the types and levels of complaints and required documentation are provided in Corporate Policy No. QA-013, "Procedures to Address Customer Complaints". Client complaints are recorded as a type of NCM in the Clouseau database, which are summarized in the monthly QA Reports to Management (see Section 9.6 for more about the monthly QA reports).

9.3 Corrective Actions

Corrective actions are measures taken to rectify conditions adverse to quality and, where possible, to prevent their reoccurrence. Investigations of potential problems and corrective actions should be timely, determine the root cause, and evaluate any propagation of the error or problem. Whenever a systematic error is discovered that affects the accuracy or defensibility of results reported to STL's clients, Corporate QA involvement followed by client notification would be part of the corrective action.

Corrective actions should be implemented with an understanding of the technology and work activities associated with the quality element, with appropriate training of STL associates and vendors, and should be monitored for progress and success. Depending on the nature of the problem, the corrective action employed may be formal or informal. In either case, occurrence of the problem, the corrective action employed, and verification that the problem has been eliminated must be documented properly. On-the-spot actions are used to correct minor problems, such as recalibration, retuning, or a minor repair (e.g., replacement of a minor part) of a malfunctioning instrument or the correction of poor analytical technique being used by an analyst. These occurrences are documented in the appropriate injection, run, or analysis logbooks. Similarly, routine instrument maintenance, malfunctions, and power failures are also documented in the appropriate instrument maintenance logbooks. These events do not require a formal NCM process, provided reported analytical results are not affected. Corrective actions specific to quality controls for analytical methods are discussed in the operational-specific SOPs.

9.3.1 Monitoring Corrective Actions

The QA department, either in the Clouseau database or in paper files maintains all formal corrective action documentation. The QA department reviews all corrective actions and selects one or more of the significant corrective actions for inclusion in the annual systems audit. The QA department may also implement a spot assessment audit. The purpose of these 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.

9.4 Internal Audits

Internal audits are performed to assess the degree of adherence to established policies, procedures and standards. STL personnel who are independent of the area being evaluated conduct these assessments. Audits can identify areas for improvement with regard to compliance with policies, procedures and standards. Audits also provide a means for correction prior to system failure.

Audits and assessments are generally conducted through the use of checklists and relevant reference documents. The findings of all audits and assessments are documented as is the laboratory response and any corrective actions. Follow-up checks are performed and the status of implementation of corrective actions is documented for all categories of audits and assessments. This cycle continues until all issues are closed.

9.4.1 Audit Types and Frequency

The following types of audits are performed at STL North Canton:

Figure 9.4.1-1 Audit Types and Frequency

Audit Type	Performed By	Frequency
Systems Audits	QA Department or designee	Annual per lab section
Data Audits	QA Department	Target of 5% of all report packages
Spot Assessment	QA Department or designee	As needed to monitor specific issues
Proficiency Testing	QA Department or designee	Two samples per year per program as required by NELAC

9.4.2 Systems Audits

Facility systems audits are comprehensive technical and systems evaluations covering each operational and support area at least once per year. Generally, a rotating schedule is established throughout the year to ensure adequate coverage of all areas. This schedule can change as situations in the lab warrant. The objectives and schedule of the audit are communicated to the lab groups being assessed in advance of the audit. At the completion of the audit, a debriefing is held to outline the findings, including identification of positive performance, to discuss areas of deficiencies, and to answer questions. The audit report issued by the QA Manager within 21 calendar days of the audit. The audit report is addressed to the area supervisor and/or manager, and copied to the General

Manager and Laboratory Director. Written audit responses are required within 21 calendar days of the date of the audit report. The audit response from the lab areas must follow the format of the original audit report, and is sent from the respondents to all individuals copied on the audit report. Where a corrective action requires longer than 21 days to complete, the target date for the corrective action is stated and evidence of corrective action is submitted to the QA department in the agreed upon time frame.

9.4.3 Data Audits

Data audits are routinely performed and documented to ensure that project records meet project requirements as described in method SOPs, project plans, or other documented requirements. The data audit is used to identify any lab errors that may have occurred. Significant issues found in the course of the audit are brought to the attention of appropriate personnel for clarification, and overseeing correction of final reports if necessary. The target frequency of QA data audits is 5% of reports. Data audits include spot-checking manual integrations to determine if they are appropriate and documented according to policy QA-011. Errors found in client project reports are revised and the revision sent to the client (also see Section 8.9.5).

9.4.4 Spot Assessments

Spot assessments, equivalent to special audits in the STL QMP, are conducted on as needed basis, generally as a follow up to specific issues such as client complaints, validator concerns, corrective actions, control chart or NCM trends, proficiency testing results, data audits, or external audit issues. Spot assessments are focused on a specific issue. The frequency, report format, distribution, and timeframes are tailored to address the nature of the issue.

9.4.5 Proficiency Testing

Proficiency testing samples (PTs) are analyzed to verify the ability of the laboratory to correctly identify and quantitate compounds in PT samples. PT samples may be supplied internally or externally as single-blind or double-blind samples. They can be used to assess if a deficiency has been corrected, they can be used to document the proficiency of the analyst performing the analysis, or they can be used to assess the overall performance of an analytical method.

PT samples are handled and tested in the same manner as environmental samples - it is not acceptable to run multiple replicates that would not otherwise be performed, it is not acceptable to average multiple results, and PT results cannot be shared among labs in advance of the close of the study. PT test sample data is archived using the same requirements as for project and raw data record retention.

9.4.5.1 External PT Samples

STL North Canton participates in a number of PT studies, as shown in Table 9.4-1. The primary one being the NELAC PT program, which involves a minimum of two PT rounds each year for NELAC field of testing for which the lab is maintaining certification. In addition, under the 12/99 SDWA requirements, the laboratory also analyzes annually a PT sample for each drinking water method, where more than one method is used for a given analyte.

9.4.5.2 Internal PT Samples

Each STL facility performing chemical analyses also participates in a double-blind performance evaluation annually. An external vendor is contracted to submit double blind samples to the STL labs. Both the level of customer service and the accuracy of the test results are assessed objectively by the external contractor. The PT contractor provides a detailed report to the Corporate QA Manager and to each of the STL facilities.

9.5 External Audits

Clients and external regulatory authorities regularly audit STL North Canton. STL is available for these audits, and makes every effort to provide the auditors with the personnel, documentation and assistance they require. STL recommends that all audits be scheduled with the QA department so that all necessary personnel are available on the day of the audit. All deficiencies reported to the laboratory must be responded to within the time frame specified by the auditors. It is the responsibility of the QA Manager to coordinate the response to the audit report. The development and implementation of the corrective actions is the responsibility of the operations management of the affected areas and must be approved by the laboratory operations and management prior to submitting the final response. It is the responsibility of the QA Manager or designee to verify implementation of the corrective actions and inform the responsible manager of the closure of all deficiencies from the audit.

9.6 Management Reviews

9.6.1 Quality Reports to Management

A monthly QA report is prepared by the QA Manager and forwarded to the Laboratory Director, the General Manager, and the Corporate QA Manager. The reports include metrics (i.e., frequency and number of revised reports, frequency and number of client complaints) to assess the effectiveness of the Quality System. The contents of the monthly report include:

- Audits
  Results of internal systems audits performed

Results of external systems audits hosted
Data audits performed, percent of total packages per month plus any issues

- Revised Reports / Client Complaints
  Frequency of revised reports
  Total number of client complaints, issues, and resolution
- Certification / Parameter Changes
- Proficiency Testing
  Score for each PT as a percentage of maximum score
  Note repeat failures and/or significant problems
- Miscellaneous QA and Operational Issues
  Narrative outlying improvements, regulatory compliance issues, general concerns, and assistance required from management

This information is compiled by the Corporate QA Manager together with similar information from and about other STL laboratories, which is then presented in a report to the STL Chief Operating Officer.

9.6.2 Management Systems Review

Annually at a minimum, the laboratory management will evaluate the status of the quality systems in the laboratory to ensure the procedures and policies are in place and they are adhered to. The Laboratory Director, Operations Manager, Customer Service Manager, and Quality Assurance Manager regularly meet to evaluate quality system(s). Management systems reviews are accomplished by the QA Manager based on monthly quality assurance reporting, goal setting and an annual LQM review. The evaluation(s) shall consider:

- The suitability of policies and procedures
- Reports from managerial and supervisory personnel
- The outcome of recent internal audits
- Corrective and preventative actions
- Assessments by external bodies
- The results of interlaboratory comparisons and proficiency tests
- Status of QA documents
- Reviews of QA related requirements in RFPs, SOWs, SAPs, and QAPjPs
- Changes in the volume and type of work and the effects on QA systems
- Client feedback
- Complaints
- Quality control activities
- Resources and staff training

STL North Canton
Laboratory Quality Manual

Table Section

TABLE 2.4-1
LQM Source Documents Requirements Matrix

STL LQM	EPA QA/R-2	ANSI/ASQC E4-1994	NQA-1[1]	5700.6C[2]	ANSI N 13.30	ANSI/ASQC Q2-1991[3]
1.0 Management Commitment and Organization	1 Management and Organization	2.1 Management and Organization	1 Organization	9.a. General	1.1 Introduction 1.2 Purpose 1.3 Scope	5.0 Management Responsibility
2.0 Quality System and Description	2 Quality System and Description	2.2 Quality System and Description	2 Quality Assurance Program	1 Program	2.1 Special Word Usage 2.2 Specific Terms 5.1 Quality Assurance 5.2 Quality Control	5.2 Quality System
3.0 Document Control and Records Maagement	5 Documentation and Records	2.5 Documents and Records	6 Document Control 17 Quality Assurance Records	4 Documents and Records	3.6 Direct Bioassay-Record Retention 4.5 Indirect Bioassay Record Retention	8.4 Quality Documentation and Records
4.0 Staff Qualification, Orientation and Training	3 Personnel Qualification and Training	2.3 Personnel Training and Qualification		2 Personnel Training and Qualification	3.2 Personnel Preparation	14.0 Personnel
5.0 Procurement of Supplies and Services	4 Procurement of Items and Services	2.4 Procurement of Items and Services	4 Procurement Document Control 7 Control of Purchased Items and Services	7 Procurement	N/A	7.0 Quality in Procurement 13.0 Subcontracting
6.0 Computer Hardware and Software	6 Computer Hardware and Software	2.6 Computer Hardware and Software	3 Design Control 11 Test Control	N/A	N/A	ISO 9000-3[4]

TABLE 2.4-1
LQM Source Documents Requirements Matrix (cont.)

LQM	EPA QA/R-2	ANSI/ASQC E4-1994	NQA-1[1]	5700.6C[2]	ANSI N 13.30	ANSI/ASQC Q2-1991[3]
7.0 Contract Review and Project Planning	7 Planning	2.7 Planning 3.1 Planning and Scoping 3.3 Implementation of Planned Operations	2 Quality Assurance Program 3 Design Control 5 Instructions, Procedures, and Drawings 8 Identification and Control of Items 9 Control of Processes 11 Test Control 13 Handling, Storage, and Shipping	1 Program 6 Design N/A	3.1 Facility Criteria 3.4 Direct Bioassay-Performance Criteria for Service Laboratories 3.5 Direct Bioassay-Reporting Results 4.1 Indirect Bioassay-Responsibilities of the Service Laboratory Customer 4.2 Indirect Bioassay-Analytical Methodology 4.3 Indirect Bioassay-Performance Criteria for Service Laboratories 5.2 Quality Control	6.3.3 Quality Plans
8.0 Work Processes and Operations	8 Implementation of Work Processes	2.8 Implementation of Work Processes	1 Organization 5 Instructions, Procedures, and Drawings	5 Work Processes 6 Design	3.1 Facility Criteria	8.0 Laboratory Operations Quality Assurance 9.0 Control of Measuring and Test Equipment 10.0 Data Validation 15.0 Use of Statistical Methods

TABLE 2.4-1
LQM Source Documents Requirements Matrix (Cont.)

LQM	EPA QA/R-2	ANSI/ASQC E4-1994	NQA-1[1]	5700.6C[2]	ANSI N 13.30	ANSI/ASQC Q2-1991[3]
	8 Implementation of Work Processes (Continued)	3.2 Design of Data Collection Operations	10 Inspection 12 Control of Measuring and Test Equipment 14 Inspection, Test, and Operating Status	8 Inspection and Acceptance Testing		
9.0 Quality Assessment and Response	9 Assessment and Response[5]	2.9 Assessment and Response 3.4 Assessment and Response 3.5 Assessment and Verification of Data Usability	2 Quality Assurance Program 13 Handling, Storage, and Shipping 15 Control of Non-conforming Items 16 Corrective Action 18 Audits	9 Management Assessment 10 Independent Assessment	3.3 Direct Bioassay-Interpretation of Measurements 3.5 Direct Bioassay-Reporting Results 4.4 Indirect Bioassay-Reporting Results 6.1 Direct Bioassay Measurements 6.2 Indirect Bioassay Measurements	16.0 Nonconformity 17.0 Corrective Action 18.0 Auditing the Quality System
N/A	9 Quality Improvement[5]	2.10 Quality Improvement	N/A	3 Quality Improvement	N/A	N/A

Footnotes

[1] Section II, "Basic Requirements."
[2] Criterion from Section 9, "Requirements."
[3] Technically equivalent to ISO 9001.
[4] Quality Management and Quality Assurance Standards, ISO 9000, Part 3, "Guidelines for the Application of ISO 9001 to the Development, Supply and Maintenance of Software."
[5] This document has two sections numbered "9."

TABLE 2.4-2
Cross-Reference of LQM to NELAC Requirements for Quality Manuals

NELAC QUALITY MANUAL: REQUIRED ELEMENTS*	LQM, QA POLICY, AND/OR QA SOP REFERENCE
a) A quality policy statement, including objectives and commitments, by top management	LQM Chapter 1
b) The organization and management structure of the laboratory, its place in any parent organization and relevant organizational charts	LQM Chapter 1
c) The relationship between management, technical operations, support services and the quality system	LQM Chapter 1 LQM Chapter 7
d) Procedures to ensure that all records required under this chapter are retained as well as procedures for control and maintenance of documentation through a document control system which ensures that all standard operating procedures, manuals, or documents clearly indicate the time period during which the procedure or document was in force	LQM Chapter 2 LQM Chapter 3
e) Job descriptions of key staff and reference to the job descriptions of other staff	LQM Chapter 1 and Chapter 4 Separate document (hardcopy and/or electronic) provides job descriptions
f) Identification of the laboratory's approved signatories; at a minimum, the title page of the Quality Manual must have the signed concurrence, (with appropriate titles) of all responsible parties including the QA officer, technical director, and the agent who is in charge of all laboratory activities such as the laboratory director or laboratory manager	LQM Title/Approval Page
g) The laboratory's procedures for achieving traceability of measurements	LQM Chapter 8

*National Environmental Laboratory Accreditation Conference Standard, Quality Systems, July 1, 1999

TABLE 2.4-2
Cross-Reference of LQM to NELAC Requirements for Quality Manuals (cont.)

NELAC QUALITY MANUAL: REQUIRED ELEMENTS*	LQM, QA POLICY, AND/OR QA SOP REFERENCE
h) A list of all test methods under which the laboratory performs its accredited testing	LQM Table 8.2-1
i) Mechanisms for ensuring that the laboratory reviews all new work to ensure that it has the appropriate facilities and resources before commencing such work	LQM Chapter 7
j) Reference to the calibration and/or verification test procedures used	LQM Chapter 8 Tables 8.5-6 through 8.5-7
k) Procedures for handling submitted samples	LQM Sections 8.5.2 and 8.5.3
l) Reference to the major equipment and reference measurement standards used as well as the facilities and services used by the laboratory in conducting tests	Equipment list is Table 8.0-1 LQM Sections 5.2.4, 8.1, 8.5.4, 8.11 to 8.12
m) Reference to procedures for calibration, verification and maintenance of equipment	Calibrations in Tables 8.5-6 through 8.11-30 LQM Sections 8.5.4, 8.11 to 8.12
n) Reference to verification practices including interlaboratory comparisons, proficiency testing programs, use of reference materials and internal quality control schemes	LQM Section 9.4.5
o) Procedures to be followed for feedback and corrective action whenever testing discrepancies are detected, or departures from documented policies and procedures occur	LQM Chapter 9 SOP CORP-QA-0010, "Nonconformance and Corrective Action"

*National Environmental Laboratory Accreditation Conference Standard, Quality Systems, July 1, 1999

TABLE 2.4-2
Cross-Reference of LQM to NELAC Requirements for Quality Manuals (cont.)

NELAC QUALITY MANUAL: REQUIRED ELEMENTS*	LQM, QA POLICY, AND/OR QA SOP REFERENCE
p) The laboratory management arrangements for exceptionally permitting departures from documented policies and procedures or from standard specifications	LQM Section 7.4 and 9.1
q) Procedures for dealing with complaints	LQM Section 9.2
r) Procedures for protecting confidentiality and proprietary rights (including national security concerns)	LQM Section 8.9.6
s) Procedures for audits and data reviews	LQM Section 5.3.6, 8.8 and 9.4 SOP CORP-QA-0004 "Independent QA Data Review"
t) Processes/procedures for establishing that personnel are adequately experienced in the duties they are expected to carry out and/or receive any needed training	LQM Chapter 4 SOP CORP-QA-0013, "Employee Orientation and Training"
u) Process/procedures for educating and training pesonnel in their ethical and legal responsibilities including the potential punishments and penalties for improper, unethical or illegal actions	LQM Section 1.4 Policy QA-008 – Data Recording Requirements QA-010 – Maintaining Time Integrity QA-011 – Acceptable Manual Integration Practices P-T-001 – Selection of Data Points Required for Initial Calib.
v) Reference to procedures for reporting analytical results	LQM Section 8.9 Policy QA-004 "Rounding and Significant Figures" Policy QA-009 "Reporting Limits"

TABLE 2.4-2
Cross-Reference of LQM to NELAC Requirements for Quality Manuals (cont.)

NELAC QUALITY MANUAL: REQUIRED ELEMENTS*	LQM, QA POLICY, AND/OR QA SOP REFERENCE
w) A table of contents and applicable list of references, glossaries, and appendices	LQM Table of Contents List of Policies and SOPs Table 2.3-2

*National Environmental Laboratory Accreditation Conference Standard, Quality Systems, July 1, 1999

TABLE 2.4-3
STL North Canton Quality Documents and Required Approval

Quality Document	Required Approvals
Laboratory Quality Manual (LQM)	• Laboratory Director • Technical Director • Quality Assurance Manager
STL North Canton Policies	• Laboratory Director • Quality Assurance Manager
STL North Canton Standard Operating Procedures (SOPs)	• Laboratory Director • Technical Specialist • Laboratory Health and Safety Coordinator(1) • Quality Assurance Manager

(1) Required only if procedure encompasses more than standard office safety requirements.

TABLE 2.4-4
STL Quality Document Review Frequency[1]

Document Type	Frequency of Review	Responsible Party
Laboratory Quality Manual (LQM)	Every Two Years	Quality Assurance Manager
Standard Operating Procedures (SOP)	Every Two Years[2]	Quality Assurance Manger & Operations Manager

[1] SOP reviewed sheets can be utilized in lieu of changing an entire document to indicate that an SOP has been reviewed and no changes were necessary (QA-001).

[2] DOD/DOE requires an annual review of all "major" SOPs (NC-QA-0016).

TABLE 3.4-1

STL North Canton Records & Retention Schedule

Type of Record	Retention	Disposition
General Laboratory Documents		
Instrument output	5 yrs from project completion	Shred or burn
Quality control data	5 yrs from project completion	Shred or burn
Field sample data	5 yrs from project completion	Shred or burn
Final analytical reports	5 yrs from project completion	Shred or burn
Instrument logbooks	5 yrs from last entry	Shred or burn
Equipment monitoring & maintenance records	5 yrs from last entry	Shred or burn
Instrument calibration records	5 yrs from last entry	Shred or burn
Standard preparation logs	5 yrs from last entry	Shred or burn
Standards certificates	5 yrs from last entry	Shred or burn
Measurement & test equipment logs (e.g., refrig., balances, etc.)	5 yrs from last entry	Shred or burn
Method & instrument validation records	5 yrs from last entry	Shred or burn
Instrument manuals	Retain until superseded	Trash
Project management files	5 yrs from date of archival	Shred or burn
Quotes & proposals	2 yrs from date of expiration	Shred or burn
LQM, policies, & SOPs	5 yrs from date of archiving	Shred or burn
Analyst demonstrations of proficiency	5 yrs from date of archival	Shred or burn
Quality assurance audits	5 yrs from last entry	Shred or burn
Certifications & approvals	5 yrs from last entry	Shred or burn
Employee signature list	5 yrs from date of archival	Shred or burn
MDL Studies	5 yrs from last entry	Shred or burn
Performance testing studies	5 yrs from last entry	Shred or burn
QA reports to management	5 yrs from last entry	Shred or burn
Quality control charts	5 yrs from last entry	Shred or burn

TABLE 3.4-1

STL North Canton Records & Retention Schedule

Type of Record	Retention	Disposition
Environment, Health and Safety Records		
Medical records	Retain while active & 30 yrs from last entry	Shred or burn
Employee exposure & monitoring records	Retain while active & 30 yrs from last entry	Shred or burn
Workers compensation files & first report of injury	Retain while active & 30 yrs from last entry	Shred or burn
Accident logs (OSHA Form 200)	5 yrs from last entry	Shred or burn
Accident reports	5 yrs from last entry	Shred or burn
Environmental permits	5 yrs from last entry	Shred or burn
Environmental management, e.g., discharge reports	5 yrs from last entry	Shred or burn
Health & safety audits	5 yrs from last entry	Shred or burn
Chemical Hygiene Plan	5yrs from archival	
Safety Inspections	5 yrs from last entry	Shred or burn
Radioactive materials records	5 yrs from last entry	Shred or burn
NRC or state radioactive materials handling inspections	5 yrs from last entry	Shred or burn
TLD exposure records	5 yrs from last entry	Shred or burn
EH&S training	5 yrs from last entry	Shred or burn
Accounting	See Accounting and Controls Procedures Manual	
Administrative		
Personnel records (not including medical or disability records)	7 years from last entry	Shred or burn

TABLE 5.2-1
List of STL Quality-Related Items
that Require Evaluation Prior to Use

Quality-Related Item	Standard Operating Procedure for Quality Testing
Acetone	CORP-QA-0001
Dichloromethane	CORP-QA-0001
Hexane	CORP-QA-0001
Hydrochloric acid	CORP-QA-0001
Freon	CORP-QA-0001
Methanol	CORP-QA-0001
Nitric acid	CORP-QA-0001
Hydrogen Peroxide	CORP-QA-0001
Sulfuric acid	CORP-QA-0001
Toluene	CORP-QA-0001

TABLE 6-1
GALP Cross Reference To LQM

GALP Section	GALP Guidance	STL Document
8.1 Laboratory Management	8.1.1 ensure that personnel clearly understand the functions they are to perform	LQM 1.6.2, 1.6.4, and 4.0
	8.1.2 ensure that QAU monitors computer activities	LQM 9.2.2.1
	8.1.3 ensure that personnel, resources, and facilities are adequate and available as scheduled	CORP-IT-0002; LQM 1.6.1-1.6.4
	8.1.4 receive reports of QAU inspection and audit reports, and ensure corrective actions are promptly taken in response to any deficiencie	LQM 9.2.2.1
	8.1.5 approve SOPs related to the computer activities, and ensure that devations to the SOPs are documented	LQM 3.3 and 9.1.4
	8.1.6 assure that GALP provisions are followed	CORP-IT-013; LQM 6.0
8.2 Personnel	8.2.1 must have adequate education, training, and experience to perform assigned IT functions	CORP-IT-013; LQM 4.0
	8.2.2 a summary of training, experience, and job description must be maintained	LQM 4.1
	8.2.3 personnel must be of sufficient number for timely and proper operation of the computer systems	CORP-IT-0002; LQM 1.6.2
8.3 Quality Assurance Personnel	8.3.1 shall be separate and independent of IT personnel, and shall report directly to laboratory management	LQM 1.6.1
	8.3.2 shall have immediate access to the computer data, SOPs, and other records	LQM 1.61 and will be added to 9.2
	8.3.3 inspect the LIMS at intervals to ensure the integrity of LIMS raw data, and shall present inspection reports to management	LQM 9.2
	8.3.4 determine that no deviations from approved SOPs were made without proper authorization and documentation	LQM 9.1.1
	8.3.5 periodically audit raw data to ensure their integrity	9.2
	8.3.6 maintain adequate records of the QAU operations	9.2
8.4 LIMS Raw Data	8.4.1 LIMS raw data and the storage media on which they reside must be identified and documented. The documentation shall be included in the lab's SOPs.	System map is with IS Director
	8.4.2 the individual(s) responsible for entering and recording LIMS raw data must be uniquely identified, together with the date and time the data were entered	QA-008
	8.4.3 the instrument transmitting raw data must be uniquely identified in the record, together with the date and time of transmission	QA-008

TABLE 6-1
GALP Cross Reference To LQM (cont.)

	8.4.4 procedures and practices used to verify LIMS raw data must be documented in controlled SOPs	CORP-IT-0007; LQM 8.8-8.8.3
8.5 Software	8.5.1 SOPs shall be established for: a. software development b. software testing c. change control d. version control e. maintaining historical file	CORP-IT-013; CORP-IT-0001; CORP-IT-0007
	8.5.2 documentation shall be maintained for: a. software description & functional requirements b. algorithms and formulas c. testing and quality assurance	CORP-IT-013
	8.5.3 all documentation is readily available in the facility where the software is used and SOPs are readily available where procedures are performed	CORP-IT-013 includes this statement, but we are on a WAN and documentation is not duplicated at each lab
	8.5.4 a historical file of software and documentaiton shall be retained	CORP-IT-0001, Sect 4.14.1
8.6 Security	Laboratory management shall ensure that security practices are adequate to assure the integrity of data	LQM 6.2 [more detail can be added to this section]; CORP-IT-0005; CORP-IT-013
8.7 Hardware	8.7.1 must be of adequate design and capacity, and a documented description maintained	LQM 6.1; CORP-IT-013
	8.7.2 must be installed in accordance with manufacturer's recommendations, and undergo documented acceptance testing as described in a laboratory SOP	CORP-IT-0001; CORP-IT-013; LQM 6.1
	8.7.3 testing, maintenance, and repair must be described in a laboratory SOP	CORP-IT-013 includes this statement
8.8 Comprehensive Testing	Management shall ensure that comprehensive testing shall be documented at least every 24 months or more frequently as a result of software changes.	CORP-IT-0001; LQM 6.3.4
8.9 Records Retention	Procedures must be in place for the retention of LIMS raw data and documentation and records pertaining to LIMS	QMP; LQM 3.4-3.5
8.10 Facilities	8.10.1 the environmental conditions of the facility housing the LIMS must be controlled to protect against data loss	LQM 6.2
	8.10.2 environmental conditions for storing LIMS raw data and records must be adequate	LQM 6.2

TABLE 6-1
GALP Cross Reference To LQM (cont.)

8.11 SOPs	8.11.1 SOPs, as described above, must be maintained and readily available where the procedure is performed	LQM 3.1-3.2; SOP Index
	8.11.2 SOPs must be reviewed periodically to ensure that they are accurate	LQM 3.3
	8.11.3 SOPs must be authorized and controlled, with all changes subject to the same approvals and control	LQM 3.3
	8.11.4 an historical file of SOPs must be maintained	LQM 3.5

Table 6-2
Computer Software Listing

Producer	Software
Microsoft	Windows NT 4.0
	Windows 95
	Windows for Workgroups 3.1.1
	Exchange 5.0
	Outlook 98
	Office 97 SR2
	Internet Explorer 4.01
IBM	Client Access v.3 SF51967
WRQ	Reflection X 7.1
	Target 3.4
	Envision 3.4
Adobe	Acrobat
McAffee	VirusScan 4.0.2

Table 6-3
Computer Hardware Listing

Model:	Gateway E-3110				
Computer Name	**Serial Number**	**Current User**	**Location**	**Jack Number**	**Subnet Port**
CANP3160	0009864777		146	120	3B9
CANP3009	0009864773	LeesonD	272	154	3A7
CANP4031	0009864776	RichmanJ	262	152	41C10
CANP3165	0009863565	WetChem	346	335	3B3
CANP3166	0009864786	KravetzB	272	322	3B2
CANP1026	0009864772	MSVOA	148	121	1B2
CANP3170	0009864782	HerronD	252	170	3B4
CANP4213	0009863562	TothR	346	269	41D5
CANP4042	0009893049	HaueterL	298	382	43B1
CANP3180	0009863563	Extractions	338	270	3A6
CANP3182	0009893048	SiegfriedA	132	317	3A10
CANP4012	0009893047	BlakemanC	258	375	43B4
CANP4035	0009893052	MingerT	120	140	42C9
CANP4014	0009864787	DiamentS	268	203	42A9
CANP4015	0009864774	BruceM	380	239	42B8
CANP4017	0007986916	UplingerJ	104	112	42D6
CANP4004	0007986920	BotimerB	110	145	42C3
CANP4066	0007986917	SheaK	108	144	42C5
CANP4046	0007986919		122	103	42D1
CANP4216	0007986918		106	115	42B3
CANP4199	0009059960		118	116	42D1
CANP4021	0009864771	CampbellS	136	129	42A12
CANP4022	0009863561	JacksonS	134	132	41C5
CANP4023	0009864775		258	151	41C9
CANP4024	0009863054	HeakinT	112	119	41A3
CANP4068	0009893462	EzzoL	300	126	41D3
CANP4029	0009864781	OmearaP	330	256	41B5
CANP4032	0009893050	HemmerichM	266	202	41C2
CANP4034	0009893053	HustonS	114	329	43A5
CANP4040	0009864770	GirardB	252	172	3A11
CANP4041	0009864785	DaleyM	252	163	42C9

Table 6-3
Computer Hardware Listing

Model:	Gateway E-3110				
Computer Name	**Serial Number**	**Current User**	**Location**	**Jack Number**	**Subnet Port**
CANP4050	0009864783	FrankL	258	150	42B4
CANP2022	0009864780		390	247	2B12
CANP4117	0009864779	StillerT	126	130	41C3
CANP4221	0009864778	BuzashK	252	185	42A3
CANP3169	0009864784		338	388	3B5
CANP4114	0009893051	HulaT	394	246	41D7
CANP2025	0009863564	GC_Semi	390	342	2B8
CANP1035	0013397604		146	215	1A10
CANP1036	0013397601	QuayleR	146	217	1B3
CANP1037	0013397603				
CANP4055	0012929095		106		
CANP4056	0012929097	MeansR	116	143	42C7
CANP4177	0013224794	McCormickA	216	303	43A11
CANP4178	0013224796	WoodG	212	305	43A10
CANP4187	0013224795	DanfordA	212	156	41A10
CANP4188	0013224793	PohlD	230	306	43A8
CANP4189	0013224792	KuziorK	216	186	43B2
CANP4203	0013279039		276	379	41A2
CANP4204	0013279041	BeldingB	276	173	41B12
CANP4205	0013279040		276	206	41B11
CANP1034	0013397602	MillerE	372	244	1B1
CANP4058	0012929100	BotimerB	112		
Model:	**Gateway E-4200**				
		RisdenR	384		
CANP2006	0013750359		390		
3	0013750358				
1	0013750357				
4	0013750356				
5	0013750355				
Model:	**IBM ThinkPads, 2635-HGU**				

Table 6-3
Computer Hardware Listing

Model:	Gateway E-3110				
Computer Name	Serial Number	Current User	Location	Jack Number	Subnet Port
Computer Name	Serial Number	Current User	Location	Jack Number	Subnet Port
CANP4182	8671445666	HulaD	226	196	3D11
CANP4183	8671445670	HeakinD	230	182	42C11
CANP4184	8671445668	McGregorR	224	195	42D4
CANP4185	8671445669	SmithJ	220	189	41A5
CANP4186	8671445667	StraitB	220	304	43A9
Model:	HP Chemstations				
Computer Name	Serial Number	Current User	Location	Jack Number	Subnet Port
CANP1029	US64757194				
CANP1031	US74651733				
CANP1030	US73351217				
CANP1032					
CANP3006	U563253596		390		
CANP2010	U571355728		394		
CANP4071			390		
CANP4013					
CANP4079			346		
CANP4176					
CANP4179			350		
CANP4020			346		
CANP1033					
CANP2021					
CANP3030			354	263	
Model:	Compaq 4/66				
Computer Name	Serial Number	Current User	Location	Jack Number	Subnet Port
CANP4081					
CANP4175					
CANP4130					
CANP4201					
CANP4038					

Table 6-3
Computer Hardware Listing

Model:	Gateway E-3110				
Computer Name	**Serial Number**	**Current User**	**Location**	**Jack Number**	**Subnet Port**
CANP4039					
CANP3007					
CANP4133			304		
CANP4131			304		
CANP4180		HaueterB			
Model:	**Dell Laptops**				
Computer Name	**Serial Number**	**Current User**	**Location**	**Jack Number**	**Subnet Port**
CANP4011		OprandiC	204	157	
CANP4044		GirardS	280	207	
CANP4037		GreenwellB	284		
CANP4047		JohnsonO	270	177	
Model:	**XTRA-PC's**				
Computer Name	**Model type**	**Current User**	**Location**	**Jack Number**	**Subnet Port**
NCA34	Caliber				
NCA35	Cumulus				
	Cumulus	StillerM			
NCA40	Hyundai				
CANP4080	LEK				
CANP4053	LEK				
CANP4120	LEK				
CANP3082	Digital				
CANP4057	Digital				
CANP4010	MicroXperts				
CANP4170	ASI				
CANP4155	Digital				
HP-UX 10.20	HP-Apollo 735	Unix			
HP-UX 10.20	HP-Apollo 735	Unix			

Table 8.0 –1
STL North Canton
Instrument List

Instrument Type	Manufacturer	Model	Instrument ID #	Date Purchased
GC/MS	Finnigan	Incos XL	503	11/27/92
	Hewlett Packard	5971A	UX2	09/96
	Hewlett Packard	5971A	UX3	09/96
	Agilent (formerly HP)	5973	UX11	10/00
	Hewlett Packard	5973	UX10	Leased
	Hewlett Packard	5973	UX8	Leased
	Hewlett Packard	5973	UX9	Leased
	Hewlett Packard	5973	UX7	Leased
AUTOSAMPLER	OI Analytical	4552	UX10 (MSV)	Leased
	OI Analytical	4552	UX8 (MSV)	Leased
	OI Analytical	4552	UX9 (MSV)	Leased
	Tekmar	ACS 2016	UX3 (MSV)	
	OI Analytical	4552	UX7 (MSV)	Leased
	OI Analytical	4552	503 (MSV)	
	OI Analytical	4552	UX11 (MSV)	11/3/00
	OI Analytical	4552	UX2 (MSV)	
	OI Analytical	4552	O (GCV)	
	Varian	Archon	A (GCV)	
	OI Analytical	4552	Z (GCV)	
	Varian	Archon	P (GCV)	
	Tekmar	2016	Spare - not in service.	
GC/MS (Semivolatiles)	Hewlett Packard	5973/6890		Leased
	Hewlett Packard	5973/6890		Leased
	Hewlett Packard	5973/6890		Leased
	Hewlett Packard	5973/6890		Leased
HPLC	Hewlett Packard	HPLC 1100		12/14/98
	Waters	600E - UV Fluorescence		09/15/92
Purge & Trap	OI Analytical	4560	UX10 (MSV)	Leased
	OI Analytical	4560	UX8 (MSV)	Leased
	OI Analytical	4560	UX9 (MSV)	Leased
	Tekmar	LSC2000	UX3 (MSV)	
	OI Analytical	4560	UX7 (MSV)	Leased
	OI Analytical	4560	503 (MSV)	
	OI Analytical	4560	UX11 (MSV)	11/3/00
Purge & Trap	OI Analytical	4560	UX2 (MSV)	
	Tekmar	3000	O (GCV)	

Table 8.0 –1
STL North Canton
Instrument List

Instrument Type	Manufacturer	Model	Instrument ID #	Date Purchased
	OI Analytical	4560	A (GCV)	
	Tekmar	LSC2000	Z (GCV)	
	Tekmar	3000	Spare – not in service	
	Tekmar	3000	P (GCV)	Leased
Gas Chromatograph	Tracor	540 PID/FID	A (GCV)	04/01/89
	Tracor	540 PID/FID	Z (GCV)	01/26/89
	Tracor	540 HALL/PID		10/22/93
	Tracor	540 HALL/PID		04/08/89
	Hewlett Packard	6890w/Dual PID/HALL	O (GCV)	08/97
	Hewlett Packard	6890 PID/HALL	P (GCV)	
	Hewlett Packard	5890A Dual FPD	PO-1 (GCS)	04/01/89
	Hewlett Packard	5890A Dual FID Y-splitter Series II	R (GCS)	10/31/90
	Hewlett Packard	5890A Dual ECD	K (GCS)	01/26/89
	Hewlett Packard	6890 EPC & Dual ECD Y-splitter	1 (GCS)	12/98
	Hewlett Packard	6890 EPC & Dual ECD Y-splitter	2 (GCS)	12/98
	Hewlett Packard	6890 EPC & Dual ECD Y-splitter	3 (GCS)	12/98
	Hewlett Packard	6890 EPC & Dual ECD Y-splitter	4 (GCS)	12/99
	Hewlett Packard	6890 EPC & Dual ECD Y-splitter	5 (GCS)	12/99
	Hewlett Packard	6890 FID	(GCS)	2000
ICP	Thermal Jarrell Ash	Trace Analyzer		02/01/94
	Thermal Jarrell Ash	Trace Analyzer		02/01/94
ICP-MS	Perkin Elmer	Elan 6100	I7	04/01
Metals/Mercury	Leeman	PS200 II		10/20/99
	Leeman	Hydra AF gold plus		12/2000
	Leeman	Hydra AF Gold+, Model#112-00067-1	H3	09/19/01
Metals/GFAA	Varian	SpectrAA-400		02/08/90
TRAACS	Bran & Luebbe	800		05/07/93
	Bran & Luebbe	800		04/01/89
TOX	Mitsubishi	TOX-10E		11/06/89
TOX	EuroGlass	1200 ThermoGlas w/ Autosampler		

Table 8.0 –1
STL North Canton
Instrument List

Instrument Type	Manufacturer	Model	Instrument ID #	Date Purchased
Turbidimeter	HF Scientific	Micro 100		2001
BOD	Labtronics, Inc.	BOD Magic		1999
Block Digester	Andrews	2210 Phenol		04/20/99
	Andrews	2205 Ammonia		04/20/99
	Lachat	BD46 TKN		1992
Autotitrator	Mantech	PC-Titrate		2001
Conductivity	Mantech	4310		11/13/89
Cyanide	Midi Serial # 1000-99-01	PRG-2520-BL		04/20/99
D.O. Meter	YSI	52C E		01/01/93
Flashpoint	Petrolab	Petrotest	199443	04/20/99
GPC Extractions	ABC (O.I. Analytical)	Model # 1002B		06/99
Oil-In-Water	Buck Scientific	HC404		02/20/90
pH Meter	Orion	250A		12/01/85
	Orion	520A		
TOC	OI	1010		4/98
UV/VIS Spectrophotometers	Milton Roy	Spectronic 401		08/13/93
	Spectronic 20	Genesys		8/98
Discrete Analyzer	Kone	Konelab		2001
Ion Chromatography	Dionex	DX-320		2001
	Dionex	DX-120		1/99
Residual Chlorine Meter	Hanna	HI 93701		06/00

Table 8.2-2 Standard Operating Procedures

SOP Number	SOP Title	Method	Rev#	Revision Date
	WET CHEMISTRY			
NC-WC-0006	Alkalinity (Total) *(Also select NC-WC-0003)*	EPA310.1 SM2320B	4	02/06/01
NC-WC-0003	Alkalinity - Carbonate, Bicarbonate, and Hydroxide *(Also select NC-WC-0006)*	SM2320B SM4500-CO_2 D	2.1	02/08/01
NC-WC-0012	Alkalinity, Phenolphthalein	EPA310.1 SM2320B	2.1	08/17/00
NC-WC-0018	Carbon, Total Organic (TOC - Analysis for Non-Waters)	Walkley-Black	2.1	03/30/01
NC-WC-0017	Carbon, Total Organic (TOC)	EPA415.1 SW9060 SM5310D	2	06/01/99
NC-WC-0023	Cation-Exchange Capacity - ***Scheduled Test* requires LD approval**	SW9081	2.1	03/30/01
NC-WC-0013	Chloride (Automated Ferricyanide)	EPA325.2 SW9251	2	06/09/98
NC-WC-0020	Chloride (Titrimetric) - ***Scheduled Test***	EPA325.3 SW9252A	2	04/18/96
NC-WC-0021	Chlorine Total (Residual) and Free	EPA330.5 SM4500-CIG	1.1	12/05/00
NC-WC-0024	Chromium, Hexavalent (Colorimetric)	SW7196A SM3500-CR	3.1	10/24/01
NC-WC-0079	Conductivity (Specific Conductance)	EPA120.1 SM2510B SW9050	1	01/16/97
NC-WC-0031	Cyanide, Automated Pyridine-Barbituric Acid *(analysis - also select NC-WC-0032)*	SW9012A EPA335.1, 335.2 SM4500CN-I CLP ILM03.0	7	05/31/01
NC-WC-0032	Cyanide, Distillation *(prep - also select NC-WC-0031)*	SW9012A EPA335.1, 335.2 SM4500CN-I SM4500CN-E CLP ILM03.0	8.2	05/31/01
NC-WC-0033	Cyanide, Reactive **Requires LD Approval**	SW846 7.3.3.2	2	10/11/00

Table 8.2-2 Standard Operating Procedures

SOP Number	SOP Title	Method	Rev#	Revision Date
NC-WC-0070	Decanting Procedure for Aqueous Sample	N/A	0	06/13/95
NC-WC-0008	Demand, Biochemical Oxygen (BOD), Carbonaceous (CBOD)	SM5210B EPA405.1	3.1	04/28/00
NC-WC-0083	Demand, Chemical Oxygen (COD - Low Level) **Requires LD approval**	SM5220D EPA410.4	1.1	03/19/01
NC-WC-0005	Demand, Chemical Oxygen (COD - Titrimetric) **Requires LD approval**	SM5220C	2	06/09/98
NC-WC-0019	Demand, Chemical Oxygen (COD) (Colorimetric)	SM5220D EPA410.4	5	03/16/00
NC-WC-0034	Flashpoint (Closed Cup)	ASTM D93-85 SW846 Method 1010	0	10/24/97
NC-WC-0035	Fluoride (ISE) --- *(analysis, all matrices)* - ***Scheduled Test***	EPA 340.2	2	07/19/01
NC-WC-0071	Gravity, Specific	SM2710F	1	04/09/99
NC-WC-0067	Halogens, Total Organic (TOX)	SW9020A EPA450.1	2	03/09/99
NC-WC-0036	Hardness, Total (mg/L as $CaCO_3$)	EPA130.2	3	04/19/99
CORP-WC-0003	HEM/SGT-HEM by Method 1664	EPA1664A	1	11/15/99 Change form 01/24/00
NC-WC-0045	Hydrocarbons - TRPH and O&G by IR (Analysis Procedure - All Matrices)	EPA418.1; 413.2 SW9071A SM5520C	1.1	08/28/01
NC-WC-0047	Hydrocarbons, TRPH and O&G by IR (Solid Soxhlet Extraction)	SW9071A	1	06/09/98
NC-WC-0048	Hydrocarbons, TRPH and O&G by IR (Water Extraction)	EPA418.1 EPA413.2 SW9070	3.1	03/30/01
NC-WC-0049	Hydrocarbons, TRPH by IR (Solid Sonication)	SW3550A SW9071A	1	06/09/98
NC-WC-0084	Ion Chromatography, Determination of Inorganic anions by	EPA300.0	3	10/03/00
NC-WC-0025	Iron, Ferrous	SM3500-Fe D	2	05/18/99

Table 8.2-2 Standard Operating Procedures

SOP Number	SOP Title	Method	Rev#	Revision Date
NC-WC-0001	Nitrite, Nitrate/Nitrite, Nitrate Automated	EPA353.2	2	12/28/98
NC-WC-0038	Nitrogen, Ammonia (ISE) ----- *(water)*	EPA350.3	4	03/13/00
NC-WC-0039	Nitrogen, Ammonia Distillation/ Titration *(solid)* - ***Scheduled Test***	EPA350.2	2.1	03/19/01
NC-WC-0040	Nitrogen, Total Kjeldahl(TKN)	EPA351.3	1.1	03/30/01
NC-WC-0041	Nitrogen, Total Organic (TON)	EPA350.2; 351.3	0	06/04/98
NC-WC-0043	Oil&Grease & TRPH, Gravimetric Solid *(prep and analysis)*	SW9071A SM5520B	1	02/12/99
NC-WC-0044	Oil&Grease & TRPH, Gravimetric Water *(prep and analysis)* *(Also see TRPH and O&G by IR)*	EPA413.1 SM5520B SM5520F SW9070	1.1	03/19/01
NC-WC-0046	Paint Filter	SW9095	0	06/08/95
NC-WC-0010	pH Electrometric Method	SW9040B SW9045C EPA150.1	4.1	11/28/00
NC-WC-0009	pH Paper Method	SW9041A	1.1	02/07/01
NC-WC-0007	Phenolics (Manual Spectrophotometer) - ***Scheduled Test***	EPA420.1 SW9065	2.1	03/12/01
NC-WC-0050	Phosphorous: Total, Ortho, and Organic - ***Scheduled Test***	EPA365.2/365.3 SM4500-P	2.4	05/15/01
NC-WC-0054	Solids, Total & Volatile Suspended (TSS & VSS)	EPA160.2 SM2540D	0.1	10/24/97
NC-WC-0055	Solids, Total Dissolved (TDS)	EPA160.1 SM2540C	1	02/17/99
NC-WC-0004	Solids, Total, Percent Moisture, Ash and Total Volatile Solids	EPA160.3/160.4 SM2540E ASTMD2216-90 ASTMD1553-83 ILM0 3.0 & 4.0 OLM01.9 & 3.1	3	08/04/00
NC-WC-0086	Alkaline Digestion for Hexavalent Chromium	SW846 3060A,ASTM Method D1498	1	09/07/01
NC-WC-0087	Spreadsheet Upload SOP, Wet Chemistry	NA	1	03/09/99

Table 8.2-2 Standard Operating Procedures

SOP Number	SOP Title	Method	Rev#	Revision Date
NC-WC-0060	Sulfide - *Scheduled Test*	SW846 9030A EPA 376.1	1.0	11/20/00
NC-WC-0061	Sulfide, Reactive	SW846 7.3.4.2	1	10/11/00
NC-WC-0068	Turbidity	EPA180.1	3	05/02/01
	METALS			
NC-MT-0001	Mercury, Prep and Analysis of Hg in H_20 by cold vapor Atomic Fluororescence	1631B MCAWW 245.7	1	04/05/01
NC-MT-0002	Inductively Coupled Plasma-Mass Spectrometry	EPA 6020 and 200.8	2.0	10/01/01
NC-MT-0010	Hardness by Calculation	SM2340B, SW846 Method 6010B	1.0	07/12/01
CORP-MT-0003NC	Graphite Furnace Atomic Absorption Spectroscopy (Thallium Only)	SW846 7000A MCAWW 200	2.2	10/04/00
CORP-MT-0001NC	Inductively Coupled Plasma –Atomic Emission Spectroscopy, Spectrometric Method for Trace Elements **Update II**	6010A 200.7 EPA 40CFR 136	2	10/27/97
CORP-MT-0001NC	Inductively Coupled Plasma-Atomic Emission Spectroscopy, Spectrometirc Method for Trace Element Analyses - **UPDATE III**	6010B 200.7	3.2	01/19/01
CORP-MT-0002NC	Inductively Coupled Plasma –Atomic Emission Spectroscopy	200.7 CLP-M SOW ILMO3.0, SOW ILM04.0	1.1	04/09/98
CORP-MT-0005NC	Mercury in Aqueous Samples by Cold Vapor Atomic Absorption (Prep and Analysis)	SW846 7470A MCAWW 245.1	2.3	05/15/01
CORP-MT-0006NC	Mercury in Aqueous Samples by Cold Vapor Atomic Absorption (Prep and Analysis)	245.1 CLP-M SOW ILMO3.0, SOW ILM04.0	1.1	12/05/00
CORP-MT-0007NC	Mercury in Solid Samples by Cold Vapor Atomic Absorption Spectroscopy (Prep and Analysis)	SW846 7471A MCAWW 245.5	2.3	05./15/01
CORP-MT-0008NC	Mercury in Solid Samples by Cold Vapor Atomic Absorption Spectroscopy (Prep and Analysis)	245.5 CLP-M SOW ILMO3.0, SOW ILM04.0	1.1	08/27/01

Table 8.2-2 Standard Operating Procedures

SOP Number	SOP Title	Method	Rev#	Revision Date
INORGANIC PREP				
CORP-IP-0003NC	Acid Digestion for Aqueous Samples by SW846 and MCAWW 200 Series Methods	SW846 and MCAWW 200 Series Methods	1.3	09/25/01
CORP-IP-0004NC	Toxicity Characteristic Leaching Procedure and Synthetic Precipitation Leaching Procedure	SW-846 Method 1311, SW-846 Method 1312	1.1	10/10/00
CORP-IP-0001	Acid Digestion of Waters and Soils, CLP SOW ILM03.0	SOW ILM03.0	1	06/28/99
CORP-IP-0002NC	Acid Digestion for Soil Samples by SW846 Method 3050B	SW846 and 3050B	2.2	09/25/01
NC-IP-0009	DI Leachate Procedure for Solids	Internal (RAS) ASTM D3987-85	0	08/12/98
NC-IP-0007	GFAA Prep for Total Metals (Aqueous)	SW3020A; 7040; 7060;7211;7740; 7761; & EPA	0	03/12/96
NC-IP-0001	Grinding & Sieving of Solid Matrices	N/A	1	05/19/99
NC-IP-0003	ICP & FLAA Prep for Total Metals (Aqueous)	EPA200.7;3005A SW3010A	2	05/21/99
ORGANIC PREP				
CORP-OP-0001NC	Extraction and Cleanup of Organic Compounds From Waters and Soils **UPDATE III**	SW846 3500-ser; 3600-ser, 8151A, and 600 - series	3.8	05/23/01
CORP-OP-0001NC	Extraction and Cleanup of Organic Compounds from Waters and Soils - **UPDATE II**	SW846 3500-ser; 3600-ser; 8150;8151; and 600-series	2.5	01/09/01
NC-OP-0022	Extractable residue (lipids) from fish tissue		1	02/21/01
NC-OP-0021	Wipe Extraction Method for PCBs	Internal	1	02/21/01
GC/MS				
NC-MS-0014	Extraction and Analysis of Semivolatiles (BNA) Operation-Specific Standard Operating Procedure	EPA CLP OLM04.2	0	05/15/00
CORP-MS-0001NC	GC/MS Semivolatiles Analysis – **Update II**	8270B	1.3	05/09/97

Table 8.2-2 Standard Operating Procedures

SOP Number	SOP Title	Method	Rev#	Revision Date
NC-MS-0009	Analysis of Semivolatile Organics by USEPA CLP Statement of Work OLM03.1and OLM03.2	OLM03.1/OLM03.2	1	08/10/00
CORP-MS-0002NC	GC/MS Volatile Organics Analysis – **UPDATE II**	8240B, 8260A	1.3	09/12/97
CORP-MS-0001NC	GC/MS Analysis Based on Method 8270C and 625 - **UPDATE III**	8270C and 625	2.4	05/29/01
CORP-MS-0002NC	Determination of Volatile Organics by GC/MS – **(Note: Update II and Update III are in one SOP)**	8260B, 8260A, and 624	2.3	05/23/01
NC-MS-0013	Analysis of Semivolatile Organics by USEPA CLP Statement of Work OLC02.1	OLC02.1	0	11/29/99
NC-MS-0015	Analysis of Polynuclear Aromatic Hydrocarbons by Selective Ion Monitoring	8270C	0	10/16/00
NC-MS-0016	GCMS Volatile Organic Analysis by EPA CLP SOW OLM04.2	OLM04.2	0	09/21/00
NC-MS-0011	Analysis of Volatile Organics by USEPA CLP Statement of Work OLM02.1	OLM02.1	0	11/17/99
NC-MS-0010	Analysis of Volatile Organics by USEPA CLP Statement of Work OLM03.1 and OLM03.2.	OLM03.1/OLM03.2	0	04/07/98
	GC			
CORP-GC-0001	Gas Chromatographic Analysis – **UPDATE II**	8000A, 8010B, 8020A, 8021A, 8080A, 8081, 8150B, 8151, SW846	2	01/31/96
CORP-GC-0001NC	Gas Chromatographic Analysis - **UPDATE III**	8000B, 8021B, 8081A, 8082, 608, 8151A, 8310, 610, 8141A and SW846 and Wisconsin DRO	5.6	05/25/01
NC-GC-0019	Azeotropic Distillation and Analysis of Water-Soluble VOCs in Waters and	SW5031-Proposed 8015A-Modified	0	05/22/97

Table 8.2-2 Standard Operating Procedures

SOP Number	SOP Title	Method	Rev#	Revision Date
	Solids - **Requires LD Approval**			
NC-GC-0003	GC Halogenated/Aromatic VOCs	EPA601 EPA602	2	05/02/01
NC-GC-0018	GC VOC in Water, Purge & Trap - **Requires LD Approval**	465D/465E	0	06/15/95
NC-GC-0027	Analysis of Chlorinated Pesticides and PCBs by USEPA CLP Statement of Work OLM03.1 and OLM03.2	OLM03.1/OLM03.2	2	08/10/00
NC-GC-0025	TPH as Gasoline	8015B-Modified	4	05/23/01
	SAMPLE CONTROL			
NC-SC-0002	Evidentiary SOP	N/A	0	06/16/95
NC-SC-0007	Sample Identification SOP	N/A	1	10/03/95
NC-SC-0006	Sample Procurement Protocol	N/A	1	05/20/98
NC-SC-0005	Sample Receiving and Sample Control	N/A	6.1	06/14/01
NC-SC-0010	Sample Shipment for Dioxin Analysis	N/A	2	03/10/99
	QUALITY ASSURANCE			
NC-QA-0024	Control Chart Generation SOP	N/A	0	07/06/98
QA-008	Data Recording Requirements	N/A	2	10/05/98
QA-017	Data Recording Requirements	N/A	0	07/07/99
CORP-QA-0013	Employee Orientation and Training	N/A	1	12/15/98
NC-QA-0022	Equipment Maintenance	N/A	1.0	06/15/01
NC-QA-0015	Equipment Monitoring and Thermometer Calibration	N/A	5	02/13/01
QA-009	Establishment of Reporting Limits	N/A	3	01/01/99
NC-QA-0014	Glassware Washing	N/A	4	06/22/01
NC-QA-0013	Inventory/Warehouse Control	N/A	1	03/19/01
NC-QA-0020	Laboratory Holding Blanks	N/A	1.0	06/15/01
CORP-QA-0014	Laboratory Internal Systems Evaluation	N/A	1	08/01/99
NC-QA-0009	Laboratory and Sample Security	N/A	2	02/13/01
QA-005	Method Detection Limits	N/A	3	05/01/99
NC-QA-0021	Method Detection Limits and Instrument Detection Limits, Evaluation	N/A	3	10/04/00

Table 8.2-2 Standard Operating Procedures

SOP Number	SOP Title	Method	Rev#	Revision Date
	of (Navy)			
NC-QA-0004	Micropipet Calibration	N/A	3	07/27/01
CORP-QA-0010	Nonconformance and Corrective Action	N/A	2	06/15/99
CORP-QA-0004	Independent QA Data Review	N/A	2	10/12/98
QA-014	Instrument Detection Limit, Determination of	N/A	1	12/30/99
QA-003	STL® Quality Control Program Change form 11/22/99	N/A	3	10/11/01
CORP-QA-0001	Quality Testing of Solvents, Acids and Reagents (QRI Program)	N/A	3	12/15/98
NC-QA-0025	Performance Checks on Spectronic Model21 and Model1001 Spectrophotometers	N/A	1	05/21/99
NC-QA-0027	Preparation and Mangement of Standard Operating Procedures	N/A	1	01/10/01
QA-013	Procedures to Address Customer Complaints	N/A	0	06/30/97
NC-QA-0023	Reagent Water	N/A	0	06/04/97
NC-QA-0019	Records Information Management	N/A	3	03/19/01
CORP-QA-0012	Selection and Evaluation of Subcontractor Laboratories	N/A	1	02/05/98
CORP-QA-0015	Selection of Data Points Required for an Initial Calibration Curve	N/A	0	09/01/99
NC-QA-0012	Shipping Department SOP	N/A	2	04/27/99
QA-001	Standard Operating Procedures, Preparation and Management of SOPs Change form 01/01/00	N/A	2	06/01/98
NC-QA-0017	Standards and Reagents	N/A	3	02/14/01
NC-QA-0018	Statistical Evaluation of Data and Development of Control Charts	N/A	6	07/30/01
NC-QA-0016	Supplemental Practices for Navy Project Work	N/A	8	02/14/01
QA-018	Vendor Approval	N/A	0	08/13/99
QA-019	Vendor Review and Oversight	N/A	0	08/13/99

Table 8.2-2 Standard Operating Procedures

SOP Number	SOP Title	Method	Rev#	Revision Date
INFORMATION TECHNOLOGY				
CORP-IT-0002	Information Technology Service Request	N/A	0	10/03/97
CORP-IT-0005	LIMS User Profile Setup and Maintenance	N/A	0	07/22/97
CORP-IT-0001	Software and Hardware Change Management	N/A	0	07/21/97
CORP-IT-014	Software and Hardware Licensing, Security and Backup	N/A	0	07/21/97
CORP-IT-013 (POLICY)	Software Quality Assurance	N/A	0	06/02/97
CORP-IT-0007	Software Testing, Validation and Verification	N/A	0	06/13/97
CORP-IT-0008	Tracking and Management of Client Deliverables	N/A	0	01/17/00
HEALTH AND SAFETY				
NC-HS-0001	Hazardous Waste Management	N/A	2	07/03/98

TABLE 8.2-3
Wet Chemistry Methods

Analytical Parameters	Matrix	Fields of Testing			
		SDWA	CWA	RCRA (SW846)	Other
Alkalinity	Water	SM 2320B	EPA 310.1	---	---
	Waste	---	---	---	---
	Solid	---	310.1 (M)	---	---
Biochemical Oxygen Demand	Water	---	EPA 405.1	---	---
	Waste	---	---	---	---
Bromide	Water	EPA 300.0	EPA 300.0	EPA 9056	---
	Waste	---	EPA 300.0	EPA 9056	---
	Solid	---	EPA 300.0 (M)	EPA 9056	---
Chemical Oxygen Demand	Water	---	EPA 410.4	---	---
	Waste	---	EPA 410.4	---	---
Chloride	Water	EPA 300.0 EPA 325.2	EPA 300.0 EPA 325.2	EPA 9056 EPA 9252	EPA 325.2
	Waste	---	EPA 300.0	EPA 9056	---
	Solid	---	EPA 300.0 (M)	EPA 9056 EPA 9252(M)	---
Chromium, Hexavalent	Water	---	EPA 3500-Cr-D	EPA 7196A	---
	Waste	---	EPA 3500-Cr-D	EPA 7196A	---
	Solid	---	---	---	---
Specific Conductance	Water	SM 2510B	EPA 120.1	EPA 9050A	---
	Waste	---	EPA 120.1	EPA 9050A	---
	Solid	---	---	EPA 9050A	---
Chlorine, Residual	Water	---	EPA 330.5	---	---
	Waste	---	---	---	---
	Solid	---	---	---	---

Analytical Parameters	Matrix	Fields of Testing			
		SDWA	CWA	RCRA (SW846)	Other
Cyanide (Amenable)	Water	---	EPA 335.1	EPA 9012A	---
	Waste	---	---	---	---
	Solid	---	---	EPA 9012A	---
Cyanide (Reactive)	Water	---	---	Section 7.3[1]	---
	Waste	---	---	Section 7.3[1]	---
	Solid	---	---	Section 7.3[1]	---
Cyanide (Total)	Water	EPA 335.4	SM 4500-CN C & E EPA 335.3, EPA 335.2, and EPA 335.4	EPA 9012A	---
	Waste	---	---	EPA 9012A	---
	Solid	---	---	EPA 9012A	---
Cyanide (Weak and Dissociable)	Water	---	SM 4500-CN I	---	ASTM D-2036-81
	Waste	---	---	---	ASTM D-2036-81 (M)
	Solid	---	---	---	ASTM D-2036-81
Fluoride	Water	EPA 300.0	EPA 300.0 EPA 340.2	EPA 9056	---
	Waste	---	EPA 340.2 (M) EPA 300.0 (M)	EPA 9056	---
	Solid	---	EPA 340.2 (M) EPA 300.0 (M)	EPA 9056	---
Ignitability	Waste	---	---	EPA 1010	---
	Solid	---	---	EPA 1010	---
Iron, Ferrous & Ferric	Water	---	SM 3500 FE D	---	---
	Waste	---	----	---	---

Analytical Parameters	Matrix	Fields of Testing			
		SDWA	CWA	RCRA (SW846)	Other
	Solid	---	---	---	---
Hardness	Water	EPA 130.2	EPA 130.2	---	SM 2345B
	Waste	---	---	---	---
Moisture	Solid	---	---	EPA 160.2 (M) ASTM D2216	---
Nitrogen, Ammonia	Water	---	EPA 350.1	---	EPA 350.2
	Waste	---	EPA 350.1	---	EPA 350.2
	Solid	---	EPA 350.1	---	EPA 350.2
Nitrite (NO_2)	Water	EPA 300.0 EPA 353.2	EPA 300.0 EPA 353.2	EPA 9056	---
	Waste	---	EPA 300.0 (M)	EPA 9056	---
	Solid	---	EPA 300.0 (M)	EPA 9056	---
Nitrate (NO_3)	Water	EPA 300.0 EPA 353.2	EPA 300.0 EPA 353.2	EPA 9056	---
	Waste	---	EPA 300.0 (M)	EPA 9056	---
	Solid	---	EPA 300.0 (M) EPA 353.2 (M)	---	---
Nitrate plus Nitrite	Water	EPA 300.0 EPA 353.2	EPA 300.0 EPA 353.2	EPA 9056	---
	Waste	---	EPA 300.0 EPA 353.2	EPA 9056	---
Total Kjeldahl Nitrogen (TKN)	Water	---	EPA 351.3 EPA 351.2	---	---
	Waste	---	EPA 351.3 EPA 351.2	---	---
	Solid	---	EPA 351.3 EPA 351.2	EPA 9071B	EPA 413.1
Oil and Grease	Water	---	EPA 1664A	EPA 9071B	---

Analytical Parameters	Matrix	Fields of Testing			
		SDWA	CWA	RCRA (SW846)	Other
(Hexane Extractable Material)	Waste	---	EPA 1664A	EPA 9071B	---
	Solid	---	---	EPA 9071B	---
Ortho-phosphate $o\text{-}PO_4$	Water	EPA 300.0	EPA 300.0 EPA 365.3 EPA 365.2	EPA 9056	---
	Waste	---	EPA 300.0 (M)	EPA 9056	
	Solid	---	EPA 300.0 (M) EPA 365.3(M) EPA 365.2 (M)	EPA 9056	---
pH	Water	EPA 150.1	EPA 150.1	EPA 9040B	---
	Waste	---	---	EPA 9045C	---
	Solid	---	---	EPA 9045C	---
Phenolics	Water	---	EPA 420.1	---	---
	Waste	---	---	EPA 9065 EPA 9066	---
	Solid	---	---	EPA 9065 EPA 9066	---
Phosphorus (Total)	Water	EPA 365.3	EPA 365.3 EPA 365.2	---	---
	Waste	---	EPA 365.3 EPA 365.2	---	---
	Solid	---	EPA 365.3 EPA 365.2	---	---
Sulfate (SO_4)	Water	EPA 300.0 EPA 375.2	EPA 300.0 EPA 375.4	EPA 9056 EPA 9038	---
	Waste	---	EPA 300.0 (M) EPA 375.4	EPA 9056 EPA 9038	---
	Solid	---	EPA 300.0 (M)	EPA 9056 EPA 9038 (M)	---
Sulfide	Water	---	EPA 376.1	EPA 9030A	---

Analytical Parameters	Matrix	Fields of Testing			
		SDWA	CWA	RCRA (SW846)	Other
Sulfide (Reactive)	Water	---	---	Section 7.3[1]	---
Sulfide (Reactive)	Waste	---	---	Section 7.3[1]	---
	Solid	---	---	Section 7.3[1]	---
Total Organic Carbon (TOC)	Water	---	EPA 415.1	EPA 9060	---
	Waste	---	---	EPA 9060	---
	Solid	---	EPA 415.1 (M)	EPA 9060 (M)	Walkley-Black
Total Organic Halides (TOX)	Water	---	---	EPA 9020B	EPA 450.1
	Waste	---	---	---	---
	Solid	---	---	EPA 9020B	---
Total Petroleum Hydro-carbons	Water	---	EPA 1664A (SGT-HEM)	EPA 9071A EPA 9071B	EPA 413.1 EPA 418.1
	Waste	---	EPA 1664A (SGT-HEM)	EPA 9071A EPA 9071B	EPA 413.1 EPA 418.1
	Solid	---	---	EPA 9071A EPA 9071B	EPA 413.2 EPA 418.1
Total Solids	Water	---	EPA 160.3	---	2540B
	Waste	---	EPA 160.3	---	---
	Solid	---	EPA 160.3 (M)	---	---
Total Dissolved Solids	Water	SM 2540C	EPA 160.1	---	2540C
Total Suspended Solids	Water	EPA 160.2	EPA 160.2	---	2540C
Volatile and Volatile Suspended Solids	Water	---	EPA 160.4	---	---

Analytical Parameters	Matrix	Fields of Testing			
		SDWA	CWA	RCRA (SW846)	Other
Settleable Solids	Water	---	EPA 160.5	---	EPA 2540 F
Turbidity	Water	EPA 180.1	EPA 180.1	---	---

Footnotes

(1) The EPA released a memo to discontinue the use of reactive cyanide and sulfide.

TABLE 8.2-3 (cont.)
Methods for Mercury by Cold Vapor Atomic Absorption

Analytical Parameters	Matrix	Fields of Testing			
		SDWA	CWA	RCRA (SW846)	Other
Mercury (CVAA)	Water	EPA 245.1	EPA 245.1	EPA 7470A	---
	TCLP Leachate	---	---	EPA 7470A	---
	Waste	---	---	EPA 7471A	---
	Solid	---	EPA 254.5	EPA 7471A	---

TABLE 8.2-3 (cont.)
Methods for Mercury by Cold Vapor Atomic Fluororescence

Analytical Parameters	Matrix	Fields of Testing			
		SDWA	CWA	RCRA (SW846)	Other
Mercury, Low Level (CVAFS)	Water	EPA 245.7	EPA 245.7	---	EPA 1631B
	TCLP Leachate	---	---	---	---
	Waste	---	---	---	---
	Solid	---	---	---	---

TABLE 8.2-3 (cont.)
Methods for Metals by Graphite Furnace Atomic Absorption

Analytical Parameters	Matrix	Fields of Testing			
		SDWA	CWA	RCRA (SW846)	Other
Thallium	Water	SM 3113B	EPA 279.2	EPA 7841	---
	Waste	---	---	EPA 7841	---

Analytical Parameters	Matrix	Fields of Testing			
		SDWA	CWA	RCRA (SW846)	Other
	Solid	---	EPA 279.2	EPA 7841	---

TABLE 8.2-3 (cont.)
Methods for Metals by ICP

Analytical Parameters	Matrix	Fields of Testing			
		SDWA	CWA	RCRA (SW846)	Other
Aluminum	Water	EPA 200.7	EPA 200.7	EPA 6010B	---
	Waste	---	---	EPA 6010B	---
	Solid	---	EPA 200.7	EPA 6010B	---
Antimony [1]	Water	---	EPA 200.7	EPA 6010B	---
	Waste	---	---	EPA 6010B	---
	Solid	---	EPA 200.7	EPA 6010B	---
Arsenic [1]	Water	EPA 200.7	EPA 200.7	EPA 6010B	---
	Waste	---	---	EPA 6010B	---
	Solid	---	EPA 200.7	EPA 6010B	---
Barium	Water	EPA 200.7	EPA 200.7	EPA 6010B	---
	Waste	---	---	EPA 6010B	---
	Solid	---	EPA 200.7	EPA 6010B	---
Beryllium	Water	EPA 200.7	EPA 200.7	EPA 6010B	---
	Waste	---	---	EPA 6010B	---
	Solid	---	EPA 200.7	EPA 6010B	---
Boron	Water	EPA 200.7	EPA 200.7	EPA 6010B	---
	Waste	---	---	EPA 6010B	---
	Solid	---	EPA 200.7	EPA 6010B	---
Calcium	Water	EPA 200.7	EPA 200.7	EPA 6010B	---
	Waste	---	---	EPA 6010B	---
	Solid	---	EPA 200.7	EPA 6010B	---
Cadmium [1]	Water	EPA 200.7	EPA 200.7	EPA 6010B	---
	Waste	---	---	EPA 6010B	---
	Solid	---	EPA 200.7	EPA 6010B	---
Cobalt	Water	EPA 200.7	EPA 200.7	EPA 6010B	---

Analytical Parameters	Matrix	Fields of Testing			
		SDWA	CWA	RCRA (SW846)	Other
	Waste	---	---	EPA 6010B	---
	Solid	---	EPA 200.7	EPA 6010B	---
Chromium	Water	EPA 200.7	EPA 200.7	EPA 6010B	---
	Waste	---	---	EPA 6010B	---
	Solid	---	EPA 200.7	EPA 6010B	---
Copper	Water	EPA 200.7	EPA 200.7	EPA 6010B	---
	Waste	---	---	EPA 6010B	---
	Solid	---	EPA 200.7	EPA 6010B	---
Iron	Water	EPA 200.7	EPA 200.7	EPA 6010B	
	Waste	---	---	EPA 6010B	---
	Solid	---	EPA 200.7	EPA 6010B	---
Lead [1]	Water	---	EPA 200.7	EPA 6010B	---
	Waste	---	---	EPA 6010B	---
	Solid	---	EPA 200.7	EPA 6010B	---
Lithium	Water	EPA 200.7	EPA 200.7	EPA 6010B	---
	Waste	---	---	EPA 6010B	---
	Solid	---	EPA 200.7	EPA 6010B	---
Magnesium	Water	EPA 200.7	EPA 200.7	EPA 6010B	---
	Waste	---	---	EPA 6010B	---
	Solid	---	EPA 200.7	EPA 6010B	---
Manganese	Water	EPA 200.7	EPA 200.7	EPA 6010B	---
	Waste	---	---	EPA 6010B	---
	Solid	---	EPA 200.7	EPA 6010B	---
Molybdenum	Water	EPA 200.7	EPA 200.7	EPA 6010B	---
	Waste	---	---	EPA 6010B	---
	Solid	---	EPA 200.7	EPA 6010B	---
Nickel	Water	EPA 200.7	EPA 200.7	EPA 6010B	---
	Waste	---	---	EPA 6010B	---
	Solid	---	EPA 200.7	EPA 6010B	---
Potassium	Water	EPA 200.7	EPA 200.7	EPA 6010B	---

Analytical Parameters	Matrix	Fields of Testing			
		SDWA	CWA	RCRA (SW846)	Other
	Waste	---	---	EPA 6010B	---
	Solid	---	---	EPA 6010B	---
Selenium [1]	Water	---	EPA 200.7	EPA 6010B	---
	Waste	---	---	EPA 6010B	---
	Solid	---	EPA 200.7	EPA 6010B	---
Silver	Water	EPA 200.7	EPA 200.7	EPA 6010B	---
	Waste	---	---	EPA 6010B	---
	Solid	---	EPA 200.7	EPA 6010B	---
Sodium	Water	EPA 200.7	EPA 200.7	EPA 6010B	---
	Waste	---	---	EPA 6010B	---
	Solid	---	---	EPA 6010B	---
Tin	Water	EPA 200.7	EPA 200.7	EPA 6010B	---
	Waste	---	---	EPA 6010B	---
	Solid	---	EPA 200.7	EPA 6010B	---
Thallium [1]	Water	---	EPA 200.7	EPA 6010B	---
	Waste	---	---	EPA 6010B	---
	Solid	---	EPA 200.7	EPA 6010B	---
Titanium	Water	EPA 200.7	EPA 200.7	EPA 6010B	---
	Waste	---	---	EPA 6010B	---
	Solid	---	---	EPA 6010B	---
Vanadium	Water	EPA 200.7	EPA 200.7	EPA 6010B	---
	Waste	---	---	EPA 6010B	---
	Solid	---	---	EPA 6010B	---
Zinc	Water	EPA 200.7	EPA 200.7	EPA 6010B	---
	Waste	---	---	EPA 6010B	---
	Solid	---	EPA 200.7---	EPA 6010B	---

Footnotes

[1] These metals are usually analyzed by Trace ICP.

TABLE 8.2-3 (cont.)
Metals Sample Preparation Methods

Analytical Parameters	Matrix	Fields of Testing			
		SDWA	CWA	RCRA (SW846)	Other

Analytical Parameters	Matrix	Fields of Testing			
		SDWA	CWA	RCRA (SW846)	Other
Toxicity Characteristic Leaching Procedure (TCLP)	Water	---	---	EPA 1311	---
	Waste	---	---	EPA 1311	---
	Solid	---	---	EPA 1311	---
ICP Metals	Water	EPA 200.7	EPA 200.7	EPA 3005A EPA 3010A	---
	TCLP Leachate	---	---	EPA 3010A	---
	Waste	---	---	EPA 3050B	---
	Solid	---	---	EPA 3050B	---
ICPMS Metals	Water	EPA 200.8	EPA 200.8	EPA 3010A	---
	TCLP	---	---	EPA 3010A	---
	Waste	---	---	EPA 3050B	---
	Solid	---	---	EPA 3050B	---
CVAA Mercury	Water	EPA 245.1	EPA 245.1	EPA 7470A	---
	TCLP Leachate	---	---	EPA 7470A	---
	Waste	---	---	EPA 7471A	---
	Solid	---	---	EPA 7471A	
CVAFS Mercury Low Level	Water	EPA 245.7	EPA 245.7	---	EPA 1631B
	TCLP	---	---	---	---
	Waste	---	---	---	---
	Solid	---	---	---	---
GFAA Metals	Water	Sample preparation procedures are detailed in the determinative EPA for each element.	Sample preparation procedures are detailed in the determinative EPA for each element.	EPA 3020A	---
	TCLP Leachate	---	---	EPA 3020A	---
	Waste	---	---	EPA 3020A	---
	Solid	---	---	EPA 3050B	---

TABLE 8.2-3 (cont.)

Organic Sample Preparation Methods

Analytical Parameters	Matrix	Fields of Testing			
		SDWA	CWA	RCRA (SW846)	Other
Volatiles by GC/MS	Water	---	EPA 624	EPA 5030B	---
	Waste	---	---	EPA 5030B EPA 5035	---
	Solid	---	---	EPA 5035	---
Halogenated Volatiles by GC	Water	---	EPA 601	EPA 5030B	---
	Waste	---	---	EPA 5030B EPA 5035	---
	Solid	---	---	EPA 5035	---
Aromatic Volatiles by GC	Water	---	EPA 602	EPA 5030B	---
	Waste	---	---	EPA 5030B EPA 5035	---
	Solid	---	---	EPA 5035	---
Semivolatiles by GC/MS	Water	---	EPA 625	EPA 3510C EPA 3520C	---
	TCLP Leachate	---	---	EPA 3510C EPA 3520C	---
	Waste	---	---	EPA 3550B EPA 3580A	---
	Solid	---	---	EPA 3550B EPA 3580A	---
Pesticides/ PCBs by GC	Water	---	EPA 608	EPA 3510C EPA 3520C	---
	TCLP Leachate	---	---	EPA 3510C EPA 3520C	---
	Waste	---	---	EPA 3550B EPA 3580A	---
	Solid	---	---	EPA 3550B	---
Pesticides (Organophos-phorus) by GC	Water	---	---	EPA 3510C EPA 3520C	---
	TCLP Leachate	---	---	EPA 3510C EPA 3520C	---
	Waste	---	---	EPA 3540C	---
	Solid	---	---	EPA 3540B	---

Analytical Parameters	Matrix	Fields of Testing			
		SDWA	CWA	RCRA (SW846)	Other
PAHs by GC	Water	---	---	EPA 3510C EPA 3520C	---
	Solid	---	---	EPA 3550B	---
PAHs by HPLC	Water	---	---	EPA 3510C EPA 3520C	---
	Solid	---	---	EPA 3550B	---
Herbicides by GC	Water	---	EPA 615	EPA 8151A	---
	Waste	---	---	EPA 8151A	---
	Solid	---	---	EPA 8151A	---
Total Petroleum Hydrocarbons (gasoline range) by GC	Water	---	---	EPA 5030B	---
	Waste	---	---	EPA 5030B EPA 5035	---
	Solid	---	---	EPA 5035 EPA 5035	---
Total Petroleum Hydrocarbons (diesel range) by GC	Water	---	---	EPA 3510C EPA 3520C	---
	TCLP Leachate	---	---	EPA 3510C EPA 3520C	---
	Waste	---	---	EPA 3550B EPA 3580A	---
	Solid	---	---	EPA 3550B	---

TABLE 8.2-3 (cont.)
Organic Methods

Analytical Parameters	Matrix	Fields of Testing			
		SDWA	CWA	RCRA (SW846)	Other
Volatiles by GC/MS	Water	---	EPA 624	EPA 8260B	---
	Waste	---	---	EPA 8260B	---
	Solid	---	---	EPA 8260B	---

Analytical Parameters	Matrix	Fields of Testing			
		SDWA	CWA	RCRA (SW846)	Other
Halogenated Volatiles by GC	Water	---	EPA 601	EPA 8021B	---
	Waste	---	---	EPA 8021B	---
	Solid	---	---	EPA 8021B	---
Aromatic Volatiles by GC	Water	---	EPA 602	EPA 8021B	---
	Waste	---	---	EPA 8021B	---
	Solid	---	---	EPA 8021B	---
Semivolatiles by GC/MS	Water	---	EPA 625	EPA 8270C	
	Waste	---	---	EPA 8270C	---
	Solid	---	---	EPA 8270C	---

Analytical Parameters	Matrix	Fields of Testing			
		SDWA	CWA	RCRA (SW846)	Other
Pesticides/ PCBs by GC	Water	---	EPA 608	Pesticides 8081A PCBs 8082	---
	TCLP Leachate	---	---	Pesticides 8081A PCBs 8082	---
	Waste	---	---	Pesticides 8081A PCBs 8082	---
	Solid	---	---	Pesticides 8081A PCBs 8082	---
Pesticides (Organophos-phorus) by GC	Water	---	---	EPA 8141A	---
	Waste	---	---	EPA 8141A	---
	Solid	---	---	EPA 8141A	---
PAHs by GC	Water	---	---	EPA 8310	---
	Waste	---	---	EPA 8310	---
	Solid	---	---	EPA 8310	---
Phenoxyacid Herbicides by GC	Water	---	EPA 615	EPA 8151A	---
	TCLP Leachate	---	---	EPA 8151A	---

Analytical Parameters	Matrix	Fields of Testing			
		SDWA	CWA	RCRA (SW846)	Other
	Waste	---	---	EPA 8151A	---
	Solid	---	---	EPA 8151A	---
Gasoline Range Organics by GC	Water	---	---	EPA 8015B (M)	API Method CA LUFT WV GRO WI GRO
	Waste	---	---	EPA 8015B (M)	---
	Solid	---	---	EPA 8015B (M)	API Method CA LUFT WV GRO WI GRO
Total Petroleum Hydrocarbons (diesel range) by GC/FID	Water	---	---	EPA 8015B (M)	EPA API WI DRO WV DRO
	Waste	---	---	EPA 8015B (M)	---
Total Petroleum Hydrocarbons (diesel range) by GC/FID (con't.)	Solid	---	---	EPA 8015B (M)	API Method CA LUFT WI DRO WV DRO
Hydrocarbons by GC/FID	Water	---	---	EPA 8015B	---
	Solid	---	---	EPA 8015B	---

TABLE 8.2-3 (cont.)
Methods for Metals by ICP/MS

Analytical Parameters	Matrix	Fields of Testing			
		SDWA	CWA	RCRA (SW846)	Other
Aluminum	Water	EPA 200.8	EPA 200.8	EPA 6020	---
	Waste	---	---	EPA 6020	---
	Solid	---	---	EPA 6020	---
Antimony [1]	Water	EPA 200.8	EPA 200.8	EPA 6020	---
	Waste	---	---	EPA 6020	---
	Solid	---	---	EPA 6020	---
Arsenic [1]	Water	EPA 200.8	EPA 200.8	EPA 6020	---
	Waste	---	---	EPA 6020	---
	Solid	---	---	EPA 6020	---
Barium	Water	EPA 200.8	EPA 200.8	EPA 6020	---
	Waste	---	---	EPA 6020	---
	Solid	---	---	EPA 6020	---
Beryllium	Water	EPA 200.8	EPA 200.8	EPA 6020	---
	Waste	---	---	EPA 6020	---
	Solid	---	---	EPA 6020	---
Cadmium [1]	Water	EPA 200.8	EPA 200.8	EPA 6020	---
	Waste	---	---	EPA 6020	---
	Solid	---	---	EPA 6020	---
Cobalt	Water	EPA 200.8	EPA 200.8	EPA 6020	---
	Waste	---	---	EPA 6020	---
	Solid	---	---	EPA 6020	---
Chromium	Water	EPA 200.8	EPA 200.8	EPA 6020	---
	Waste	---	---	EPA 6020	---
	Solid	---	---	EPA 6020	---
Copper	Water	EPA 200.8	EPA 200.8	EPA 6020	---
	Waste	---	---	EPA 6020	---
	Solid	---	---	EPA 6020	---
Lead [1]	Water	EPA 200.8	EPA 200.8	EPA 6020	---

Analytical Parameters	Matrix	Fields of Testing			
		SDWA	CWA	RCRA (SW846)	Other
	Waste	---	---	EPA 6020	---
	Solid	---	---	EPA 6020	---
Manganese	Water	EPA 200.8	EPA 200.8	EPA 6020	---
	Waste	---	---	EPA 6020	---
	Solid	---	---	EPA 6020	---
Molybdenum	Water	EPA 200.8	EPA 200.8	EPA 6020	---
	Waste	---	---	EPA 6020	---
	Solid	---	---	EPA 6020	---
Nickel	Water	EPA 200.8	EPA 200.8	EPA 6020	---
	Waste	---	---	EPA 6020	---
	Solid	---	---	EPA 6020	---
Selenium [1]	Water	EPA 200.8	EPA 200.8	EPA 6020	---
	Waste	---	---	EPA 6020	---
	Solid	---	---	EPA 6020	---
Silver	Water	EPA 200.8	EPA 200.8	EPA 6020	---
	Waste	---	---	EPA 6020	---
	Solid	---	---	EPA 6020	---
Tin	Water	EPA 200.8	EPA 200.8	EPA 6020	---
	Waste	---	---	EPA 6020	---
	Solid	---	---	EPA 6020	---
Thallium [1]	Water	EPA 200.8	EPA 200.8	EPA 6020	---
	Waste	---	---	EPA 6020	---
	Solid	---	---	EPA 6020	---
Vandium	Water	EPA 200.8	EPA 200.8	EPA 6020	
	Solid	---	---	EPA 6020	
	Waste	---	---	EPA 6020	
Zinc	Water	EPA 200.8	EPA 200.8	EPA 6020	---
	Waste	---	---	EPA 6020	---
	Solid	---	---	EPA 6020	---

Footnotes

TABLE 8.2-4-1
General Chemistry
Reporting Limits (RL), Method Detection Limits (MDL),
and Control Limits (LCS, MS/MSD, and RPD)(1)

310.1							LCS				MS		
#	Compound	RL	Units	MDL	Units	T	LCL	UCL	RPD	T	LCL	UCL	RPD
3041	Total Alkalinity	5	mg/L	4.5	mg/L	C	80	120	20	C	80	120	20

300.0/9056							LCS				MS		
#	Compound	RL	Units	MDL	Units	T	LCL	UCL	RPD	T	LCL	UCL	RPD
315	Bromide	0.5	mg/L	0.086	mg/L	C	90	110	20	C	90	110	20
512	Chloride	1	mg/L	0.13	mg/L	C	90	110	20	C	59	132	20
2680	Nitrate	0.1	mg/L	0.015	mg/L	C	90	110	20	C	74	129	21
1422	Fluoride	1	mg/L	0.017	mg/L	C	89	110	20	C	82	111	20
2363	Sulfate	1	mg/L	0.15	mg/L	C	90	110	20	C	56	135	83
3624	ortho-Phosphate	0.5	mg/L	0.017	mg/L	C	83	118	24	C	22	177	48
2682	Nitrite	0.1	mg/L	0.022	mg/L	C	89	110	20	C	52	152	27

405.1							LCS				MS		
#	Compound	RL	Units	MDL	Units	T	LCL	UCL	RPD	T	LCL	UCL	RPD
3137	Biochemical Oxygen Demand	2	mg/L	2	mg/L	C	64	124	28	C	70	130	20

410.4							LCS				MS		
#	Compound	RL	Units	MDL	Units	T	LCL	UCL	RPD	T	LCL	UCL	RPD
472	Chemical Oxygen Demand (COD)	10	mg/L	7	mg/L	C	86	117	30	C	76	112	20

9252							LCS				MS		
#	Compound	RL	Units	MDL	Units	T	LCL	UCL	RPD	T	LCL	UCL	RPD
512	Chloride	3	mg/L	1.5	mg/L	C	90	110	20	C	90	110	20

325.2							LCS				MS		
#	Compound	RL	Units	MDL	Units	T	LCL	UCL	RPD	T	LCL	UCL	RPD
3089	Chloride - Automated	1	mg/L	0.38	mg/L	C	90	115	21	C	42	152	28

325.2							LCS				MS		
#	Compound	RL	Units	MDL	Units	T	LCL	UCL	RPD	T	LCL	UCL	RPD
512	Chloride	3	mg/L	1.5	mg/L	C	90	110	20	C	90	110	20

330.5							LCS				MS		
#	Compound	RL	Units	MDL	Units	T	LCL	UCL	RPD	T	LCL	UCL	RPD
517	Total Residual Chlorine	0.2	mg/L				75	125	20		75	125	20

TABLE 8.2-4-1
General Chemistry
Reporting Limits (RL), Method Detection Limits (MDL),
and Control Limits (LCS, MS/MSD, and RPD)[1]

	9012A					LCS				MS			
#	Compound	RL	Units	MDL	Units	T	LCL	UCL	RPD	T	LCL	UCL	RPD
667	Total Cyanide	0.01	mg/L	0.003	mg/L	C	61	115	32	C	25	134	99
				3									

	335.2					LCS				MS			
#	Compound	RL	Units	MDL	Units	T	LCL	UCL	RPD	T	LCL	UCL	RPD
667	Total Cyanide	0.01	mg/L	0.003	mg/L	C	61	115	32	C	25	134	99
				3									

	340.2					LCS				MS			
#	Compound	RL	Units	MDL	Units	T	LCL	UCL	RPD	T	LCL	UCL	RPD
1422	Fluoride	0.1	mg/L			C	81	110	20	C	72	120	20

	130.2					LCS				MS			
#	Compound	RL	Units	MDL	Units	T	LCL	UCL	RPD	T	LCL	UCL	RPD
1469	Hardness, as CaCO3	5	mg/L	1.3	mg/L	C	90	110	20	C	18	153	20

	1664A					LCS				MS			
#	Compound	RL	Units	MDL	Units	T	LCL	UCL	RPD	T	LCL	UCL	RPD
3506	n-Hexane Extractable Material	5	mg/L	1.3	mg/L		79	114	20		79	114	18
3507	n-Hexane Extractable Material, SGT	10	mg/L	3.3	mg/L		66	114	20		66	114	24

	7196A					LCS				MS			
#	Compound	RL	Units	MDL	Units	T	LCL	UCL	RPD	T	LCL	UCL	RPD
630	Hexavalent Chromium	0.02	mg/L	0.002	mg/L	C	89	119	20	C	57	138	27

	3500 Cr D					LCS				MS			
#	Compound	RL	Units	MDL	Units	T	LCL	UCL	RPD	T	LCL	UCL	RPD
630	Hexavalent Chromium	0.02	mg/L	0.002	mg/L	C	89	119	20	C	57	138	27

	353.2					LCS				MS			
#	Compound	RL	Units	MDL	Units	T	LCL	UCL	RPD	T	LCL	UCL	RPD
2682	Nitrite	0.1	mg/L	0.03	mg/L	C	85	115	20	C	85	115	20
2680	Nitrate	0.1	mg/L	0.03	mg/L	C	85	115	20	C	85	115	20
2681	Nitrate/Nitrite	0.1	mg/L	0.03	mg/L	C	85	115	20	C	85	115	20

TABLE 8.2-4-1
General Chemistry
Reporting Limits (RL), Method Detection Limits (MDL),
and Control Limits (LCS, MS/MSD, and RPD)[1]

	350.2					LCS				MS		
#	Compound	RL	Units	MDL	Units	T	LCL	UCL	RPD	T	LCL	UCL	RPD
1986	Nitrogen, as Ammonia	1	mg/L	0.48	mg/L	C	80	120	20	C	80	120	20

	9065					LCS				MS		
#	Compound	RL	Units	MDL	Units	T	LCL	UCL	RPD	T	LCL	UCL	RPD
2163	Total Phenols	0.02	mg/L	0.011	mg/L	C	57	142	64	C	10	153	99

	9045B, C					LCS				MS		
#	Compound	RL	Units	MDL	Units	T	LCL	UCL	RPD	T	LCL	UCL	RPD
2204	pH (solid)		No Units				90	110	20		90	110	20

	4500					LCS				MS		
#	Compound	RL	Units	MDL	Units	T	LCL	UCL	RPD	T	LCL	UCL	RPD
2196	Phosphorus as Orthophosphate	0.1	mg/L	0.031	mg/L	C	89	115	20	C	90	110	20

	365.2					LCS				MS		
#	Compound	RL	Units	MDL	Units	T	LCL	UCL	RPD	T	LCL	UCL	RPD
3624	ortho-Phosphate	0.1	mg/L	0.031	mg/L	C	89	115	20	C	90	110	20
2196	Phosphorus as Orthophosphate	0.1	mg/L	0.031	mg/L	C	89	115	20	C	90	110	20
3651	Dissolved ortho-Phosphate	0.1	mg/L	0.031	mg/L	C	89	115	20	C	90	110	20

	120.1					LCS				MS		
#	Compound	RL	Units	MDL	Units	T	LCL	UCL	RPD	T	LCL	UCL	RPD
2667	Specific Conductance	1	umhos/cm				75	125	20		75	125	20

	9050A					LCS				MS		
#	Compound	RL	Units	MDL	Units	T	LCL	UCL	RPD	T	LCL	UCL	RPD
2667	Specific Conductance	1	umhos/cm				75	125	20		75	125	20

	160.1					LCS				MS		
#	Compound	RL	Units	MDL	Units	T	LCL	UCL	RPD	T	LCL	UCL	RPD
3639	Dissolved Filterable Solids (Residue)	10	mg/L	8.3	mg/L	C	80	120	20		80	120	20
2687	Total Dissolved Solids	10	mg/L	8.3	mg/L		72	131	20		90	110	20

TABLE 8.2-4-1
General Chemistry
Reporting Limits (RL), Method Detection Limits (MDL),
and Control Limits (LCS, MS/MSD, and RPD)[1]

	375.4					LCS				MS			
#	Compound	RL	Units	MDL	Units	T	LCL	UCL	RPD	T	LCL	UCL	RPD
2363	Sulfate	5	mg/L	1.4	mg/L	C	90	110	20	C	90	110	20

	9038					LCS				MS			
#	Compound	RL	Units	MDL	Units	T	LCL	UCL	RPD	T	LCL	UCL	RPD
2363	Sulfate	5	mg/L	1.4	mg/L	C	90	110	20	C	90	110	20

	351.3					LCS				MS			
#	Compound	RL	Units	MDL	Units	T	LCL	UCL	RPD	T	LCL	UCL	RPD
1992	Total Kjeldahl Nitrogen	1	mg/L	0.621	mg/L	C	69	130	37	C	80	120	20

	376.1					LCS				MS			
#	Compound	RL	Units	MDL	Units	T	LCL	UCL	RPD	T	LCL	UCL	RPD
2366	Total Sulfide	1	mg/L	0.92	mg/L	C	72	110	20	C	80	120	20

	9030A					LCS				MS			
#	Compound	RL	Units	MDL	Units	T	LCL	UCL	RPD	T	LCL	UCL	RPD
2366	Total Sulfide	1	mg/L	0.92	mg/L	C	72	110	20	C	80	120	20

	9030A					LCS				MS			
#	Compound	RL	Units	MDL	Units	T	LCL	UCL	RPD	T	LCL	UCL	RPD
3658	Acid-insoluble Sulfide	1	mg/L	0.92	mg/L	C	75	110	20	C	80	120	20

	415.1					LCS				MS			
#	Compound	RL	Units	MDL	Units	T	LCL	UCL	RPD	T	LCL	UCL	RPD
470	Total Organic Carbon	1	mg/L	0.18	mg/L	C	88	110	20	C	80	110	20

	9060					LCS				MS			
#	Compound	RL	Units	MDL	Units	T	LCL	UCL	RPD	T	LCL	UCL	RPD
470	Total Organic Carbon	1	mg/L	0.18	mg/L	C	88	110	20	C	80	110	20

	9020B					LCS				MS			
#	Compound	RL	Units	MDL	Units	T	LCL	UCL	RPD	T	LCL	UCL	RPD
1468	Total Organic Halogens	30	ug/L	13	ug/L	C	67	124	53	C	10	178	99

	180.1					LCS				MS			
#	Compound	RL	Units	MDL	Units	T	LCL	UCL	RPD	T	LCL	UCL	RPD
2604	Turbidity	0.5	NTU				90	110	20		90	110	20

TABLE 8.2-4-2
Metals – ICP,CVAA, GFAA (6000, 7000, 200 Series)
Reporting Limits (RL), Method Detection Limits (MDL),
and Control Limits (LCS, MS/MSD, and RPD)[1]

Trace	Water					LCS			MS		
#	Compound	RL	Units	MDL	Units	LCL	UCL	RPD	LCL	UCL	RPD
128	Antimony	0.01	mg/L	2.2	ug/L	80	120	20	75	125	20
140	Arsenic	0.01	mg/L	4.1	ug/L	80	120	20	75	125	20
411	Cadmium	0.002	mg/L	0.28	ug/L	80	120	20	75	125	20
2952	Chromium	0.005	mg/L	1.4	ug/L	80	120	20	75	125	20
637	Cobalt	0.007	mg/L	1.3	ug/L	80	120	20	75	125	20
1605	Lead	0.003	mg/L	2.5	ug/L	80	120	20	75	125	20
1906	Molybdenum	0.01	mg/L	2.6	ug/L	80	120	20	75	125	20
2281	Selenium	0.005	mg/L	4.5	ug/L	80	120	20	75	125	20
2285	Silver	0.005	mg/L	1.5	ug/L	80	120	20	75	125	20
2477	Thallium	0.01	mg/L	5	ug/L	80	120	20	75	125	20
2607	Vanadium	0.007	mg/L	0.82	ug/L	80	120	20	75	125	20
Trace	Solid					LCS			MS		
#	Compound	RL	Units	MDL	Units	LCL	UCL	RPD	LCL	UCL	RPD
128	Antimony	1	mg/kg	0.49	mg/kg	80	120	20	75	125	20
140	Arsenic	1	mg/kg	0.3	mg/kg	80	120	20	75	125	20
411	Cadmium	0.2	mg/kg	0.043	mg/kg	80	120	20	75	125	20
2952	Chromium	0.5	mg/kg	0.38	mg/kg	80	120	20	75	125	20
637	Cobalt	5	mg/kg	0.15	mg/kg	80	120	20	75	125	20
1605	Lead	0.3	mg/kg	0.24	mg/kg	80	120	20	75	125	20
1906	Molybdenum	1	mg/kg	0.18	mg/kg	80	120	20	75	125	20
2281	Selenium	0.5	mg/kg	0.31	mg/kg	80	120	20	75	125	20
2285	Silver	0.5	mg/kg	0.15	mg/kg	80	120	20	75	125	20
2477	Thallium	1	mg/kg	0.5	mg/kg	80	120	20	75	125	20
2607	Vanadium	5	mg/kg	0.13	mg/kg	80	120	20	75	125	20

TABLE 8.2-4-2
Metals – ICP,CVAA, GFAA (6000, 7000, 200 Series)
Reporting Limits (RL), Method Detection Limits (MDL),
and Control Limits (LCS, MS/MSD, and RPD)[1]

ICP	Water					LCS			MS		
#	Compound	RL	Units	MDL	Units	LCL	UCL	RPD	LCL	UCL	RPD
88	Aluminum	0.2	mg/L	28	ug/L	80	120	20	75	125	20
128	Antimony	0.06	mg/L	2.2	ug/L	80	120	20	75	125	20
140	Arsenic	0.3	mg/L	4.1	ug/L	80	120	20	75	125	20
194	Barium	0.2	mg/L	3	ug/L	80	120	20	75	125	20
222	Beryllium	0.005	mg/L	0.54	ug/L	80	120	20	75	125	20
313	Boron	0.2	mg/L	21	ug/L	80	120	20	75	125	20
411	Cadmium	0.005	mg/L	0.28	ug/L	80	120	20	75	125	20
413	Calcium	5	mg/L	250	ug/L	80	120	20	75	125	20
2952	Chromium	0.01	mg/L	1.4	ug/L	80	120	20	75	125	20
637	Cobalt	0.05	mg/L	1.3	ug/L	80	120	20	75	125	20
643	Copper	0.025	mg/L	4.2	ug/L	80	120	20	75	125	20
1539	Iron	0.1	mg/L	88	ug/L	77	127	20	75	125	20
1605	Lead	0.1	mg/L	2.5	ug/L	80	120	20	75	125	20
1618	Magnesium	5	mg/L	30	ug/L	80	120	20	75	125	20
1659	Manganese	0.015	mg/L	0.9	ug/L	80	120	20	75	125	20
1906	Molybdenum	0.04	mg/L	2.6	ug/L	80	120	20	75	125	20
1956	Nickel	0.04	mg/L	2.2	ug/L	80	120	20	75	125	20
2214	Potassium	5	mg/L	41	ug/L	80	120	20	75	125	20
2281	Selenium	0.25	mg/L	4.5	ug/L	80	120	20	75	125	20
2285	Silver	0.01	mg/L	1.5	ug/L	80	120	20	75	125	20
2315	Sodium	5	mg/L	630	ug/L	80	120	20	75	125	20
2477	Thallium	2	mg/L	5	ug/L	80	120	20	75	125	20
2479	Tin	0.1	mg/L	8	ug/L	80	120	20	75	125	20
2482	Titanium	0.05	mg/L	6.3	ug/L	80	120	20	75	125	20
2607	Vanadium	0.05	mg/L	0.82	ug/L	80	120	20	75	125	20
2649	Zinc	0.02	mg/L	12	ug/L	80	120	20	75	125	20

TABLE 8.2-4-2
Metals – ICP,CVAA, GFAA (6000, 7000, 200 Series)
Reporting Limits (RL), Method Detection Limits (MDL),
and Control Limits (LCS, MS/MSD, and RPD)[1]

ICP	Solid					LCS			MS		
#	Compound	RL	Units	MDL	Units	LCL	UCL	RPD	LCL	UCL	RPD
88	Aluminum	20	mg/kg	1.1	mg/kg	80	120	20	75	125	20
128	Antimony	6	mg/kg	0.49	mg/kg	80	120	20	75	125	20
140	Arsenic	30	mg/kg	0.3	mg/kg	80	120	20	75	125	20
194	Barium	20	mg/kg	0.13	mg/kg	80	120	20	75	125	20
222	Beryllium	0.5	mg/kg	0.046	mg/kg	80	120	20	75	125	20
313	Boron	20	mg/kg	1.2	mg/kg	80	120	20	75	125	20
411	Cadmium	0.5	mg/kg	0.043	mg/kg	80	120	20	75	125	20
413	Calcium	500	mg/kg	37	mg/kg	80	120	20	75	125	20
2952	Chromium	1	mg/kg	0.38	mg/kg	80	120	20	75	125	20
637	Cobalt	5	mg/kg	0.15	mg/kg	80	120	20	75	125	20
643	Copper	2.5	mg/kg	0.27	mg/kg	80	120	20	75	125	20
1539	Iron	10	mg/kg	6.6	mg/kg	73	137	20	75	125	20
1605	Lead	10	mg/kg	0.24	mg/kg	80	120	20	75	125	20
1618	Magnesium	500	mg/kg	12	mg/kg	80	120	20	75	125	20
1659	Manganese	1.5	mg/kg	0.15	mg/kg	80	120	20	75	125	20
1906	Molybdenum	4	mg/kg	0.18	mg/kg	80	120	20	75	125	20
1956	Nickel	4	mg/kg	0.27	mg/kg	80	120	20	75	125	20
2214	Potassium	500	mg/kg	5.1	mg/kg	80	120	20	75	125	20
2281	Selenium	25	mg/kg	0.31	mg/kg	80	120	20	75	125	20
2285	Silver	1	mg/kg	0.15	mg/kg	80	120	20	75	125	20
2315	Sodium	500	mg/kg	50	mg/kg	80	120	20	75	125	20
2477	Thallium	200	mg/kg	0.5	mg/kg	80	120	20	75	125	20
2479	Tin	10	mg/kg	0.66	mg/kg	80	120	20	75	125	20
2482	Titanium	5	mg/kg	0.53	mg/kg	80	120	20	75	125	20
2607	Vanadium	5	mg/kg	0.13	mg/kg	80	120	20	75	125	20
2649	Zinc	2	mg/kg	1.2	mg/kg	80	120	20	75	125	20
	Water					LCS			MS		
#	Compound	RL	Units	MDL	Units	LCL	UCL	RPD	LCL	UCL	RPD
1701	Mercury	0.0002	mg/L	0.13	ug/L	70	118	20	53	135	20
	Solid					LCS			MS		
#	Compound	RL	Units	MDL	Units	LCL	UCL	RPD	LCL	UCL	RPD
1701	Mercury	0.1	mg/kg	0.0084	mg/kg	52	127	20	10	209	20

TABLE 8.2-4-2
Metals – ICP,CVAA, GFAA (6000, 7000, 200 Series)
Reporting Limits (RL), Method Detection Limits (MDL),
and Control Limits (LCS, MS/MSD, and RPD)[1]

	Water					LCS			MS		
#	Compound	RL	Units	MDL	Units	LCL	UCL	RPD	LCL	UCL	RPD
2477	Thallium	0.01	mg/L	1.7	ug/L	80	120	20	75	125	20

	Solid					LCS			MS		
#	Compound	RL	Units	MDL	Units	LCL	UCL	RPD	LCL	UCL	RPD
2477	Thallium	1	mg/kg	0.12	ug/L	80	120	20	75	125	20

Table 8.2-4-2
Metals – ICPMS, 6020
Reporting Limits (RL), Method Detection Limits (MDL),
and Control Limits (LCS, MS/MSD, and RPD)

	Water					LCS			MS		
#	Compound	RL	Units	MDL	Units	LCL	UCL	RPD	LCL	UCL	RPD
88	Aluminum	50	ug/L	11	ug/L	80	120	20	70	130	20
128	Antimony	2	ug/L	0.066	ug/L	80	120	20	70	130	20
140	Arsenic	5	ug/L	0.35	ug/L	80	120	20	70	130	20
194	Barium	1	ug/L	0.16	ug/L	80	120	20	70	130	20
222	Beryllium	1	ug/L	0.21	ug/L	80	120	20	70	130	20
411	Cadmium	1	ug/L	0.05	ug/L	80	120	20	70	130	20
2952	Chromium	2	ug/L	0.18	ug/L	80	120	20	70	130	20
637	Cobalt	1	ug/L	0.025	ug/L	80	120	20	70	130	20
643	Copper	2	ug/L	0.6	ug/L	80	120	20	70	130	20
1539	Iron	20	ug/L	9	ug/L	80	120	20	80	120	20
1605	Lead	1	ug/L	0.15	ug/L	80	120	20	70	130	20
1659	Manganese	1	ug/L	0.23	ug/L	80	120	20	70	130	20
1906	Molybdenum*	2	ug/L	0.046	ug/L	80	120	20	70	130	20
1956	Nickel	2	ug/L	0.13	ug/L	80	120	20	70	130	20
2281	Selenium*	5	ug/L	1.2	ug/L	80	120	20	70	130	20
2285	Silver	1	ug/L	0.054	ug/L	80	120	20	70	130	20
2477	Thallium	1	ug/L	0.018	ug/L	80	120	20	70	130	20
2479	Tin*	10	ug/L	1.1	ug/L	80	120	20	70	130	20
2607	Vanadium*	5	ug/L	0.15	ug/L	80	120	20	70	130	20
2649	Zinc	10	ug/L	5	ug/L	80	120	20	70	130	20

* Molybdenum, Selenium, Tin, and Vanadium are not on the 6020 method list

Table 8.2-4-2
Metals – ICPMS, 6020
Reporting Limits (RL), Method Detection Limits (MDL),
and Control Limits (LCS, MS/MSD, and RPD)

Solid					LCS			MS		
#	Compound	RL	Units	MDL	Units	LCL	UCL	RPD	LCL	UCL	RPD
128	Antimony	0.2	mg/kg	0.0053	mg/kg	80	120	20	70	130	20
140	Arsenic	0.5	mg/kg	0.01	mg/kg	74	110	20	70	130	20
194	Barium	0.1	mg/kg	0.038	mg/kg	80	120	20	70	130	20
222	Beryllium	0.1	mg/kg	0.015	mg/kg	80	120	20	70	130	20
411	Cadmium	0.1	mg/kg	0.012	mg/kg	75	110	20	70	130	20
2952	Chromium	0.2	mg/kg	0.025	mg/kg	80	120	20	70	130	20
637	Cobalt	0.1	mg/kg	0.0046	mg/kg	80	120	20	70	130	20
643	Copper	0.2	mg/kg	0.026	mg/kg	80	120	20	70	130	20
1605	Lead	0.1	mg/kg	0.014	mg/kg	80	120	20	70	130	20
1659	Manganese	0.1	mg/kg	0.079	mg/kg	80	120	20	70	130	20
1906	Molybdenum*	0.2	mg/kg	0.012	mg/kg	80	120	20	70	130	20
1956	Nickel	0.1	mg/kg	0.034	mg/kg	80	120	20	70	130	20
2281	Selenium*	0.5	mg/kg	0.056	mg/kg	74	110	20	70	130	20
2285	Silver	0.1	mg/kg	0.022	mg/kg	80	120	20	70	130	20
2477	Thallium	0.1	mg/kg	0.0032	mg/kg	76	110	20	70	130	20
2479	Tin*	1	mg/kg	0.4	mg/kg	80	120	20	70	130	20
2607	Vanadium*	0.5	mg/kg	0.021	mg/kg	80	120	20	70	130	20
2649	Zinc	1	mg/kg	0.31	mg/kg	70	110	20	70	130	20

* Molybdenum, Selenium, Tin, and Vanadium are not on the 6020 method list

Table 8.2-4-2
Metals – ICPMS, 200.8
Reporting Limits (RL), Method Detection Limits (MDL),
and Control Limits (LCS, MS/MSD, and RPD)

	Water					LCS			MS		
#	Compound	RL	Units	MDL	Units	LCL	UCL	RPD	LCL	UCL	RPD
88	Aluminum	50	ug/L	11	ug/L	85	115	20	70	130	20
128	Antimony	2	ug/L	0.066	ug/L	85	115	20	70	130	20
140	Arsenic	5	ug/L	0.35	ug/L	85	115	20	70	130	20
194	Barium	1	ug/L	0.16	ug/L	85	115	20	70	130	20
222	Beryllium	1	ug/L	0.21	ug/L	85	115	20	70	130	20
411	Cadmium	1	ug/L	0.05	ug/L	85	115	20	70	130	20
2952	Chromium	2	ug/L	0.18	ug/L	85	115	20	70	130	20
637	Cobalt	1	ug/L	0.025	ug/L	85	115	20	70	130	20
643	Copper	2	ug/L	0.6	ug/L	85	115	20	70	130	20
1539	Iron	20	ug/L	9	ug/L	0	0	0	0	0	0
1605	Lead	1	ug/L	0.15	ug/L	85	115	20	70	130	20
1659	Manganese	1	ug/L	0.23	ug/L	85	115	20	70	130	20
1906	Molybdenum	2	ug/L	0.046	ug/L	85	115	20	70	130	20
1956	Nickel	2	ug/L	0.13	ug/L	85	115	20	70	130	20
2281	Selenium	5	ug/L	1.2	ug/L	85	115	20	70	130	20
2285	Silver	1	ug/L	0.054	ug/L	85	115	20	70	130	20
2477	Thallium	1	ug/L	0.018	ug/L	85	115	20	70	130	20
2479	Tin*	10	ug/L	1.1	ug/L	85	115	20	70	130	20
2607	Vanadium	5	ug/L	0.15	ug/L	85	115	20	70	130	20
2649	Zinc	10	ug/L	5	ug/L	85	115	20	70	130	20

*Tin is not on the 200.8 method list.

Table 8.2-4-2
Metals – Low-Level Mercury, 1631B & 245.7
Reporting Limits (RL), Method Detection Limits (MDL),
and Control Limits (LCS, MS/MSD, and RPD)

1631B	Water					LCS			MS		
#	Compound	RL	Units	MDL	Units	LCL	UCL	RPD	LCL	UCL	RPD
1701	Mercury	0.5	ng/L	0.12	ng/L	77	125	18	71	125	24

245.7	Water					LCS			MS		
#	Compound	RL	Units	MDL	Units	LCL	UCL	RPD	LCL	UCL	RPD
1701	Mercury	5	ng/L	0.906	ng/L	76	111	18	76	111	18

TABLE 8.2-4-3
MS Semivolatiles – Method 8270C
Reporting Limits (RL), Method Detection Limits (MDL),
and Control Limits (LCS, MS/MSD, and RPD)[1]

	Water						LCS				MS		
#	Compound	RL	Units	MDL	Units	T	LCL	UCL	RPD	T	LCL	UCL	RPD
1	Acenaphthene	10	ug/L	2.7	ug/L	C	39	118	35	C	26	118	35
5	Acenaphthylene	10	ug/L	2.7	ug/L		48	101	21		48	96	21
122	Anthracene	10	ug/L	0.89	ug/L		56	105	18		52	101	18
202	Benzo(a)anthracene	10	ug/L	2.8	ug/L		56	109	16		52	110	16
205	Benzo(b)fluoranthene	10	ug/L	2.6	ug/L		52	108	20		48	107	20
208	Benzo(k)fluoranthene	10	ug/L	1.2	ug/L		53	112	20		53	109	20
210	Benzo(ghi)perylene	10	ug/L	3.3	ug/L		45	115	17		48	109	17
211	Benzo(a)pyrene	10	ug/L	3	ug/L		50	100	18		47	98	18
289	bis(2-chloro ethoxy)methane	10	ug/L	2.6	ug/L		39	109	26		40	101	40
293	bis(2-Chloroethyl) ether	10	ug/L	2.1	ug/L		45	103	26		36	104	26
302	bis(2-Ethylhexyl) phthalate	10	ug/L	2.1	ug/L		56	127	23		44	133	23
348	4-Bromophenyl phenyl ether	10	ug/L	1	ug/L		57	114	17		56	110	17
403	Butyl benzyl phthalate	10	ug/L	1.9	ug/L		53	113	18		46	115	18
2751	Carbazole	10	ug/L	1.1	ug/L		37	114	21		42	115	21
518	4-Chloroaniline	10	ug/L	2.8	ug/L		19	82	41		13	71	41
578	4-Chloro-3-methylphenol	10	ug/L	1.2	ug/L	C	29	124	55	C	21	124	55
589	2-Chloronaphthalene	10	ug/L	2.5	ug/L		51	106	25		46	104	25
600	2-Chlorophenol	10	ug/L	1.6	ug/L	C	19	124	43	C	19	124	43
602	4-Chlorophenyl phenyl ether	10	ug/L	1.3	ug/L		57	114	19		55	110	19
633	Chrysene	10	ug/L	0.88	ug/L		59	112	16		54	115	16
860	Dibenz(a,h)anthracene	10	ug/L	1.2	ug/L		50	112	18		49	110	18
863	Dibenzofuran	10	ug/L	2.8	ug/L		55	107	20		53	104	20
891	Di-n-butyl phthalate	10	ug/L	1.1	ug/L		59	108	17		53	109	17
904	1,2-Dichlorobenzene	10	ug/L	0.86	ug/L		39	90	31		33	91	29
907	1,3-Dichlorobenzene	10	ug/L	1	ug/L		34	85	31		30	86	31
910	1,4-Dichlorobenzene	10	ug/L	0.89	ug/L	C	28	110	36	C	18	110	36
918	3,3'-Dichlorobenzidine	50	ug/L	1.1	ug/L		20	76	36		10	71	36
971	2,4-Dichlorophenol	10	ug/L	1	ug/L		48	101	26		43	103	26

TABLE 8.2-4-3
MS Semivolatiles – Method 8270C
Reporting Limits (RL), Method Detection Limits (MDL),
and Control Limits (LCS, MS/MSD, and RPD)[1]

	Water						LCS				MS		
#	Compound	RL	Units	MDL	Units	T	LCL	UCL	RPD	T	LCL	UCL	RPD
1082	Diethyl phthalate	10	ug/L	3.2	ug/L		48	112	20		36	117	20
1145	2,4-Dimethylphenol	10	ug/L	1.1	ug/L		10	88	28		10	88	28
1149	Dimethyl phthalate	10	ug/L	3.7	ug/L		46	117	22		32	124	22
1167	4,6-Dinitro-2-methylphenol	50	ug/L	7.5	ug/L		37	137	24		46	123	24
1187	2,4-Dinitrophenol	50	ug/L	13	ug/L		21	143	32		30	133	32
1191	2,4-Dinitrotoluene	10	ug/L	0.8	ug/L	C	47	131	32	C	31	131	32
1193	2,6-Dinitrotoluene	10	ug/L	2.8	ug/L		62	114	16		58	109	16
1162	Di-n-octyl phthalate	10	ug/L	2	ug/L		49	127	22		46	124	22
1414	Fluoranthene	10	ug/L	0.94	ug/L		53	116	19		51	113	19
1417	Fluorene	10	ug/L	2.9	ug/L		57	107	18		54	105	19
1482	Hexachlorobenzene	10	ug/L	1.8	ug/L		57	128	22		36	132	22
1489	Hexachlorobutadiene	10	ug/L	1.2	ug/L		36	116	32		18	116	32
1492	Hexachloro-cyclopentadiene	50	ug/L	3.4	ug/L		10	81	59		10	45	59
1497	Hexachloroethane	10	ug/L	2.3	ug/L		30	110	33		18	110	33
1535	Indeno(1,2,3-cd)pyrene	10	ug/L	1.2	ug/L		49	114	19		48	113	19
1566	Isophorone	10	ug/L	2.7	ug/L		48	103	25		42	102	25
1829	2-Methylnaphthalene	10	ug/L	0.92	ug/L		49	98	28		39	102	28
1851	2-Methylphenol	10	ug/L	1.1	ug/L		33	115	31		29	115	31
1857	4-Methylphenol	10	ug/L	1.7	ug/L		29	144	33		25	144	33
1932	Naphthalene	10	ug/L	0.72	ug/L		46	95	26		39	96	26
1960	2-Nitroaniline	50	ug/L	1.4	ug/L		55	119	17		44	116	17
1964	3-Nitroaniline	50	ug/L	2	ug/L		33	107	23		20	102	23
1968	4-Nitroaniline	50	ug/L	1.2	ug/L		32	106	26		25	95	26
1972	Nitrobenzene	10	ug/L	2.6	ug/L		45	130	50		10	211	50
1998	2-Nitrophenol	10	ug/L	0.99	ug/L		43	104	26		35	104	26
2001	4-Nitrophenol	50	ug/L	4.8	ug/L	C	19	144	34	C	10	145	34
2028	N-Nitrosodiphenylamine	10	ug/L	0.91	ug/L		47	112	18		53	99	18
2024	N-Nitrosodi-n-propylamine	10	ug/L	1	ug/L	C	30	115	36	C	18	115	36
2118	Pentachlorophenol	10	ug/L	0.58	ug/L	C	10	140	56	C	10	140	56
2154	Phenanthrene	10	ug/L	2.4	ug/L		58	110	18		55	109	18
2155	Phenol	10	ug/L	1.3	ug/L	C	10	131	43	C	10	131	43
301	2,2'-Oxybis(1-Chloropropane)	10	ug/L	1.3	ug/L								
2252	Pyrene	10	ug/L	1.4	ug/L	C	46	130	31	C	27	138	31

TABLE 8.2-4-3
MS Semivolatiles – Method 8270C
Reporting Limits (RL), Method Detection Limits (MDL),
and Control Limits (LCS, MS/MSD, and RPD)[1]

	Water					LCS				MS			
#	Compound	RL	Units	MDL	Units	T	LCL	UCL	RPD	T	LCL	UCL	RPD
2515	1,2,4-Trichlorobenzene	10	ug/L	2.5	ug/L	C	31	110	37	C	22	110	37
2555	2,4,5-Trichlorophenol	10	ug/L	1.1	ug/L		41	125	22		24	143	22
2559	2,4,6-Trichlorophenol	10	ug/L	1.3	ug/L		46	135	27		36	135	27
1425	2-Fluorobiphenyl					X	30	110		X	30	110	
1426	2-Fluorophenol					X	13	110		X	13	110	
2512	2,4,6-Tribromophenol					X	21	122		X	21	122	
2736	Nitrobenzene-d5					X	32	112		X	32	112	
2737	Phenol-d5					X	10	113		X	10	113	
2738	Terphenyl-d14					X	10	144		X	10	144	

TABLE 8.2-4-3
MS Semivolatiles – Method 8270C
Reporting Limits (RL), Method Detection Limits (MDL),
and Control Limits (LCS, MS/MSD, and RPD)[1]

Solid							LCS				MS		
#	Compound	RL	Units	MDL	Units	T	LCL	UCL	RPD	T	LCL	UCL	RPD
1	Acenaphthene	330	ug/kg	35	ug/kg	C	44	108	44	C	13	133	44
5	Acenaphthylene	330	ug/kg	35	ug/kg		48	107	36		35	111	36
122	Anthracene	330	ug/kg	37	ug/kg		49	114	38		35	115	38
202	Benzo(a)anthracene	330	ug/kg	35	ug/kg		49	116	35		30	122	35
205	Benzo(b)fluoranthene	330	ug/kg	35	ug/kg		44	117	38		28	121	38
208	Benzo(k)fluoranthene	330	ug/kg	41	ug/kg		43	116	36		28	121	36
210	Benzo(ghi)perylene	330	ug/kg	45	ug/kg		44	121	38		17	126	38
211	Benzo(a)pyrene	330	ug/kg	33	ug/kg		46	109	36		26	114	36
289	bis(2-Chloroethoxy)-methane	330	ug/kg	35	ug/kg		42	109	40		34	109	40
293	bis(2-Chloroethyl) ether	330	ug/kg	33	ug/kg		45	100	52		29	104	52
302	bis(2-Ethylhexyl) phthalate	330	ug/kg	65	ug/kg		44	125	38		21	130	38
348	4-Bromophenyl phenyl ether	330	ug/kg	35	ug/kg		45	120	38		33	124	38
403	Butyl benzyl phthalate	330	ug/kg	44	ug/kg		46	111	37		26	119	37
2751	Carbazole	330	ug/kg	42	ug/kg		48	133	37		38	126	37
518	4-Chloroaniline	330	ug/kg	33	ug/kg		14	70	67		10	73	67
578	4-Chloro-3-methylphenol	330	ug/kg	31	ug/kg	C	43	110	55	C	17	128	55
589	2-Chloronaphthalene	330	ug/kg	32	ug/kg		49	111	36		37	114	36
600	2-Chlorophenol	330	ug/kg	28	ug/kg	C	43	110	54	C	17	116	54
602	4-Chlorophenyl phenyl ether	330	ug/kg	36	ug/kg		47	120	36		33	128	36
633	Chrysene	330	ug/kg	50	ug/kg		53	115	36		28	126	36
860	Dibenz(a,h)anthracene	330	ug/kg	38	ug/kg		49	119	37		26	123	37
863	Dibenzofuran	330	ug/kg	36	ug/kg		48	113	36		36	119	36
891	Di-n-butyl phthalate	330	ug/kg	59	ug/kg		48	115	36		33	117	36
904	1,2-Dichlorobenzene	330	ug/kg	29	ug/kg		49	100	40		32	104	40
907	1,3-Dichlorobenzene	330	ug/kg	32	ug/kg		47	96	44		29	99	44
910	1,4-Dichlorobenzene	330	ug/kg	36	ug/kg	C	38	100	59	C	18	110	59
918	3,3'-Dichlorobenzidine	1600	ug/kg	140	ug/kg		15	80	79		10	76	79
971	2,4-Dichlorophenol	330	ug/kg	45	ug/kg		46	111	42		31	120	42
1082	Diethyl phthalate	330	ug/kg	38	ug/kg		47	116	36		32	118	36
1145	2,4-Dimethylphenol	330	ug/kg	57	ug/kg		34	108	56		18	118	56
1149	Dimethyl phthalate	330	ug/kg	36	ug/kg		48	118	38		34	120	38
1167	4,6-Dinitro-2-	1600	ug/kg	180	ug/kg		25	137	45		13	126	45

TABLE 8.2-4-3
MS Semivolatiles – Method 8270C
Reporting Limits (RL), Method Detection Limits (MDL),
and Control Limits (LCS, MS/MSD, and RPD)[1]

	Solid						LCS				MS		
#	Compound	RL	Units	MDL	Units	T	LCL	UCL	RPD	T	LCL	UCL	RPD
	methylphenol												
1187	2,4-Dinitrophenol	1600	ug/kg	150	ug/kg		10	143	50		10	141	50
1191	2,4-Dinitrotoluene	330	ug/kg	41	ug/kg	C	48	111	45	C	10	171	45
1193	2,6-Dinitrotoluene	330	ug/kg	30	ug/kg		50	122	38		36	123	38
1162	Di-n-octyl phthalate	330	ug/kg	50	ug/kg		40	121	39		21	130	39
1414	Fluoranthene	330	ug/kg	38	ug/kg		46	124	42		24	138	42
1417	Fluorene	330	ug/kg	29	ug/kg		48	114	37		32	123	37
1482	Hexachlorobenzene	330	ug/kg	41	ug/kg		44	126	29		39	127	29
1489	Hexachlorobutadiene	330	ug/kg	31	ug/kg		36	110	41		31	110	41
1492	Hexachloro-cyclopentadiene	1600	ug/kg	150	ug/kg		10	126	90		10	102	90
1497	Hexachloroethane	330	ug/kg	40	ug/kg		30	110	40		23	110	40
1535	Indeno(1,2,3-cd)pyrene	330	ug/kg	42	ug/kg		47	125	41		27	123	41
1566	Isophorone	330	ug/kg	32	ug/kg		41	102	37		27	107	37
1829	2-Methylnaphthalene	330	ug/kg	33	ug/kg		46	105	43		33	112	43
1851	2-Methylphenol	330	ug/kg	37	ug/kg		41	102	39		33	113	39
1857	4-Methylphenol	330	ug/kg	27	ug/kg		40	110	34		33	118	34
1932	Naphthalene	330	ug/kg	35	ug/kg		48	101	38		34	107	38
1960	2-Nitroaniline	1600	ug/kg	33	ug/kg		45	123	33		30	124	33
1964	3-Nitroaniline	1600	ug/kg	33	ug/kg		26	119	53		10	105	53
1968	4-Nitroaniline	1600	ug/kg	47	ug/kg		34	122	69		10	105	69
1972	Nitrobenzene	330	ug/kg	32	ug/kg		35	112	36		33	112	36
1998	2-Nitrophenol	330	ug/kg	44	ug/kg		43	110	39		29	112	39
2001	4-Nitrophenol	1600	ug/kg	350	ug/kg	C	22	128	64	C	10	148	64
2028	N-Nitrosodiphenylamine	330	ug/kg	37	ug/kg		50	118	42		35	118	42
2024	N-Nitrosodi-n-propylamine	330	ug/kg	31	ug/kg	C	38	110	50	C	12	128	50
2118	Pentachlorophenol	330	ug/kg	34	ug/kg	C	10	123	87	C	10	144	87
2154	Phenanthrene	330	ug/kg	43	ug/kg		50	117	39		32	126	39
2155	Phenol	330	ug/kg	35	ug/kg	C	35	110	50	C	10	148	50
301	2,2'-Oxybis(1-Chloropropane)	330	ug/kg	93	ug/kg								
2252	Pyrene	330	ug/kg	57	ug/kg	C	42	122	66	C	10	218	66
2515	1,2,4-Trichlorobenzene	330	ug/kg	38	ug/kg	C	45	110	54	C	16	121	54
2555	2,4,5-Trichlorophenol	330	ug/kg	69	ug/kg		39	117	29		29	125	29
2559	2,4,6-Trichlorophenol	330	ug/kg	57	ug/kg		40	110	60		21	126	60
1425	2-Fluorobiphenyl					X	43	110		X	43	110	
1426	2-Fluorophenol					X	11	116		X	11	116	

TABLE 8.2-4-3
MS Semivolatiles – Method 8270C
Reporting Limits (RL), Method Detection Limits (MDL),
and Control Limits (LCS, MS/MSD, and RPD)[1]

Solid						LCS				MS			
#	Compound	RL	Units	MDL	Units	T	LCL	UCL	RPD	T	LCL	UCL	RPD
2512	2,4,6-Tribromophenol					X	35	116		X	35	116	
2736	Nitrobenzene-d5					X	42	110		X	42	110	
2737	Phenol-d5					X	25	115		X	25	115	
2738	Terphenyl-d14					X	37	137		X	37	137	

TABLE 8.2-4-4
MS Semivolatiles – Method 625
Reporting Limits (RL), Method Detection Limits (MDL),
and Control Limits (LCS, MS/MSD, and RPD)[1]

	Water					LCS				MS			
#	Compound	RL	Units	MDL	Units	T	LCL	UCL	RPD	T	LCL	UCL	RPD
1	Acenaphthene	10	ug/L	2.7	ug/L	C	48	113		C	45	112	
5	Acenaphthylene	10	ug/L	2.7	ug/L	C	47	111		C	30	122	
122	Anthracene	10	ug/L	0.89	ug/L	C	53	107		C	18	133	
202	Benzo(a)anthracene	10	ug/L	2.8	ug/L	C	53	110		C	17	130	
205	Benzo(b)fluoranthene	10	ug/L	2.6	ug/L	C	43	110		C	29	108	
208	Benzo(k)fluoranthene	10	ug/L	1.2	ug/L	C	36	136		C	23	127	
210	Benzo(ghi)perylene	10	ug/L	3.3	ug/L	C	17	120		C	10	128	
211	Benzo(a)pyrene	10	ug/L	3	ug/L	C	28	114		C	13	126	
289	bis(2-Chloroethoxy)-methane	10	ug/L	2.6	ug/L	C	50	106		C	29	130	
293	bis(2-Chloroethyl) ether	10	ug/L	2.1	ug/L	C	44	95		C	29	103	
298	bis(2-Chloroisopropyl) ether	10	ug/L	1.3	ug/L	C	21	122		C	19	150	
302	bis(2-Ethylhexyl) phthalate	10	ug/L	2.1	ug/L	C	30	123		C	12	145	
348	4-Bromophenyl phenyl ether	10	ug/L	1	ug/L	C	52	114		C	32	128	
403	Butyl benzyl phthalate	10	ug/L	1.9	ug/L								
578	4-Chloro-3-methylphenol	10	ug/L	1.2	ug/L	C	30	117		C	21	120	
589	2-Chloronaphthalene	10	ug/L	2.5	ug/L	C	43	111		C	27	125	
600	2-Chlorophenol	10	ug/L	1.6	ug/L	C	35	111		C	10	134	
602	4-Chlorophenyl phenyl ether	10	ug/L	1.3	ug/L	C	48	124		C	33	131	
633	Chrysene	10	ug/L	0.88	ug/L	C	54	120		C	20	138	
860	Dibenz(a,h)anthracene	10	ug/L	1.2	ug/L								
891	Di-n-butyl phthalate	10	ug/L	1.1	ug/L	C	44	115		C	24	126	
904	1,2-Dichlorobenzene	10	ug/L	0.86	ug/L	C	43	95		C	28	103	
907	1,3-Dichlorobenzene	10	ug/L	1	ug/L	C	41	92		C	31	97	
910	1,4-Dichlorobenzene	10	ug/L	0.89	ug/L	C	37	92		C	33	90	
918	3,3'-Dichlorobenzidine	10	ug/L	1.1	ug/L	C	19	137		C	10	139	
971	2,4-Dichlorophenol	10	ug/L	1	ug/L	C	33	93		C	34	100	
1082	Diethyl phthalate	10	ug/L	3.2	ug/L	C	16	129		C	27	110	
1145	2,4-Dimethylphenol	10	ug/L	1.1	ug/L	C	23	74		C	11	78	
1149	Dimethyl phthalate	10	ug/L	3.7	ug/L	C	10	115		C	10	101	

TABLE 8.2-4-4
MS Semivolatiles – Method 625
Reporting Limits (RL), Method Detection Limits (MDL),
and Control Limits (LCS, MS/MSD, and RPD)[1]

	Water					LCS				MS			
#	Compound	RL	Units	MDL	Units	T	LCL	UCL	RPD	T	LCL	UCL	RPD
2942	2-Methyl-4,6-dinitrophenol	50	ug/L	7.5	ug/L								
1187	2,4-Dinitrophenol	50	ug/L	13	ug/L	C	10	96		C	10	114	
1191	2,4-Dinitrotoluene	10	ug/L	0.8	ug/L	C	37	131		C	37	124	
1193	2,6-Dinitrotoluene	10	ug/L	2.8	ug/L	C	40	121		C	23	130	
1162	Di-n-octyl phthalate	10	ug/L	2	ug/L	C	25	123		C	31	110	
1414	Fluoranthene	10	ug/L	0.94	ug/L	C	35	131		C	26	123	
1417	Fluorene	10	ug/L	2.9	ug/L	C	45	124		C	32	122	
1482	Hexachlorobenzene	10	ug/L	1.8	ug/L	C	53	110		C	34	129	
1489	Hexachlorobutadiene	10	ug/L	1.2	ug/L	C	45	96		C	31	109	
1497	Hexachloroethane	10	ug/L	2.3	ug/L	C	38	90		C	32	95	
1535	Indeno(1,2,3-cd)pyrene	10	ug/L	1.2	ug/L	C	28	112		C	13	127	
1566	Isophorone	10	ug/L	2.7	ug/L	C	44	105		C	19	133	
1932	Naphthalene	10	ug/L	0.72	ug/L	C	44	99		C	32	111	
1972	Nitrobenzene	10	ug/L	2.6	ug/L	C	44	102		C	34	114	
1998	2-Nitrophenol	10	ug/L	0.99	ug/L	C	10	99		C	19	107	
2001	4-Nitrophenol	50	ug/L	4.8	ug/L	C	10	128		C	10	144	
2024	N-Nitrosodi-n-propylamine	10	ug/L	1	ug/L	C	31	113		C	10	230	
2118	Pentachlorophenol	10	ug/L	0.58	ug/L	C	10	113		C	10	122	
2154	Phenanthrene	10	ug/L	2.4	ug/L	C	50	109		C	30	120	
2155	Phenol	10	ug/L	1.3	ug/L	C	12	116		C	10	107	
2252	Pyrene	10	ug/L	1.4	ug/L	C	39	159		C	15	185	
2515	1,2,4-Trichlorobenzene	10	ug/L	2.5	ug/L	C	39	136		C	40	133	
2559	2,4,6-Trichlorophenol	10	ug/L	1.3	ug/L	C	22	102		C	18	118	
1425	2-Fluorobiphenyl					X	38	106		X	38	106	
1426	2-Fluorophenol					X	17	106		X	17	106	
2512	2,4,6-Tribromophenol					X	13	145		X	13	145	
2736	Nitrobenzene-d5					X	36	148		X	36	148	
2737	Phenol-d5					X	15	126		X	15	126	
2738	Terphenyl-d14					X	10	169		X	10	169	

TABLE 8.2-4-5
MS Volatiles – Method 8260B
Reporting Limits (RL), Method Detection Limits (MDL),
and Control Limits (LCS MS/MSD, and RPD)[1]

Water - 25 mL						LCS				MS			
#	Compound	RL	Units	MDL	Units	T	LCL	UCL	RPD	T	LCL	UCL	RPD
196	Benzene	1	ug/L	0.63	ug/L	C	80	116	20	C	76	118	20
318	Bromobenzene	1	ug/L	0.75	ug/L								
321	Bromochloromethane	1	ug/L	0.59	ug/L								
323	Bromodichloromethane	1	ug/L	0.52	ug/L								
340	Bromoform	1	ug/L	0.24	ug/L								
343	Bromomethane	2	ug/L	1.2	ug/L								
393	n-Butylbenzene	1	ug/L	0.75	ug/L								
395	sec-Butylbenzene	1	ug/L	0.76	ug/L								
398	tert-Butylbenzene	1	ug/L	0.81	ug/L								
463	Carbon tetrachloride	1	ug/L	0.55	ug/L								
521	Chlorobenzene	1	ug/L	0.72	ug/L	C	76	117	20	C	76	117	20
534	Chlorodibromomethane	1	ug/L	0.2	ug/L								
550	Chloroethane	2	ug/L	0.82	ug/L								
569	Chloroform	1	ug/L	0.71	ug/L								
574	Chloromethane	2	ug/L	0.73	ug/L								
614	2-Chlorotoluene	1	ug/L	0.77	ug/L								
617	4-Chlorotoluene	1	ug/L	0.8	ug/L								
539	1,2-Dibromo-3-chloropropane	2	ug/L	0.26	ug/L								
870	1,2-Dibromoethane	1	ug/L	0.41	ug/L								
888	Dibromomethane	1	ug/L	0.46	ug/L								
904	1,2-Dichlorobenzene	1	ug/L	0.68	ug/L								
907	1,3-Dichlorobenzene	1	ug/L	0.8	ug/L								
910	1,4-Dichlorobenzene	1	ug/L	0.64	ug/L								
924	Dichlorodifluoro-methane	2	ug/L	0.55	ug/L								
933	1,1-Dichloroethane	1	ug/L	0.63	ug/L								
936	1,2-Dichloroethane	1	ug/L	0.57	ug/L								
948	cis-1,2-Dichloroethene	0.5	ug/L	0.21	ug/L								
950	trans-1,2-Dichloroethene	0.5	ug/L	0.27	ug/L								
943	1,1-Dichloroethene	1	ug/L	0.67	ug/L	C	63	130	20	C	62	130	20
986	1,2-Dichloropropane	1	ug/L	0.65	ug/L								
989	1,3-Dichloropropane	1	ug/L	0.4	ug/L								
990	2,2-Dichloropropane	1	ug/L	0.27	ug/L								
996	1,1-Dichloropropene	1	ug/L	0.51	ug/L								
1332	Ethylbenzene	1	ug/L	0.64	ug/L								

TABLE 8.2-4-5
MS Volatiles – Method 8260B
Reporting Limits (RL), Method Detection Limits (MDL),
and Control Limits (LCS MS/MSD, and RPD)[1]

Water - 25 mL						LCS				MS			
#	Compound	RL	Units	MDL	Units	T	LCL	UCL	RPD	T	LCL	UCL	RPD
1489	Hexachlorobutadiene	1	ug/L	0.75	ug/L								
1578	Isopropylbenzene	1	ug/L	0.67	ug/L								
1590	p-Isopropyltoluene	1	ug/L	0.75	ug/L								
1811	Methylene chloride	1	ug/L	0.19	ug/L								
1932	Naphthalene	1	ug/L	0.79	ug/L								
2247	n-Propylbenzene	1	ug/L	0.86	ug/L								
2355	Styrene	1	ug/L	0.65	ug/L								
2437	1,1,1,2-Tetrachloroethane	1	ug/L	0.89	ug/L								
2439	1,1,2,2-Tetrachloroethane	1	ug/L	0.39	ug/L								
2445	Tetrachloroethene	1	ug/L	0.74	ug/L								
2489	Toluene	1	ug/L	0.75	ug/L	C	74	119	20	C	70	119	20
2514	1,2,3-Trichlorobenzene	1	ug/L	0.81	ug/L								
2515	1,2,4-Trichlorobenzene	1	ug/L	0.66	ug/L								
2518	1,1,1-Trichloroethane	1	ug/L	0.71	ug/L								
2522	1,1,2-Trichloroethane	1	ug/L	0.42	ug/L								
2525	Trichloroethene	1	ug/L	0.62	ug/L	C	75	122	20	C	62	130	20
1428	Trichlorofluoromethane	2	ug/L	1.8	ug/L								
2563	1,2,3-Trichloropropane	1	ug/L	0.49	ug/L								
2587	1,2,4-Trimethylbenzene	1	ug/L	0.69	ug/L								
2592	1,3,5-Trimethylbenzene	1	ug/L	0.76	ug/L								
2613	Vinyl chloride	2	ug/L	0.66	ug/L								
2940	m-Xylene & p-Xylene	1	ug/L	0.53	ug/L								
2623	o-Xylene	0.5	ug/L	0.34	ug/L								
337	4-Bromofluorobenzene					X	74	116		X	74	116	
2735	1,2-Dichloroethane-d4					X	61	128		X	61	128	
2740	Toluene-d8					X	76	110		X	76	110	
2863	Dibromofluoromethane					X	73	122		X	73	122	

TABLE 8.2-4-5
MS Volatiles – Method 8260B
Reporting Limits (RL), Method Detection Limits (MDL),
and Control Limits (LCS, MS/MSD, and RPD)[1]

	Water - 5 mL					LCS				MS			
#	Compound	RL	Units	MDL	Units	T	LCL	UCL	RPD	T	LCL	UCL	RPD
196	Benzene	5	ug/L	0.63	ug/L	C	79	116	20	C	73	123	11
318	Bromobenzene	5	ug/L	0.63	ug/L								
321	Bromochloromethane	5	ug/L	0.63	ug/L								
323	Bromodichloromethane	5	ug/L	0.63	ug/L		90	114	18		90	114	18
340	Bromoform	5	ug/L	0.5	ug/L		71	118	34		71	118	34
343	Bromomethane	10	ug/L	1.2	ug/L		47	160	22		47	160	22
393	n-Butylbenzene	5	ug/L	0.75	ug/L								
395	sec-Butylbenzene	5	ug/L	0.76	ug/L								
398	tert-Butylbenzene	5	ug/L	0.81	ug/L								
463	Carbon tetrachloride	5	ug/L	0.62	ug/L		72	133	20		61	143	14
521	Chlorobenzene	5	ug/L	0.72	ug/L	C	81	115	20	C	70	122	14
534	Chlorodibromomethane	5	ug/L	0.57	ug/L								
550	Chloroethane	10	ug/L	0.92	ug/L		80	118	17		80	118	17
569	Chloroform	5	ug/L	0.71	ug/L		81	122	20		65	131	20
574	Chloromethane	10	ug/L	0.73	ug/L		61	129	20		61	129	20
614	2-Chlorotoluene	5	ug/L	0.77	ug/L								
617	4-Chlorotoluene	5	ug/L	0.8	ug/L								
539	1,2-Dibromo-3-chloropropane	10	ug/L	3.1	ug/L								
870	1,2-Dibromoethane	5	ug/L	0.63	ug/L								
888	Dibromomethane	5	ug/L	0.84	ug/L								
904	1,2-Dichlorobenzene	5	ug/L	0.68	ug/L								
907	1,3-Dichlorobenzene	5	ug/L	0.8	ug/L								
910	1,4-Dichlorobenzene	5	ug/L	0.64	ug/L								
924	Dichlorodifluoro-methane	10	ug/L	0.88	ug/L								
933	1,1-Dichloroethane	5	ug/L	0.63	ug/L		87	120	22		87	120	22
936	1,2-Dichloroethane	5	ug/L	0.57	ug/L		73	127	20		67	132	18
948	cis-1,2-Dichloroethene	2.5	ug/L	0.27	ug/L		50	150	50		50	150	50
950	trans-1,2-Dichloroethene	2.5	ug/L	0.8	ug/L		70	130	50		70	130	50
943	1,1-Dichloroethene	5	ug/L	0.67	ug/L	C	65	119	20	C	57	138	15
986	1,2-Dichloropropane	5	ug/L	0.65	ug/L		91	113	18		91	113	18
989	1,3-Dichloropropane	5	ug/L	0.69	ug/L								
990	2,2-Dichloropropane	5	ug/L	1.1	ug/L								
996	1,1-Dichloropropene	5	ug/L	0.64	ug/L								
1332	Ethylbenzene	5	ug/L	0.84	ug/L		90	116	18		90	116	18

TABLE 8.2-4-5
MS Volatiles – Method 8260B
Reporting Limits (RL), Method Detection Limits (MDL),
and Control Limits (LCS, MS/MSD, and RPD)[1]

Water - 5 mL						LCS				MS			
#	Compound	RL	Units	MDL	Units	T	LCL	UCL	RPD	T	LCL	UCL	RPD
1489	Hexachlorobutadiene	5	ug/L	0.75	ug/L								
1578	Isopropylbenzene	5	ug/L	0.67	ug/L								
1590	p-Isopropyltoluene	5	ug/L	0.75	ug/L								
1811	Methylene chloride	5	ug/L	1.4	ug/L		81	134	27		81	134	27
1932	Naphthalene	5	ug/L	0.98	ug/L								
2247	n-Propylbenzene	5	ug/L	0.86	ug/L								
2355	Styrene	5	ug/L	0.65	ug/L		81	113	18		81	113	18
2437	1,1,1,2-Tetrachloroethane	5	ug/L	0.89	ug/L								
2439	1,1,2,2-Tetrachloroethane	5	ug/L	0.7	ug/L		80	127	24		80	127	24
2445	Tetrachloroethene	5	ug/L	0.74	ug/L		78	131	20		70	130	14
2489	Toluene	5	ug/L	0.75	ug/L	C	76	119	20	C	67	129	14
2514	1,2,3-Trichlorobenzene	5	ug/L	0.81	ug/L								
2515	1,2,4-Trichlorobenzene	5	ug/L	0.87	ug/L								
2518	1,1,1-Trichloroethane	5	ug/L	0.71	ug/L		91	113	17		91	113	17
2522	1,1,2-Trichloroethane	5	ug/L	0.54	ug/L		81	117	20		81	117	20
2525	Trichloroethene	5	ug/L	0.74	ug/L	C	80	122	20	C	58	141	17
1428	Trichlorofluoromethane	10	ug/L	1.8	ug/L								
2563	1,2,3-Trichloropropane	5	ug/L	0.68	ug/L								
2587	1,2,4-Trimethylbenzene	5	ug/L	0.69	ug/L								
2592	1,3,5-Trimethylbenzene	5	ug/L	0.76	ug/L								
2613	Vinyl chloride	10	ug/L	0.66	ug/L		53	134	20		51	133	18
2940	m-Xylene & p-Xylene	5	ug/L	1.4	ug/L								
2623	o-Xylene	2.5	ug/L	0.56	ug/L								
337	4-Bromofluorobenzene					X	80	114		X	80	114	
2735	1,2-Dichloroethane-d4					X	77	120		X	77	120	
2740	Toluene-d8					X	78	111		X	78	111	
2863	Dibromofluoromethane					X	78	110		X	78	110	

TABLE 8.2-4-5
MS Volatiles – Method 8260B
Reporting Limits (RL), Method Detection Limits (MDL),
and Control Limits (LCS, MS/MSD, and RPD)[1]

Encore #	Compound	RL	Units	MDL	Units	LCS T	LCS LCL	LCS UCL	LCS RPD	MS T	MS LCL	MS UCL	MS RPD
196	Benzene	250	ug/kg	25	ug/kg	C	75	129	20	C	55	138	20
318	Bromobenzene	250	ug/kg	26	ug/kg								
321	Bromochloromethane	250	ug/kg	29	ug/kg								
323	Bromodichloromethane	250	ug/kg	27	ug/kg		35	155	21		35	155	21
340	Bromoform	250	ug/kg	49	ug/kg		45	169	22		45	169	22
343	Bromomethane	500	ug/kg	34	ug/kg		10	242	11		10	242	11
393	n-Butylbenzene	250	ug/kg	27	ug/kg								
395	sec-Butylbenzene	250	ug/kg	35	ug/kg								
398	tert-Butylbenzene	250	ug/kg	26	ug/kg								
463	Carbon tetrachloride	250	ug/kg	22	ug/kg		66	141	55		39	149	55
521	Chlorobenzene	250	ug/kg	25	ug/kg	C	75	127	22	C	49	139	22
534	Chlorodibromomethane	250	ug/kg	24	ug/kg								
550	Chloroethane	500	ug/kg	39	ug/kg		82	114	11		82	114	11
569	Chloroform	250	ug/kg	19	ug/kg		77	126	17		52	140	17
574	Chloromethane	500	ug/kg	36	ug/kg		10	273	18		10	273	18
614	2-Chlorotoluene	250	ug/kg	26	ug/kg								
617	4-Chlorotoluene	250	ug/kg	29	ug/kg								
539	1,2-Dibromo-3-chloropropane	500	ug/kg	29	ug/kg								
870	1,2-Dibromoethane	250	ug/kg	28	ug/kg								
888	Dibromomethane	250	ug/kg	33	ug/kg								
904	1,2-Dichlorobenzene	250	ug/kg	28	ug/kg								
907	1,3-Dichlorobenzene	250	ug/kg	28	ug/kg								
910	1,4-Dichlorobenzene	250	ug/kg	27	ug/kg								
924	Dichlorodifluoro-methane	500	ug/kg	34	ug/kg								
933	1,1-Dichloroethane	250	ug/kg	26	ug/kg		59	155	12		33	137	14
936	1,2-Dichloroethane	250	ug/kg	25	ug/kg		76	127	41		44	145	41
948	cis-1,2-Dichloroethene	125	ug/kg	26	ug/kg		50	150	50		50	150	50
950	trans-1,2-Dichloroethene	125	ug/kg	17	ug/kg		54	156	10		54	156	10
943	1,1-Dichloroethene	250	ug/kg	52	ug/kg	C	55	142	27	C	43	147	27
986	1,2-Dichloropropane	250	ug/kg	31	ug/kg		10	210	13		10	210	13
989	1,3-Dichloropropane	250	ug/kg	25	ug/kg								
990	2,2-Dichloropropane	250	ug/kg	21	ug/kg								
996	1,1-Dichloropropene	250	ug/kg	24	ug/kg								
1332	Ethylbenzene	250	ug/kg	41	ug/kg		37	162	14		37	162	14

TABLE 8.2-4-5
MS Volatiles – Method 8260B
Reporting Limits (RL), Method Detection Limits (MDL),
and Control Limits (LCS, MS/MSD, and RPD)[1]

Encore #	Compound	RL	Units	MDL	Units	LCS T	LCS LCL	LCS UCL	LCS RPD	MS T	MS LCL	MS UCL	MS RPD
1489	Hexachlorobutadiene	250	ug/kg	46	ug/kg								
1578	Isopropylbenzene	250	ug/kg	55	ug/kg								
1590	p-Isopropyltoluene	250	ug/kg	26	ug/kg								
1811	Methylene chloride	250	ug/kg	64	ug/kg		10	221	22		10	221	22
1932	Naphthalene	250	ug/kg	28	ug/kg								
2247	n-Propylbenzene	250	ug/kg	39	ug/kg								
2355	Styrene	250	ug/kg	47	ug/kg		79	100	10		79	110	10
2437	1,1,1,2-Tetrachloroethane	250	ug/kg	30	ug/kg								
2439	1,1,2,2-Tetrachloroethane	250	ug/kg	29	ug/kg		46	157	24		46	157	24
2445	Tetrachloroethene	250	ug/kg	20	ug/kg		68	136	22		39	154	22
2489	Toluene	250	ug/kg	23	ug/kg	C	71	130	24	C	46	147	24
2514	1,2,3-Trichlorobenzene	250	ug/kg	34	ug/kg								
2515	1,2,4-Trichlorobenzene	250	ug/kg	39	ug/kg								
2518	1,1,1-Trichloroethane	250	ug/kg	23	ug/kg		52	162	12		52	162	12
2522	1,1,2-Trichloroethane	250	ug/kg	27	ug/kg		52	150	19		52	150	19
2525	Trichloroethene	250	ug/kg	27	ug/kg	C	70	131	23	C	46	143	23
1428	Trichlorofluoromethane	500	ug/kg	38	ug/kg								
2563	1,2,3-Trichloropropane	250	ug/kg	30	ug/kg								
2587	1,2,4-Trimethylbenzene	250	ug/kg	13	ug/kg								
2592	1,3,5-Trimethylbenzene	250	ug/kg	26	ug/kg								
2613	Vinyl chloride	500	ug/kg	66	ug/kg		41	138	43		29	150	43
2940	m-Xylene & p-Xylene	250	ug/kg	42	ug/kg								
2623	o-Xylene	125	ug/kg	29	ug/kg								
337	4-Bromofluorobenzene					X	47	158		X	47	158	
2735	1,2-Dichloroethane-d4					X	61	130		X	61	130	
2740	Toluene-d8					X	60	143		X	60	143	
2863	Dibromofluoromethane					X	59	138		X	59	138	

TABLE 8.2-4-5
MS Volatiles – Method 8260B
Reporting Limits (RL), Method Detection Limits (MDL),
and Control Limits (LCS, MS/MSD, and RPD)[1]

Low Level Encore						LCS				MS		
#	Compound	RL	Units	MDL	Units	T	LCL	UCL	RPD	T	LCL	UCL	RPD
196	Benzene	5	ug/kg	0.1	ug/kg	C	75	129	20	C	55	138	20
318	Bromobenzene	5	ug/kg	0.94	ug/kg								
321	Bromochloromethane	5	ug/kg	0.51	ug/kg								
323	Bromodichloromethane	5	ug/kg	0.079	ug/kg		35	155	21		35	155	21
340	Bromoform	5	ug/kg	0.51	ug/kg		45	169	22		45	169	22
343	Bromomethane	10	ug/kg	0.13	ug/kg		10	242	11		10	242	11
393	n-Butylbenzene	5	ug/kg	1.3	ug/kg								
395	sec-Butylbenzene	5	ug/kg	1.3	ug/kg								
398	tert-Butylbenzene	5	ug/kg	1.1	ug/kg								
463	Carbon tetrachloride	5	ug/kg	0.1	ug/kg		66	141	55		39	149	55
521	Chlorobenzene	5	ug/kg	0.87	ug/kg	C	75	127	22	C	49	139	22
534	Chlorodibromomethane	5	ug/kg	0.24	ug/kg								
550	Chloroethane	10	ug/kg	0.19	ug/kg		82	114	11		82	114	11
569	Chloroform	5	ug/kg	0.062	ug/kg		77	126	17		52	140	17
574	Chloromethane	10	ug/kg	0.66	ug/kg		10	273	18		10	273	18
614	2-Chlorotoluene	5	ug/kg	1.2	ug/kg								
617	4-Chlorotoluene	5	ug/kg	1.2	ug/kg								
539	1,2-Dibromo-3-chloropropane	10	ug/kg	0.38	ug/kg								
870	1,2-Dibromoethane	5	ug/kg	1.8	ug/kg								
888	Dibromomethane	5	ug/kg	0.065	ug/kg								
904	1,2-Dichlorobenzene	5	ug/kg	0.99	ug/kg								
907	1,3-Dichlorobenzene	5	ug/kg	1	ug/kg								
910	1,4-Dichlorobenzene	5	ug/kg	1.1	ug/kg								
924	Dichlorodifluoro-methane	10	ug/kg	0.15	ug/kg								
933	1,1-Dichloroethane	5	ug/kg	0.1	ug/kg		59	155	12		33	137	14
936	1,2-Dichloroethane	5	ug/kg	0.088	ug/kg		76	127	41		44	145	41
948	cis-1,2-Dichloroethene	2.5	ug/kg	0.24	ug/kg		50	150	50		50	150	50
950	trans-1,2-Dichloroethene	2.5	ug/kg	0.21	ug/kg		54	156	10		54	156	10
943	1,1-Dichloroethene	5	ug/kg	0.2	ug/kg	C	55	142	27	C	43	147	27
986	1,2-Dichloropropane	5	ug/kg	0.13	ug/kg		10	210	13		10	210	13
989	1,3-Dichloropropane	5	ug/kg	0.097	ug/kg								
990	2,2-Dichloropropane	5	ug/kg	0.5	ug/kg								
996	1,1-Dichloropropene	5	ug/kg	0.1	ug/kg								
1332	Ethylbenzene	5	ug/kg	0.99	ug/kg		37	162	14		37	162	14

TABLE 8.2-4-5
MS Volatiles – Method 8260B
Reporting Limits (RL), Method Detection Limits (MDL),
and Control Limits (LCS, MS/MSD, and RPD)[1]

	Low Level Encore					LCS				MS			
#	Compound	RL	Units	MDL	Units	T	LCL	UCL	RPD	T	LCL	UCL	RPD
1489	Hexachlorobutadiene	5	ug/kg	1.3	ug/kg								
1578	Isopropylbenzene	5	ug/kg	0.99	ug/kg								
1590	p-Isopropyltoluene	5	ug/kg	1.3	ug/kg								
1811	Methylene chloride	5	ug/kg	0.3	ug/kg		10	221	22		10	221	22
1932	Naphthalene	5	ug/kg	0.86	ug/kg								
2247	n-Propylbenzene	5	ug/kg	1.4	ug/kg								
2355	Styrene	5	ug/kg	1	ug/kg		79	100	10		79	110	10
2437	1,1,1,2-Tetrachloroethane	5	ug/kg	0.23	ug/kg								
2439	1,1,2,2-Tetrachloroethane	5	ug/kg	0.61	ug/kg		46	157	24		46	157	24
2445	Tetrachloroethene	5	ug/kg	0.7	ug/kg		68	136	22		39	154	22
2489	Toluene	5	ug/kg	0.67	ug/kg	C	71	130	24	C	46	147	24
2514	1,2,3-Trichlorobenzene	5	ug/kg	0.79	ug/kg								
2515	1,2,4-Trichlorobenzene	5	ug/kg	0.69	ug/kg								
2518	1,1,1-Trichloroethane	5	ug/kg	0.062	ug/kg		52	162	12		52	162	12
2522	1,1,2-Trichloroethane	5	ug/kg	1.2	ug/kg		52	150	19		52	150	19
2525	Trichloroethene	5	ug/kg	0.1	ug/kg	C	70	131	23	C	46	143	23
1428	Trichlorofluoromethane	10	ug/kg	0.1	ug/kg								
2563	1,2,3-Trichloropropane	5	ug/kg	0.69	ug/kg								
2587	1,2,4-Trimethylbenzene	5	ug/kg	1.2	ug/kg								
2592	1,3,5-Trimethylbenzene	5	ug/kg	1.2	ug/kg								
2613	Vinyl chloride	10	ug/kg	0.5	ug/kg		41	138	43		29	150	43
2940	m-Xylene & p-Xylene	5	ug/kg	2	ug/kg								
2623	o-Xylene	2.5	ug/kg	0.98	ug/kg								
337	4-Bromofluorobenzene					X	47	158		X	47	158	
2735	1,2-Dichloroethane-d4					X	61	130		X	61	130	
2740	Toluene-d8					X	60	143		X	60	143	
2863	Dibromofluoromethane					X	59	138		X	59	138	

TABLE 8.2-4-5
MS Volatiles – Method 8260B
Reporting Limits (RL), Method Detection Limits (MDL),
and Control Limits (LCS, MS/MSD, and RPD)[1]

#	Frozen Encore Compound	RL	Units	MDL	Units	LCS T	LCS LCL	LCS UCL	LCS RPD	MS T	MS LCL	MS UCL	MS RPD
196	Benzene	5	ug/kg	0.63	ug/kg	C	75	129	20	C	55	138	20
318	Bromobenzene	5	ug/kg	0.75	ug/kg								
321	Bromochloromethane	5	ug/kg	0.63	ug/kg								
323	Bromodichloromethane	5	ug/kg	0.65	ug/kg		35	155	21		35	155	21
340	Bromoform	5	ug/kg	0.5	ug/kg		45	169	22		45	169	22
343	Bromomethane	10	ug/kg	1.2	ug/kg		10	242	11		10	242	11
393	n-Butylbenzene	5	ug/kg	0.75	ug/kg								
395	sec-Butylbenzene	5	ug/kg	0.76	ug/kg								
398	tert-Butylbenzene	5	ug/kg	0.81	ug/kg								
463	Carbon tetrachloride	5	ug/kg	0.62	ug/kg		66	141	55		39	149	55
521	Chlorobenzene	5	ug/kg	0.72	ug/kg	C	75	127	22	C	49	139	22
534	Chlorodibromomethane	5	ug/kg	0.57	ug/kg								
550	Chloroethane	10	ug/kg	0.92	ug/kg		82	114	11		82	114	11
569	Chloroform	5	ug/kg	0.71	ug/kg		77	126	17		52	140	17
574	Chloromethane	10	ug/kg	0.73	ug/kg		10	273	18		10	273	18
614	2-Chlorotoluene	5	ug/kg	0.77	ug/kg								
617	4-Chlorotoluene	5	ug/kg	0.8	ug/kg								
539	1,2-Dibromo-3-chloropropane	10	ug/kg	3.1	ug/kg								
870	1,2-Dibromoethane	5	ug/kg	0.63	ug/kg								
888	Dibromomethane	5	ug/kg	0.84	ug/kg								
904	1,2-Dichlorobenzene	5	ug/kg	0.68	ug/kg								
907	1,3-Dichlorobenzene	5	ug/kg	0.8	ug/kg								
910	1,4-Dichlorobenzene	5	ug/kg	0.64	ug/kg								
924	Dichlorodifluoro-methane	10	ug/kg	0.88	ug/kg								
933	1,1-Dichloroethane	5	ug/kg	0.63	ug/kg		59	155	12		33	137	14
936	1,2-Dichloroethane	5	ug/kg	0.57	ug/kg		76	127	41		44	145	41
948	cis-1,2-Dichloroethene	2.5	ug/kg	0.27	ug/kg		50	150	50		50	150	50
950	trans-1,2-Dichloroethene	2.5	ug/kg	0.8	ug/kg		54	156	10		54	156	10
943	1,1-Dichloroethene	5	ug/kg	0.67	ug/kg	C	55	142	27	C	43	147	27
986	1,2-Dichloropropane	5	ug/kg	0.65	ug/kg		10	210	13		10	210	13
989	1,3-Dichloropropane	5	ug/kg	0.69	ug/kg								
990	2,2-Dichloropropane	5	ug/kg	1.1	ug/kg								
996	1,1-Dichloropropene	5	ug/kg	0.64	ug/kg								
1332	Ethylbenzene	5	ug/kg	0.84	ug/kg		37	162	14		37	162	14

TABLE 8.2-4-5
MS Volatiles – Method 8260B
Reporting Limits (RL), Method Detection Limits (MDL),
and Control Limits (LCS, MS/MSD, and RPD)[1]

Frozen Encore						LCS				MS			
#	Compound	RL	Units	MDL	Units	T	LCL	UCL	RPD	T	LCL	UCL	RPD
1489	Hexachlorobutadiene	5	ug/kg	0.75	ug/kg								
1578	Isopropylbenzene	5	ug/kg	0.67	ug/kg								
1590	p-Isopropyltoluene	5	ug/kg	0.75	ug/kg								
1811	Methylene chloride	5	ug/kg	1.4	ug/kg		10	221	22		10	221	22
1932	Naphthalene	5	ug/kg	0.98	ug/kg								
2247	n-Propylbenzene	5	ug/kg	0.86	ug/kg								
2355	Styrene	5	ug/kg	0.65	ug/kg		79	100	10		79	110	10
2437	1,1,1,2-Tetrachloroethane	5	ug/kg	0.89	ug/kg								
2439	1,1,2,2-Tetrachloroethane	5	ug/kg	0.7	ug/kg		46	157	24		46	157	24
2445	Tetrachloroethene	5	ug/kg	0.74	ug/kg		68	136	22		39	154	22
2489	Toluene	5	ug/kg	0.75	ug/kg	C	71	130	24	C	46	147	24
2514	1,2,3-Trichlorobenzene	5	ug/kg	0.81	ug/kg								
2515	1,2,4-Trichlorobenzene	5	ug/kg	0.87	ug/kg								
2518	1,1,1-Trichloroethane	5	ug/kg	0.71	ug/kg		52	162	12		52	162	12
2522	1,1,2-Trichloroethane	5	ug/kg	0.54	ug/kg		52	150	19		52	150	19
2525	Trichloroethene	5	ug/kg	0.7	ug/kg	C	70	131	23	C	46	143	23
1428	Trichlorofluoromethane	10	ug/kg	1.8	ug/kg								
2563	1,2,3-Trichloropropane	5	ug/kg	0.68	ug/kg								
2587	1,2,4-Trimethylbenzene	5	ug/kg	0.69	ug/kg								
2592	1,3,5-Trimethylbenzene	5	ug/kg	0.76	ug/kg								
2613	Vinyl chloride	10	ug/kg	0.66	ug/kg		41	138	43		29	150	43
2940	m-Xylene & p-Xylene	5	ug/kg	1.4	ug/kg								
2623	o-Xylene	2.5	ug/kg	0.56	ug/kg								
337	4-Bromofluorobenzene					X	47	158		X	47	158	
2735	1,2-Dichloroethane-d4					X	61	130		X	61	130	
2740	Toluene-d8					X	60	143		X	60	143	
2863	Dibromofluoromethane					X	59	138		X	59	138	

TABLE 8.2-4-6
MS Volatiles – Method 624
Reporting Limits (RL), Method Detection Limits (MDL),
and Control Limits (LCS, MS/MSD, and RPD)[1]

Water						LCS				MS		
#	Compound	RL	Units	MDL	Units	T	LCL	UCL	RPD	T	LCL	UCL	RPD
196	Benzene	5	ug/L	0.63	ug/L	C	79	125		C	83	123	
323	Bromodichloro-methane	5	ug/L	0.63	ug/L	C	65	144		C	57	153	
340	Bromoform	5	ug/L	0.5	ug/L	C	46	151		C	47	165	
343	Bromomethane	10	ug/L	1.2	ug/L	C	47	160		C	76	150	
463	Carbon tetrachloride	5	ug/L	0.62	ug/L	C	63	141		C	73	135	
521	Chlorobenzene	5	ug/L	0.72	ug/L	C	81	121		C	85	120	
535	Dibromochloro-methane	5	ug/L	0.57	ug/L	C	58	140		C	60	146	
550	Chloroethane	10	ug/L	0.92	ug/L	C	38	170		C	71	154	
568	2-Chloroethyl vinyl ether	--	ug/L	1.4	ug/L	C	27	166		C	10	153	
569	Chloroform	5	ug/L	0.71	ug/L	C	70	141		C	10	204	
574	Chloromethane	10	ug/L	0.73	ug/L	C	36	158		C	44	151	
904	1,2-Dichlorobenzene	5	ug/L	0.68	ug/L	C	18	190		C	18	190	
907	1,3-Dichlorobenzene	5	ug/L	0.8	ug/L	C	59	156		C	59	156	
910	1,4-Dichlorobenzene	5	ug/L	0.64	ug/L	C	18	190		C	18	190	
933	1,1-Dichloroethane	5	ug/L	0.63	ug/L	C	64	145		C	53	148	
936	1,2-Dichloroethane	5	ug/L	0.57	ug/L	C	66	150		C	52	167	
950	trans-1,2-Dichloroethene	2.5	ug/L	0.8	ug/L	C	58	140		C	55	139	
943	1,1-Dichloroethene	5	ug/L	0.67	ug/L	C	70	143		C	55	142	
986	1,2-Dichloropropane	5	ug/L	0.65	ug/L	C	71	142		C	64	146	
998	cis-1,3-Dichloropropene	5	ug/L	0.61	ug/L	C	59	140		C	52	135	
1000	trans-1,3-Dichloropropene	5	ug/L	0.59	ug/L	C	53	148		C	50	144	
1332	Ethylbenzene	5	ug/L	0.84	ug/L	C	70	140		C	72	140	
1811	Methylene chloride	10	ug/L	1.4	ug/L	C	72	144		C	62	152	
2439	1,1,2,2-Tetrachloroethane	5	ug/L	0.7	ug/L	C	52	153		C	53	149	
2445	Tetrachloroethene	5	ug/L	0.74	ug/L	C	67	136		C	67	141	
2489	Toluene	5	ug/L	0.75	ug/L	C	79	122		C	82	122	
2518	1,1,1-Trichloroethane	5	ug/L	0.71	ug/L	C	67	137		C	33	171	
2522	1,1,2-Trichloroethane	5	ug/L	0.54	ug/L	C	70	140		C	73	138	
2525	Trichloroethene	5	ug/L	0.74	ug/L	C	76	121		C	77	124	

TABLE 8.2-4-6
MS Volatiles – Method 624
Reporting Limits (RL), Method Detection Limits (MDL),
and Control Limits (LCS, MS/MSD, and RPD)[1]

Water						LCS				MS			
#	Compound	RL	Units	MDL	Units	T	LCL	UCL	RPD	T	LCL	UCL	RPD
1428	Trichlorofluoro-methane	10	ug/L	1.8	ug/L	C	17	181		C	17	181	
2613	Vinyl chloride	10	ug/L	0.66	ug/L	C	47	156		C	48	161	
2730	Bromofluorobenzene					X	86	115		X	86	115	
2735	1,2-Dichloro-ethane-d4					X	76	114		X	76	114	
2740	Toluene-d8					X	88	110		X	88	110	

TABLE 8.2-4-7
GC Semivolatiles – Method 8081A
Reporting Limits (RL), Method Detection Limits (MDL), and Control Limits (LCS, MS/MSD, and RPD)[1]

	Water					LCS				MS			
#	Compound	RL	Units	MDL	Units	T	LCL	UCL	RPD	T	LCL	UCL	RPD
60	Aldrin	0.05	ug/L	0.0064	ug/L	C	62	120	33	C	19	131	33
226	alpha-BHC	0.05	ug/L	0.0059	ug/L		48	130	54		30	139	54
228	beta-BHC	0.05	ug/L	0.0062	ug/L		47	127	38		19	152	38
230	delta-BHC	0.05	ug/L	0.006	ug/L		34	147	44		26	150	44
232	gamma-BHC (Lindane)	0.05	ug/L	0.0062	ug/L	C	49	137	22	C	30	148	22
497	alpha-Chlordane	0.05	ug/L	0.0067	ug/L		52	140	41		33	142	41
499	gamma-Chlordane	0.05	ug/L	0.0065	ug/L		47	143	28		35	143	28
770	4,4'-DDD	0.05	ug/L	0.0064	ug/L		44	158	39		42	158	39
777	4,4'-DDE	0.05	ug/L	0.0074	ug/L		32	157	39		35	134	39
780	4,4'-DDT	0.05	ug/L	0.0072	ug/L	C	60	140	50	C	24	145	50
1052	Dieldrin	0.05	ug/L	0.0069	ug/L	C	68	130	37	C	35	141	37
1236	Endosulfan I	0.05	ug/L	0.0069	ug/L		27	120	36		25	120	36
1239	Endosulfan II	0.05	ug/L	0.0066	ug/L		33	127	52		35	127	52
1241	Endosulfan sulfate	0.05	ug/L	0.007	ug/L		44	144	40		45	142	40
1270	Endrin	0.05	ug/L	0.0087	ug/L	C	46	137	40	C	28	148	40
1277	Endrin aldehyde	0.05	ug/L	0.0064	ug/L		42	142	54		16	158	54
1279	Endrin ketone	0.05	ug/L	0.0066	ug/L		44	149	44		35	156	44
1470	Heptachlor	0.05	ug/L	0.0071	ug/L	C	57	124	32	C	25	135	32
1479	Heptachlor epoxide	0.05	ug/L	0.0068	ug/L		53	135	31		38	138	31
1741	Methoxychlor	0.1	ug/L	0.0084	ug/L		12	154	29		13	154	29
2499	Toxaphene	2	ug/L	0.23	ug/L								
2732	Decachloro-biphenyl					X	10	147		X	10	147	
2739	Tetrachloro-m-xylene					X	39	130		X	39	130	

TABLE 8.2-4-7
GC Semivolatiles – 8081A
Reporting Limits (RL), Method Detection Limits (MDL), and Control Limits (LCS, MS/MSD, and RPD)[1]

Solid						LCS				MS			
#	Compound	RL	Units	MDL	Units	T	LCL	UCL	RPD	T	LCL	UCL	RPD
60	Aldrin	1.7	ug/kg	0.55	ug/kg	C	39	122	40	C	33	122	40
226	alpha-BHC	1.7	ug/kg	0.46	ug/kg		33	130	40		30	130	40
228	beta-BHC	1.7	ug/kg	0.46	ug/kg		51	110	43		18	116	43
230	delta-BHC	1.7	ug/kg	0.48	ug/kg		19	142	34		16	142	34
232	gamma-BHC (Lindane)	1.7	ug/kg	0.57	ug/kg	C	47	130	36	C	33	130	36
497	alpha-Chlordane	1.7	ug/kg	0.59	ug/kg		39	145	65		26	145	65
499	gamma-Chlordane	1.7	ug/kg	0.57	ug/kg		33	154	36		31	154	36
770	4,4'-DDD	1.7	ug/kg	0.79	ug/kg		39	157	35		19	157	35
777	4,4'-DDE	1.7	ug/kg	0.57	ug/kg		26	157	39		49	157	39
780	4,4'-DDT	1.7	ug/kg	1.3	ug/kg	C	35	144	42	C	23	144	42
1052	Dieldrin	1.7	ug/kg	1.5	ug/kg	C	45	128	33	C	33	133	33
1236	Endosulfan I	1.7	ug/kg	0.67	ug/kg		24	113	41		17	113	41
1239	Endosulfan II	1.7	ug/kg	0.38	ug/kg		35	124	27		21	129	27
1241	Endosulfan sulfate	1.7	ug/kg	0.28	ug/kg		36	139	34		22	139	34
1270	Endrin	1.7	ug/kg	0.61	ug/kg	C	47	133	38	C	33	138	38
1277	Endrin aldehyde	1.7	ug/kg	1.5	ug/kg		27	130	29		18	153	29
1279	Endrin ketone	1.7	ug/kg	0.26	ug/kg		49	137	32		34	137	32
1470	Heptachlor	1.7	ug/kg	0.61	ug/kg	C	39	126	44	C	32	128	44
1479	Heptachlor epoxide	1.7	ug/kg	0.66	ug/kg		46	125	43		33	148	43
1741	Methoxychlor	3.3	ug/kg	0.82	ug/kg		24	161	41		25	164	41
2499	Toxaphene	67	ug/kg	6.9	ug/kg								
2732	Decachloro-biphenyl					X	18	145		X	18	145	
2739	Tetrachloro-m-xylene					X	31	131		X	31	131	

TABLE 8.2-4-8
GC Semivolatiles – Method 8082
Reporting Limits (RL), Method Detection Limits (MDL),
and Control Limits (LCS, MS/MSD, and RPD)[1]

	Water					LCS				MS			
#	Compound	RL	Units	MDL	Units	T	LCL	UCL	RPD	T	LCL	UCL	RPD
2082	Aroclor 1016	1	ug/L	0.018	ug/L	C	61	118	20	C	56	119	20
2085	Aroclor 1221	1	ug/L	0.094	ug/L								
2088	Aroclor 1232	1	ug/L	0.16	ug/L								
2091	Aroclor 1242	1	ug/L	0.3	ug/L								
2094	Aroclor 1248	1	ug/L	0.22	ug/L								
2097	Aroclor 1254	1	ug/L	0.096	ug/L								
2100	Aroclor 1260	1	ug/L	0.065	ug/L	C	61	124	27	C	31	138	27
2732	Decachlorobi-phenyl					X	24	128		X	24	128	
2739	Tetrachloro-m-xylene					X	45	120		X	45	120	

	Solid					LCS				MS			
#	Compound	RL	Units	MDL	Units	T	LCL	UCL	RPD	T	LCL	UCL	RPD
2082	Aroclor 1016	33	ug/kg	5.3	ug/kg	C	49	122	39	C	26	144	39
2085	Aroclor 1221	33	ug/kg	19	ug/kg								
2088	Aroclor 1232	33	ug/kg	11	ug/kg								
2091	Aroclor 1242	33	ug/kg	18	ug/kg								
2094	Aroclor 1248	33	ug/kg	4.6	ug/kg								
2097	Aroclor 1254	33	ug/kg	20	ug/kg								
2100	Aroclor 1260	33	ug/kg	7.4	ug/kg	C	51	127	33	C	37	138	33
2732	Decachloro-biphenyl					X	23	141		X	23	141	
2739	Tetrachloro-m-xylene					X	31	127		X	31	127	

TABLE 8.2-4-9
GC Semivolatiles – Method 608
Reporting Limits (RL), Method Detection Limits (MDL),
and Control Limits (LCS, MS/MSD, and RPD)[1]

Water						LCS				MS			
#	Compound	RL	Units	MDL	Units	T	LCL	UCL	RPD	T	LCL	UCL	RPD
60	Aldrin	0.05	ug/L	0.0064	ug/L	C	60	117	29	C	54	120	40
226	alpha-BHC	0.05	ug/L	0.0059	ug/L		54	130	38	C	37	134	24
228	beta-BHC	0.05	ug/L	0.0062	ug/L		29	147	60	C	17	147	32
230	delta-BHC	0.05	ug/L	0.006	ug/L		35	140	55	C	19	140	36
232	gamma-BHC (Lindane)	0.05	ug/L	0.0062	ug/L	C	63	122	29	C	48	135	51
476	Chlordane (technical)	0.5	ug/L	0.075	ug/L								
770	4,4'-DDD	0.05	ug/L	0.0064	ug/L		56	135	38	C	31	141	28
777	4,4'-DDE	0.05	ug/L	0.0074	ug/L		63	115	26	C	30	145	27
780	4,4'-DDT	0.05	ug/L	0.0072	ug/L	C	55	128	36	C	48	154	47
1052	Dieldrin	0.05	ug/L	0.0069	ug/L	C	63	122	25	C	54	143	32
1236	Endosulfan I	0.05	ug/L	0.0069	ug/L		60	129	34	C	45	153	25
1239	Endosulfan II	0.05	ug/L	0.0066	ug/L		41	147	53	C	10	202	61
1241	Endosulfan sulfate	0.05	ug/L	0.007	ug/L		59	134	38	C	26	144	27
1270	Endrin	0.05	ug/L	0.0087	ug/L	C	48	129	41	C	64	142	39
1277	Endrin aldehyde	0.05	ug/L	0.0064	ug/L								
1470	Heptachlor	0.05	ug/L	0.0071	ug/L	C	56	125	34	C	56	158	36
1479	Heptachlor epoxide	0.05	ug/L	0.0068	ug/L		61	133	36	C	37	142	21
2080	PCB-1016	1	ug/L	0.18	ug/L								
2083	PCB-1221	1	ug/L	0.094	ug/L								
2086	PCB-1232	1	ug/L	0.16	ug/L								
2089	PCB-1242	1	ug/L	0.3	ug/L								
2092	PCB-1248	1	ug/L	0.22	ug/L								
2095	PCB-1254	1	ug/L	0.096	ug/L								
2098	PCB-1260	1	ug/L	0.065	ug/L								
2499	Toxaphene	2	ug/L	0.23	ug/L								
2732	Decachloro-biphenyl					X	10	116	74	X	10	116	74
2739	Tetrachloro-m-xylene					X	10	130	60	X	10	130	60

TABLE 8.2-4-10
GC Semivolatiles – Method 8310
Reporting Limits (RL), Method Detection Limits (MDL),
and Control Limits (LCS, MS/MSD, and RPD)[1]

Water							LCS				MS		
#	Compound	RL	Units	MDL	Units	T	LCL	UCL	RPD	T	LCL	UCL	RPD
1	Acenaphthene	1	ug/L	0.85	ug/L		10	101	48		10	124	50
5	Acenaphthylene	1	ug/L	0.64	ug/L		10	99	49		10	139	50
122	Anthracene	2	ug/L	1.2	ug/L		18	126	59		10	126	50
202	Benzo(a)anthracene	0.1	ug/L	0.096	ug/L		44	116	36		12	135	50
205	Benzo(b)fluoranthene	0.1	ug/L	0.098	ug/L		39	125	43		10	150	50
208	Benzo(k)fluoranthene	0.05	ug/L	0.047	ug/L		38	124	43		10	159	50
210	Benzo(ghi)perylene	0.1	ug/L	0.1	ug/L		23	116	54		10	116	50
211	Benzo(a)pyrene	0.1	ug/L	0.091	ug/L	C	22	128	64	C	10	128	50
633	Chrysene	0.1	ug/L	0.094	ug/L	C	38	118	40	C	10	199	50
860	Dibenz(a,h)anthracene	0.1	ug/L	0.096	ug/L		22	110	54		10	110	50
1414	Fluoranthene	0.1	ug/L	0.098	ug/L	C	43	102	29	C	14	123	50
1417	Fluorene	1	ug/L	0.011	ug/L		13	100	43		10	142	50
1535	Indeno(1,2,3-cd)pyrene	0.1	ug/L	0.093	ug/L	C	36	116	44	C	10	116	50
1932	Naphthalene	2	ug/L	0.024	ug/L	C	10	90	46	C	10	122	50
2154	Phenanthrene	1	ug/L	0.027	ug/L	C	28	113	43	C	10	155	50
2252	Pyrene	0.1	ug/L	0.086	ug/L		38	118	40		10	140	50
213	Benzo(e)pyrene					X	39	182		X	39	182	
2738	Terphenyl-d14					X	33	120		X	33	120	

Solid							LCS				MS		
#	Compound	RL	Units	MDL	Units	T	LCL	UCL	RPD	T	LCL	UCL	RPD
1	Acenaphthene	100	ug/kg	50	ug/kg		15	105	45		10	124	50
5	Acenaphthylene	100	ug/kg	36	ug/kg		10	101	47		10	130	50
122	Anthracene	100	ug/kg	26	ug/kg		37	130	47		10	126	50
202	Benzo(a)anthracene	5	ug/kg	3.5	ug/kg		52	105	27		12	135	50
205	Benzo(b)fluoranthene	5	ug/kg	2.6	ug/kg		46	118	36		10	150	50
208	Benzo(k)fluoranthene	1.7	ug/kg	0.96	ug/kg		48	114	33		10	159	50
210	Benzo(ghi)perylene	10	ug/kg	6.5	ug/kg		37	127	45		10	116	50
211	Benzo(a)pyrene	5	ug/kg	2.1	ug/kg	C	23	126	52	C	10	128	50
633	Chrysene	5	ug/kg	3.1	ug/kg	C	51	107	28	C	10	199	50
860	Dibenz(a,h)anthracene	5	ug/kg	1.9	ug/kg		43	125	41		10	110	50
1414	Fluoranthene	10	ug/kg	5.3	ug/kg	C	46	103	29	C	14	123	50
1417	Fluorene	100	ug/kg	1.7	ug/kg		21	104	42		10	142	50
1535	Indeno(1,2,3-cd)pyrene	5	ug/kg	4.2	ug/kg	C	41	121	40	C	10	116	50
1932	Naphthalene	100	ug/kg	14	ug/kg	C	10	99	48	C	10	122	50
2154	Phenanthrene	100	ug/kg	2.4	ug/kg	C	36	113	39	C	10	155	50
2252	Pyrene	5	ug/kg	4.8	ug/kg		51	108	29		10	140	50
213	Benzo(e)pyrene					X	25	176		X	25	176	
2738	Terphenyl-d14					X	10	141		X	10	141	

TABLE 8.2-4-11
GC Semivolatiles – Method 610
Reporting Limits (RL), Method Detection Limits (MDL),
and Control Limits (LCS, MS/MSD, and RPD)[1]

	Water						LCS				MS		
#	Compound	RL	Units	MDL	Units	T	LCL	UCL	RPD	T	LCL	UCL	RPD
1	Acenaphthene	1	ug/L	0.85	ug/L		10	101	48		10	124	50
5	Acenaphthylene	1	ug/L	0.64	ug/L		10	99	49		10	139	50
122	Anthracene	2	ug/L	1.2	ug/L		18	126	59		10	126	50
202	Benzo(a)anthracene	0.1	ug/L	0.096	ug/L		44	116	36		12	135	50
205	Benzo(b)fluoranthene	0.1	ug/L	0.098	ug/L		39	125	43		10	150	50
208	Benzo(k)fluoranthene	0.05	ug/L	0.047	ug/L		38	124	43		10	159	50
210	Benzo(ghi)perylene	0.1	ug/L	0.1	ug/L		23	116	54		10	116	50
211	Benzo(a)pyrene	0.1	ug/L	0.091	ug/L	C	22	128	64	C	10	128	50
633	Chrysene	0.1	ug/L	0.094	ug/L	C	38	118	40	C	10	199	50
2669	Dibenzo(a,h)anthracene	0.1	ug/L	0.096	ug/L								
1414	Fluoranthene	0.1	ug/L	0.098	ug/L	C	43	102	29	C	14	123	50
1417	Fluorene	1	ug/L	0.011	ug/L		13	100	43		10	142	50
1535	Indeno(1,2,3-cd)pyrene	0.1	ug/L	0.093	ug/L	C	36	116	44	C	10	116	50
1932	Naphthalene	2	ug/L	0.024	ug/L	C	10	90	46	C	10	122	50
2154	Phenanthrene	1	ug/L	0.027	ug/L	C	28	113	43	C	10	155	50
2252	Pyrene	0.1	ug/L	0.086	ug/L		38	118	40		10	140	50
213	Benzo(e)pyrene					X	39	182		X	39	182	
2738	Terphenyl-d14					X	33	120		X	33	120	

TABLE 8.2-4-12
GC Semivolatiles – Method 8141A
Reporting Limits (RL), Method Detection Limits (MDL),
and Control Limits (LCS, MS/MSD, and RPD)[1]

	Water					LCS				MS			
#	Compound	RL	Units	MDL	Units	T	LCL	UCL	RPD	T	LCL	UCL	RPD
1099	Dimethoate	1	ug/L	0.26	ug/L	C	49	124	99	C	55	121	56
1225	Disulfoton	1	ug/L	0.61	ug/L	C	45	112	99	C	45	116	61
1372	Famphur	1	ug/L	0.31	ug/L	C	37	127	99	C	46	123	56
1831	Methyl parathion	1	ug/L	0.68	ug/L	C	45	116	99	C	47	119	57
2062	Parathion	1	ug/L	0.28	ug/L	C	47	119	99	C	53	115	54
2170	Phorate	1	ug/L	0.57	ug/L	C	40	110	99	C	43	112	68
2459	Tetraethyldithiopyro-phosphate	1	ug/L	0.33	ug/L	C	46	118	99	C	50	117	64
1086	Thionazin	1	ug/L	0.32	ug/L	C	41	117	99	C	46	113	70
2569	O,O,O-Triethyl phosphorothioate	1	ug/L	0.36	ug/L	C	38	116	99	C	48	110	65
2600	Triphenyl phosphate					X	42	135		X	42	135	
	Solid					**LCS**				**MS**			
#	**Compound**	**RL**	**Units**	**MDL**	**Units**	**T**	**LCL**	**UCL**	**RPD**	**T**	**LCL**	**UCL**	**RPD**
1099	Dimethoate	33	ug/kg	5.1	ug/kg	C	51	142	44	C	32	196	43
1225	Disulfoton	33	ug/kg	5.2	ug/kg	C	40	115	71	C	37	122	99
1372	Famphur	33	ug/kg	4.5	ug/kg	C	42	137	48	C	34	165	47
1831	Methyl parathion	33	ug/kg	6.6	ug/kg	C	45	130	48	C	50	140	50
2062	Parathion	33	ug/kg	4	ug/kg	C	42	137	48	C	46	148	52
2170	Phorate	33	ug/kg	5.5	ug/kg	C	41	113	47	C	37	123	63
2459	Tetraethyldithio-pyrophosphate	33	ug/kg	11	ug/kg	C	41	126	49	C	50	131	68
1086	Thionazin	33	ug/kg	4.1	ug/kg	C	40	128	47	C	48	139	53
2569	O,O,O-Triethyl phosphorothioate	33	ug/kg	3.5	ug/kg	C	40	120	47	C	50	121	48
2600	Triphenyl phosphate					X	54	143		X	54	143	

TABLE 8.2-4-13
GC Semivolatiles – Method 8151A
Reporting Limits (RL), Method Detection Limits (MDL), and Control Limits (LCS, MS/MSD, and RPD)[1]

Water						LCS				MS			
#	Compound	RL	Units	MDL	Units	T	LCL	UCL	RPD	T	LCL	UCL	RPD
690	2,4-D	4	ug/L	0.077	ug/L	C	26	108	20	C	52	102	18
753	Dalapon	2	ug/L	0.39	ug/L		30	130	50		30	130	50
766	2,4-DB	4	ug/L	0.33	ug/L		28	141	50		28	141	50
897	Dicamba	2	ug/L	0.059	ug/L		30	130	50		30	130	50
975	Dichlorprop	4	ug/L	0.089	ug/L		30	130	50		30	130	50
1195	Dinoseb	0.6	ug/L	0.013	ug/L		74	98	50		74	98	50
1661	MCPA	400	ug/L	8	ug/L		30	130	50		30	130	50
1680	MCPP	400	ug/L	25	ug/L		30	130	50		30	130	50
2291	2,4,5-TP (Silvex)	1	ug/L	0.036	ug/L								
2384	2,4,5-T	1	ug/L	0.043	ug/L	C	41	109	20	C	67	95	16
2924	2,4-Dichlorophenylacetic acid					X	36	109		X	36	109	

Solid						LCS				MS			
#	Compound	RL	Units	MDL	Units	T	LCL	UCL	RPD	T	LCL	UCL	RPD
690	2,4-D	80	ug/kg	53	ug/kg	C	10	110	62	C	10	113	62
753	Dalapon	40	ug/kg	37	ug/kg		30	130	50		30	130	50
766	2,4-DB	80	ug/kg	39	ug/kg		38	119	30		38	119	
897	Dicamba	40	ug/kg	15	ug/kg		30	130	50		30	130	50
975	Dichlorprop	80	ug/kg	10	ug/kg		30	130	50		30	130	50
1195	Dinoseb	12	ug/kg	12	ug/kg		74	98	50		74	98	50
1661	MCPA	8000	ug/kg	2300	ug/kg		30	130	50		30	130	50
1680	MCPP	8000	ug/kg	2700	ug/kg		30	130	50		30	130	50
2291	2,4,5-TP (Silvex)	20	ug/kg	3.9	ug/kg								
2384	2,4,5-T	20	ug/kg	3.1	ug/kg	C	17	117	66	C	10	122	66
2924	2,4-Dichlorophenylacetic acid					X	10	115		X	10	115	

TABLE 8.2-4-14
GC Volatiles - Method 8021B
Reporting Limits (RL), Method Detection Limits (MDL),
and Control Limits (LCS, MS/MSD, and RPD)[1]

Water - 5mL Purge						LCS				MS			
#	Compound	RL	Units	MDL	Units	T	LCL	UCL	RPD	T	LCL	UCL	RPD
196	Benzene	1	ug/L	0.29	ug/L	C	73	134	20	C	55	161	25
318	Bromobenzene	1	ug/L	0.33	ug/L								
321	Bromochloromethane	1	ug/L	0.4	ug/L								
323	Bromodichloromethane	1	ug/L	0.59	ug/L								
340	Bromoform	1	ug/L	0.33	ug/L								
343	Bromomethane	1	ug/L	0.55	ug/L								
393	n-Butylbenzene	1	ug/L	0.56	ug/L								
395	sec-Butylbenzene	1	ug/L	0.1	ug/L								
398	tert-Butylbenzene	1	ug/L	0.83	ug/L								
463	Carbon tetrachloride	1	ug/L	0.55	ug/L								
521	Chlorobenzene	1	ug/L	0.48	ug/L	C	61	134	20	C	42	147	23
534	Chlorodibromomethane	1	ug/L	0.34	ug/L								
550	Chloroethane	1	ug/L	0.7	ug/L								
569	Chloroform	1	ug/L	0.4	ug/L								
574	Chloromethane	1	ug/L	0.52	ug/L								
614	2-Chlorotoluene	1	ug/L	0.53	ug/L								
617	4-Chlorotoluene	1	ug/L	0.66	ug/L								
3260	1,2-Dibromo-3-chloropropane (DBCP)	1	ug/L	0.6	ug/L								
3261	1,2-Dibromoethane (EDB)	1	ug/L	0.43	ug/L								
888	Dibromomethane	1	ug/L	0.29	ug/L								
904	1,2-Dichlorobenzene	1	ug/L	0.48	ug/L								
907	1,3-Dichlorobenzene	1	ug/L	0.43	ug/L								
910	1,4-Dichlorobenzene	1	ug/L	0.57	ug/L								
924	Dichlorodifluoromethane	1	ug/L	0.29	ug/L								
933	1,1-Dichloroethane	1	ug/L	0.45	ug/L								
936	1,2-Dichloroethane	1	ug/L	0.32	ug/L								
948	cis-1,2-Dichloroethene	1	ug/L	0.39	ug/L								
950	trans-1,2-Dichloroethene	1	ug/L	0.43	ug/L								
943	1,1-Dichloroethene	1	ug/L	0.46	ug/L	C	35	127	20	C	14	151	28
986	1,2-Dichloropropane	1	ug/L	0.36	ug/L								
989	1,3-Dichloropropane	1	ug/L	0.48	ug/L								
990	2,2-Dichloropropane	1	ug/L	0.32	ug/L								
998	cis-1,3-Dichloropropene	1	ug/L	0.39	ug/L								
1000	trans-1,3-Dichloropropene	1	ug/L	0.37	ug/L								
996	1,1-Dichloropropene	1	ug/L	0.62	ug/L								
1332	Ethylbenzene	1	ug/L	0.68	ug/L								

TABLE 8.2-4-14
GC Volatiles - Method 8021B
Reporting Limits (RL), Method Detection Limits (MDL),
and Control Limits (LCS, MS/MSD, and RPD)[1]

Water - 5mL Purge						LCS				MS			
#	Compound	RL	Units	MDL	Units	T	LCL	UCL	RPD	T	LCL	UCL	RPD
1489	Hexachlorobutadiene	1	ug/L	0.62	ug/L								
1578	Isopropylbenzene	1	ug/L	0.83	ug/L								
1590	p-Isopropyltoluene	1	ug/L	0.7	ug/L								
1811	Methylene chloride	5	ug/L	0.48	ug/L								
1932	Naphthalene	1	ug/L	0.3	ug/L								
2247	n-Propylbenzene	1	ug/L	0.76	ug/L								
2355	Styrene	1	ug/L	0.4	ug/L								
2437	1,1,1,2-Tetrachloroethane	1	ug/L	0.35	ug/L								
2439	1,1,2,2-Tetrachloroethane	1	ug/L	0.97	ug/L								
2445	Tetrachloroethene	1	ug/L	0.72	ug/L								
2489	Toluene	1	ug/L	0.56	ug/L	C	71	132	20	C	55	159	25
2514	1,2,3-Trichlorobenzene	1	ug/L	0.32	ug/L								
2515	1,2,4-Trichlorobenzene	1	ug/L	0.25	ug/L								
2518	1,1,1-Trichloroethane	1	ug/L	0.46	ug/L								
2522	1,1,2-Trichloroethane	1	ug/L	0.4	ug/L								
2525	Trichloroethene	1	ug/L	0.61	ug/L	C	58	131	20	C	10	229	41
1428	Trichlorofluoromethane	1	ug/L	0.34	ug/L								
2563	1,2,3-Trichloropropane	1	ug/L	0.51	ug/L								
2587	1,2,4-Trimethylbenzene	1	ug/L	0.58	ug/L								
2592	1,3,5-Trimethylbenzene	1	ug/L	0.49	ug/L								
2613	Vinyl chloride	1	ug/L	0.71	ug/L								
2627	Xylenes (total)	1	ug/L	0.93	ug/L								
2734	1,4-Dichlorobutane					X	50	150		X	50	150	
2741	Trifluorotoluene					X	50	150		X	50	150	

TABLE 8.2-4-14
GC Volatiles - Method 8021B
Reporting Limits (RL), Method Detection Limits (MDL),
and Control Limits (LCS, MS/MSD, and RPD)[1]

Solid - 5 mL						LCS				MS			
#	Compound	RL	Units	MDL	Units	T	LCL	UCL	RPD	T	LCL	UCL	RPD
196	Benzene	1	ug/kg	0.12	ug/kg	C	69	132	20	C	62	150	33
318	Bromobenzene	1	ug/kg	0.39	ug/kg								
321	Bromochloromethane	1	ug/kg	0.37	ug/kg								
323	Bromodichloromethane	1	ug/kg	0.31	ug/kg								
340	Bromoform	1	ug/kg	0.74	ug/kg								
343	Bromomethane	1	ug/kg	0.22	ug/kg								
393	n-Butylbenzene	1	ug/kg	0.46	ug/kg								
395	sec-Butylbenzene	1	ug/kg	0.47	ug/kg								
398	tert-Butylbenzene	1	ug/kg	0.57	ug/kg								
463	Carbon tetrachloride	1	ug/kg	0.14	ug/kg								
521	Chlorobenzene	1	ug/kg	0.6	ug/kg	C	51	131	20	C	10	142	32
534	Chlorodibromomethane	1	ug/kg	0.52	ug/kg								
550	Chloroethane	1	ug/kg	0.18	ug/kg								
569	Chloroform	1	ug/kg	0.13	ug/kg								
574	Chloromethane	1	ug/kg	0.99	ug/kg								
614	2-Chlorotoluene	1	ug/kg	0.42	ug/kg								
617	4-Chlorotoluene	1	ug/kg	0.53	ug/kg								
3260	1,2-Dibromo-3-chloropropane (DBCP)	1	ug/kg										
3261	1,2-Dibromoethane (EDB)	1	ug/kg	0.24	ug/kg								
888	Dibromomethane	1	ug/kg	0.12	ug/kg								
904	1,2-Dichlorobenzene	1	ug/kg	0.55	ug/kg								
907	1,3-Dichlorobenzene	1	ug/kg	0.48	ug/kg								
910	1,4-Dichlorobenzene	1	ug/kg	0.62	ug/kg								
924	Dichlorodifluoromethane	1	ug/kg	0.31	ug/kg								
933	1,1-Dichloroethane	1	ug/kg	0.11	ug/kg								
936	1,2-Dichloroethane	1	ug/kg	0.12	ug/kg								
948	cis-1,2-Dichloroethene	1	ug/kg	0.11	ug/kg								
950	trans-1,2-Dichloroethene	1	ug/kg	0.11	ug/kg								
943	1,1-Dichloroethene	1	ug/kg	0.28	ug/kg	C	29	140	20	C	10	203	38
986	1,2-Dichloropropane	1	ug/kg	0.2	ug/kg								
989	1,3-Dichloropropane	1	ug/kg	0.42	ug/kg								
990	2,2-Dichloropropane	1	ug/kg	0.22	ug/kg								
998	cis-1,3-Dichloropropene	1	ug/kg	0.29	ug/kg								
1000	trans-1,3-Dichloropropene	1	ug/kg	0.38	ug/kg								
996	1,1-Dichloropropene	1	ug/kg	0.14	ug/kg								
1332	Ethylbenzene	1	ug/kg	0.38	ug/kg								

TABLE 8.2-4-14
GC Volatiles - Method 8021B
Reporting Limits (RL), Method Detection Limits (MDL),
and Control Limits (LCS, MS/MSD, and RPD)[1]

Solid - 5 mL						LCS				MS			
#	Compound	RL	Units	MDL	Units	T	LCL	UCL	RPD	T	LCL	UCL	RPD
1489	Hexachlorobutadiene	1	ug/kg	0.5	ug/kg								
1578	Isopropylbenzene	1	ug/kg	0.52	ug/kg								
1590	p-Isopropyltoluene	1	ug/kg	0.65	ug/kg								
1811	Methylene chloride	5	ug/kg	0.25	ug/kg								
1932	Naphthalene	1	ug/kg	0.39	ug/kg								
2247	n-Propylbenzene	1	ug/kg	0.44	ug/kg								
2355	Styrene	1	ug/kg	0.31	ug/kg								
2437	1,1,1,2-Tetrachloroethane	1	ug/kg	0.55	ug/kg								
2439	1,1,2,2-Tetrachloroethane	1	ug/kg	0.19	ug/kg								
2445	Tetrachloroethene	1	ug/kg	0.42	ug/kg								
2489	Toluene	1	ug/kg	0.17	ug/kg	C	66	129	20	C	50	142	40
2514	1,2,3-Trichlorobenzene	1	ug/kg	0.54	ug/kg								
2515	1,2,4-Trichlorobenzene	1	ug/kg	0.53	ug/kg								
2518	1,1,1-Trichloroethane	1	ug/kg	0.15	ug/kg								
2522	1,1,2-Trichloroethane	1	ug/kg	0.52	ug/kg								
2525	Trichloroethene	1	ug/kg	0.18	ug/kg	C	10	216	20	C	10	216	45
1428	Trichlorofluoromethane	1	ug/kg	0.13	ug/kg								
2563	1,2,3-Trichloropropane	1	ug/kg	0.68	ug/kg								
2587	1,2,4-Trimethylbenzene	1	ug/kg	0.33	ug/kg								
2592	1,3,5-Trimethylbenzene	1	ug/kg	0.32	ug/kg								
2613	Vinyl chloride	1	ug/kg	0.48	ug/kg								
2627	Xylenes (total)	1	ug/kg	0.39	ug/kg								
2734	1,4-Dichlorobutane					X	50	150		X	50	150	
2741	Trifluorotoluene					X	50	150		X	50	150	

TABLE 8.2-4-14
GC Volatiles – Method 8021B
Reporting Limits (RL), Method Detection Limits (MDL),
and Control Limits (LCS, MS/MSD, and RPD)[1]

Solid - Encore						LCS				MS			
#	Compound	RL	Units	MDL	Units	T	LCL	UCL	RPD	T	LCL	UCL	RPD
196	Benzene	50	ug/kg	4.6	ug/kg	C	69	132	20	C	62	150	33
318	Bromobenzene	50	ug/kg	9.7	ug/kg								
321	Bromochloromethane	50	ug/kg	9.7	ug/kg								
323	Bromodichloromethane	50	ug/kg	5.5	ug/kg								
340	Bromoform	50	ug/kg	8.9	ug/kg								
343	Bromomethane	50	ug/kg	15	ug/kg								
393	n-Butylbenzene	50	ug/kg	5.9	ug/kg								
395	sec-Butylbenzene	50	ug/kg	5.5	ug/kg								
398	tert-Butylbenzene	50	ug/kg	5.6	ug/kg								
463	Carbon tetrachloride	50	ug/kg	16	ug/kg								
521	Chlorobenzene	50	ug/kg	6.7	ug/kg	C	51	131	20	C	10	142	32
534	Chlorodibromomethane	50	ug/kg	7.5	ug/kg								
550	Chloroethane	50	ug/kg	24	ug/kg								
569	Chloroform	50	ug/kg	6.9	ug/kg								
574	Chloromethane	50	ug/kg	8.4	ug/kg								
614	2-Chlorotoluene	50	ug/kg	9	ug/kg								
617	4-Chlorotoluene	50	ug/kg	11	ug/kg								
3260	1,2-Dibromo-3-chloropropane (DBCP)	50	ug/kg										
3261	1,2-Dibromoethane (EDB)	50	ug/kg	8.9	ug/kg								
888	Dibromomethane	50	ug/kg	6.3	ug/kg								
904	1,2-Dichlorobenzene	50	ug/kg	6.3	ug/kg								
907	1,3-Dichlorobenzene	50	ug/kg	8	ug/kg								
910	1,4-Dichlorobenzene	50	ug/kg	9.1	ug/kg								
924	Dichlorodifluoromethane	50	ug/kg	7.1	ug/kg								
933	1,1-Dichloroethane	50	ug/kg	8.7	ug/kg								
936	1,2-Dichloroethane	50	ug/kg	11	ug/kg								
948	cis-1,2-Dichloroethene	50	ug/kg	15	ug/kg								
950	trans-1,2-Dichloroethene	50	ug/kg	8.4	ug/kg								
943	1,1-Dichloroethene	50	ug/kg	6	ug/kg	C	29	140	20	C	10	203	38
986	1,2-Dichloropropane	50	ug/kg	9.5	ug/kg								
989	1,3-Dichloropropane	50	ug/kg	0.42	ug/kg								
990	2,2-Dichloropropane	50	ug/kg	15	ug/kg								
998	cis-1,3-Dichloropropene	50	ug/kg	7.3	ug/kg								
1000	trans-1,3-Dichloropropene	50	ug/kg	4.9	ug/kg								
996	1,1-Dichloropropene	50	ug/kg	5.4	ug/kg								
1332	Ethylbenzene	50	ug/kg	5.5	ug/kg								

TABLE 8.2-4-14
GC Volatiles – Method 8021B
Reporting Limits (RL), Method Detection Limits (MDL),
and Control Limits (LCS, MS/MSD, and RPD)[1]

Solid - Encore						LCS				MS			
#	Compound	RL	Units	MDL	Units	T	LCL	UCL	RPD	T	LCL	UCL	RPD
1489	Hexachlorobutadiene	50	ug/kg	7.5	ug/kg								
1578	Isopropylbenzene	50	ug/kg	4.7	ug/kg								
1590	p-Isopropyltoluene	50	ug/kg	11	ug/kg								
1811	Methylene chloride	250	ug/kg	8.8	ug/kg								
1932	Naphthalene	250	ug/kg	5.6	ug/kg								
2247	n-Propylbenzene	50	ug/kg	5.1	ug/kg								
2355	Styrene	50	ug/kg	10	ug/kg								
2437	1,1,1,2-Tetrachloroethane	50	ug/kg	7.2	ug/kg								
2439	1,1,2,2-Tetrachloroethane	50	ug/kg	16	ug/kg								
2445	Tetrachloroethene	50	ug/kg	6.8	ug/kg								
2489	Toluene	50	ug/kg	4.8	ug/kg	C	66	129	20	C	50	142	40
2514	1,2,3-Trichlorobenzene	50	ug/kg	16	ug/kg								
2515	1,2,4-Trichlorobenzene	50	ug/kg	7.5	ug/kg								
2518	1,1,1-Trichloroethane	50	ug/kg	9	ug/kg								
2522	1,1,2-Trichloroethane	50	ug/kg	5.1	ug/kg								
2525	Trichloroethene	50	ug/kg	9	ug/kg	C	10	216	20	C	10	216	45
1428	Trichlorofluoromethane	50	ug/kg	8.5	ug/kg								
2563	1,2,3-Trichloropropane	50	ug/kg	0.68	ug/kg								
2587	1,2,4-Trimethylbenzene	50	ug/kg	5.9	ug/kg								
2592	1,3,5-Trimethylbenzene	50	ug/kg	7.8	ug/kg								
2613	Vinyl chloride	50	ug/kg	1.23	ug/kg								
2627	Xylenes (total)	50	ug/kg	1.23	ug/kg								
2734	1,4-Dichlorobutane					X	50	150		X	50	150	
2741	Trifluorotoluene					X	50	150		X	50	150	

TABLE 8.2-4-14
GC Volatiles – Method 8021B
Reporting Limits (RL), Method Detection Limits (MDL), and Control Limits (LCS, MS/MSD, and RPD)[1]

Low Level Solid Encore						LCS				MS			
#	Compound	RL	Units	MDL	Units	T	LCL	UCL	RPD	T	LCL	UCL	RPD
196	Benzene	1	ug/kg	0.27	ug/kg	C	69	132	20	C	62	150	33
318	Bromobenzene	1	ug/kg	0.45	ug/kg								
321	Bromochloromethane	1	ug/kg	0.17	ug/kg								
323	Bromodichloromethane	1	ug/kg	0.23	ug/kg								
340	Bromoform	1	ug/kg	0.35	ug/kg								
343	Bromomethane	1	ug/kg	0.16	ug/kg								
393	n-Butylbenzene	1	ug/kg	0.45	ug/kg								
395	sec-Butylbenzene	1	ug/kg	0.47	ug/kg								
398	tert-Butylbenzene	1	ug/kg	0.47	ug/kg								
463	Carbon tetrachloride	1	ug/kg	0.4	ug/kg								
521	Chlorobenzene	1	ug/kg	0.42	ug/kg	C	51	131	20	C	10	142	32
534	Chlorodibromomethane	1	ug/kg	0.32	ug/kg								
550	Chloroethane	1	ug/kg	0.15	ug/kg								
569	Chloroform	1	ug/kg	0.26	ug/kg								
574	Chloromethane	1	ug/kg	0.15	ug/kg								
614	2-Chlorotoluene	1	ug/kg	0.41	ug/kg								
617	4-Chlorotoluene	1	ug/kg	0.52	ug/kg								
3260	1,2-Dibromo-3-chloropropane (DBCP)	1	ug/kg										
3261	1,2-Dibromoethane (EDB)	1	ug/kg	0.24	ug/kg								
888	Dibromomethane	1	ug/kg	0.24	ug/kg								
904	1,2-Dichlorobenzene	1	ug/kg	0.43	ug/kg								
907	1,3-Dichlorobenzene	1	ug/kg	0.45	ug/kg								
910	1,4-Dichlorobenzene	1	ug/kg	0.96	ug/kg								
924	Dichlorodifluoromethane	1	ug/kg	0.18	ug/kg								
933	1,1-Dichloroethane	1	ug/kg	0.18	ug/kg								
936	1,2-Dichloroethane	1	ug/kg	0.21	ug/kg								
948	cis-1,2-Dichloroethene	1	ug/kg	0.67	ug/kg								
950	trans-1,2-Dichloroethene	1	ug/kg	0.21	ug/kg								
943	1,1-Dichloroethene	1	ug/kg	0.2	ug/kg	C	29	140	20	C	10	203	38
986	1,2-Dichloropropane	1	ug/kg	0.2	ug/kg								
989	1,3-Dichloropropane	1	ug/kg	0.17	ug/kg								
990	2,2-Dichloropropane	1	ug/kg	0.45	ug/kg								
998	cis-1,3-Dichloropropene	1	ug/kg	0.55	ug/kg								
1000	trans-1,3-Dichloropropene	1	ug/kg	0.54	ug/kg								
996	1,1-Dichloropropene	1	ug/kg	0.4	ug/kg								
1332	Ethylbenzene	1	ug/kg	0.65	ug/kg								

TABLE 8.2-4-14
GC Volatiles – Method 8021B
Reporting Limits (RL), Method Detection Limits (MDL),
and Control Limits (LCS, MS/MSD, and RPD)[1]

Low Level Solid Encore						LCS				MS			
#	Compound	RL	Units	MDL	Units	T	LCL	UCL	RPD	T	LCL	UCL	RPD
1489	Hexachlorobutadiene	1	ug/kg	0.48	ug/kg								
1578	Isopropylbenzene	1	ug/kg	0.56	ug/kg								
1590	p-Isopropyltoluene	1	ug/kg	0.96	ug/kg								
1811	Methylene chloride	5	ug/kg	0.15	ug/kg								
1932	Naphthalene	1	ug/kg	0.71	ug/kg								
2247	n-Propylbenzene	1	ug/kg	0.43	ug/kg								
2355	Styrene	1	ug/kg	0.31	ug/kg								
2437	1,1,1,2-Tetrachloroethane	1	ug/kg	0.18	ug/kg								
2439	1,1,2,2-Tetrachloroethane	1	ug/kg	0.88	ug/kg								
2445	Tetrachloroethene	1	ug/kg	0.42	ug/kg								
2489	Toluene	1	ug/kg	0.42	ug/kg	C	66	129	20	C	50	142	40
2514	1,2,3-Trichlorobenzene	1	ug/kg	0.88	ug/kg								
2515	1,2,4-Trichlorobenzene	1	ug/kg	0.44	ug/kg								
2518	1,1,1-Trichloroethane	1	ug/kg	0.22	ug/kg								
2522	1,1,2-Trichloroethane	1	ug/kg	0.31	ug/kg								
2525	Trichloroethene	1	ug/kg	0.38	ug/kg	C	10	216	20	C	10	216	45
1428	Trichlorofluoromethane	1	ug/kg	0.17	ug/kg								
2563	1,2,3-Trichloropropane	1	ug/kg	0.68	ug/kg								
2587	1,2,4-Trimethylbenzene	1	ug/kg	0.82	ug/kg								
2592	1,3,5-Trimethylbenzene	1	ug/kg	0.32	ug/kg								
2613	Vinyl chloride	1	ug/kg	0.14	ug/kg								
2627	Xylenes (total)	1	ug/kg	0.39	ug/kg								
2734	1,4-Dichlorobutane					X	50	150		X	50	150	
2741	Trifluorotoluene					X	50	150		X	50	150	

TABLE 8.2-4-14
GC Volatiles – Method 8021B
Reporting Limits (RL), Method Detection Limits (MDL),
and Control Limits (LCS, MS/MSD, and RPD)[1]

Frozen Encore						LCS				MS			
#	Compound	RL	Units	MDL	Units	T	LCL	UCL	RPD	T	LCL	UCL	RPD
196	Benzene	1	ug/kg	0.12	ug/kg	C	69	132	20	C	62	150	33
318	Bromobenzene	1	ug/kg	0.39	ug/kg								
321	Bromochloromethane	1	ug/kg	0.37	ug/kg								
323	Bromodichloromethane	1	ug/kg	0.31	ug/kg								
340	Bromoform	1	ug/kg	0.74	ug/kg								
343	Bromomethane	1	ug/kg	0.22	ug/kg								
393	n-Butylbenzene	1	ug/kg	0.46	ug/kg								
395	sec-Butylbenzene	1	ug/kg	0.47	ug/kg								
398	tert-Butylbenzene	1	ug/kg	0.57	ug/kg								
463	Carbon tetrachloride	1	ug/kg	0.14	ug/kg								
521	Chlorobenzene	1	ug/kg	0.6	ug/kg	C	51	131	20	C	10	142	32
534	Chlorodibromomethane	1	ug/kg	0.52	ug/kg								
550	Chloroethane	1	ug/kg	0.18	ug/kg								
569	Chloroform	1	ug/kg	0.13	ug/kg								
574	Chloromethane	1	ug/kg	0.99	ug/kg								
614	2-Chlorotoluene	1	ug/kg	0.42	ug/kg								
617	4-Chlorotoluene	1	ug/kg	0.53	ug/kg								
3260	1,2-Dibromo-3-chloropropane (DBCP)	1	ug/kg										
3261	1,2-Dibromoethane (EDB)	1	ug/kg	0.24	ug/kg								
888	Dibromomethane	1	ug/kg	0.12	ug/kg								
904	1,2-Dichlorobenzene	1	ug/kg	0.55	ug/kg								
907	1,3-Dichlorobenzene	1	ug/kg	0.48	ug/kg								
910	1,4-Dichlorobenzene	1	ug/kg	0.62	ug/kg								
924	Dichlorodifluoromethane	1	ug/kg	0.31	ug/kg								
933	1,1-Dichloroethane	1	ug/kg	0.11	ug/kg								
936	1,2-Dichloroethane	1	ug/kg	0.12	ug/kg								
948	cis-1,2-Dichloroethene	1	ug/kg	0.11	ug/kg								
950	trans-1,2-Dichloroethene	1	ug/kg	0.11	ug/kg								
943	1,1-Dichloroethene	1	ug/kg	0.28	ug/kg	C	29	140	20	C	10	203	38
986	1,2-Dichloropropane	1	ug/kg	0.2	ug/kg								
989	1,3-Dichloropropane	1	ug/kg	0.42	ug/kg								
990	2,2-Dichloropropane	1	ug/kg	0.22	ug/kg								
998	cis-1,3-Dichloropropene	1	ug/kg	0.29	ug/kg								
1000	trans-1,3-Dichloropropene	1	ug/kg	0.38	ug/kg								
996	1,1-Dichloropropene	1	ug/kg	0.14	ug/kg								
1332	Ethylbenzene	1	ug/kg	0.38	ug/kg								

TABLE 8.2-4-14
GC Volatiles – Method 8021B
Reporting Limits (RL), Method Detection Limits (MDL),
and Control Limits (LCS, MS/MSD, and RPD)[1]

Frozen Encore #	Compound	RL	Units	MDL	Units	LCS T	LCS LCL	LCS UCL	LCS RPD	MS T	MS LCL	MS UCL	MS RPD
1489	Hexachlorobutadiene	1	ug/kg	0.5	ug/kg								
1578	Isopropylbenzene	1	ug/kg	0.52	ug/kg								
1590	p-Isopropyltoluene	1	ug/kg	0.65	ug/kg								
1811	Methylene chloride	5	ug/kg	0.25	ug/kg								
1932	Naphthalene	1	ug/kg	0.39	ug/kg								
2247	n-Propylbenzene	1	ug/kg	0.44	ug/kg								
2355	Styrene	1	ug/kg	0.31	ug/kg								
2437	1,1,1,2-Tetrachloroethane	1	ug/kg	0.55	ug/kg								
2439	1,1,2,2-Tetrachloroethane	1	ug/kg	0.19	ug/kg								
2445	Tetrachloroethene	1	ug/kg	0.42	ug/kg								
2489	Toluene	1	ug/kg	0.17	ug/kg	C	66	129	20	C	50	142	40
2514	1,2,3-Trichlorobenzene	1	ug/kg	0.54	ug/kg								
2515	1,2,4-Trichlorobenzene	1	ug/kg	0.53	ug/kg								
2518	1,1,1-Trichloroethane	1	ug/kg	0.15	ug/kg								
2522	1,1,2-Trichloroethane	1	ug/kg	0.52	ug/kg								
2525	Trichloroethene	1	ug/kg	0.18	ug/kg	C	10	216	20	C	10	216	45
1428	Trichlorofluoromethane	1	ug/kg	0.13	ug/kg								
2563	1,2,3-Trichloropropane	1	ug/kg	0.68	ug/kg								
2587	1,2,4-Trimethylbenzene	1	ug/kg	0.33	ug/kg								
2592	1,3,5-Trimethylbenzene	1	ug/kg	0.32	ug/kg								
2613	Vinyl chloride	1	ug/kg	0.48	ug/kg								
2627	Xylenes (total)	1	ug/kg	0.39	ug/kg								
2734	1,4-Dichlorobutane					X	50	150		X	50	150	
2741	Trifluorotoluene					X	50	150		X	50	150	

TABLE 8.2-4-15
GC Volatiles – Method 601
Reporting Limits (RL), Method Detection Limits (MDL),
and Control Limits (LCS, MS/MSD, and RPD)[1]

Water						LCS				MS			
#	Compound	RL	Units	MDL	Units	T	LCL	UCL	RPD	T	LCL	UCL	RPD
323	Bromodichloromethane	1	ug/L	0.59	ug/L	C	10	225		C	10	225	
340	Bromoform	1	ug/L	0.33	ug/L	C	36	158		C	36	158	
343	Bromomethane	1	ug/L	0.55	ug/L	C	10	196		C	10	196	
463	Carbon tetrachloride	1	ug/L	0.55	ug/L	C	30	157		C	30	157	
521	Chlorobenzene	1	ug/L	0.48	ug/L	C	61	134		C	42	147	
535	Dibromochloromethane	1	ug/L	0.34	ug/L	C	51	157		C	51	157	
550	Chloroethane	1	ug/L	0.7	ug/L	C	10	181		C	10	181	
568	2-Chloroethyl vinyl ether	5	ug/L	0.35	ug/L	C	10	226		C	10	226	
569	Chloroform	1	ug/L	0.4	ug/L	C	26	177		C	26	177	
574	Chloromethane	1	ug/L	0.52	ug/L	C	10	209		C	10	209	
904	1,2-Dichlorobenzene	1	ug/L	0.48	ug/L	C	31	146		C	31	146	
907	1,3-Dichlorobenzene	1	ug/L	0.43	ug/L	C	27	147		C	27	147	
910	1,4-Dichlorobenzene	1	ug/L	0.57	ug/L	C	34	170		C	34	170	
924	Dichlorodifluoromethane	1	ug/L	0.29	ug/L								
933	1,1-Dichloroethane	1	ug/L	0.45	ug/L	C	21	167		C	21	167	
936	1,2-Dichloroethane	1	ug/L	0.32	ug/L	C	36	158		C	36	158	
950	trans-1,2-Dichloroethene	1	ug/L	0.43	ug/L	C	10	166		C	10	166	
943	1,1-Dichloroethene	1	ug/L	0.46	ug/L	C	35	127		C	14	151	
986	1,2-Dichloropropane	1	ug/L	0.36	ug/L	C	43	171		C	43	171	
998	cis-1,3-Dichloropropene	1	ug/L	0.39	ug/L	C	30	150		C	30	150	
1000	trans-1,3-Dichloropropene	1	ug/L	0.37	ug/L	C	22	178		C	22	178	
1811	Methylene chloride	5	ug/L	0.48	ug/L	C	10	153		C	10	153	
2439	1,1,2,2-Tetrachloroethane	1	ug/L	0.97	ug/L	C	28	212		C	28	212	
2445	Tetrachloroethene	1	ug/L	0.72	ug/L	C	10	212		C	10	212	
2518	1,1,1-Trichloroethane	1	ug/L	0.46	ug/L	C	32	171		C	32	171	
2522	1,1,2-Trichloroethane	1	ug/L	0.4	ug/L	C	49	155		C	49	155	
2525	Trichloroethene	1	ug/L	0.61	ug/L	C	58	131		C	10	229	
1428	Trichlorofluoromethane	1	ug/L	0.34	ug/L	C	10	157		C	10	157	
2613	Vinyl chloride	1	ug/L	0.71	ug/L	C	10	205		C	10	205	
2734	1,4-Dichlorobutane					X	76	145		X	76	145	
2741	Trifluorotoluene					X	76	121		X	76	121	
2760	Fluorobenzene					X	92	114		X	92	114	

TABLE 8.2-4-15
GC Volatiles – Method 602
Reporting Limits (RL), Method Detection Limits (MDL),
and Control Limits (LCS MS/MSD, and RPD)[1]

602 #	Water Compound	RL	Units	MDL	Units	LCS T	LCS LCL	LCS UCL	LCS RPD	MS T	MS LCL	MS UCL	MS RPD
196	Benzene	1	ug/L	0.29	ug/L	C	73	134	25	C	55	161	25
521	Chlorobenzene	1	ug/L	0.48	ug/L								
904	1,2-Dichlorobenzene	1	ug/L	0.48	ug/L								
907	1,3-Dichlorobenzene	1	ug/L	0.43	ug/L								
910	1,4-Dichlorobenzene	1	ug/L	0.57	ug/L								
1332	Ethylbenzene	1	ug/L	0.68	ug/L	C	51	142	20	C	51	142	10
2489	Toluene	1	ug/L	0.56	ug/L	C	71	132	25	C	55	159	25
321	Bromochloromethane					X	54	112		X	54	112	
2734	1,4-Dichlorobutane					X	76	145		X	76	145	
2741	Trifluorotoluene					X	76	121		X	76	121	
2760	Fluorobenzene					X	92	114		X	84	112	

TABLE 8.2-4-16
Total Petroleum Hydrocarbons, Diesel & Gasoline Range Organics
Reporting Limits (RL), Method Detection Limits (MDL),
and Control Limits (LCS, MS/MSD, and RPD)(1)

							LCS				MS		
DRO - Water													
#	Compound	RL	Units	MDL	Units	T	LCL	UCL	RPD	T	LCL	UCL	RPD
3052	TPH (as Diesel)	100	ug/L	92	ug/L	C	66	111	36	C	70	130	50
2976	C9 (nonane)					X	10	110		X	10	110	
DRO - Solid							LCS				MS		
#	Compound	RL	Units	MDL	Units	T	LCL	UCL	RPD	T	LCL	UCL	RPD
3052	TPH (as Diesel)	10	mg/kg	2.5	mg/kg	C	64	125	30	C	70	130	50
2976	C9 (nonane)					X	10	110		X	10	110	
GRO - Water							LCS				MS		
#	Compound	RL	Units	MDL	Units	T	LCL	UCL	RPD	T	LCL	UCL	RPD
2861	Gasoline Range Organics	100	ug/L	13	ug/L								
2909	TPH (as Gasoline)	100	ug/L	13	ug/L	C	70	130	77	C	10	116	77
4977	TPH (Purgeables)	100	ug/L	13	ug/L								
GRO - Solid							LCS				MS		
#	Compound	RL	Units	MDL	Units	T	LCL	UCL	RPD	T	LCL	UCL	RPD
2861	Gasoline Range Organics	100	ug/kg	17	ug/kg								
2909	TPH (as Gasoline)	100	ug/kg	17	ug/kg	C	38	120	49	C	10	114	49
4977	TPH (Purgeables)	100	ug/kg	17	ug/kg								

Legend

c = Control Analyte/Compound
x = Surrogate
(1) = The latest MDLs, RLs, and Control Limits will be utilized at the time of sample analysis

TABLE 8.4-1
Field Quality Control Samples

Type	Applicability		Accuracy and Precision Application	Introduced By
	Inorganic	Organic		
Trip Blank (volatiles)	No	Yes	Accuracy	Supplier of Containers
Field Blank	Yes	Yes	Accuracy	Field Sampler
Rinsate Blank	Yes	Yes	Accuracy	Field Sampler
Collocated Sample	Yes	Yes	Precision	Field Sampler
Split Sample	Yes	Yes	Precision	Field Sampler
Field Duplicate	Yes	Yes	Precision	Field Sampler
Field Matrix Spike	Yes	Yes	Accuracy	Field Sampler

TABLE 8.4-2
Laboratory Quality Control Samples

Type	Frequency	Applicability		Accuracy and Precision Application	Introduced By
		Inorganic	Organic		
Analytical Spike	As specified in methods, or as needed	Yes	No	Accuracy	Analyst/ Prep
Duplicate	1 out of 20 or at least 1/month/run	Yes	Yes	Precision	Analyst/ Prep
Instrument Blank	As specified methods, or as needed	Yes	Yes	Accuracy	Analyst
Interference Check Sample	As specified in methods	Yes	No	Accuracy	Analyst
Internal Standard	Each sample and standard	Yes	Yes	Both	Analyst/ Prep
Laboratory Control Sample	1 per each group of samples processed up to 20 samples.	Yes	Yes	Accuracy	Analyst/ Prep
Matrix Spike	1 per each group of samples processed up to 20 samples.	Yes	Yes	Accuracy	Analyst/ Prep
Matrix Spike Duplicate	1 per each group of samples processed up to 20 samples.	Yes	Yes	Both	Analyst/ Prep
Method Blank	1 per each group of samples processed up to 20 samples.	Yes	Yes	Accuracy	Analyst/ Prep
Surrogate	All standards, method blanks, LCS, and samples.	No	Yes Method Dependent	Accuracy	Analyst/ Prep
Yield Monitor	Operation-specific	Yes	No	Accuracy	Prep

TABLE 8.4-3
Laboratory Performance Quality Control Samples

Sample/Measurement	Purpose
Method Blanks	Demonstrates that the laboratory systems (*e.g.*, glassware cleaning procedures) and laboratory reagents used for the preparation and analysis of samples have not contributed to a false positive or negative measurement.
Instrument Blank	Demonstrates that the analytical system has not contributed to a false positive or negative measurement.
Laboratory Control Sample	Demonstrates the laboratory's ability to perform an analysis within the performance requirements of the method.

TABLE 8.4-4
Matrix Specific Quality Control Samples

Quality Control Sample	Purpose
Duplicate Samples	Estimates the ability of the laboratory to obtain precise measurements on a sample. This measure is dependent on the homogeneity of the sample being duplicated. Solid samples often portray poor sample homogeneity and therefore often have poor duplication with regards to the sample result.
Matrix Spike Sample	Estimates the ability of the laboratory to obtain accurate measurements on a sample. The measure is dependent on the bias a sample matrix may cause regarding a given analyte.
Matrix Spike Duplicate Sample	In addition to verifying the accuracy of the matrix spike sample, the matrix spike duplicate can be used with the matrix spike sample as a measure of precision by calculating the relative percent difference (RPD).

Inorganic Laboratory Quality Control Samples

Analysis	QC Sample	Method	NPDES[1]	Method	RCRA (SW846)[2]
Alkalinity	Method Blank	310.1 2320B	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Concentration must be less than the reporting limit Corrective Action: Rerun all samples associated with unacceptable blank	—	Not Applicable
	Laboratory Control Sample	310.1 2320B	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Percent recovery must be within laboratory control limits Corrective Action: If not within laboratory control limits, rerun all associated samples	—	Not Applicable
	Matrix Spike	310.1 2320B	Not Applicable	—	Not Applicable
	Matrix Spike Duplicate	310.1 2320B	Not Applicable	—	Not Applicable
	Duplicate	310.1 2320B	Frequency: 1 per batch of 10 samples Criteria 310.1: ≤ 20 % RPD[3] Criteria 2320B: ≤ 25 % RPD[3] Corrective Action: Flag data outside of limit.	—	Not Applicable

TABLE 8.4-5
Inorganic Laboratory Quality Control Samples
(Continued)

Analysis	QC Sample	Method	NPDES[1]	Method	RCRA (SW846)[2]
Ammonia	Method Blank	350.1 350.2	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Concentration less than reporting limit Corrective Action: Rerun all samples associated with unacceptable blank	—	Not Applicable
	Laboratory Control Sample	350.1 350.2	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Percent recovery must be within laboratory control limits Corrective Action: If not within control limits, rerun all associated samples	—	Not Applicable
	Matrix Spike	350.1 350.2	Frequency: 1 per 10 samples, minimum of one per batch of samples processed Criteria: Percent recovery must be within laboratory control limits Corrective Action: Flag data outside of limit	—	Not Applicable
	Matrix Spike Duplicate	350.1 350.2	Not Applicable	—	Not Applicable
	Duplicate	350.1 350.2	Not Applicable	—	Not Applicable

TABLE 8.4-5
Inorganic Laboratory Quality Control Samples
(Continued)

Analysis	QC Sample	Method	NPDES[1]	Method	RCRA (SW846)[2]
Ammonia (TKN)	Method Blank	351.2 351.3	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Concentration must be less than the reporting limit Corrective Action: Rerun all samples associated with unacceptable blank	—	Not Applicable
	Laboratory Control Sample	351.2 351.3	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Percent recovery must be within laboratory control limits Corrective Action: If not within laboratory control limits, rerun all associated samples	—	Not Applicable
	Matrix Spike	351.2 351.3	Frequency: 1 per 10 samples, minimum of one per batch of samples processed Criteria: Must be within laboratory control limits Corrective Action: Flag data outside of limit	—	Not Applicable
	Matrix Spike Duplicate	351.2 351.3	Not Applicable	—	Not Applicable
	Duplicate	351.2 351.3	Not Applicable	—	Not Applicable

TABLE 8.4-5
Inorganic Laboratory Quality Control Samples
(Continued)

Analysis	QC Sample	Method	NPDES[1]	Method	RCRA (SW846)[2]
BOD	Method Blank	405.1	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Concentration must be less than the reporting limit Corrective Action: Rerun all samples associated with unacceptable blank	—	Not Applicable
	Laboratory Control Sample	405.1	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Percent recovery must be within laboratory control limits Corrective Action: If not within laboratory control limits, rerun all associated samples	—	Not Applicable
	Matrix Spike	405.1	Not Applicable	—	Not Applicable
	Matrix Spike Duplicate	405.1	Not Applicable	—	Not Applicable
	Duplicate	405.1	Not Applicable	—	Not Applicable

TABLE 8.4-5
Inorganic Laboratory Quality Control Samples
(Continued)

Analysis	QC Sample	Method	NPDES[1]	Method	RCRA (SW846)[2]
Bromide	Method Blank	300.0[5]	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Concentration must be less than the reporting limit Corrective Action: Rerun all samples associated with unacceptable blank	9056	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Concentration must be less than the reporting limit Corrective Action: Rerun all samples associated with unacceptable blank
	Laboratory Control Sample	300.0[5]	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Percent recovery must be within laboratory control limits Corrective Action: If not within control limits, rerun all associated samples	9056	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Percent recovery must be within laboratory control limits Corrective Action: If not within control limits, rerun all associated samples
	Matrix Spike	300.0[5]	Frequency: 1 per 10 samples, minimum of one per batch of samples processed Criteria: Percent recovery must be within laboratory control limits Corrective Action: Flag data outside of limit	9056	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Percent recovery must be within laboratory control limits Corrective Action: Flag data associated with MS outside of limit
	Matrix Spike Duplicate	300.0[5]	Not Applicable	9056	Not Applicable
	Duplicate	300.0[5]	Methods 300.0: Not Applicable Frequency: Method D1246: 1 with each batch of samples processed not to exceed 20 samples	9056	Frequency: 1 with each batch of samples processed Criteria: RPD[3] must be within laboratory control limits Corrective Action: Flag data associated with duplicates outside of laboratory RPD[3] limits

TABLE 8.4-5
Inorganic Laboratory Quality Control Samples
(Continued)

Analysis	QC Sample	Method	NPDES[1]	Method	RCRA (SW846)[2]
Chemical Oxygen Demand (COD)	Method Blank	410.4	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Concentration must be less than the reporting limit Corrective Action: Rerun all samples associated with unacceptable blank	—	Not Applicable
	Laboratory Control Sample	410.4	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Percent recovery must be within laboratory control limits Corrective Action: If not within laboratory control limits, rerun all associated samples	—	Not Applicable
	Matrix Spike	410.4	Frequency: 1 per 10 samples, minimum of one per batch of samples processed Criteria: Must be within laboratory control limits Corrective Action: Flag data outside of limit	—	Not Applicable
	Matrix Spike Duplicate	410.4	Not Applicable	—	Not Applicable
	Duplicate	410.4	Not Applicable	—	Not Applicable

TABLE 8.4-5
Inorganic Laboratory Quality Control Samples
(Continued)

Analysis	QC Sample	Method	NPDES[1]	Method	RCRA (SW846)[2]
Chloride	Method Blank	300.0[5] 325.2 325.3	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Concentration must be less than the reporting limit Corrective Action: Rerun all samples associated with unacceptable blank	9056 9252	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Concentration must be less than the reporting limit Corrective Action: Rerun all samples associated with unacceptable blank
	Laboratory Control Sample	300.0[5] 325.2 325.3	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Percent recovery must be within laboratory control limits Corrective Action: If not within control limits, rerun all associated samples	9056 9252	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Percent recovery must be within laboratory control limits Corrective Action: If not within laboratory control limits, rerun all associated samples
	Matrix Spike	300.0[5] 325.2 325.3	Frequency: 1 per 10 samples, minimum of one per batch of samples processed Criteria: Percent recovery must be within laboratory control limits Corrective Action: Flag data outside of limit	9056 9252	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Percent recovery must be within laboratory control limits Methods 9251Corrective Action: If not within laboratory control limits, rerun all associated samples Method 9056/9253 Corrective Action: Flag data associated with MS outside of limits

TABLE 8.4-5
Inorganic Laboratory Quality Control Samples
(Continued)

Analysis	QC Sample	Method	NPDES[1]	Method	RCRA (SW846)[2]
Chloride (continued)	Matrix Spike Duplicate	300.0[5] 325.2 325.3	Not Applicable	9056 9252	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Percent recovery must be within laboratory control limits/< 20 % RPD[3] Corrective Action: Flag data outside of limit Method 9056: MSD is not applicable
	Duplicate	300.0[5] 325.2 325.3	Methods 300.0, 325.1, 325.2, 325.3: Not Applicable Method 4500-Cl E: Frequency: 1 with each batch of samples processed not to exceed 20 samples	9056 9252	Method 9056/9253: Frequency: 1 with each batch of samples processed Criteria: RPD[3] must be within laboratory control limits Corrective Action: Flag data associated with duplicates outside of laboratory RPD[3] limits
Chlorine, Residual	Method Blank	330.5	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Concentration must be less than the reporting limit Corrective Action: Rerun all samples associated with unacceptable blank	—	Not Applicable

TABLE 8.4-5
Inorganic Laboratory Quality Control Samples
(Continued)

Analysis	QC Sample	Method	NPDES[1]	Method	RCRA (SW846)[2]
Chlorine, Residual (continued)	Laboratory Control Sample	330.5	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Percent recovery must be within laboratory control limits Corrective Action: If not within laboratory control limits, rerun all associated samples	—	Not Applicable
	Matrix Spike	330.5	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Must be within laboratory control limits Corrective Action: Flag data outside of limit	—	Not Applicable
	Matrix Spike Duplicate	330.5	Not Applicable	—	Not Applicable
	Duplicate	330.5	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: ≤ 20 % RPD[3] Corrective Action: Flag data outside of limit.	—	Water

TABLE 8.4-5
Inorganic Laboratory Quality Control Samples
(Continued)

Analysis	QC Sample	Method	NPDES[1]	Method	RCRA (SW846)[2]
Chromium (Cr^{+6})	Method Blank	3500 Cr-D	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Concentration must be less than the reporting limit Corrective Action: Rerun all samples associated with unacceptable blank	7196A	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Concentration less than reporting limit Corrective Action: Rerun all samples associated with unacceptable blank
	Laboratory Control Sample	3500 Cr-D	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Percent recovery must be within laboratory control limits Corrective Action: If not within laboratory control limits, rerun all associated samples	7196A	Frequency: 1 with each batch of samples processed not to exceed 20 samples prepped Criteria: percent recovery for water must be within ± 15 % and for solids must be within ± 20% Corrective Action: Rerun all samples associated with unacceptable LCS
	Matrix Spike	3500 Cr-D	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Must be within laboratory QC limits Corrective Action: Flag data outside of limit	3060A 7196A	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Advisory limits are 75% - 125% recovery Corrective Action: Flag data associated with unacceptable Matrix Spike
	Matrix Spike Duplicate	3500 Cr-D	Not Applicable	7196A	Not Applicable

TABLE 8.4-5
Inorganic Laboratory Quality Control Samples
(Continued)

Analysis	QC Sample	Method	NPDES[1]	Method	RCRA (SW846)[2]
Chromium (Cr^{+6}) (continued)	Duplicate	3500 Cr-D	Not Applicable	7196A	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: ≤ 20 % RPD[3] limit Corrective Action: Flag data outside of limit.
Conductivity, Specific	Method Blank	120.1	Not Applicable	9050A	Not Applicable
Conductivity, Specific (continued)	Laboratory Control Sample	120.1	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Percent recovery must be within laboratory control limits Corrective Action: If not within laboratory control limits, rerun all associated samples	9050A	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Percent recovery must be within laboratory control limits Corrective Action: If not within laboratory control limits, rerun all associated samples
	Matrix Spike	120.1	Not Applicable	9050A	Not Applicable
	Matrix Spike Duplicate	120.1	Not Applicable	9050A	Not Applicable
	Duplicate	120.1	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: ≤ 20 % RPD[3] Corrective Action: Flag data outside of limit.	9050A	Frequency: 1 with each batch of samples processed not to exceed 10 samples

TABLE 8.4-5
Inorganic Laboratory Quality Control Samples
(Continued)

Analysis	QC Sample	Method	NPDES[1]	Method	RCRA (SW846)[2]
Cyanide (Amenable)	Method Blank	335.1	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Concentration must be less than the reporting limit Corrective Action: Rerun all samples associated with unacceptable blank	9012A	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Concentration less than reporting limit Corrective Action: Rerun all samples associated with unacceptable blank
Cyanide (Amenable) (continued)	Laboratory Control Sample	335.1	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Percent recovery must be within laboratory control limits Corrective Action: If not within laboratory control limits, rerun all associated samples	9012A	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Percent recovery must be within laboratory control limits Corrective Action: Rerun all samples associated with unacceptable LCS
	Matrix Spike	335.1	Frequency: 1 per 10 samples, minimum of one per batch of samples processed Criteria: Percent recovery must be within laboratory control limits Corrective Action: Flag data outside of limit	9012A	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Advisory limits are 75% - 125% recovery Corrective Action: Flag data associated with unacceptable Matrix Spike
	Matrix Spike Duplicate	335.1	Not Applicable	9012A	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Advisory limits are 75% - 125% recovery Corrective Action: Flag data associated with unacceptable Matrix Spike

TABLE 8.4-5
Inorganic Laboratory Quality Control Samples
(Continued)

Analysis	QC Sample	Method	NPDES[1]	Method	RCRA (SW846)[2]
	Duplicate	335.1	Not Applicable	9012A	Not Applicable
Cyanide (Total)	Method Blank	335.2 335.3 335.4 4500-CN E	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Concentration must be less than the reporting limit Corrective Action: Rerun all samples associated with unacceptable blank	9012A	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Concentration less than reporting limit Corrective Action: Rerun all samples associated with unacceptable blank
	Laboratory Control Sample	335.2 335.3 335.4 4500-CN E	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Percent recovery must be within laboratory control limits Corrective Action: If not within laboratory control limits, rerun all associated samples	9012A	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Percent recovery must be within laboratory control limits Corrective Action: Rerun all samples associated with unacceptable LCS
	Matrix Spike	335.2 335.3 335.4 4500-CN E	Frequency: 1 per 10 samples, minimum of one per batch of samples processed Criteria: Percent recovery must be within laboratory control limits Corrective Action: Flag data outside of limit	9012A	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Advisory limit is 75% - 125% recovery Corrective Action: Flag data associated with unacceptable Matrix Spike

TABLE 8.4-5
Inorganic Laboratory Quality Control Samples
(Continued)

Analysis	QC Sample	Method	NPDES[1]	Method	RCRA (SW846)[2]
Cyanide (Total) (continued)	Matrix Spike Duplicate	335.2 335.3 335.4 4500-CN E	Not Applicable	9012A	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Limit is 75% - 125% recovery Corrective Action: Flag data associated with unacceptable Matrix Spike
	Duplicate	335.2 335.3 335.4	Methods 335.2, 335.3: Not Applicable Method 4500-CN E: Frequecny: 1 with each batch of samples processed not to exceed 20 samples Criteria: ≤ 20 % RPD[3] Corrective Action: Flag data outside of limit.	9012A	Not Applicable
Flashpoint	Method Blank	—	Not Applicable	1010	Not Applicable
	Laboratory Control Sample	—	Not Applicable	1010	Not Applicable
	Matrix Spike	—	Not Applicable	1010	Not Applicable
	Matrix Spike Duplicate	—	Not Applicable	1010	Not Applicable

TABLE 8.4-5
Inorganic Laboratory Quality Control Samples
(Continued)

Analysis	QC Sample	Method	NPDES[1]	Method	RCRA (SW846)[2]
Flashpoint (continued)	Duplicate	—	Not Applicable	1010	Frequency: 1 per batch of ≤20 samples Criteria: RPD[3] must be ≤ 20% Corrective Action: Flag data associated with unacceptable Duplicate
Fluoride	Method Blank	300.0[5] 340.2	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Concentration must be less than the reporting limit Corrective Action: Rerun all samples associated with unacceptable blank	9056	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Concentration must be less than the reporting limit Corrective Action: Rerun all samples associated with unacceptable
	Laboratory Control Sample	300.0[5] 340.2	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Percent recovery must be within laboratory control limits Corrective Action: If not within laboratory control limits, rerun all associated samples	9056	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Percent recovery must be within laboratory control limits Corrective Action: If not within control limits, rerun all associated samples

TABLE 8.4-5
Inorganic Laboratory Quality Control Samples
(Continued)

Analysis	QC Sample	Method	NPDES[1]	Method	RCRA (SW846)[2]
Fluoride (continued)	Matrix Spike	300.0[5] 340.2	Frequency: 1 per 10 samples by IC Criteria: Must be within laboratory QC limits Corrective Action: Flag data outside of limit	9056	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Percent recovery must be within laboratory control limits Corrective Action: Flag data associated with outside of limit
	Matrix Spike Duplicate	300.0[5] 340.2	Not Applicable	9056	Not Applicable
	Duplicate	300.0[5] 340.2	Not Applicable	9056	Frequency: 1 with each batch of samples processed Criteria: RPD[3] must be within laboratory control limits Corrective Action: Flag data associated with duplicates outside of laboratory RPD[3] limits
Hardness	Method Blank	130.2 2340B	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Concentration must be less than the reporting limit Corrective Action: Rerun all samples associated with unacceptable blank	—	Not Applicable

TABLE 8.4-5
Inorganic Laboratory Quality Control Samples
(Continued)

Analysis	QC Sample	Method	NPDES[1]	Method	RCRA (SW846)[2]
Hardness (continued)	Laboratory Control Sample	130.2 2340B	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Percent recovery must be within laboratory control limits Corrective Action: If not within laboratory control limits, rerun all associated samples	—	Not Applicable
	Matrix Spike	130.2 2340B	Method 130.2, Not Applicable Method 2340B: Frequency, Criteria, and Corrective Action: See ICP Metals Method 200.7 Requirements	—	Not Applicable
	Matrix Spike Duplicate	130.2 2340B	Method 130.2, Not Applicable Method 2340B: Frequency, Criteria, and Corrective Action: See ICP Metals Method 200.7 Requirements	—	Not Applicable
	Duplicate	130.2 2340B	Frequency: 1 per 20 samples, minimum of one per batch of samples processed Criteria: RPD[3] must be within laboratory control limits Corrective Action: Flag data outside of limit	—	Not Applicable

TABLE 8.4-5
Inorganic Laboratory Quality Control Samples
(Continued)

Analysis	QC Sample	Method	NPDES[3]	Method	RCRA (SW846)[2]
Iron, Ferrous & Ferric	Method Blank	3500-Fe D	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Concentration must be less than the reporting limit Corrective Action: Rerun all samples associated with unacceptable blank	—	Not Applicable
	Laboratory Control Sample	3500-Fe D	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Percent recovery must be within laboratory control limits Corrective Action: If not within laboratory control limits, rerun all associated samples	—	Not Applicable
	Matrix Spike	3500-Fe D	Frequency: 1 every 10 samples Criteria: Must be within laboratory QC limits Corrective Action: Flag associated data outside of limit	—	Not Applicable
	Matrix Spike Duplicate	3500-Fe D	Not Applicable	—	Not Applicable

TABLE 8.4-5
Inorganic Laboratory Quality Control Samples
(Continued)

Analysis	QC Sample	Method	NPDES[1]	Method	RCRA (SW846)[2]
Iron, Ferrous & Ferric (continued)	Duplicate	3500-Fe D	Frequency: 1 per batch of 20 samples Criteria: Must be within laboratory QC limits Corrective Action: Flag associated data outside of limit	—	Not Applicable
Nitrate	Method Blank	300.0[5] 353.2	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Concentration must be less than the reporting limit Corrective Action: Rerun all samples associated with unacceptable blank	9056	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Concentration must be less than the reporting limit
	Laboratory Control Sample	300.0[5] 353.2	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Percent recovery must be within laboratory control limits Corrective Action: If not within laboratory control limits, rerun all associated samples	9056	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Percent recovery must be within laboratory control limits Corrective Action: If not within laboratory control limits, rerun all associated samples

TABLE 8.4-5

Inorganic Laboratory Quality Control Samples (Continued)

Analysis	QC Sample	Method	NPDES[1]	Method	RCRA (SW846)[2]
Nitrate (continued)	Matrix Spike	300.0[5] 353.2	Frequency: 1 per 10 samples, minimum of one per batch of samples processed Criteria: Must be within laboratory control limits Corrective Action: Flag data outside of limit	9056	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Percent recovery must be within laboratory control limits Corrective Action: If not within laboratory control limits, flag all associated samples
	Matrix Spike Duplicate	300.0[5] 353.2	Not Applicable	9056	Not applicable
	Duplicate	300.0[5] 353.2	Not Applicable	9056	Frequency: 1 per 10 samples Criteria: RPD[3] must be within laboratory control limits Corrective Action: If not within laboratory control limits, flag all associated samples

TABLE 8.4-5
Inorganic Laboratory Quality Control Samples
(Continued)

Analysis	QC Sample	Method	NPDES[1]	Method	RCRA (SW846)[2]
Nitrite	Method Blank	300.0[5] 353.2	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Concentration must be less than the reporting limit Corrective Action: Rerun all samples associated with unacceptable blank	9056	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Concentration must be less than the reporting limit Corrective Action: Rerun all samples associated with unacceptable blank
	Laboratory Control Sample	300.0[5] 354.1 353.2	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Percent recovery must be within laboratory control limits Corrective Action: If not within laboratory control limits, rerun all associated samples	9056	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Percent recovery must be within laboratory control limits Corrective Action: If not within laboratory control limits, rerun all associated samples
	Matrix Spike	300.0[5] 354.1 353.2	Frequency: 1 per 10 samples, minimum of one per batch of samples processed Criteria: Must be within laboratory control limits Corrective Action: Flag data outside of limit	9056	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Percent recovery must be within laboratory control limits Corrective Action: If not within laboratory control limits, flag all associated samples
	Matrix Spike Duplicate	300.0[5] 354.1 353.2	Not Applicable	9056	Not Applicable

TABLE 8.4-5
Inorganic Laboratory Quality Control Samples
(Continued)

Analysis	QC Sample	Method	NPDES[1]	Method	RCRA (SW846)[2]
Nitrite (continued)	Duplicate	300.0[5] 354.1 353.2	Not Applicable	9056	Frequency: 1 per 10 samples Criteria: RPD[3] must be within laboratory control limits Corrective Action: If not within laboratory control limits, flag all associated samples
Nitrate-Nitrite	Method Blank	353.2	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Concentration must be less than the reporting limit Corrective Action: Rerun all samples associated with unacceptable blank	—	Not Applicable
	Laboratory Control Sample	353.2	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Percent recovery must be within laboratory control limits Corrective Action: If not within laboratory control limits, rerun all associated samples	—	Not Applicable

TABLE 8.4-5
Inorganic Laboratory Quality Control Samples
(Continued)

Analysis	QC Sample	Method	NPDES[1]	Method	RCRA (SW846)[2]
Nitrate-Nitrite (continued)	Matrix Spike	353.2	Frequency: 1 per 20 samples, minimum of one per batch of samples processed Criteria: Percent recovery must be within laboratory control limits Corrective Action: Flag data outside of limit	—	Not Applicable
	Matrix Spike Duplicate	353.2	Not Applicable	—	Not Applicable
	Duplicate	353.2	Not Applicable	—	Not Applicable

TABLE 8.4-5
Inorganic Laboratory Quality Control Samples
(Continued)

Analysis	QC Sample	Method	NPDES[1]	Method	RCRA (SW846)[2]
pH	Method Blank	150.1	Not Applicable	9040B 9045C	Not Applicable
	Laboratory Control Sample	150.1	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Sample provided by external source, must be within ± 0.05 pH units Corrective Action: If not within laboratory control limits, rerun all associated samples	9040B 9045C	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Sample provided by external source, must be within ± 0.05 pH units Corrective Action: If not within laboratory control limits, rerun all associated samples
	Matrix Spike	150.1	Not Applicable	9040B 9045C	Not Applicable
	Matrix Spike Duplicate	150.1	Not Applicable	9040B 9045C	Not Applicable

TABLE 8.4-5
Inorganic Laboratory Quality Control Samples
(Continued)

Analysis	QC Sample	Method	NPDES[1]	Method	RCRA (SW846)[2]
pH (continued)	Duplicate	150.1	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: ≤ 20 % RPD[3] limit Corrective Action: Flag data outside of limit.	9040B 9045C	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Advisory limits are ≤ 20% RPD[3] Corrective Action: Flag data associated with unacceptable Duplicate
Phenolics	Method Blank	420.1	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Concentration must be less than the reporting limit Corrective Action: Rerun all samples associated with unacceptable blank	9065 9066	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Concentration less than reporting limit Corrective Action: Rerun all samples associated with unacceptable blank
	Laboratory Control Sample	420.1	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Percent recovery must be within laboratory control limits Corrective Action: If not within laboratory control limits, rerun all associated samples	9065 9066	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Percent recovery must be within laboratory control limits Corrective Action: If not within laboratory control limits, rerun all associated samples

TABLE 8.4-5
Inorganic Laboratory Quality Control Samples
(Continued)

Analysis	QC Sample	Method	NPDES[1]	Method	RCRA (SW846)[2]
Phenolics (continued)	Matrix Spike	420.1	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Percent recovery must be within laboratory control limits Corrective Action: Flag data associated with unacceptable Matrix Spike	9065 9066	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Percent recovery must be within laboratory control limits Corrective Action: Flag associated data
	Matrix Spike Duplicate	420.1	Not Applicable	9065 9066	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Percent recovery must be within laboratory control limits Corrective Action: Flag associated data
	Duplicate	420.1	Not Applicable	9065 9066	Not Applicable
Phosphate	Method Blank	---	Not Applicable	9056	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Concentration less than reporting limit Corrective Action: Rerun all samples associated with unacceptable blank
	Laboratory Control Sample	---	Not Applicable	9056	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Percent recovery must be within laboratory control limits Corrective Action: If not within laboratory control limits, rerun all associated samples

TABLE 8.4-5
Inorganic Laboratory Quality Control Samples
(Continued)

Analysis	QC Sample	Method	NPDES[1]	Method	RCRA (SW846)[2]
Phosphate (continued)	Matrix Spike	---	Not Applicable	9056	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Percent recovery must be within laboratory control limits Corrective Action: Flag associated data associated with MS outside of limits
	Matrix Spike Duplicate	---	Not Applicable	9056	Not Applicable
	Duplicate	---	Not Applicable	9056	Frequency: 1 with each batch of samples processed Criteria: RPD[3] must be within laboratory control limits Corrective Action: Flag data associated with duplicates outside of laboratory RPD[3] limits
Phosphorus (Total and Ortho-phosphate)	Method Blank	300.0[4,5] 365.2 365.3	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Concentration must be less than the reporting limit Corrective Action: Rerun all samples associated with unacceptable blank	—	Not Applicable

TABLE 8.4-5
Inorganic Laboratory Quality Control Samples
(Continued)

Analysis	QC Sample	Method	NPDES[1]	Method	RCRA (SW846)[2]
Phosphorus (Total and Ortho-phosphate) (continued)	Laboratory Control Sample	300.0[4,5] 365.2 365.3	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Percent recovery must be within laboratory control limits Corrective Action: If not within laboratory control limits, rerun all associated samples	—	Not Applicable
	Matrix Spike	300.0[4,5] 365.2 365.3	Frequency: 1 per 10 samples Criteria: Must be within laboratory QC limits Corrective Action: Flag data outside of limit	—	Not Applicable
	Matrix Spike Duplicate	300.0[4,5] 365.2 365.3	Not Applicable	—	Not Applicable
	Duplicate	300.0[4,5] 365.2 365.3	Not Applicable	—	Not Applicable
Reactivity (Cyanide and Sulfide)	Method Blank	---	Not Applicable	Chapter 7[6] Sections 7.3.3.2 and 7.3.4.2	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Concentration must be less than the reporting limit Corrective Action: Rerun all samples associated with unacceptable blank

TABLE 8.4-5
Inorganic Laboratory Quality Control Samples
(Continued)

Analysis	QC Sample	Method	NPDES[1]	Method	RCRA (SW846)[2]
Reactivity (Cyanide and Sulfide) (continued)	Laboratory Control Sample	—	Not Applicable	Chapter 7[6] Sections 7.3.3.2 and 7.3.4.2	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Percent recovery must be within laboratory control limits Corrective Action: Rerun all samples associated with unacceptable LCS
	Matrix Spike	---	Not Applicable	Chapter 7[6] Sections 7.3.3.2 and 7.3.4.2	Follow QC sample requirements of determinative method
	Matrix Spike Duplicate	---	Not Applicable	Chapter 7[6] Sections 7.3.3.2 and 7.3.4.2	Follow QC sample requirements of determinative method
	Duplicate	---	Not Applicable	Chapter 7[6] Sections 7.3.3.2 and 7.3.4.2	Not Applicable
Solids	Method Blank	160.1 160.2 160.3 160.4 160.5	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Concentration must be less than the reporting limit Corrective Action: If analyte level in method blank is ≥ RL for the analyte of interest in the sample, all associated samples with reportable levels of analyte are reprepared and reanalyzed.	—	Not Applicable

TABLE 8.4-5
Inorganic Laboratory Quality Control Samples
(Continued)

Analysis	QC Sample	Method	NPDES[1]	Method	RCRA (SW846)[2]
Solids	Method Blank	160.1 160.2 160.3 160.4 160.5	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Concentration must be less than the reporting limit Corrective Action: If analyte level in method blank is ≥ RL for the analyte of interest in the sample, all associated samples with reportable levels of analyte are reprepared and reanalyzed.	—	Not Applicable
Solids (continued)	Laboratory Control Sample	160.1 160.2 160.3 160.4 160.5	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Percent recovery must be within laboratory control limits Corrective Action: If not within laboratory control limits, reprepare and rerun all associated samples	—	Not Applicable
	Matrix Spike	160.1 160.2 160.3 160.4 160.5	Not Applicable	—	Not Applicable
	Matrix Spike Duplicate	160.1 160.2 160.3 160.4 160.5	Not Applicable	—	Not Applicable
	Duplicate	160.1 160.2 160.3 160.4 160.5	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Sample results should agree within 20% if both the sample and sample duplicate results are > 5 X RL Corrective Action: Flag data	—	Not Applicable

Analysis	QC Sample	Method	NPDES[1]	Method	RCRA (SW846)[2]
			outside of limit.		

TABLE 8.4-5
Inorganic Laboratory Quality Control Samples
(Continued)

Analysis	QC Sample	Method	NPDES[1]	Method	RCRA (SW846)[2]
Sulfate	Method Blank	300.0[5] 375.4	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Concentration must be less than the reporting limit Corrective Action: Rerun all samples associated with unacceptable blank	9038 9056	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Concentration must be less than the reporting limit Corrective Action: Rerun all samples associated with unacceptable blank
	Laboratory Control Sample	300.0[5] 375.4	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Percent recovery must be within laboratory control limits Corrective Action: If not within laboratory control limits, rerun all associated samples	9038 9056	Frequency: 1 with each batch of samples processed not to exceed 20 samples Method 9038 Criteria: Percent recovery must be within ± 15 % Method 9056 Criteria: Percent recovery must be within laboratory control limits Corrective Action: Rerun all samples associated with unacceptable LCS (ICV)
	Matrix Spike	300.0[5] 375.4	Frequency: 1 per 10 samples, minimum of one per batch of samples processed Criteria: Percent recovery must be within laboratory control limits Corrective Action: Flag data outside of limit	9038 9056	Frequency: 1 with each batch of samples processed not to exceed 10 samples (9038) or 20 samples (9056) Method 9038 Criteria: Limits are 75% - 125% recovery Method 9056 Criteria: Percent recovery must be within laboratory 0control limits Corrective Action: Flag data associated with unacceptable Matrix Spike
	Matrix Spike Duplicate	300.0[5] 375.4	Not Applicable	9038 9056	Not Applicable

TABLE 8.4-5
Inorganic Laboratory Quality Control Samples
(Continued)

Analysis	QC Sample	Method	NPDES[1]	Method	RCRA (SW846)[2]
Sulfate (continued)	Duplicate	300.0[5] 375.4	Not Applicable	9038 9056	Frequency: 1 with each batch of samples processed Criteria: RPD[3] must be within laboratory control limits Corrective Action: Flag data associated with duplicates outside of laboratory RPD[3] limits
Sulfide	Method Blank	376.2	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Concentration must be less than the reporting limit Corrective Action: Rerun all samples associated with unacceptable blank	9030A	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Concentration must be less than the reporting limit Corrective Action: Rerun all samples associated with unacceptable blank
	Laboratory Control Sample	376.2	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Percent recovery must be within laboratory control limits Corrective Action: If not within laboratory control limits, rerun all associated samples	9030A	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Percent recovery must be within laboratory control limits Corrective Action: Flag associated data

TABLE 8.4-5
Inorganic Laboratory Quality Control Samples
(Continued)

Analysis	QC Sample	Method	NPDES[1]	Method	RCRA (SW846)[2]
Sulfide (continued)	Matrix Spike	376.2	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Percent recovery must be within laboratory control limits Corrective Action: Flag data outside of limit	9030A	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Percent recovery must be within laboratory control limits Corrective Action: Flag associated data
	Matrix Spike Duplicate	376.2	Not Applicable	9030A	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Percent recovery must be within laboratory control limits Corrective Action: Flag associated data Method 9034: Not Applicable
	Duplicate	376.2	Not Applicable	9030A	Not Applicable

TABLE 8.4-5
Inorganic Laboratory Quality Control Samples
(Continued)

Analysis	QC Sample	Method	NPDES[1]	Method	RCRA (SW846)[2]
Total Organic Carbon (TOC)	Method Blank	415.1	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Concentration must be less than the reporting limit Corrective Action: Rerun all samples associated with unacceptable blank	9060 Walkley-Black	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Concentration less than reporting limit Corrective Action: Rerun all samples associated with unacceptable blank
	Laboratory Control Sample	415.1	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Percent recovery must be within laboratory control limits Corrective Action: If not within laboratory control limits, rerun all associated samples	9060 Walkley-Black	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: percent recovery must be within laboratory control limits Corrective Action: Rerun all samples associated with unacceptable LCS
	Matrix Spike	415.1	Frequency: 1 per 10 samples, minimum of one per batch of samples processed Criteria: Percent recovery must be within laboratory control limits Corrective Action: Flag data outside of limit	9060 Walkley-Black	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Percent recovery must be within laboratory control limits Corrective Action: Reanalyze if sample remaining. If not, flag data associated with unacceptable Matrix Spike

TABLE 8.4-5
Inorganic Laboratory Quality Control Samples
(Continued)

Analysis	QC Sample	Method	NPDES[1]	Method	RCRA (SW846)[2]
Total Organic Carbon (TOC) (continued)	Matrix Spike Duplicate	415.1	Not Applicable	9060 Walkley-Black	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Percent recovery must be within laboratory control limits Corrective Action: Reanalyze if sample remaining. If not, flag data associated with unacceptable Matrix Spike Duplicate
	Duplicate	415.1	Not Applicable	9060 Walkley-Black	Not Applicable
Total Organic Halides (TOX)	Method Blank	450.1 [5]	Frequency: 1 with each set of 8 samples Criteria: Concentration less than reporting limit Corrective Action: Rerun all samples associated with unacceptable blank	9020B	Frequency: Run in duplicate between each group of 8 analytical determinations Criteria: Concentration less than reporting limit or less than 2 X MDL or RL whichever is lower Corrective Action: Rerun all samples associated with unacceptable blank
	Laboratory Control Sample	450.1 [5]	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Percent recovery of analyte must be within laboratory control limits Corrective Action: Rerun all samples associated with unacceptable LCS (ICV)	9020B	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Percent recovery of analyte must be within 90-110% Corrective Action: Rerun all samples associated with unacceptable LCS (ICV)

TABLE 8.4-5
Inorganic Laboratory Quality Control Samples
(Continued)

Analysis	QC Sample	Method	NPDES[1]	Method	RCRA (SW846)[2]
Total Organic Halides (TOX) (continued)	Matrix Spike	450.1 [5]	Frequency: 1 per 10 samples, minimum of one per batch of samples processed Criteria: Must be within laboratory control limits Corrective Action: Reanalyze if sample remaining. If not, flag data with unacceptable Matrix Spike	9020B	Frequency: 1 per batch of 10 samples Criteria: Must be within laboratory control limits Corrective Action: Flag data associated with unacceptable Matrix Spike SOP NO. CORP-WC-0001
	Matrix Spike Duplicate	450.1 [5]	Not Applicable	9020B	Not Applicable
	Duplicate	450.1 [5]	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: ≤ 20 % RPD[3] limit Corrective Action: Flag data outside of limit.	9020B	Frequency: All samples will be analyzed in duplicate Criteria: ≤ 20 % RPD[3] limit if both the sample and sample duplicate results are > 10 X MDL. Corrective Action: Flag data outside of limit. SOP NO. CORP-WC-0001
Turbidity	Method Blank	180.1	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Concentration must be less than the reporting limit Corrective Action: Rerun all samples associated with unacceptable blank	—	Not Applicable

TABLE 8.4-5
Inorganic Laboratory Quality Control Samples
(Continued)

Analysis	QC Sample	Method	NPDES[1]	Method	RCRA (SW846)[2]
Turbidity (continued)	Laboratory Control Sample	180.1	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Percent recovery must be within laboratory control limits Corrective Action: If not within laboratory control limits, rerun all associated samples	—	Not Applicable
	Matrix Spike	180.1	Not applicable	—	Not Applicable
	Matrix Spike Duplicate	180.1	Not Applicable	—	Not Applicable
	Duplicate	180.1	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Must be within laboratory QC limits Corrective Action: Flag data outside of limit Not Applicable.	—	Not Applicable
Water Content	Method Blank	—	Not Applicable	—	Not Applicable
	Laboratory Control Sample	—	Not Applicable	—	Not Applicable
	Matrix Spike	—	Not Applicable	—	Not Applicable

TABLE 8.4-5
Inorganic Laboratory Quality Control Samples
(Continued)

Analysis	QC Sample	Method	NPDES[1]	Method	RCRA (SW846)[2]
Water Content (continued)	Matrix Spike Duplicate	—	Not Applicable	—	Not Applicable
	Duplicate	—	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: ≤ 20 % RPD[3] Corrective Action: Flag data outside of limit.	—	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: ≤ 20 % RPD[3] limit Corrective Action: Reanalyze if sample remaining. If not, flag data outside of limit.
GFAA Metals and Mercury by CVAA & CVAFS	Method Blank	200 series 1631B (5)	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Concentration less than reporting limit Corrective Action: Rerun all samples associated with unacceptable blank SOP NO. CORP-MT-0003	7000A series	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Concentration less than reporting limit Corrective Action: Rerun all samples associated with unacceptable blank SOP NO. CORP-MT-0003
	Laboratory Control Sample	200 series 1631B (5)	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: percent recovery of analyte must be within ± 20 % Corrective Action: Rerun all samples associated with unacceptable LCS SOP NO. CORP-MT-0003	7000A series	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: percent recovery of analyte must be within ± 20 % Corrective Action: Rerun all samples associated with unacceptable LCS SOP NO. CORP-MT-0003

TABLE 8.4-5
Inorganic Laboratory Quality Control Samples
(Continued)

Analysis	QC Sample	Method	NPDES[1]	Method	RCRA (SW846)[2]
GFAA Metals and Mercury by CVAA & CVAFS (continued)	Matrix Spike	200 series 1631B (5)	Frequency: with each batch of samples processed not to exceed 20 samples Criteria: Recovery must be within 75-125 % Corrective Action: Flag data associated with unacceptable MS. (See SOP NO. CORP-MT-0003 for detailed corrective action procedure and for other QC procedures.)	7000A series	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Recovery must be within 75-125 % Corrective Action: Flag data associated with unacceptable MS. (See SOP NO. CORP-MT-0003 for detailed corrective action procedure and for other QC procedures.)
	Matrix Spike Duplicate	200 series 1631B (5)	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Recovery must be within 75-125 %, RPD[3] must be within 20 % Corrective Action: Flag data associated with unacceptable MSD SOP NO. CORP-MT-0003	7000A series	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Recovery must be within 75-125 %, RPD[3] must be within 20 % Corrective Action: Flag data associated with unacceptable MSD SOP NO. CORP-MT-0003
	Duplicate	200 series 1631B (5)	Not Applicable	7000A series	Not Applicable
	Post Digestion Spikes	200 series 1631B (5)	Post Digestion Spike is conducted on all samples	7000A series	Post Digestion Spike is conducted on all samples
ICP Metals	Method Blank	200.7 200.8	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Concentration less than reporting limit Corrective Action: Rerun all samples associated with unacceptable blank SOP NO. CORP-MT-0001	6010B 6020	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Concentration less than reporting limit Corrective Action: Rerun all samples associated with unacceptable blank SOP NO. CORP-MT-0001

TABLE 8.4-5
Inorganic Laboratory Quality Control Samples
(Continued)

Analysis	QC Sample	Method	NPDES[1]	Method	RCRA (SW846)[2]
ICP Metals (continued)	Laboratory Control Sample	200.7 200.8	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: percent recovery of analyte must be ± 85-115% Corrective Action: Rerun all samples associated with unacceptable LCS SOP NO. CORP-MT-0001	6010B 6020	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: percent recovery of analyte must be ± 20 % Corrective Action: Rerun all samples associated with unacceptable LCS SOP NO. CORP-MT-0001
	Matrix Spike	200.7 200.8	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Limits for percent recovery are 75-125% Corrective Action: Flag data associated with unacceptable Matrix Spike SOP NO. CORP-MT-0001	6010B 6020	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Limits for percent recovery are 75-125% Corrective Action: Flag data associated with unacceptable Matrix Spike SOP NO. CORP-MT-0001
	Matrix Spike Duplicate	200.7 200.8	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Limits for percent recovery are 75-125%, RPD[3] must be within 20 % Corrective Action: Flag data associated with unacceptable Matrix Spike SOP NO. CORP-MT-0001	6010B 6020	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Limits for percent recovery are 75-125%, RPD[3] must be within 20 % Corrective Action: Flag data associated with unacceptable Matrix Spike SOP NO. CORP-MT-0001

TABLE 8.4-5
Inorganic Laboratory Quality Control Samples
(Continued)

Analysis	QC Sample	Method	NPDES[1]	Method	RCRA (SW846)[2]
ICP Metals (continued)	Duplicate	200.7 200.8	Not Applicable	6010B 6020	Not Applicable
	Serial Dilution	200.7 200.8	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: 10 % Difference Corrective Action: Flag data associated with unacceptable Serial Dilution SOP NO. CORP-MT-0001	6010B 6020	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: 10 % Difference Corrective Action: Flag data associated with unacceptable Serial Dilution SOP NO. CORP-MT-0001

Footnotes

(1) National Pollutant Discharge Elimination System

(2) Resource Conservation and Recovery Act, Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, (SW-846), Third Edition, September 1986. Contains Final Update I (July 1992), Final Update IIA (August 1993), Final Update II (September 1994), Final Update IIB (January 1995), and Final Update III (December 1996).

(3) RPD-Relative Percent Difference

(4) Orthophosphate only

(5) Method not listed in 40 CFR Part 136.

(6) Current promulgated method is a Guidance Method Only, SW-846, Final Update III, Rev.3, 12/96.

TABLE 8.4-6[7]
Organic Laboratory Quality Control Samples

Analysis	QC Sample	Method	NPDES[1]	Method	RCRA (SW846)[2]
Aromatic Volatiles by GC	Method Blank	602	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Concentration less than reporting limit Corrective Action: Rerun all samples associated with unacceptable blank	8021B	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Concentration less than reporting limit Corrective Action: Rerun all samples associated with unacceptable blank
	Laboratory Control Sample	602	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: percent recovery must be within acceptance limits given in method for each analyte Corrective Action: Rerun all samples associated with unacceptable LCS	8021B	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: percent recovery for each analyte must be within laboratory acceptance limits Corrective Action: Rerun all samples associated with unacceptable LCS
	Matrix Spike	602	Frequency: 1 per 10 samples from each site or 1 per month, whichever is more frequent Criteria: percent recovery for each analyte should be within advisory limits given in method Corrective Action: Flag data associated with unacceptable Matrix Spike	8021B	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: percent recovery for each analyte should be within laboratory acceptance limits Corrective Action: Flag data associated with unacceptable Matrix Spike

TABLE 8.4-6
Organic Laboratory Quality Control Samples
(Continued)

Analysis	QC Sample	Method	NPDES[1]	Method	RCRA (SW846)[2]
Aromatic Volatiles by GC (continued)	Matrix Spike Duplicate	602	Not Applicable	8021B	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: percent recovery for each analyte should be within laboratory acceptance limits Corrective Action: Flag data associated with unacceptable Matrix Spike
	Duplicate	602	Not Applicable	8021B	Not Applicable
	Surrogates	602	Surrogates spiked into method blank and all samples (QC included) Method Blank Criteria and LCS: All surrogates must be within laboratory established control limits before sample analysis may proceed. Sample Criteria: Re-extract samples or flag sample data not meeting surrogate criteria	8021B	Surrogates spiked into method blank and all samples (QC included) Method Blank Criteria and LCS: All surrogates must be within laboratory established control limits before sample analysis may proceed. Sample Criteria: Reprepare and reanlayze samples or flag sample data not meeting surrogate criteria
	Internal Standards	602	Optional: Internal standards are added to the method blank and all samples (QC included). If used, same compounds as used for surrogates may be appropriate.	8021B	Optional: Internal standards are added to the method blank and all samples (QC included). If used, same compounds as used for surrogates may be appropriate.

TABLE 8.4-6
Organic Laboratory Quality Control Samples
(Continued)

Analysis	QC Sample	Method	NPDES[1]	Method	RCRA (SW846)[2]
Halogenated Volatiles Volatiles by GC	Method Blank	--	Not Applicable	8021B	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Concentration less than reporting limit Corrective Action: Rerun all samples associated with unacceptable blank
	Laboratory Control Sample	--	Not Applicable	8021B	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: percent recovery for each analyte must be within laboratory acceptance limits Corrective Action: Rerun all samples associated with unacceptable LCS

TABLE 8.4-6
Organic Laboratory Quality Control Samples
(Continued)

Analysis	QC Sample	Method	NPDES[1]	Method	RCRA (SW846) [2]
Halogenated Volatiles by GC (continued)	Matrix Spike	--	Not Applicable	8021B	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: percent recovery for each analyte should be within laboratory acceptance limits Corrective Action: Flag data associated with unacceptable Matrix Spike
	Matrix Spike Duplicate	--	Not Applicable	8021B	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: percent recovery for each analyte should be within laboratory acceptance limits Corrective Action: Flag data associated with unacceptable Matrix Spike
	Duplicate	--	Not Applicable	8021B	Not Applicable
	Surrogates	--	Not Applicable	8021B	Surrogates spiked into method blank and all samples (QC included) Method Blank Criteria and LCS: All surrogates must be within laboratory established control limits before sample analysis may proceed. Sample Criteria: Reprepare and reanalyze samples or flag sample data not meeting surrogate criteria.

TABLE 8.4-6
Organic Laboratory Quality Control Samples
(Continued)

Analysis	QC Sample	Method	NPDES[1]	Method	RCRA (SW846)[2]
Halogenated Volatiles by GC (continued)	Internal Standards	--	Not Applicable	8021B	Optional: Internal standards are added to the method blank and all samples (QC included). If used, same compounds as used for surrogates may be appropriate.
Herbicides	Method Blank	615[3]	Frequency: 1 with each extraction batch of samples not to exceed 20 samples Criteria: Concentration less than reporting limit Corrective Action: Re-extract all samples associated with unacceptable blank	8151A	Frequency: 1 with each extraction batch of samples not to exceed 20 samples Criteria: Concentration less than reporting limit Corrective Action: Re-extract all samples associated with unacceptable blank
	Laboratory Control Sample	615[3]	Frequency: 1 with each extraction batch of samples not to exceed 20 samples Criteria: Percent recovery must be within acceptance limits given in method for each analyte Corrective Action: Re-extract all samples associated with unacceptable LCS	8151A	Frequency: 1 with each extraction batch of samples not to exceed 20 samples Criteria: Percent recovery for each analyte must be within laboratory acceptance limits Corrective Action: Re-extract and reanalyze all samples associated with unacceptable LCS
	Matrix Spike	615[3]	Frequency: 1 per 10 samples from each site or 1 per month, whichever is more frequent Criteria: Percent recovery for each analyte should be within advisory limits given in method Corrective Action: Flag data associated with unacceptable Matrix Spike	8151A	Frequency: 1 with each extraction batch of samples not to exceed 20 samples Criteria: Percent recovery for each analyte should be within laboratory acceptance limits Corrective Action: Flag data associated with unacceptable Matrix Spike

TABLE 8.4-6
Organic Laboratory Quality Control Samples
(Continued)

Analysis	QC Sample	Method	NPDES[1]	Method	RCRA (SW846)[2]
Herbicides (continued)	Matrix Spike Duplicate	615[3]	Not Applicable	8151A	Frequency: 1 with each extraction batch of samples not to exceed 20 samples Criteria: percent recovery for each analyte should be within laboratory control limits Corrective Action: Flag data associated with unacceptable matrix spike sample
	Duplicate	615[3]	Not Applicable	8151A	Not Applicable
	Surrogates	615[3]	Not Applicable	8151A	Surrogates spiked into method blank and all samples (QC included) Method Blank Criteria and LCS: All surrogates must fall within laboratory established control limits before sample analysis may proceed. Sample Criteria: Re-extract and reanalyze samples or flag sample data not meeting surrogate criteria
	Internal Standards	615[3]	Not Applicable	8151A	Optional

TABLE 8.4-6
Organic Laboratory Quality Control Samples
(Continued)

Analysis	QC Sample	Method	NPDES[1]	Method	RCRA (SW846)[2]
Organo-phosphorus Pesticides	Method Blank	--	Not Applicable	8141A	Frequency: 1 with each extraction batch of samples not to exceed 20 samples Criteria: Concentration less than reporting limit Corrective Action: Rerun all samples associated with unacceptable blank
	Laboratory Control Sample	--	Not Applicable	8141A	Frequency: 1 with each extraction batch of samples not to exceed 20 samples Criteria: Percent recovery for each analyte should be within laboratory acceptance limits Corrective Action: Rerun all samples associated with unacceptable LCS
	Matrix Spike	--	Not Applicable	8141A	Frequency: 1 with each extraction batch of samples not to exceed 20 samples Criteria: Percent recovery for each analyte should be within laboratory acceptance limits Corrective Action: Flag data associated with unacceptable MS
	Matrix Spike Duplicate	--	Not Applicable	8141A	Frequency: 1 with each extraction batch of samples not to exceed 20 samples Criteria: Percent recovery for each analyte should be within laboratory acceptance limits Corrective Action: Flag data associated with unacceptable MS

Analysis	QC Sample	Method	NPDES[1]	Method	RCRA (SW846) [2]
	Duplicate	--	Not Applicable	8141A	Not Applicable

TABLE 8.4-6
Organic Laboratory Quality Control Samples
(Continued)

Analysis	QC Sample	Method	NPDES[1]	Method	RCRA (SW846) [2]
Organo-phosphorus Pesticides (continued)	Surrogates	--	Not Applicable	8141A	Surrogates spiked into method blank and all samples (QC included) Method Blank and LCS Criteria: Results must fall within laboratory-established control limits Sample Criteria: Re-extract and reanalyze samples or flag sample data not meeting surrogate criteria
PAHs by GC and HPLC	Method Blank	610	Frequency: 1 with each extraction batch of samples not to exceed 20 samples Criteria: Concentration less than reporting limit Corrective Action: Rerun all samples associated with unacceptable blank	8310	Frequency: 1 with each extraction batch of samples not to exceed 20 samples Criteria: Concentration less than reporting limit Corrective Action: Rerun all samples associated with unacceptable blank
	Laboratory Control Sample	610	Frequency: 1 with each extraction batch of samples not to exceed 20 samples Criteria: percent recovery must be within acceptance limits given in method for each analyte Corrective Action: Rerun all samples associated with unacceptable LCS	8310	Frequency: 1 with each extraction batch of samples not to exceed 20 samples Criteria: percent recovery for each analyte must be within laboratory acceptance limits Corrective Action: Rerun all samples associated with unacceptable LCS

TABLE 8.4-6
Organic Laboratory Quality Control Samples
(Continued)

Analysis	QC Sample	Method	NPDES[1]	Method	RCRA (SW846)[2]
PAHs by GC and HPLC (continued)	Matrix Spike	610	Frequency: 1 per 10 samples from each site or 1 per month, whichever is more frequent Criteria: percent recovery for each analyte should be within advisory limits given in method Corrective Action: Flag data associated with unacceptable Matrix Spike	8310	Frequency: 1 with each extraction batch of samples not to exceed 20 samples Criteria: percent recovery for each analyte should be within laboratory acceptance limits Corrective Action: Flag data associated with unacceptable Matrix Spike
	Matrix Spike Duplicate	610	Not Applicable	8310	Frequency: 1 with each extraction batch of samples not to exceed 20 samples Criteria: percent recovery for each analyte should be within laboratory acceptance limits Corrective Action: Flag data associated with unacceptable Matrix Spike
	Duplicate	610	Not Applicable	8310	Not Applicable
	Surrogates	610	Not specified in method	8310	Surrogates spiked into method blank and all samples (QC included) Method Blank Criteria and LCS: Results must fall within laboratory established control limits Sample Criteria: Re-extract and reanalyze samples or flag sample data not meeting surrogate criteria
	Internal Standards	610	Optional	8310	Optional

TABLE 8.4-6
Organic Laboratory Quality Control Samples
(Continued)

Analysis	QC Sample	Method	NPDES[1]	Method	RCRA (SW846)[2]
Pesticides/ PCBs	Method Blank	608	Frequency: 1 with each extraction batch of samples not to exceed 20 samples Criteria: Concentration less than reporting limit Corrective Action: Rerun all samples associated with unacceptable blank	8081A 8082	Frequency: 1 with each extraction batch of samples not to exceed 20 samples Criteria: Concentration less than reporting limit Corrective Action: Reprepare and reanalyze all samples associated with unacceptable blank
	Laboratory Control Sample	608	Frequency: 1 with each extraction batch of samples not to exceed 20 samples Criteria: percent recovery must be within acceptance limits given in method for each analyte Corrective Action: Rerun all samples associated with unacceptable LCS	8081A 8082	Frequency: 1 with each extraction batch of samples not to exceed 20 samples Criteria: percent recovery for each analyte must be within laboratory acceptance limits Corrective Action: Rerun all samples associated with unacceptable LCS
	Matrix Spike	608	Frequency: 1 per 10 samples from each site or 1 per month, whichever is more frequent Criteria: percent recovery for each analyte should be within advisory limits given in method Corrective Action: Flag data associated with unacceptable Matrix Spike	8081A 8082	Frequency: 1 with each extraction batch of samples not to exceed 20 samples Criteria: percent recovery for each analyte should be within laboratory acceptance limits Corrective Action: Flag data associated with unacceptable Matrix Spike

TABLE 8.4-6
Organic Laboratory Quality Control Samples
(Continued)

Analysis	QC Sample	Method	NPDES[1]	Method	RCRA (SW846)[2]
Pesticides/ PCBs (continued)	Matrix Spike Duplicate	608	Not Applicable	8081A 80882	Frequency: 1 with each extraction batch of samples not to exceed 20 samples Criteria: percent recovery for each analyte should be within laboratory acceptance limits Corrective Action: Flag data associated with unacceptable Matrix Spike
	Duplicate	608	Not Applicable	8081A 8082	Not Applicable
	Surrogates	608	Not specified in method	8081A 8082	Surrogates spiked into method blank and all samples (QC included) Method Blank Criteria and LCS: Results must fall within laboratory established control limits Sample Criteria: Re-extract and reanalyze samples or flag sample data not meeting surrogate criteria
	Internal Standards	608	Optional	8081A 8082	Optional

TABLE 8.4-6
Organic Laboratory Quality Control Samples
(Continued)

Analysis	QC Sample	Method	NPDES[1]	Method	RCRA (SW846)[2]
Petroleum Hydro-carbons/Oil and Grease	Method Blank	413.1 418.1	Frequency: 1 with each extraction batch of samples not to exceed 20 samples Criteria: Concentration less than reporting limit Corrective Action: Rerun all samples associated with unacceptable blank Method 413.1: Not Applicable	9070 9071A	Frequency: 1 with each extraction batch of samples not to exceed 20 samples Criteria: Concentration less than reporting limit Corrective Action: Rerun all samples associated with unacceptable blank

TABLE 8,4-6
Organic Laboratory Quality Control Samples
(Continued)

Analysis	QC Sample	Method	NPDES[1]	Method	RCRA (SW846)[2]
Petroleum Hydro-carbons/Oil and Grease (continued)	Laboratory Control Sample	413.1 418.1	Frequency: 1 with each extraction batch of samples not to exceed 20 samples Criteria: Percent recovery must be within laboratory control limits Corrective Action: If not within laboratory control limits, rerun all associated samples Method 413.1: Not Applicable	9070 9071A	Frequency: 1 with each extraction batch of samples not to exceed 20 samples Criteria: Percent recovery must be within ± 20% Corrective Action: Rerun all samples associated with unacceptable LCS
	Matrix Spike	413.1 418.1	Frequency: 1 with each extraction batch of samples not to exceed 20 samples Criteria: Percent recovery must be within laboratory control limits Corrective Action: Flag associated data Method 413.1: Not Applicable	9070 9071A	Frequency: 1 with each extraction batch of samples not to exceed 20 samples Criteria: Percent recovery must be within laboratory control limits Corrective Action: Flag associated data
	Matrix Spike Duplicate	413.1 418.1	Not Applicable	9070 9071A	Frequency: 1 with each extraction batch of samples not to exceed 20 samples Criteria: Percent recovery must be within laboratory control limits Corrective Action: Flag associated Method 9071: Not Applicable

TABLE 8.4-6
Organic Laboratory Quality Control Samples
(Continued)

Analysis	QC Sample	Method	NPDES[1]	Method	RCRA (SW846)[2]
Petroleum Hydro-carbons/Oil and Grease (continued)	Duplicate	413.1 418.1	Not Applicable	9070 9071A	Frequency: 1 with each extraction batch of samples not to exceed 20 samples Criteria: Percent recovery must be within laboratory control limits Corrective Action: Flag associated Method 9070: Not Applicable
	Surrogates	413.1 418.1	Not Applicable	9070 9071A	Not Applicable
	Internal Standards	413.1 418.1 1664A	Not Applicable	9070 9071A 9071B	Not Applicable
Petroleum Hydro-carbons	Method Blank	1664A[4]	Frequency: 1 with each preparation batch Criteria: Concentration must be less than the reporting limit Corrective Action: Rerun all samples associated with unacceptable blank	9071B	Frequency: 1 with each preparation batch Criteria: Concentration must be less than the reporting limit Corrective Action: Rerun all samples associated with unacceptable blank

TABLE 8.4-6
Organic Laboratory Quality Control Samples
(Continued)

Analysis	QC Sample	Method	NPDES[1]	Method	RCRA (SW846)[2]
Petroleum Hydro-carbons (continued)	Laboratory Control Sample	1664A	Frequency: 1 with each analytical batch Criteria: Waters - See limits in SOP, NC-WC-0084 Soils - Percent recovery must be within laboratory control limits Corrective Action: Rerun all samples associated with unacceptable LCS	9071B	Frequency: 1 with each analytical batch Criteria: Waters - See limits in SOP, NC-WC-0084 Soils - Percent recovery must be within laboratory control limits Corrective Action: Rerun all samples associated with unacceptable LCS
	Matrix Spike	1664A	Frequency: 1 with every 10 samples per site Criteria: See percent recovery limits in SOP, NC-WC-0084 Corrective Action: See SOP, NC-WC-0084	9071B	Frequency: 1 with every 10 samples per site Criteria: See percent recovery limits in SOP, NC-WC-0084 Corrective Action: See SOP, NC-WC-0084
	Matrix Spike Duplicate	1664A	Frequency: 1 with every 10 samples per site Criteria: See percent recovery and RPD limits in SOP, NC-WC-0084 Corrective Action: See NC-WC-0084	9071B	Frequency: 1 with every 10 samples per site Criteria: See percent recovery and RPD limits in SOP, NC-WC-0084 Corrective Action: See NC-WC-0084
	Duplicate	1664A	Not Applicable	9071B	Not Applicable

TABLE 8.4-6
Organic Laboratory Quality Control Samples
(Continued)

Analysis	QC Sample	Method	NPDES[1]	Method	RCRA (SW846)[2]
Purgeable Halocarbons by GC	Method Blank	601	Frequency: 1 with each extraction batch of samples not to exceed 20 samples Criteria: Concentration less than reporting limit Corrective Action: Rerun all samples associated with unacceptable blank	8021B	Frequency: 1 with each extraction batch of samples not to exceed 20 samples Criteria: Concentration less than reporting limit Corrective Action: Rerun all samples associated with unacceptable blank
	Laboratory Control Sample	601	Frequency: 1 with each extraction batch of samples not to exceed 20 samples Criteria: percent recovery must be within acceptance limits given in method for each analyte Corrective Action: Rerun all samples associated with unacceptable LCS	8021B	Frequency: 1 with each extraction batch of samples not to exceed 20 samples Criteria: percent recovery for each analyte must be within laboratory acceptance limits Corrective Action: Rerun all samples associated with unacceptable LCS
	Matrix Spike	601	Frequency: 1 per 10 samples from each site or 1 per month, whichever is more frequent Criteria: percent recovery for each analyte should be within advisory limits given in method Corrective Action: Flag data associated with unacceptable Matrix Spike	8021B	Frequency: 1 with each extraction batch of samples not to exceed 20 samples Criteria: percent recovery for each analyte should be within laboratory acceptance limits Corrective Action: Flag data associated with unacceptable Matrix Spike

TABLE 8.4-6
Organic Laboratory Quality Control Samples
(Continued)

Analysis	QC Sample	Method	NPDES[1]	Method	RCRA (SW846)[2]
Purgeable Halocarbons by GC (continued)	Matrix Spike Duplicate	601	Not Applicable	8021B	Frequency: 1 with each extraction batch of samples not to exceed 20 samples Criteria: percent recovery for each analyte should be within laboratory acceptance limits Corrective Action: Flag data associated with unacceptable Matrix Spike
	Duplicate	601	Not Applicable	8021B	Not Applicable
	Surrogates	601	Surrogates spiked into method blank and all samples (QC included) Method Blank Criteria and LCS: All surrogates must be within laboratory established control limits before sample analysis may proceed. Sample Criteria: Re-extract samples or flag sample data not meeting surrogate criteria	8021B	Surrogates spiked into method blank and all samples (QC included) Method Blank Criteria and LCS: All surrogates must be within laboratory established control limits before sample analysis may proceed. Sample Criteria: Re-extract and reanalyze samples or flag sample data not meeting surrogate criteria
	Internal Standards	601	Optional: Internal standards are added to the method blank and all samples (QC included). If used, same compounds as used for surrogates may be appropriate.	8021B	Optional: Internal standards are added to the method blank and all samples (QC included). If used, same compounds as used for surrogates may be appropriate.

TABLE 8.4-6
Organic Laboratory Quality Control Samples
(Continued)

Analysis	QC Sample	Method	NPDES[1]	Method	RCRA (SW846)[2]
Semivolatiles	Method Blank	625	Frequency: 1 with each extraction batch of samples not to exceed 20 samples Criteria: Concentration less than reporting limit Corrective Action: Rerun all samples associated with unacceptable blank	8270C	Frequency: 1 with each extraction batch of samples not to exceed 20 samples Criteria: Concentration less than reporting limit Corrective Action: Reextract and reanalyze all samples associated with unacceptable blank
	Laboratory Control Sample	625	Frequency: 1 with each extraction batch of samples not to exceed 20 samples Criteria: percent recovery must be within acceptance limits given in method for each analyte Corrective Action: Rerun all samples associated with unacceptable LCS	8270C	Frequency: 1 with each extraction batch of samples not to exceed 20 samples Criteria: percent recovery for each analyte must be within laboratory acceptance limits Corrective Action: Reextract and reanalyze all samples associated with unacceptable LCS
	Matrix Spike	625	Frequency: 1 with each extraction batch of samples not to exceed 20 samples Criteria: percent recovery for each analyte should be within advisory limits given in method Corrective Action: Flag data associated with unacceptable Matrix Spike	8270C	Frequency: 1 with each extraction batch of samples not to exceed 20 samples Criteria: percent recovery for each analyte should be within laboratory acceptance limits Corrective Action: Flag data associated with unacceptable Matrix Spike

TABLE 8.4-6
Organic Laboratory Quality Control Samples
(Continued)

Analysis	QC Sample	Method	NPDES[1]	Method	RCRA (SW846)[2]
Semivolatiles (continued)	Matrix Spike Duplicate	625	Not Applicable	8270C	Frequency: 1 with each extraction batch of samples not to exceed 20 samples Criteria: percent recovery for each analyte should be within laboratory acceptance limits Corrective Action: Flag data associated with unacceptable Matrix Spike
	Duplicate	625	Not Applicable	8270C	Not Applicable
	Surrogates	625	Surrogates spiked into method blank and all samples (QC included) Method Blank and LCS Criteria: All surrogates must be in control before sample analysis may proceed Sample Criteria: Re-extract samples or flag sample data not meeting surrogate criteria	8270C	Surrogates spiked into method blank and all samples (QC included) Method Blank and LCS Criteria: All surrogates must be in control before sample analysis may proceed Sample Criteria: Re-extract and reanalyze samples or flag sample data not meeting surrogate criteria
	Internal Standards	625	Frequency: Internal standards spiked into method blank and all samples (QC included) Criteria: All internal standard recoveries must be within laboratory control limits Corrective Action: Flag sample data not meeting internal standard recovery requirements	8270C	Internal Standards are added to all samples (QC samples included). Internal standard area of daily standard must be within 50% to 200% of the response in the mid level of the initial calibration standard. The retention time (RT) for any internal standard (IS) in the continuing calibration must not exceed ± 0.5 minutes from mid level initial calibration standard IS RT.

TABLE 8.4-6
Organic Laboratory Quality Control Samples
(Continued)

Analysis	QC Sample	Method	NPDES[1]	Method	RCRA (SW846)[2]
Volatiles by GC/MS	Method Blank	624	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Concentration less than reporting limit Corrective Action: Rerun all samples associated with unacceptable blank	8260B	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Concentration less than reporting limit Corrective Action: Rerun all samples associated with unacceptable blank
	Laboratory Control Sample	624	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: percent recovery for each analyte should be within advisory limits given in method Corrective Action: Flag data associated with unacceptable Matrix Spike	8260B	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: percent recovery for each analyte must be within laboratory acceptance limits Corrective Action: Rerun all samples associated with unacceptable LCS
	Matrix Spike	624	Frequency: 1 per ≤ 20 samples from each site or 1 per month, whichever is more frequent Criteria: percent recovery for each analyte should be within advisory limits given in method Corrective Action: Flag data associated with unacceptable Matrix Spike	8260B	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: percent recovery for each analyte should be within laboratory acceptance limits Corrective Action: Flag data associated with unacceptable Matrix Spike

TABLE 8.4-6
Organic Laboratory Quality Control Samples
(Continued)

Analysis	QC Sample	Method	NPDES[1]	Method	RCRA (SW846)[2]
Volatiles by GC/MS (continued)	Matrix Spike Duplicate	624	Not Applicable	8260B	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: percent recovery for each analyte should be within laboratory acceptance limits Corrective Action: Flag data associated with unacceptable Matrix Spike
	Duplicate	624	Not Applicable	8260B	Not Applicable
	Surrogates	624	Surrogates spiked into Method Blank and all samples (QC included) Method Blank Criteria: All surrogates must be in control before sample analysis may proceed. Sample Criteria: Re-extract samples or flag sample data not meeting surrogate criteria	8260B	Surrogates spiked into Method Blank and all samples (QC included) Method Blank Criteria and LCS: All surrogates must be in control before sample analysis may proceed. Sample Criteria: Re-extract and reanalyze samples or flag sample data not meeting surrogate criteria
	Internal Standards	624	Frequency: Internal standards spiked into method blank and all samples (QC included) Criteria: All internal standard recoveries must be within laboratory control limits Corrective Action: Flag sample data not meeting internal standard recovery requirements	8260B	Internal Standards are added to all samples (QC samples included). Internal standard area of daily standard must be within 50% to 200% of the response in the mid level of the initial calibration standard. The retention time (RT) for any internal standard (IS) in the continuing calibration must not exceed ± 0.5 minutes from mid level initial calibration standard IS RT.

Footnotes

(1) National Pollutant Discharge Elimination System
(2) Resource Conservation and Recovery Act, Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, (SW-846), Third Edition, September 1986. Contains Final Update I (July 1992), Final Update IIA (August 1993), Final Update II (September 1994), Final Update IIB (January 1995), and Final Update III (December 1996).
(3) Method not listed in 40 CFR Part 136.
(4) footnote deleted
(5) Method 300.0 is a proposed 40CFR method. Specific state and/or region approval is required for NPDES.
(6) EPA issued memo on the recommendation not to utilize reactive cyanide and sulfide methods
(7) STL North Canton does not perform organic drinking water methods (500 series)

TABLE 8.4-7
USEPA Contract Laboratory Program Statement of Work Quality Control Samples

Analysis	QC Sample	Method	Requirement
Cyanide, Total	Method Blank	ILM03.0 ILMO4.0	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria ILMO3.0: Concentration less than CRDL or less than 10x sample concentration Criteria ILMO4.0: If method blank is > CRDL, sample results are acceptable if they are ≥ 10-times method blank level. Corrective Action: Reprepare all samples associated with unacceptable blank
	Laboratory Control Sample	ILM03.0 ILMO4.0	Frequency: 1 with each batch of samples processed or for each SDG, whichever is more frequent Criteria: Water - 80-120% Solid - Meet control limits established for solid reference material Corrective Action: Reprepare all samples associated with unacceptable LCS
	Matrix Spike	ILM03.0 ILMO4.0	Frequency: 1 with each group of samples of a similar matrix type and concentration or for each SDG, whichever is more frequent Criteria: 75-125% unless sample result > 4x spike amount Corrective Action: Flag data associated with unacceptable Matrix Spike, perform post distillation spike at 2 x CRDL or 2x sample concentration whichever is greater
	Matrix Spike Duplicate	ILM03.0 ILMO4.0	Not Applicable
	Duplicate	ILM03.0 ILMO4.0	Frequency: 1 with each group of samples of a similar matrix type and concentration or for each SDG whichever is more frequent Criteria: RPD ≤ 20% or ± CRDL if sample or duplicate value < 5x CRDL Corrective Action: Flag all associated data associated if duplicate results outside control limits
	Surrogates	ILM03.0 ILMO4.0	Not Applicable
	Internal Standards	ILM03.0 ILMO4.0	Not Applicable

TABLE 8.4-7
USEPA Contract Laboratory Program Statement of Work Quality Control Samples (Continued)

Analysis	QC Sample	Method	Requirement
ICAP (excludes mercury)	Method Blank	ILM03.0 ILMO4.0	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria ILMO3.0: Concentration less than CRDL or less than 10x sample concentration Criteria ILMO4.0: If method blank is > CRDL, sample results are acceptable if they are ≥ 10-times method blank level. Corrective Action: Reprepare all samples associated with unacceptable blank
	Laboratory Control Sample	ILM03.0 ILMO4.0	Frequency: 1 with each batch of samples processed or for each SDG, whichever is more frequent Criteria: Water - 80-120% except silver and antimony Solid - Meet control limits established for solid reference material Corrective Action: Reprepare all samples associated with unacceptable LCS
	Matrix Spike	ILM03.0 ILMO4.0	Frequency: 1 with each group of samples of a similar matrix type and concentration or for each SDG whichever is more frequent Criteria: 75-125% unless sample result > 4x spike amount Corrective Action: Flag data associated with unacceptable Matrix perform post digestion spike at 2xCRDL or 2x sample concentration whichever is greater
	Matrix Spike Duplicate	ILM03.0 ILMO4.0	Not Applicable
	Duplicate	ILM03.0 ILMO4.0	Frequency: 1 with each group of samples of a similar matrix type and concentration or for each SDG whichever is more frequent Criteria: RPD ≤ 20% or ± CRDL if sample or duplicate value < 5x CRDL Corrective Action: Flag all data associated with duplicate results outside control limits

TABLE 8.4-7
USEPA Contract Laboratory Program Statement of Work Quality Control Samples
(Continued)

Analysis	QC Sample	Method	Requirement
ICAP (excludes mercury) (continued)	Serial Dilution	ILM03.0 ILMO4.0	Frequency: 1 with each group of samples of a similar matrix type and concentration or for each SDG whichever is more frequent Criteria: <10% D when sample concentration > 50x IDL Corrective Action: Flag all data associated with results outside control limits
	Surrogates	ILM03.0 ILMO4.0	Not Applicable
	Internal Standards	ILM03.0 ILMO4.0	Not Applicable.
GFAA (excludes mercury)	Method Blank	ILM03.0 ILMO4.0	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria ILMO3.0: Concentration less than CRDL or less than 10x sample concentration Criteria ILMO4.0: If method blank is > CRDL, sample results are acceptable if they are ≥ 10-times method blank level. Corrective Action: Reprepare all samples associated with unacceptable blank
	Laboratory Control Sample	ILM03.0 ILMO4.0	Frequency: 1 with each batch of samples processed or for each SDG, whichever is more frequent Criteria: Water - 80-120% except silver and antimony Solid - Meet control limits established for solid reference material Corrective Action: Reprepare all samples associated with unacceptable LCS
	Matrix Spike	ILM03.0 ILMO4.0	Frequency: 1 with each group of samples of a similar matrix type and concentration or for each SDG whichever is more frequent Criteria: 75-125% unless sample result > 4x spike amount Corrective Action: Flag data associated with unacceptable Matrix
	Matrix Spike Duplicate	ILM03.0 ILMO4.0	Not Applicable

TABLE 8.4-7
USEPA Contract Laboratory Program Statement of Work Quality Control Samples
(Continued)

Analysis	QC Sample	Method	Requirement
GFAA (excludes mercury) (continued)	Duplicate	ILM03.0 ILMO4.0	Frequency: 1 with each group of samples of a similar matrix type and concentration or for each SDG whichever is more frequent Criteria: RPD ≤ 20% or ± CRDL if sample or duplicate value < 5x CRDL Corrective Action: Flag all associated data associated if duplicate results outside control limits
	Analytical Spike	ILM03.0 ILMO4.0	Frequency: 1 with each sample except matrix spike Criteria: Evaluate per method requirements Corrective action: Perform per method requirements
	Surrogates	ILM03.0 ILMO4.0	Not Applicable
	Internal Standards	ILM03.0 ILMO4.0	Not Applicable.
Mercury (CVAA)	Method Blank	ILM03.0 ILMO4.0	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria ILMO3.0: Concentration less than CRDL Criteria ILMO4.0: If method blank is > CRDL, sample results are acceptable if they are ≥ 10-times method blank level. Corrective Action: Reprepare all samples associated with unacceptable blank
	Laboratory Control Sample	ILM03.0 ILMO4.0	Frequency: 1 with each batch of samples processed or for each SDG, whichever is more frequent Criteria: Water - 80-120% Solid - Meet control limits established for solid reference material Corrective Action: Reprepare all samples associated with unacceptable LCS
	Matrix Spike	ILM03.0 ILMO4.0	Frequency: 1 with each group of samples of a similar matrix type and concentration or for each SDG Criteria: 75-125% unless sample result > 4x spike amount Corrective Action: Flag data associated with unacceptable Matrix Spike

TABLE 8.4-7
USEPA Contract Laboratory Program Statement of Work Quality Control Samples
(Continued)

Analysis	QC Sample	Method	Requirement
Mercury (CVAA) (continued)	Matrix Spike Duplicate	ILM03.0 ILMO4.0	Not Applicable
	Duplicate	ILM03.0 ILMO4.0	Frequency: 1 with each group of samples of a similar matrix type and concentration or for each SDG whichever is more frequent Criteria: RPD ≤ 20% or ± CRDL if sample or duplicate value < 5x CRDL Corrective Action: Flag all associated data associated if duplicate results outside control limits
	Surrogates	ILM03.0 ILMO4.0	Not Applicable
	Internal Standards	ILM03.0 ILMO4.0	Not Applicable.

TABLE 8.4-7
USEPA Contract Laboratory Program Statement of Work Quality Control Samples (Continued)

Analysis	QC Sample	Method	Requirement
Pesticides/PCBs	Method Blank	OLM03.1	Frequency: 1 with each case of samples received (up to 20 samples), for each extraction procedure within each SDG, whichever is most frequent or whenever samples are extracted Criteria: Concentration < CRQL Corrective Action: Re-extract and reanalyze all samples associated with unacceptable blank
	Laboratory Control Sample	OLM03.1	Not Applicable.
	Matrix Spike	OLM03.1	Frequency: 1 with each case of samples received (up to 20 samples), for each extraction procedure or for each SDG, whichever is most frequent Criteria: Percent recovery for each analyte should be within advisory limits given in method Corrective Action: Flag data associated with Matrix Spike recoveries outside of advisory limits

TABLE 8.4-7
USEPA Contract Laboratory Program Statement of Work Quality Control Samples (Continued)

Analysis	QC Sample	Method	Requirement
Pesticides/PCBs (continued)	Matrix Spike Duplicate	OLM03.1	Frequency: 1 with each case of samples received (up to 20 samples), for each concentration level (soils) or for each SDG, whichever is most frequent Criteria: Percent recovery for each analyte should be within advisory limits given in method RPD between MS/MSD should be within advisory limits given in method Corrective Action: Flag data associated with Matrix Spike recoveries or RPD outside of advisory limits
	Duplicate	OLM03.1	Not Applicable
	Surrogates	OLM03.1	Frequency: Surrogates spiked onto all samples and QC samples Criteria: Percent recovery for each surrogate in samples should be within 30-150% Percent recovery for each surrogate in the method blank must be 30-150% Corrective Action: Flag unacceptable surrogate recoveries in samples Re-extract all samples associated with unacceptable surrogate recoveries in the method blank
	Internal Standards	OLM03.1	Not Applicable.
Semivolatiles by GC/MS	Method Blank	OLM03.1	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Concentration less than CRQL except phthalates which must be ≤ 5x CRQL Corrective Action: Re-extract and re-analyze all samples associated with unacceptable blank
	Laboratory Control Sample	OLM03.1	Not Applicable.

TABLE 8.4-7
USEPA Contract Laboratory Program Statement of Work Quality Control Samples (Continued)

Analysis	QC Sample	Method	Requirement
Semivolatiles by GC/MS (continued)	Matrix Spike	OLM03.1	Frequency: 1 with each case of samples received (up to 20 samples), for each concentration level (soils) or for each SDG, whichever is most frequent Criteria: Percent recovery for each analyte should be within advisory limits given in method Corrective Action: Flag data associated with Matrix Spike outside of advisory limits
	Matrix Spike Duplicate	OLM03.1	Frequency: 1 with each case of samples received (up to 20 samples), for each concentration level (soils) or for each SDG, whichever is most frequent Criteria: Percent recovery for each analyte should be within advisory limits given in method RPD between MS/MSD should be within advisory limits given in method Corrective Action: Flag data associated with Matrix Spike recoveries or RPD outside of advisory limits
	Duplicate	OLM03.1	Not Applicable
	Surrogates	OLM03.1	Frequency: Surrogates spiked onto all samples and QC samples Criteria: Percent recovery for each surrogate must be within limits given in method (one base/neutral and/or one acid surrogate may be outside of limits but not below 10%) Corrective Action: Flag data associated with unacceptable recoveries or reanalyze all samples with unacceptable surrogate recoveries as required in method
	Internal Standards	OLM03.1	Frequency: Internal Standards are spiked onto all samples and QC samples Criteria: Internal Standard areas must be within -50% to + 100% from the last daily calibration check standard Corrective Action: Reanalyze all samples with unacceptable areas

TABLE 8.4-7
USEPA Contract Laboratory Program Statement of Work Quality Control Samples (Continued)

Analysis	QC Sample	Method	Requirement
Volatiles by GC/MS	Method Blank	OLM03.1	Frequency: 1 per 12 hours Criteria: Concentration less than CRQL except methylene chloride, acetone, 2-butanone must be ≤ 5x CRQL Corrective Action: Reanalyze all samples associated with unacceptable blank
	Laboratory Control Sample	OLM03.1	Not Applicable
	Matrix Spike	OLM03.1	Frequency: 1 with each case of samples received (up to 20 samples), for each concentration level (soils) or for each SDG, whichever is most frequent Criteria: Percent recovery for each analyte should be within advisory limits given in method Corrective Action: Flag data associated with Matrix Spike outside of advisory limits

TABLE 8.4-7
USEPA Contract Laboratory Program Statement of Work Quality Control Samples
(Continued)

Analysis	QC Sample	Method	Requirement
Volatiles by GC/MS (continued)	Matrix Spike Duplicate	OLM03.1	Frequency: 1 with each case of samples received (up to 20 samples), for each concentration level (soils) or for each SDG, whichever is most frequent Criteria: Percent recovery for each analyte should be within advisory limits given in method RPD between MS/MSD should be within advisory limits given in method Corrective Action: Flag data associated with Matrix Spike recoveries or RPD outside of advisory limits
	Duplicate	OLM03.1	Not Applicable
	Surrogates	OLM03.1	Frequency: Surrogates spiked onto all samples and QC samples Criteria: Percent recovery for each surrogate must be within limits given in method Corrective Action: Reanalyze all samples with unacceptable surrogate recoveries
	Internal Standards	OLM03.1	Frequency: Internal Standards are spiked onto all samples and QC samples Criteria: Internal Standard areas must be within -50% to + 100% from the last daily calibration check standard Corrective Action: Reanalyze all samples with unacceptable Internal Standard areas
	Storage Blank	OLM03.1	Frequency: 1 per SDG Criteria: Concentration less than CRQL except methylene chloride, acetone, 2-butanone must be ≤ 5x CRQL Corrective Action: Narrate with corrective action plan

Notes:

SDG = Sample Delivery Group

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TABLE 8.5-1
Inorganic Sample Containers, Preservatives, and Holding Times

Analytical Parameters	Matrix	Minimum Sample Size[1]	NPDES[2], [3]		RCRA (SW846)[3], [4]	
			Method	Requirements	Method	Requirements
Alkalinity	Water	100 mL	310.1 2320B	250 mL plastic or glass, Cool, 4°C, 14 days	---	Not Applicable
	Solid	Not Applicable	---	Not Applicable	---	Not Applicable
	Waste	Not Applicable	---	Not Applicable	---	Not Applicable
Ammonia	Water	400 mL	350.1	500 mL plastic or glass, Cool, 4°C H_2SO_4 to pH < 2, 28 days	---	Not Applicable
	Solid	Not Applicable	---	Not Applicable	---	Not Applicable
	Waste	Not Applicable	---	Not Applicable	---	Not Applicable
Biochemical Oxygen Demand (BOD)	Water	200 mL	405.1	1000 mL plastic or glass, Cool, 4°C 48 hours	---	Not Applicable
	Solid	Not Applicable	---	Not Applicable	---	Not Applicable
	Waste	Not Applicable	---	Not Applicable	---	Not Applicable
Bromide	Water	100 mL	300.0[7]	250 mL plastic or glass, No preservative required, 28 days	9056	Cool, 4°C, analyze ASAP after collection
	Solid	Not Applicable	---	Not Applicable	---	Not Applicable
	Waste	Not Applicable	---	Not Applicable	---	Not Applicable
Chemical Oxygen Demand (COD)	Water	100 mL	410.4	250 mL glass or plastic, Cool, 4°C, H_2SO_4 to pH < 2, 28 days	---	Not Applicable
	Solid	Not Applicable	---	Not Applicable	---	Not Applicable
	Waste	Not Applicable	---	Not Applicable	---	Not Applicable

TABLE 8.5-1
Inorganic Sample Containers, Preservatives, and Holding Times (Continued)

Analytical Parameters	Matrix	Minimum Sample Size[1]	NPDES[2], [3], [7]		RCRA (SW846)[3], [4]	
			Method	Requirements	Method	Requirements
Chloride	Water	50 mL	300.0[7] 325.2 325.3	250 mL plastic or glass, No preservative required, 28 days	9056 9252	Method 9056: Cool, 4°C, analyze ASAP after collection. Method 9251/9253: 250ml plastic or glass, no preservative required, 28 days
	Solid	Not Applicable	---	Not Applicable	---	Not Applicable
	Waste	Not Applicable	---	Not Applicable	---	Not Applicable
Chlorine, Residual	Water	100 mL	330.5	250 mL glass or plastic, Cool, 4°C, analyze immediately	---	Not Applicable
	Solid	Not Applicable	---	Not Applicable	---	Not Applicable
	Waste	Not Applicable	---	Not Applicable	---	Not Applicable
Chromium (Cr^{+6})	Water	100 mL	3500 Cr-D	Method 218.4: 200 mL plastic or glass, Cool, 4°C, 24 hours Method 3500 Cr-D: 200 mL quartz, TFE, or polypropylene HNO_3 to pH <2 Cool, 4°C Analyze ASAP after collection	7196A	200 mL plastic or glass, Cool, 4°C, 24 hours
	Solid	Not Applicable	---	Not Applicable	7196A	250 mL plastic or glass, 30 days to digestion, 96 hours after digestion
	Waste	Not Applicable	---	Not Applicable	---	Not Applicable

TABLE 8.5-1
Inorganic Sample Containers, Preservatives, and Holding Times
(Continued)

Analytical Parameters	Matrix	Minimum Sample Size[1]	NPDES[2], [3], [7]		RCRA (SW846)[3], [4]	
			Method	Requirements	Method	Requirements
Conductivity	Water	100 mL	120.1	200 mL glass or plastic, Cool, 4°C, 28 days	9050A	200 mL glass or plastic, Cool, 4°C, 24 hours
	Solid	Not Applicable	---	Not Applicable	---	Not Applicable
	Waste	Not Applicable	---	Not Applicable	---	Not Applicable
Cyanide (Amenable)	Water	IL	335.1	1 liter plastic or glass, NaOH to pH >12 0.6g ascorbic acid[6] Cool, 4°C, 14 days unless sulfide is present. Then maximum holding time is 24 hours	9012A	1 liter plastic or glass, NaOH to pH >12 0.6g ascorbic acid[6] Cool, 4°C, 14 days
	Solid	50g	---	Not Applicable	9012A	Not Specified
	Waste	50g	---	Not Applicable	9012A	Not Specified
Cyanide (Total)	Water	IL	335.2 335.3 335.4 [7]	1 liter plastic or glass, NaOH to pH >12 0.6g ascorbic acid[6] Cool, 4°C, 14 days unless sulfide is present. Then maximum holding time is 24 hours	9012A	1 liter plastic or glass, NaOH to pH >12 0.6g ascorbic acid[6] Cool, 4°C, 14 days
	Solid	50g	--	Not Applicable	9012A	8 or 16 oz glass Teflon-lined lids, Cool, 4°C, 14 days

TABLE 8.5-1
Inorganic Sample Containers, Preservatives, and Holding Times
(Continued)

Analytical Parameters	Matrix	Minimum Sample Size[1]	NPDES[2], [3], [7]		RCRA (SW846)[3], [4]	
			Method	Requirements	Method	Requirements
Cyanide (Total) (continued)	Waste	50g	--	Not Applicable	9012A	8 or 16 oz glass Teflon-lined lids, Cool, 4°C
Flashpoint (Ignitability)	Liquid	Not Applicable	---	Not Applicable	1010	No requirements, 250 mL amber glass, Cool, 4°C is recommended
	Solid	Not Applicable	--	Not Applicable	---	Not Applicable
	Waste	Not Applicable	--	Not Applicable	---	Not Applicable
Fluoride	Water	300 mL	300.0[7] 340.2	500 mL plastic, No preservation required, 28 days	9056	Cool, 4°C, analyze ASAP after collection
	Solid	Not Applicable	---	Not Applicable	---	Not Applicable
	Waste	Not Applicable	---	Not Applicable	---	Not Applicable
Hardness (Total)	Water	50 mL	130.2 2340B	250 mL glass or plastic, HNO_3 to pH < 2, 6 months	---	Not Applicable
	Solid	Not Applicable	---	Not Applicable	---	Not Applicable
	Waste	Not Applicable	---	Not Applicable	---	Not Applicable
Iron (Ferrous)	Water	100 mL	3500-Fe D	1 liter glass or polyethylene container, 6 months **This test should be performed in the field.**	-	Not Applicable
	Solid	Not Applicable	-	Not Applicable	-	Not Applicable
	Waste	Not Applicable	-	Not Applicable	-	Not Applicable

TABLE 8.5-1
Inorganic Sample Containers, Preservatives, and Holding Times
(Continued)

Analytical Parameters	Matrix	Minimum Sample Size[1]	NPDES[2], [3], [7]		RCRA (SW846)[3], [4]	
			Method	Requirements	Method	Requirements
Nitrate	Water	100 mL	300.0[7] 353.2	Method 300.0: 250 mL plastic or glass, Cool, 4°C, 48 hours.	9056	Method 9056: Cool, 4°C, analyze ASAP after collection
	Solid	Not Applicable	---	Not Applicable	---	Not Applicable
	Waste	Not Applicable	---	Not Applicable	---	Not Specified
Nitrite	Water	50 mL	300.0[7] 353.2	250 mL plastic or glass Cool, 4°C, 48 hours	9056	Cool, 4°C, analyze ASAP after collection
	Solid	Not Applicable	---	Not Applicable	---	Not Applicable
	Waste	Not Applicable	---	Not Applicable	---	Not Applicable

TABLE 8.5-1
Inorganic Sample Containers, Preservatives, and Holding Times
(Continued)

Analytical Parameters	Matrix	Minimum Sample Size[1]	NPDES[2], [3], [7]		RCRA (SW846)[3], [4]	
			Method	Requirements	Method	Requirements
Nitrate-Nitrite	Water	100 mL	353.1 353.2	250 mL plastic or glass, H_2SO_4 to pH < 2, 28 days	---	Not Applicable
	Solid	Not Applicable	---	Not Applicable	---	Not Applicable
	Waste	Not Applicable	---	Not Applicable	---	Not Applicable
Ortho-phosphate	Water	50 mL	300.0[7] 365.2 365.3	100 mL plastic or glass, Filter on site Cool, 4°C, 48 hours	9056	Cool, 4°C, analyze ASAP collection
	Solid	Not Applicable	---	Not Applicable	---	Not Applicable
	Waste	Not Applicable	---	Not Applicable	---	Not Applicable
pH	Water	50 mL	150.1	100 mL plastic or glass. Analyze immediately. **This test should be performed in the field.**	9040B	100 mL plastic or glass. Analyze immediately. **This test should be performed in the field.**[8]
	Solid	Not Applicable	---	Not Applicable	9045C	4 oz glass or plastic, Cool, 4°C, Analyze as soon as possible.[8]
	Waste	Not Applicable	---	Not Applicable	9045C	4 oz glass or plastic, Cool, 4°C, Analyze as soon as possible.[8]
Phenolics	Water	100 mL	420.1	500 mL glass, Cool, 4°C, H_2SO_4 to pH < 2, 28 days	9065 9066	1 liter glass recommended, Cool, 4°C, H_2SO_4 to pH < 4, 28 days
	Solid	Not Applicable	---	Not Applicable	---	Not Applicable
	Waste	Not Applicable	---	Not Applicable	9065	Not Specified
Phosphate	Water	50 mL	---	Not Applicable	9056	Cool, 4°C, analyze ASAP collection
	Solid	Not Applicable	---	Not Applicable	9056	Not Applicable
	Waste	Not Applicable	---	Not Applicable	9056	Not Applicable

TABLE 8.5-1
Inorganic Sample Containers, Preservatives, and Holding Times
(Continued)

Analytical Parameters	Matrix	Minimum Sample Size[1]	NPDES[2], [3], [7]		RCRA (SW846)[3], [4]	
			Method	Requirements	Method	Requirements
Phosphorus (Total)	Water	50 mL	365.2 365.3	100 mL plastic or glass, H_2SO_4 to pH < 2, 28 days	---	Not Applicable
	Solid	Not Applicable	---	Not Applicable	---	Not Applicable
	Waste	Not Applicable	---	Not Applicable	---	Not Applicable
Reactivity[9] (Cyanide and Sulfide)	Liquid	10 g	---	Not Applicable	Chapter 7 Sections 7.3.3.2 and 7.3.4.2	10 oz amber glass, Cool, 4°C, no headspace, analyze as soon as possible.
	Solid	10 g	---	Not Applicable	Chapter 7 Sections 7.3.3.2 and 7.3.4.2	10 oz amber glass, Cool, 4°C, no headspace, analyze as soon as possible.
	Waste	10 g	---	Not Applicable	Chapter 7 Sections 7.3.3.2 and 7.3.4.2	10 oz amber glass, Cool, 4°C, no headspace, analyze as soon as possible.
Settleable Solids	Water	1000 mL	160.5	1000 mL plastic or glass, Cool, 4°C, 48 hours	---	Not Applicable
	Solid	Not Applicable	---	Not Applicable	---	Not Applicable
	Waste	Not Applicable	---	Not Applicable	---	Not Applicable

TABLE 8.5-1
Inorganic Sample Containers, Preservatives, and Holding Times
(Continued)

Analytical Parameters	Matrix	Minimum Sample Size[1]	NPDES[2], [3], [7]		RCRA (SW846)[3], [4]	
			Method	Requirements	Method	Requirements
Specific Conductance	Water	50 mL	120.1	250 mL plastic or glass, Cool, 4°C, 24 hours	9050A	250 mL plastic or glass, Cool, 4°C, 28 days
	Solid	Not Applicable	---	Not Applicable	---	Not Applicable
	Waste	Not Applicable	---	Not Applicable	---	Not Applicable
Sulfate (SO_4)	Water	100 mL	300.0[7] 375.4	100 mL plastic or glass, Cool, 4°C, 28 days	9056 9038	Method 9056: Cool, 4°C, analyze ASAP collection Method 9038: 200 mL plastic or glass, Cool, 4°C, 28 days
	Solid	Not Applicable	---	Not Applicable	---	Not Applicable
	Waste	100 mL	---	Not Applicable	9038	200 mL plastic or glass, Cool, 4°C, 28 days
Sulfide	Water	100 mL	376.1	500 mL plastic or glass, Cool, 4°C, Add 2 mL zinc acetate plus NaOH to pH > 9, 7 days	9030A	500 mL plastic, no headspace, Cool, 4°C, Add 4 drops of 2N zinc acetate per 100 mL of sample, adjust the pH to > 9 with 6 N NaOH solution, 7 days
	Solid	50 g	---	Not Applicable	9030A	Cool, 4°C, fill surface of solid with 2N Zinc acetate until moistened, store headspace-free
	Waste	50 g	---	Not Applicable	9030A	Cool, 4°C, fill surface of solid with 2N Zinc acetate until moistened, store headspace-free

TABLE 8.5-1
Inorganic Sample Containers, Preservatives, and Holding Times
(Continued)

Analytical Parameters	Matrix	Minimum Sample Size[1]	NPDES[2], [3], [7]		RCRA (SW846)[3], [4]	
			Method	Requirements	Method	Requirements
Total Dissolved Solids (Filterable)	Water	100 mL	160.1	250 mL plastic or glass, Cool, 4°C, 7 days	—	Not Applicable
	Solid	Not Applicable	—	Not Applicable	—	Not Applicable
	Waste	Not Applicable	—	Not Applicable	—	Not Applicable
Total Kjeldahl Nitrogen (TKN)	Water	500 mL	351.2 351.3	500 mL plastic or glass, Cool, 4°C, H_2SO_4 to pH < 2, 28 days	—	Not Applicable
	Solid	Not Applicable	—	Not Applicable	—	Not Applicable
	Waste	Not Applicable	—	Not Applicable	—	Not Applicable
Total Organic Carbon (TOC)	Water	100 mL	415.1	100 mL plastic or glass, Cool, 4°C, H_2SO_4 to pH < 2, 28 days	9060 Walkley-Black	100 mL glass or 40 mL VOA vials, Cool, 4°C, H_2SO_4 or HCl to pH < 2, 28 days
	Solid	Not Applicable	—	Not Applicable	9060 Walkley-Black	Not Specified
	Waste	Not Applicable	—	Not Applicable	9060 Walkley-Black	Not Specified

TABLE 8.5-1
Inorganic Sample Containers, Preservatives, and Holding Times
(Continued)

Analytical Parameters	Matrix	Minimum Sample Size[1]	NPDES[2], [3]		RCRA (SW846)[3], [4]	
			Method	Requirements	Method	Requirements
Total Organic Halides (TOX)	Water	100 mL	450.1 [7]	500 mL amber glass, Teflon®-lined lid, Cool, 4°C, HNO_3 to pH <2, no headspace, 28 days	9020B	500 mL amber glass, Teflon®-lined lid, Cool, 4°C, H_2SO_4 to pH < 2, no headspace, 28 days
	Solid	Not Applicable	---	Not Applicable	---	Not Applicable
	Waste	Not Applicable	---	Not Applicable	---	Not Applicable
Total Solids	Water	100 mL	160.3	250 mL plastic or glass, Cool, 4°C, 7 days	---	Not Applicable
	Solid	Not Applicable	---	Not Applicable	---	Not Applicable
	Waste	Not Applicable	---	Not Applicable	---	Not Applicable
Total Suspended Solids (Nonfilterable)	Water	100 mL	160.2	250 mL plastic or glass, Cool, 4°C, 7 days	---	Not Applicable
	Solid	Not Applicable	---	Not Applicable	---	Not Applicable
	Waste	Not Applicable	---	Not Applicable	---	Not Applicable
Turbidity	Water	50 mL	180.1	250 mL plastic or glass, Cool, 4°C, 48 hours	---	Not Applicable
	Solid	Not Applicable	---	Not Applicable	---	Not Applicable
	Waste	Not Applicable	---	Not Applicable	---	Not Applicable
Volatile Solids	Water	100 mL	160.4	250 mL plastic or glass, Cool, 4°C, 7 days	---	Not Applicable
	Solid	Not Applicable	---	Not Applicable	---	Not Applicable
	Waste	Not Applicable	---	Not Applicable	---	Not Applicable

TABLE 8.5-1
Inorganic Sample Containers, Preservatives, and Holding Times (Continued)

Analytical Parameters	Matrix	Minimum Sample Size[1]	NPDES[2], [3]		RCRA (SW846)[3], [4]	
			Method	Requirements	Method	Requirements
Water Content	Water	Not Applicable	---	Not Applicable	---	Not Applicable
	Solid	10 g	---	Refer to specific method used	---	Refer to specific method used
	Waste	10 g	---	Refer to specific method used	---	Refer to specific method used
Metals (excludes Hg)	Water	100 mL	200 series	1 liter glass or polyethylene container, HNO_3 to pH ≤ 2, 6 months	6010B, 6020, 7000A series	1 liter glass or polyethylene container, HNO_3 to pH ≤ 2, 6 months
	Solid	200 g	200 series	8 or 16 oz glass or polyethylene container storage at 4 °C	6010B, 6020, 7000A series	8 or 16 oz glass or polyethylene container, storage at 4°C, 6 months
	Waste	200 g	200 series	Not Applicable	6010B, 6020, 7000A series	8 or 16 oz glass or polyethylene container, storage at 4°C, 6 months
Mercury (CVAA) (CVAFS)	Water	100 mL	245.1 1631B[7] 245.7	1 liter glass or polyethylene container, HNO_3 to pH ≤ 2, 28 days	7470A	1 liter glass or polyethylene container, HNO_3 to pH ≤ 2, 28 days
	Solid	200 g	245.5	8 or 16 oz glass or polyethylene container, Cool, 4°C, 28 days	7471A	8 or 16 oz glass or polyethylene container, Cool, 4°C, 28 days (CORP-MT-0007)
	Waste	200 g	–	Not Applicable	7471A	8 or 16 oz glass or polyethylene container, Cool, 4°C, 28 days (CORP-MT-0007)

TABLE 8.5-1
Inorganic Sample Containers, Preservatives, and Holding Times (Continued)

Footnotes

(1) Minimum sample size indicates sample amount needed for a single analysis. Matrix spikes or duplicates will require an additional sample amount of at least this amount for each additional QC sample aliquot required.
(2) National Pollutant Discharge Elimination System - MCAWW, March 1983.
(3) Holding times are calculated from date of collection.
(4) Resource Conservation and Recovery Act, Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, (SW-846), Third Edition, September 1986. Contains Final Update I (July 1992), Final Update IIA, (August 1993), Final Update II (September 1994), Final Update IIB (January 1995), and Final Update III (December 1996).
(5) Solid matrix type includes soil, sediment, sludge and other solid materials not classified as waste.
(6) Samples to be analyzed for cyanide should be field-tested for residual chlorine. If residual chlorine is detected, ascorbic acid should be added.
(7) Method not listed in 40 CFR Part 136.
(8) If not done in the field (ASAP) per the method and requested by client, analyze in lab within 48 hours.
(9) EPA issued memo recommending not to use reactive cyanide and sulfide methods.

TABLE 8.5-2
Organic Sample Containers, Preservatives, and Holding Times

Analytical Parameters	Matrix	Minimum Sample Size[1]	NPDES[2], [3]		RCRA (SW846)[3], [4]	
			Method	Requirements	Method[6]	Requirements
Aromatic Volatiles	Water	40 mL	602	40 mL glass, VOA vial (in triplicate) with Teflon®-lined septa without headspace, Cool, 4°C, Add sodium thiosulfate if residual chlorine, 7 days with pH > 2, 14 days with pH ≤ 2	8021B	40 mL glass, VOA vial (in triplicate) with Teflon®-lined septa without headspace, Cool, 4°C, Add sodium thiosulfate if residual chlorine, 1:1 HCl to pH ≤ 2, 14 days with pH ≤ 2
	Solid[5]	5 g or 25 g	--	Not Applicable	8021B	4 or 8 oz glass with Teflon®-lined lid, Cool 4 °C, 14 days. Field preserved with sodium bisulfate solution for low level analysis, or with methanol for medium level analysis. Soil sample can also be taken by using the EnCore™ sampler and preserved in the lab within 48 hours of sampling. Maximum holding time for Encore Sampler is 48 hours (before the sample is added to methanol or sodium bisulfate).Cool, 4°C[12]
	Waste	5 g or 25 g	--	Not Applicable	8021B	4 or 8 oz glass with Teflon®-lined lid, Cool 4 °C, 14 days. Field preserved with sodium bisulfate solution for low level analysis, or with methanol for medium level analysis.

TABLE 8.5-2
Organic Sample Containers, Preservatives, and Holding Times
(Continued)

Analytical Parameters	Matrix	Minimum Sample Size[1]	NPDES[2], [3]		RCRA (SW846)[3], [4]	
			Method	Requirements	Method[6]	Requirements
Aromatic Volatiles (continued)	Waste	5 g or 25 g	--	Not Applicable	8021B	Soil sample can also be taken by using the EnCore™ sampler and preserved in the lab within 48 hours of sampling. Maximum holding time for Encore Sampler is 48 hours (before the sample is added to methanol or sodium bisulfate). Cool, 4°C[12]
Halogenated Volatiles By GC	Water	40 mL	--	Not Applicable	8021B	40 mL glass, VOA vial (in triplicate) with Teflon®-lined septa without headspace, Cool, 4°C, Add sodium thiosulfate if residual chlorine, 1:1 HCl to pH ≤ 2, 14 days
	Solid[5]	5 g or 25 g	--		8021B	4 or 8 oz glass with Teflon®-lined lid, Cool 4 °C, 14 days. Field preserved with sodium bisulfate solution for low level analysis, or with methanol for medium level analysis. Soil sample can also be taken by using the EnCore™ sampler and preserved in the lab within 48 hours of sampling. Maximum holding time for Encore Sampler is 48 hours (before the sample is added to methanol or sodium bisulfate). Cool, 4°C[12]

TABLE 8.5-2
Organic Sample Containers, Preservatives, and Holding Times
(Continued)

Analytical Parameters	Matrix	Minimum Sample Size[1]	NPDES[2],[3]		RCRA (SW846)[3],[4]	
			Method	Requirements	Method[6]	Requirements
Halogenated Volatiles (continued)	Waste	5 g or 25 g	--	Not Applicable	8021B	4 or 8 oz glass with Teflon®-lined lid, Cool 4 °C, 14 days. Field preserved with sodium bisulfate solution for low level analysis, or with methanol for medium level analysis. Soil sample can also be taken by using the EnCore™ sampler and preserved in the lab within 48 hours of sampling. Maximum holding time for Encore Sampler is 48 hours (before the sample is added to methanol or sodium bisulfate). Cool, 4°C[12]
Herbicides	Water	1L	615 [10]	1 liter amber glass with Teflon®-lined lid, Sodium thiosulfate or ascorbic acid if residual chlorine present, Cool, 4°C, Extraction, 7 days Analysis, 40 days after extraction	8151A	1 liter amber glass with Teflon®-lined lid. If residual chlorine present, add 3 mL sodium thiosulfate per gallon. Cool, 4°C, Extraction, 7 days Analysis, 40 days of the start of the extraction
	Solid	50 g	--	Not Applicable	8151A	4 or 8 oz glass widemouth with Teflon®-lined lid, Cool 4 °C, Extraction, 14 days Analysis, 40 days of the start of the extraction

TABLE 8.5-2
Organic Sample Containers, Preservatives, and Holding Times (Continued)

Analytical Parameters	Matrix	Minimum Sample Size[1]	NPDES[2], [3]		RCRA (SW846)[3], [4]	
			Method	Requirements	Method[6]	Requirements
Herbicides (continued)	Waste	50 g	--	Not Applicable	8151A	4 or 8 oz glass widemouth with Teflon®-lined lid. Cool 4 °C Extraction, 14 days Analysis, 40 days of the start of the extraction
Organo-phosphorus Pesticides	Water	1L	---	Not Applicable	8141A	1 liter amber glass with Teflon®-lined lid. If residual chlorine present, add 3 mL sodium thiosulfate per gallon. Cool, 4°C, Extraction, 7 days Analysis, 40 days of the start of the extraction
	Solid	50 g	---	Not Applicable	8141A	4 or 8 oz glass widemouth with Teflon®-lined lid Cool, 4°C, Extraction, 14 days Analysis, 40 days of the start of the extraction
	Waste	50 g	---	Not Applicable	8141A	4 or 8 oz glass widemouth with Teflon®-lined lid, Cool, 4°C, Extraction, 14 days Analysis, 40 days of the start of the extraction
PAHs by GC and HPLC	Water	1L	610	1 liter amber glass with Teflon®-lined lid, Adjust pH to 5-9 if extraction not to be done within 72 hours of sampling. Add sodium thiosulfate if residual chlorine present. Cool, 4°C, Extraction, 7 days Analysis, 40 days after extraction	8310	1 liter amber glass with Teflon®-lined lid, If residual chlorine present, add 3 mL sodium thiosulfate per gallon, Cool, 4°C, Extraction, 7 days Analysis, 40 days of the start of the extraction

TABLE 8.5-2
Organic Sample Containers, Preservatives, and Holding Times
(Continued)

Analytical Parameters	Matrix	Minimum Sample Size[1]	NPDES[2], [3]		RCRA (SW846)[3], [4]	
			Method	Requirements	Method[6]	Requirements
PAHs by GC and HPLC (continued)	Solid	50 g	---	Not Applicable	8310	4 or 8 oz glass wide mouth with Teflon®-lined lid, Cool, 4°C, Extraction, 14 days Analysis, 40 days of the start of the extraction
	Waste	50 g	---	Not Applicable	8310	4 or 8 oz glass wide mouth with Teflon®-lined lid, Cool, 4°C Extraction, 14 days Analysis, 40 days of the start of the extraction
Pesticides/ PCBs	Water	1L	608	1 liter amber glass with Teflon®-lined lid, Adjust pH to 5-9 if extraction not to be done within 72 hours of sampling. Add sodium thiosulfate if residual chlorine present and aldrin is being determined. Cool, 4°C, Extraction, 7 days Analysis, 40 days after extraction	8081A 8082	1 liter amber glass with Teflon®-lined lid, If residual chlorine present, add 3 mL 10% sodium thiosulfate per gallon, Cool, 4°C, Extraction, 7 days Analysis, 40 days of the start of the extraction
	Solid	50 g	---	Not Applicable	8081A 8082	4 or 8 oz glass wide mouth with Teflon®-lined lid, Cool, 4°C, Extraction, 14 days Analysis, 40 days of the start of the extraction
	Waste	50 g	---	Not Applicable	8081A 8082	4 or 8 oz glass wide mouth with Teflon®-lined lid, Cool, 4°C Extraction, 14 days Analysis, 40 days of the start of the extraction

TABLE 8.5-2
Organic Sample Containers, Preservatives, and Holding Times (Continued)

Analytical Parameters	Matrix	Minimum Sample Size[1]	NPDES[2], [3]		RCRA (SW846)[3], [4]	
			Method	Requirements	Method[6]	Requirements
Petroleum Hydrocarbons/ Oil and Grease	Water	1L	413.1 418.1	1 liter glass, Cool, 4°C, HCl to pH <2, 28 days	9070	1 liter glass with Cool, 4°C, HCl to pH <2, 28 days
	Solid	---	---	Not Applicable	9071A	8 oz. glass with Teflon®-lined lid, Holding Time not specified
	Waste	---	---	Not Applicable	9071A	8 oz. glass with Teflon®-lined lid, Holding Time not specified
	Water	1 L	1664A[7]	1 liter glass, Cool, 4°C HCl or H_2SO_4 to pH <2 28 days	9071B	1 liter glass, Cool, 0-4°C HCl or H_2SO_4 to pH <2 28 days
	Solid	30 g	1664A[7]	8 or 16 oz. wide mouth glass jar, Cool, 4°C, 28 days	9071B	8 or 16 oz. wide mouth glass jar, Cool, 0-4°C, 28 days
	Waste	---	---	Not Applicable	9071B	Not Applicable
Purgeable Halocarbons By GC	Water	40 mL	601	40 mL glass VOA vial (in triplicate) with Teflon®-lined septa with no headspace, Cool, 4°C, Add sodium thiosulfate if residual chlorine present, 14 days	8021B	40 mL glass VOA vial (in triplicate) with Teflon®-lined septa with no headspace, Cool, 4°C, 1:1 HCl to pH ≤ 2, sodium thiosulfate if residual chlorine present, 14 days
	Solid	5 g or 25 g	---	Not Applicable	8021B	4 or 8 oz glass with Teflon®-lined lid, Cool 4 °C, 14 days. Field preserved with sodium bisulfate solution for low level analysis, or with methanol for medium level analysis.

TABLE 8.5-2
Organic Sample Containers, Preservatives, and Holding Times
(Continued)

Analytical Parameters	Matrix	Minimum Sample Size[1]	NPDES[2],[3]		RCRA (SW846)[3],[4]	
			Method	Requirements	Method[6]	Requirements
Purgeable Halocarbons By GC (continued)	Solid	5 g or 25 g	---	Not Applicable	8021B	Soil sample can also be taken by using the EnCore™ sampler and preserved in the lab within 48 hours of sampling. Maximum holding time for Encore Sampler is 48 hours (before the sample is added to methanol or sodium bisulfate). Cool, 4°C[12]
	Waste	5 g or 25 g	---	Not Applicable	8021B	4 or 8 oz glass with Teflon®-lined lid, Cool 4 °C, 14 days. Field preserved with sodium bisulfate solution for low level analysis, or with methanol for medium level analysis. Soil sample can also be taken by using the EnCore™ sampler and preserved in the lab within 48 hours of sampling. Maximum holding time for Encore Sampler is 48 hours (before the sample is added to methanol or sodium bisulfate).Cool, 4°C[12]

TABLE 8.5-2
Organic Sample Containers, Preservatives, and Holding Times
(Continued)

Analytical Parameters	Matrix	Minimum Sample Size[1]	NPDES[2], [3]		RCRA (SW846)[3], [4]	
			Method	Requirements	Method[6]	Requirements
Semivolatiles	Water	1L	625	1 liter amber glass with Teflon®-lined lid, Cool, 4°C, Extraction, 7 days Analysis, 40 days	8270C	1 liter amber glass with Teflon®-lined lid, If residual chlorine present, add 3 mL sodium thiosulfate per gallon, Cool, 4°C, Extraction, 7 days Analysis, within 40 days of extraction
	Solid	50 g	—	Not Applicable	8270C	8 or 16 oz glass wide mouth with Teflon-lined lid, Cool, 4°C, Extraction, 14 days Analysis, within 40 days of extraction
	Waste	50 g	—	Not Applicable	8270C	8 or 16 oz glass wide mouth with Teflon®-lined lid, Cool, 4°C, Extraction, 14 days Analysis, within 40 days of extraction
Volatile Organics	Water	40 mL	624	40 mL glass, VOA vial (in triplicate) with Teflon®-lined septa without headspace, Cool, 4°C, Add sodium thiosulfate if residual chlorine, 7 days with pH > 2, 14 days with pH ≤ 2[8]	8260B	40 mL glass, VOA vial (in triplicate) with Teflon®-lined septa without headspace, Cool, 4°C, Add sodium thiosulfate if residual chlorine, 1:1 HCl to pH ≤ 2, 14 days with pH ≤ 2[9]

TABLE 8.5-2
Organic Sample Containers, Preservatives, and Holding Times (Continued)

Analytical Parameters	Matrix	Minimum Sample Size[1]	NPDES[2], [3]		RCRA (SW846)[3], [4]	
			Method	Requirements	Method[6]	Requirements
Volatile Organics (continued)	Solid[5]	5 g or 25 g	--	Not Applicable	8260B	4 or 8 oz glass with Teflon®-lined lid, Cool 4 °C, 14 days. Field preserved with sodium bisulfate solution for low level analysis, or with methanol for medium level analysis. Soil sample can also be taken by using the EnCore™ sampler and preserved in the lab within 48 hours of sampling. Maximum holding time for Encore Sampler is 48 hours (before the sample is added to methanol or sodium bisulfate). Cool, 4°C[12]
	Waste	5 g or 25 g	--	Not Applicable	8260B	4 or 8 oz glass with Teflon®-lined lid, Cool 4 °C, 14 days. Field preserved with sodium bisulfate solution for low level analysis, or with methanol for medium level analysis. Soil sample can also be taken by using the EnCore™ sampler and preserved in the lab within 48 hours of sampling. Maximum holding time for Encore Sampler is 48 hours (before the sample is added to methanol or sodium bisulfate). Cool, 4°C[12]

TABLE 8.5-2
Organic Sample Containers, Preservatives, and Holding Times
(Continued)

Footnotes

(1) Minimum sample size indicates sample amount needed for a single analysis. Matrix spikes or duplicates will require an additional sample amount of at least this amount for each additional QC sample aliquot required.
(2) National Pollutant Discharge Elimination System - 40 CFR Part 136, Appendix A.
(3) Holding times are calculated from the date of collection.
(4) Resource Conservation and Recovery Act, Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, Third Edition, September 1986. Contains Final Update I (July 1992), Final Update IIA (August 1993), Final Update II (September 1994), Final Update IIB (January 1995), and Final Update III (December 1996).
(5) Solid matrix type includes soil, sediment, sludge or other solids not classified as waste.
(6) Only one determination method is listed when separate methods are required for preparation and analysis.
(7) **Method 1664 was promulgated by the EPA with an effective date of June 14, 1999.**
(8) **For acrolein and acrylonitrile the pH should be adjusted to 4-5. This pH adjustment is not required if acrolein is not measured. Samples requiring analysis of acrolein that received no pH adjustment must be analyzed within three days of sampling.**
(9) **For acrolein and acrylonitrile the pH should be adjusted to 4-5.**
(10) Method not listed in 40 CFR Part 136.
(11) Should only be used in the presence of residual chlorine.
(12) Depending on regulatory programs, EnCore™ samplers may be preserved for up to 14 days from sampling by freezing at -5 to -12°C until analysis. Alternatively the EnCore™ sample may be transferred to a 40-ml VOA vial and preserved by freezing at -5 to -12°C until analysis. Some regulatory agencies may require 4 or 8 oz glass with Teflon®-lined lid, Cool 4°C, 14 days. This technique is not recommended, but will be supported where required. (Preservation and holding times are subject to client specifications.)

TABLE 8.5-4
Sample Containers, Preservatives, and Holding Times for USEPA Contract Laboratory Program Statement of Work

Analytical Parameters	Matrix	Minimum Sample Size	Requirements[1]
Cyanide, Total and Amenable to Chlorination	Water	500 mL	500 mL, glass or polyethylene container, 0.6 g ascorbic acid (only in presence of residual chlorine) NaOH to pH > 12, Cool, 4°C, 12 days
	Soil/Sediment	25 g	8 or 16 oz glass with Teflon-lined lids, Cool, 4°C, 12 days
ICAP and GFAA (excludes mercury)	Water	100 mL	1 liter glass or polyethylene container, HNO_3 to pH =2, 180 days
	Soil/Sediment	25 g	4 or 8 oz glass or polyethylene container, Cool, 4°C, 180 days
Mercury (CVAA)	Water	100 mL	1 liter glass or polyethylene container, HNO_3 to pH =2, 26 days
	Soil/Sediment	25 g	8 or 16 oz glass with Teflon®-lined lids, Cool, 4°C, 26 days
Pesticides/PCBs	Water	1 L	1 liter amber glass with Teflon®-lined lid, Cool, 4°C, Extraction within 5 days of sample receipt Analysis within 40 days after start of extraction
	Soil/Sediment	50 g	8 or 16 oz glass wide mouth with Teflon®-lined lid, protect from light, Cool, 4°C, Extraction within 10 days of sample receipt Analysis within 40 days after start of extraction

TABLE 8.5-4
Sample Containers, Preservatives, and Holding Times for USEPA Contract Laboratory Program Statement of Work (Continued)

Analytical Parameters	Matrix	Minimum Sample Size	Requirements[1]
Semivolatiles	Water	1L	1 liter amber glass with Teflon®-lined lid, Cool, 4°C, Extraction within 5 days of sample receipt Analysis within 40 days after start of extraction
	Soil/Sediment	50 g	8 or 16 oz glass wide mouth with Teflon®-lined lid, Cool, 4°C, Extraction within 10 days of sample receipt Analysis within 40 days after start of extraction
Volatiles	Water	40 mL	40 mL glass with Teflon®-lined lid, no entrapped air bubbles pH <2[3], Cool, 4°C, 10 days
	Soil/Sediment	25 g	4 or 8 oz glass with Teflon®-lined lids, Cool, 4°C, 10 days

Footnotes

(1) Holding times are calculated from verified time of sample receipt.

(2) Footnote deleted

(3) The OLM03.0 requirement is to acidify the sample to pH<2. The OLM01.8 requirement is to determine and report the pH of the sample to check that the sample was acidified in the field.

TABLE 8.5-5
Sample Containers, Preservatives, and Holding Times for TCLP[1] and SPLP[2]

Analytical Parameters	Matrix	Minimum Sample Size	TCLP Method 1311 and SPLP Method 1312 Requirements	
			From Field Collection to TCLP/SPLP Extraction	From TCLP/SPLP Extraction to Analysis
Mercury	Liquid Solid Waste	1L	1L glass, Cool, 4°C, 28 days	Glass or polyethylene 28 days
Metals (except mercury)	Liquid Solid Waste	1L	1L glass, Cool, 4°C, 180 days	Glass or polyethylene 180 days
Semivolatiles	Liquid Solid Waste	1L	1L glass, Cool 4°C, 14 days	1L glass Extraction of leachate within 7 days of TCLP extraction, Analyze extract within 40 days
Volatiles	Liquid Solid Waste	6 oz	4 oz glass, Cool 4°C, 14 days	40 mL glass, 14 days

Footnotes

[1] TCLP = Toxicity Characteristic Leaching Procedure
[2] SPLP = Synthetic Precipitation Leaching Procedure

TABLE 8.5-6
Periodic Equipment Calibrations

Type of Equipment	Calibration Requirements
Balances	• Must be serviced and calibrated annually by an approved vendor. • Calibration must be checked daily or before use by analyst with weight(s) classified as Class 1 (formerly termed Class S) by NIST or Class 1 traceable. Acceptance criteria vary according to weight used and accuracy of balance. Acceptance criteria must be documented in the log. • All Class 1 weights must be certified by an outside vendor every three years. • All non-Class 1 weights must be checked annually against NIST Class 1 weights annually.
Thermometers	• Working glass thermometers must be calibrated against a certified NIST thermometer at least annually as described in operation-specific SOPs. • Working non-glass thermometers must be calibrated against a certified NIST thermometer annually as described in operation-specific SOPs. • The NIST thermometer must be recertified every three years.
Refrigerators/Freezers	• Thermometers must be immersed in a liquid such as mineral oil or glycol • Temperature of units used for sample or standard storage must be checked daily as described in operation-specific SOPs. Refrigerator acceptance limits: 4°C ± 2°C Freezer acceptance limits: < - 10°C
Ovens	• Temperature of units must be checked daily or before use. • Acceptance limits vary according to use as described in operation-specific SOPs and must be documented in the temperature log.
Micropipettors	• Calibrations are checked gravimetrically as required by the operation-specific SOP. • Must be calibrated at the frequency (normally quarterly) required by the manufacturer at a minimum.
Syringes, Volumetric Glassware and Graduated Glassware	• All syringes and volumetric glassware are purchased as Class A items. • Class A items are certified by the manufacturer to be within ± 1% of the measured volume, therefore, calibration of these items by STL laboratories is not required. • All analysts are trained in the proper use and maintenance of measuring devices to ensure the measurement of standards, reagents and sample volumes are within method tolerances.

TABLE 8.5-7
Summary of Inorganic Method Calibrations

Analysis	Calibration	NPDES[1]		RCRA (SW846)[2]	
		Method	Requirement	Method	Requirement
Alkalinity	Initial	310.1 2320B	2 point calibration of pH meter (± 0.05 pH units of true value)	--	Not Applicable
	Continuing	310.1 2320B	Not Applicable	--	Not Applicable
	Ending	310.1 2320B	Not Applicable	--	Not Applicable
Ammonia	Initial	350.1	6 levels including blank, "r"[3] ≥ 0.995	--	Not Applicable
	Continuing	350.1	1 level or LCS every 10 samples ± 10% of true value	--	Not Applicable
	Ending	350.1	1 level or LCS every 10 samples ± 10% of true value	--	Not Applicable
Biochemical Oxygen Demand (BOD)	Initial	405.1	a. Winkler titration: Iodometric with standard thiosulfate b. Membrane electrode: Read in air and in water with zero dissolved oxygen	--	Not Applicable
	Continuing	405.1	Not Applicable	--	Not Applicable
	Ending	405.1	Not Applicable	--	Not Applicable

TABLE 8.5-7
Summary of Inorganic Method Calibrations
(Continued)

Analysis	Calibration	NPDES[1]		RCRA (SW846)[2]	
		Method	Requirement	Method	Requirement
Bromide	Initial	300.0[4]	5 levels plus a blank, "r"[3] ≥ 0.995	9056	5 levels plus a blank, "r"[3] ≥ 0.995
	Continuing	300.0[4]	level every 10 samples ± 10% of true value	9056	Not Applicable
	Ending	300.0[4]	Not Applicable	9056	Not Applicable
Chemical Oxygen Demand (COD)	Initial	410.4	5 levels plus a blank"r"[3] ≥ 0.995	--	Not Applicable
	Continuing	410.4	1 level every 10 samples ± 10% of true value	--	Not Applicable
	Ending	410.4	1 level ± 10% of true value	--	Not Applicable

TABLE 8.5-7
Summary of Inorganic Method Calibrations
(Continued)

Analysis	Calibration	NPDES[1]		RCRA (SW846)[2]	
		Method	Requirement	Method	Requirement
Chloride	Initial	300.0[4] 325.2	5 levels plus blank "r"[3] ≥ 0.995	9056 9252	Method 9056: 3 levels plus a blank Method 9252: 5 levels plus blank "r"[3] ≥ 0.995
	Continuing	300.0[4] 325.2	1 level every 10 samples ± 10% of true value	9056 9252	Method 9056: 1 per batch of 20 samples, ± 10% of true value Method 9252: 1 level every 10 samples ± 10% of true value
	Ending	300.0[4] 325.2	1 level every 10 samples ± 10% of true value	9056 9252	Method 9056 : Not Applicable Method 9252: 1 level ± 10% of true value
Chromium Cr^{+6}	Initial	3500 Cr-D	3 levels plus blank	7196A	5 levels plus blank "r"[3] ≥ 0.995
	Continuing	3500 Cr-D	1 level every 10 samples ± 10% of true value	7196A	1 level every 10 samples ± 15%
	Ending	3500 Cr-D	1 level ± 10% of true value	7196A	1 level ± 15%

TABLE 8.5-7
Summary of Inorganic Method Calibrations
(Continued)

Analysis	Calibration	NPDES[1]		RCRA (SW846)[2]	
		Method	Requirement	Method	Requirement
Chlorine, Residual	Initial	330.5	Standardize titrant	--	Not Applicable
	Continuing	330.5	Not Applicable	--	Not Applicable
	Ending	330.5	Not Applicable	--	Not Applicable
Conductivity	Initial	120.1	Standard KCl solution	9050A	1 level to determine cell constant
	Continuing	120.1	Not Applicable	9050A	Not Applicable
	Ending	120.1	Not Applicable	9050A	Not Applicable
Cyanide (Amenable)	Initial	335.1	7 levels plus blank "r"[3] ≥ 0.995	9012A	7 levels plus blank "r"[3] ≥ 0.995
	Continuing	335.1	1 level every 10 samples ± 10% of true	9012A	1 mid-level every 10 samples ± 15% of true value
	Ending	335.1	1 level ± 10 % of true value	9012A	± 15% of true value
Cyanide (Total)	Initial	335.1 335.2 335.3 335.4	7 levels plus blank "r"[3] ≥ 0.995	9012A	7 levels plus blank "r"[3] ≥ 0.995
	Continuing	335.1 335.2 335.3 335.4	1 mid-level every 10 samples ± 10 % of true value	9012A	1 mid-level every 10 samples ± 15% of true value
	Ending	335.1 335.2 335.3 335.4	1 mid-level ± 10 % of true value	9012A	± 15% of true value

TABLE 8.5-7
Summary of Inorganic Method Calibrations
(Continued)

Analysis	Calibration	NPDES[1]		RCRA (SW846)[2]	
		Method	Requirement	Method	Requirement
Flashpoint	Initial	--	Not Applicable	1010	p-Xylene reference standard must have flashpoint of 27.2°C ± 1.1°C
	Continuing	--	Not Applicable	1010	Not Applicable
	Ending	--	Not Applicable	1010	Not Applicable
Fluoride	Initial	300.0[4] 340.2	Method 300.0: 5 levels plus a blank, "r"[3] ≥ 0.995 Method 340.2: 6 levels "r"[3] ≥ 0.995	9056	3 levels plus a blank
	Continuing	300.0[4] 340.2	1 mid-level every 10 samples ± 10% of true value	9056	1 per batch of 20 samples ± 10% of true value
	Ending	300.0[4] 340.2	1 mid-level ± 10% of true value	9056	Not Applicable
Hardness	Initial	130.2 2340B	Method 130.2: Standardize titrant Method 2340B: See ICP Metals 200.7	--	Not Applicable
	Continuing	130.2 2340B	Method 130.2: Not Applicable Method 2340B: See ICP Metals 200.7	--	Not Applicable
	Ending	130.2 2340B	Method 130.2: Not Applicable Method 2340B: See ICP Metals 200.7	--	Not Applicable
Iron (Ferrous)	Initial	3500-Fe D	3 levels plus a blank, "r"[3] ≥ 0.995	-	Not Applicable
	Continuing	3500-Fe D	1 mid-level every 10 samples ± 10% of true value	-	Not Applicable
	Ending	3500-Fe D	1 mid-level ± 10% of true value	-	Not Applicable

TABLE 8.5-7
Summary of Inorganic Method Calibrations
(Continued)

Analysis	Calibration	NPDES[1]		RCRA (SW846)[2]	
		Method	Requirement	Method	Requirement
Nitrate	Initial	300.0[4] 353.2	5 levels plus a blank "r"[3] ≥ 0.995	9056	3 levels plus a blank
	Continuing	300.0[4] 353.2	1 mid-level every 10 samples ± 10% of true value	9056	1 per batch of 20 samples, ± 10% of true value
	Ending	300.0[4] 353.2	1 mid-level ± 10% of true value	9056	Not Applicable
Nitrite	Initial	300.0[4] 353.2	5 levels plus a blank "r"[3] ≥ 0.995	9056	3 levels plus a blank
	Continuing	300.0[4] 353.2	1 mid-level every 10 samples ± 10% of true value	9056	1 per batch of 20 samples, ± 10% of true value
	Ending	300.0[4] 353.2	1 mid-level ± 10% of true value	9056	Not Applicable
Nitrate-Nitrite	Initial	300.0[4] 353.2	5 levels plus blank "r"[3] ≥ 0.995	--	Not Applicable
	Continuing	300.0[4] 353.2	1 level every 10 samples ± 10% of true value	--	Not Applicable
	Ending	300.0[4] 353.2	1 mid-level ± 10% of true value	--	Not Applicable

TABLE 8.5-7
Summary of Inorganic Method Calibrations
(Continued)

Analysis	Calibration	NPDES[1]		RCRA (SW846)[2]	
		Method	Requirement	Method	Requirement
Phosphorus (total and Ortho-phosphate)	Initial	300.0[4] 365.2 365.3	Method 300.0/365.3: 3 levels plus a blank Method 365.2: 8 levels plus a blank	--	Not Applicable
	Continuing	300.0[4] 365.2 365.3	Method 300.0/365.3: 1 level every 10 samples ± 10% of true value Method 365.2: Blank and 2 standards with each series of samples, ± 2% of true value or recalibrate	--	Not Applicable
	Ending	300.0[4] 365.2 365.3	Method 300.0/365.3: ± 10% of true value Method 365.2: Not Applicable	--	Not Applicable
pH	Initial	150.1	2 level calibration that bracket the expected pH of the sample (± 0.05 pH units of true value)	9040B 9045C	2 point calibration (± 0.05 pH units of true value)
	Continuing	150.1	1 buffer check every 10 samples ± 5% of true value	9040B 9045C	Not Applicable
	Other	150.1	Third point check	9040B 9045C	Third point check

TABLE 8.5-7
Summary of Inorganic Method Calibrations
(Continued)

Analysis	Calibration	NPDES[1]		RCRA (SW846)[2]	
		Method	Requirement	Method	Requirement
pH (continued)	Ending	150.1	1 buffer check ± 5% of true value	9040B 9045C	Not Applicable
Phenolics	Initial	420.1	5 levels plus a blank "r"[3] ≥ 0.995	9065 9066	5 levels plus a blank "r"[3] 0.995
	Continuing	420.1	1 mid-level every 10 samples ± 10% true value	9065 9066	1 mid-level ± 15% true value
	Ending	420.1	1 mid-level ± 10% true value	9065 9066	1 mid-level ± 15% true value
Phosphate	Initial	---	Not Applicable	9056	3 levels plus a blank
	Continuing	---	Not Applicable	9056	1 per batch of 20 samples, ± 15% of true value
	Ending	---	Not Applicable	9056	Not Applicable
Reactivity [5]	Initial	--	Not Applicable	Chap 7	See Total Cyanide and Sulfide
	Continuing	--	Not Applicable		
	Ending	--	Not Applicable		
Settleable Solids	Initial	160.5	Not Applicable	--	Not Applicable
	Continuing	160.5	Not Applicable	--	Not Applicable
	Ending	160.5	Not Applicable	--	Not Applicable

TABLE 8.5-7
Summary of Inorganic Method Calibrations
(Continued)

Analysis	Calibration	NPDES[1]		RCRA (SW846)[2]	
		Method	Requirement	Method	Requirement
Specific Conductance	Initial	120.1	Standardize meter with 0.01 M KCl	9050A	Not Applicable
	Continuing	120.1	1 level every 10 samples ± 10% of true value	9050A	Not Applicable
	Ending	120.1	1 level ± 10% of true value	9050A	Not Applicable
Sulfate	Initial	300.0[4] 375.4	Method 300.0: 5 levels plus blank "r"[3] ≥ 0.995 Method 375.4: 3 levels plus blank "r"[3] ≥ 0.995	9038 9056	Method 9038: 3 levels plus a blank for every hour of continuous sample analysis. Method 9056: 3 levels plus a blank
	Continuing	300.0[4] 375.4	Method 300.0: 1 mid-level after every 10 samples ± 10% of true value Method 375.4: 1 level every 3 or 4 samples ± 10% of true value	9038 9056	Method 9038: Independent-prepared check standard every 15 samples Method 9056: 1 per batch of 20 samples, ± 10% of true value
	Ending	300.0[4] 375.4	± 10% of true value	9038 9056	Not Applicable
Sulfide	Initial	376.1 376.2	Method 376.1: This is a titration method. Therefore, calibrations are not applicable. Method 376.2: 5 levels plus a blank "r" [3] ≥ 0.995	9030B 9034	This is a colorimetric titration. Therefore, calibration is not applicable.

TABLE 8.5-7
Summary of Inorganic Method Calibrations
(Continued)

Analysis	Calibration	NPDES[1]		RCRA (SW846)[2]	
		Method	Requirement	Method	Requirement
Sulfide (continued)	Continuing	376.1 376.2	Method 376.1: Not Applicable Method 376.2: 1 level every 10 samples ± 10% of true value	9030B 9034	This is a colorimetric titration. Therefore, calibration is not applicable.
	Ending	376.1 376.2	Method 376.1: Not Applicable Method 376.2: ± 10% of true value	9030B 9034	This is a colorimetric titration. Therefore, calibration is not applicable.
Total Dissolved Solids	Initial	160.1	This is a gravimetric determination. Calibrate balance prior to analysis	--	Not Applicable
	Continuing	160.1		--	Not Applicable
	Ending	160.1		--	Not Applicable

TABLE 8.5-7
Summary of Inorganic Method Calibrations
(Continued)

Analysis	Calibration	NPDES[1]		RCRA (SW846)[2]	
		Method	Requirement	Method	Requirement
Total Kjeldahl Nitrogen (TKN)	Initial	351.2 351.3	Method 351.2: 5 levels plus blank "r"[3] ≥ 0.995 Method 351.3: Titrimetric: Standardize titrant Colorimetric: 7 levels plus blank	--	Not Applicable
	Continuing	351.2 351.3	Method 351.2: 1 mid-level every 10 samples ± 10% of true value Method 351.3: Not Applicable	--	Not Applicable
	Ending	351.2 351.3	Method 351.2: ± 10% of true value Method 351.3: Not Applicable	--	Not Applicable
Total Organic Carbon (TOC)	Initial	415.1	3 levels plus blank	9060	3 levels plus blank "r"[3] ≥ 0.995
	Continuing	415.1	1 mid-level every 10 samples ± 15% of true value	9060	1 mid-level every 10 samples ± 15% of true value
	Ending	415.1	± 15% of true value	9060	± 15% of true value

TABLE 8.5-7
Summary of Inorganic Method Calibrations
(Continued)

Analysis	Calibration	NPDES[1]		RCRA (SW846)[2]	
		Method	Requirement	Method	Requirement
Total Organic Halides (TOX)	Initial	SM 5320B [4] 450.1 [4]	Method 5320B: 7 levels plus a blank ± 10% of true value Method 450.1: Daily instrument calibration standard and blank in duplicate ± 10% of true value (calibration std.) Verify with independently-prepared check standard	9020B	Daily instrument calibration standard and blank in duplicate ± 10% of true value (calibration std.) Verify with independently-prepared check standard – ICV ± 10% SOP NO. CORP-WC-0001
	Continuing	SM 5320B [4] 450.1 [4]	± 10% of true value	9020B	CCV ± 10% of true value SOP NO. CORP-WC-0001
	Ending	SM 5320B [4] 450.1 [4]	± 10% of true value	9020B	CCV ± 10% of true value SOP NO. CORP-WC-0001
Total Solids	Initial	160.3	This is a gravimetric determination. Calibrate balance before use.	--	Not Applicable
	Continuing	160.3		--	Not Applicable
	Ending	160.3		--	Not Applicable
Total Suspended Solids (Nonfilterable)	Initial	160.2	This is a gravimetric determination. Calibrate balance before use.	--	Not Applicable
	Continuing	160.2		--	Not Applicable
	Ending	160.2		--	Not Applicable

TABLE 8.5-7
Summary of Inorganic Method Calibrations
(Continued)

Analysis	Calibration	NPDES[1]		RCRA (SW846)[2]	
		Method	Requirement	Method	Requirement
Turbidity	Initial	180.1	Minimum of 1 level in each instrument range Follow manufacturer's instructions	--	Not Applicable
	Continuing	180.1	Not Applicable	--	Not Applicable
	Ending	180.1	Not Applicable	--	Not Applicable
Volatile Solids	Initial	160.4	This is a gravimetric determination. Calibrate balance before use.	--	Not Applicable
	Continuing	160.4		--	Not Applicable
	Ending	160.4		--	Not Applicable
Water Content	Initial	--	Calibrate Balance	--	Calibrate Balance
	Continuing	--	Not Applicable	--	Not Applicable
	Ending	--	Not Applicable	--	Not Applicable
GFAA Metals (excludes Hg)	Initial	200 series	3 levels plus blank ICV ± 10% of true value $r^{n(3)} \geq 0.995$	7000A series	3 levels plus blank ICV ± 10% of true value $r^{n(3)} \geq 0.995$
	Continuing	200 series	Every 10 samples ± 10% of true value	7000A series	Every 10 samples ± 20% of true value
	Ending	200 series	± 10% of true value	7000A series	± 20% of true value
	Other	200 series	Annually - Instrument detection limits	7000A	Annually - Instrument detection limits

TABLE 8.5-7
Summary of Inorganic Method Calibrations
(Continued)

Analysis	Calibration	NPDES[1]		RCRA (SW846)[2]	
		Method	Requirement	Method	Requirement
ICP & ICP/MS Metals (excludes Hg)	Initial	200.7 200.8	1 level and blank Rerun high calibration standard: verify quantitation at ± 5% of true value,ICV RSD < 3% from replicate	6010B 6020	1 level and blank Rerun high calibration standard: verify quantitation at ± 5% of true value, ICV RSD < 5% from replicate
	Continuing	200.7 200.8	Every 10 samples ± 5% of true value CCV RSD < 5% from replicate	6010B 6020	Mid-level calibration standard Every 10 samples ± 10% of true value CCV RSD < 5% from replicate
	Ending	200.7 200.8	± 5% of true value CCV RSD < 5% from replicate	6010B 6020	Mid-level calibration standard ± 10% of true value CCV RSD < 5% from replicate
	Other	200.7 200.8	ICSA, ICSAB: Analyze at beginning of run. For ICSA, AB criteria see SOP Annually: ICP interelement correction factors Instrument detection limits	6010B 6020	ICSA, ICSAB: Analyze at beginning of run. For ICSA, AB criteria see SOP Annually: ICP interelement correction factors Instrument detection limits

TABLE 8.5-7
Summary of Inorganic Method Calibrations
(Continued)

Analysis	Calibration	NPDES[1]		RCRA (SW846)[2]	
		Method	Requirement	Method	Requirement
Mercury by CVAA & CVAFS	Initial	245.1 245.7 1631B[4]	5 levels plus blank ICV ± 10% of true value "r"[3] ≥ 0.995	7470A 7471A	5 levels plus blank ICV ± 10% of true value "r"[3] ≥ 0.995
	Continuing	245.1 245.7 1631B[4]	Daily or every 10 samples, whichever is more frequent ± 20% of true value	7470A 7471A	Every 10 samples ± 20% of true value
	Ending	245.1 245.7 1631B[4]	± 20% of true value	7470A 7471A	± 20% of original prepared standard
	Other	245.1 245.7 1631B[4]	Annually: - Instrument detection limits	7470A 7471A	Annually - Instrument detection limits

Footnotes

(1) National Pollutant Discharge Elimination System

(2) Resource Conservation and Recovery Act, Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, (SW-846), Third Edition, September 1986. Contains Final Update I (July 1992), Final Update IIA (August 1993), Final Update II (September 1994), Final Update IIB (January 1995), and Final Update III (December, 1996).

(3) "r" = correlation coefficient

(4) Method not listed in 40 CFR Part 136.

(5) EPA memo recommended that reactive cyanide and sulfide are not used.

TABLE 8.5-8
Summary of Organic Method Calibrations

Analytical Parameter	Calibration	NPDES[1]		RCRA (SW846)[2]	
		Method	Requirement	Method	Requirement
Aromatic Volatiles by GC	Initial	602	Minimum of 3 levels If % RSD < 10%, use avg RF. Otherwise, calibration curve employed	8021B	Minimum of 5 levels If % RSD < 20%, use avg RF. Otherwise, calibration curve employed.
	Continuing	602	Analyze QC check sample and evaluate per method requirements	8021B	Mid-level calibration standard analyzed every 10 samples. % D ≤ 15%, gases 20% D. Evaluate per SOP
	Ending	602	Not Applicable	8021B	Mid-level calibration standard % D ≤ 15%. Evaluate per SOP
	Other	602	Not Applicable	8021B	Not Applicable

TABLE 8.5-8
Summary of Organic Method Calibrations
(Continued)

Analytical Parameter	Calibration	NPDES[1]		RCRA (SW846)[2]	
		Method	Requirement	Method	Requirement
Herbicides by GC	Initial	615[9]	Minimum of 3 levels If % RSD < 10%, use avg RF. Otherwise, calibration curve employed	8151A	Minimum of 5 levels If % RSD < 20%, use avg RF. Otherwise, calibration curve employed.
	Continuing	615[9]	1 or more calibration standards analyzed daily % D ± 15% of predicted response	8151A	Mid-level calibration standard analyzed every 10 samples. % D < 15% of predicted response for any analyte quantitated and reported.
	Ending	615[9]	Not Applicable	8151A	Mid-level calibration standard. % D < 15% of predicted response for any analyte quantitated and reported.
	Other	615[9]	Not Applicable	8151A	Not Applicable

TABLE 8.5-8
Summary of Organic Method Calibrations
(Continued)

Analytical Parameter	Calibration	NPDES[1]		RCRA (SW846)[2]	
		Method	Requirement	Method	Requirement
Polyaromatic Hydrocarbons by GC or HPLC	Initial	610	Minimum of 3 levels If % RSD < 10%, use avg RF. Otherwise, calibration curve employed	8310	Minimum of 5 levels If % RSD < 20%, use avg RF. Otherwise, calibration curve employed.
	Continuing	610	1 or more calibration standards analyzed daily % D ± 15% of predicted response	8310	Mid-level calibration standard analyzed every 10 samples. % D < 15% of predicted response for any analyte quantitated and reported.
	Ending	610	Not Applicable	8310	Mid-level calibration standard. % D ± 15% of predicted response for any analyte quantitated and reported.
	Other	610	Not Applicable	8310	Not Applicable
Pesticides/ PCBs by GC	Initial	608	Minimum of 3 levels If % RSD < 10%, use avg RF. Otherwise, calibration curve employed	8081A 8082	Minimum of 5 levels. If % RSD < 20%, use avg RF. Otherwise, calibration curve employed. (See SOP No. CORP-GC-0001)
	Continuing	608	1 or more calibration standards analyzed daily % D ± 15% of predicted response	8081A 8082	Mid-level calibration standard analyzed every 10 samples. % D < 15% of predicted response for any analyte quantitated and reported.

TABLE 8.5-8
Summary of Organic Method Calibrations
(Continued)

Analytical Parameter	Calibration	NPDES[1]		RCRA (SW846)[2]	
		Method	Requirement	Method	Requirement
Pesticides/ PCBs by GC (continued)	Ending	608	Not Applicable	8081A 8082	Mid-level calibration standard. % D < 15% of predicted response for any analyte quantitated and reported.
	Other	608	Not Applicable	8081A 8082	Not Applicable
Petroleum Hydrocarbons/ Oil and Grease	Initial	413.1 418.1	Method 413.1: This is a gravimetric determination. Calibrate balance before use. 418.1: 3 levels plus a blank "r" ≥ 0.995	9070 9071A 9071B	This is a gravimetric determination. Calibrate balance before use
	Continuing	413.1 418.1	Not Applicable	9070 9071A 9071B	Not Applicable
	Ending	413.1 418.1	Not Applicable	9070 9071B 9071A	Not Applicable
	Initial	1664A	Calibrate analytical balance at 2 mg and 1000 mg Calibration must be ± 10% at 2 mg and ± 0.5% at 1000 mg or recalibrate balance	9071B	Calibrate analytical balance at 2 mg and 1000 mg Calibration must be ± 10% at 2 mg and ± 0.5% at 1000 mg or recalibrate balance
	Continuing	1664A	Not Applicable	9071B	Not Applicable
	Ending	1664A	Not Applicable	9071B	Not Applicable
Organophos-phorous Pesticides by GC	Initial	--	Not Applicable	8141A	Minimum of 5 levels If % RSD < 20%, use avg RF. Otherwise, calibration curve employed.

TABLE 8.5-8
Summary of Organic Method Calibrations
(Continued)

Analytical Parameter	Calibration	NPDES[1]		RCRA (SW846)[2]	
		Method	Requirement	Method	Requirement
Organophos-phorous Pesticides by GC (continued)	Continuing	--	Not Applicable	8141A	Mid-level calibration standard analyzed every 10 samples. % D < 15% of predicted response for any analyte quantitated and reported.
	Ending	--	Not Applicable	8141A	Mid-level calibration standard % D < 15% of predicted response for any analyte quantitated and reported.
	Other	--	Not Applicable	8141A	Not Applicable
Purgeable Halocarbons by GC	Initial	601	Minimum of 3 levels If % RSD < 10%, use avg RF. Otherwise, calibration curve employed	8021B	Minimum of 5 levels If % RSD < 20%, use avg RF. Otherwise, calibration curve employed.
	Continuing	601	Analyze QC check sample and evaluate per method requirements	8021B	Mid-level calibration standard analyzed every 10 samples. % D < 15%, gases 20% D Evaluate per SOP
	Ending	601	Not Applicable	8021B	Mid-level calibration standard % D <15% Evaluate per SOP
	Other	601	Not Applicable	8021B	Not Applicable

TABLE 8.5-8
Summary of Organic Method Calibrations
(Continued)

Analytical Parameter	Calibration	NPDES[1]		RCRA (SW846)[2]	
		Method	Requirement	Method	Requirement
Halogenated Volatiles by GC	Initial	--	Not Applicable	8021B	Minimum of 5 levels If % RSD < 20%, use avg RF. Otherwise, calibration curve employed.
	Continuing	--	Not Applicable	8021B	Mid-level calibration standard analyzed every 10 samples. % D < 15% of predicted response for any analyte quantitated and reported.
	Ending	--	Not Applicable	8021B	Mid-level calibration standard % D < 15% of predicted response for any analyte quantitated and reported.
	Other	--	Not Applicable	8021B	Not Applicable
Semivolatiles	Initial	625	Minimum of 3 levels, lowest near but above MDL If % RSD ≤ 35%, use avg RF Otherwise calibration curve employed.	8270C	Minimum of 5 levels, % RSD for RF for CCCs[4] < 30% SPCCs[5]: RF > 0.050
	Continuing	625	1 level every 24 hours Acceptance criteria are found in the method and SOP	8270C	Mid-level standard every 12 hours (after tuning) %D for CCCs[4] < 20 % between RF from standard and avg RF from initial SPCCs[5]: RF > 0.050

TABLE 8.5-8
Summary of Organic Method Calibrations
(Continued)

Analytical Parameter	Calibration	NPDES[1]		RCRA (SW846)[2]	
		Method	Requirement	Method	Requirement
Semivolatiles (continued)	Ending	625	Not Applicable	8270C	Not Applicable
	Other	625	DFTPP[7] tuning every 24 hours before standard or sample runs.	8270C	DFTPP[7] tuning at the beginning of every 12 hour shift.
Volatiles	Initial	624	Minimum of 3 levels, lowest near but above MDL If % RSD ≤ 35%, use avg RF Otherwise calibration curve employed.	8260B	Minimum of 5 levels, %RSD for RF for CCCs[4] < 30.0% SPCCs[5]: RF ≥ 0.300 for Chlorobenzene and 1,1,2,2-tetrachloroethane, Chloromethane and 1,1-dichloroethane, and RF > 0.100 for Bromoform

TABLE 8.5-8
Summary of Organic Method Calibrations
(Continued)

Analytical Parameter	Calibration	NPDES[1]		RCRA (SW846)[2]	
		Method	Requirement	Method	Requirement
Volatiles (continued)	Continuing	624	1 level every 24 hours Acceptance criteria are found in the method and SOP	8260B	Mid-level standard every 12 hours (after tuning) %Drift for CCCs[4] < 20.0% between RF from standard and avg RF from initial SPCCs[5]: RF ≥ 0.300 for Chlorobenzene and 1,1,2,2-tetrachloroethane, Chloromethane and 1,1-dichloroethane, and RF > 0.100 for Bromoform
	Ending	624	Not Applicable	8260B	Not Applicable
	Other	624	BFB[6] tuning at the beginning of every 24 hour shift.	8260B	BFB[6] tuning at the beginning of every 12 hour shift.

Footnotes

(1) National Pollutant Discharge Elimination System
(2) Resource Conservation and Recovery Act, Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, (SW-846), Third Edition, September 1986. Contains Final Update I (July 1992), Final Update IIA (August 1993), Final Update II (September 1994), Final Update IIB (January 1995), and Final Update III (December 1996).
(3) TCDD - 2,3,7,8-Tetrachlorodibenzo-p-dioxin
(4) CCC - Continuing Calibration Compounds
(5) SPCC - System Performance Check Compound
(6) BFB - Bromofluorobenzene
(7) DFTPP - Decafluorotriphenylphosphine
(8) Footnote deleted
(9) Method not listed in 40 CFR Part 136.

TABLE 8.5-9
Summary of USEPA Contract Laboratory Program Statement of Work
Method Calibrations

Analytical Parameter	Calibration	Method	Requirement
Cyanide, Total	Initial	ILM03.0 ILMO4.0	Minimum 5 levels plus blank "r" ≥ 0.995
	Continuing	ILM03.0 ILMO4.0	1 mid-level every 10 samples ± 15 % of true value
	Ending	ILM03.0 ILMO4.0	± 15 % of true value
	Other	ILM03.0 ILMO4.0	Not Applicable
ICAP (excludes mercury)	Initial	ILM03.0 ILMO4.0	1 level and blank ICV: ± 10% of true
	Continuing	ILM03.0 ILMO4.0	Mid-level calibration standard Every 10 samples ± 10% of true value
	Ending	ILM03.0 ILMO4.0	Mid-level calibration standard ± 10% of true value
	Other	ILM03.0 ILMO4.0	ILMO3.0: ICSA, ICSAB: Analyze at beginning and end or every 8 hours whichever is more frequent ILMO3.0: CRI: Beginning and end of each run, and every 8 hours for all analytes at 2x CRDL or 2x IDL whichever is greater, except for Al, Ba, Ca, Fe, Mg, Na, K ILMO4.0: ICSA, ICSAB: Analyze at beginning and end of run, but not before ICV. Must be analyzed every 20 analytical samples per ICP run. ILMO4.0: CRI: Beginning and end of each run and every 20 analytical samples per ICP run. CRI must be immediately followed by ICS analysis. <u>Quarterly:</u> Instrument detection limits Linear Range Verification <u>Annually:</u> ICP interelement correction factors

TABLE 8.5-9
Summary of USEPA Contract Laboratory Program Statement of Work
Method Calibrations
(Continued)

Analytical Parameter	Calibration	Method	Requirement
GFAA (excludes Hg)	Initial	ILM03.0 ILMO4.0	Minimum 3 levels plus blank ICV: ± 10%
	Continuing	ILM03.0 ILMO4.0	Every 10 samples ± 10% of true value
	Ending	ILM03.0 ILMO4.0	± 10% of true value
	Other	ILM03.0 ILMO4.0	CRA: Beginning of every analytical run (no acceptance criteria) Quarterly - Instrument detection limits
Mercury (CVAA)	Initial	ILM03.0 ILMO4.0	Minimum 3 levels plus blank "r"(4) ≥ 0.995 ICV: ± 20%
	Continuing	ILM03.0 ILMO4.0	Every 10 samples ± 20% of true value
	Ending	ILM03.0 ILMO4.0	± 20% of true value
	Other	ILM03.0 ILMO4.0	Quarterly - Instrument detection limits ILMO3.0: CRA not required. ILMO4.0: CRA: Beginning of every analytical run (no acceptance criteria)
Pesticides/PCBs	Initial	OLM03.1	3 levels for single component analytes, 1 level for multicomponent analytes RSD must be ≤ 20% except α-BHC and δ-BHC at 25% (allow up to 2 target analytes to be 20% ≤ 30%)
	Continuing	OLM03.1	Instrument Blank and midpoint calibration or PEM every 12 hours % D: ± 25% of predicted response

TABLE 8.5-9
Summary of USEPA Contract Laboratory Program Statement of Work
Method Calibrations
(Continued)

Analytical Parameter	Calibration	Method	Requirement
Pesticides/PCBs (continued)	Ending	OLM03.1	Instrument Blank and midpoint calibration or PEM
	Other	OLM03.1	Resolution Check Mixture ≥ 60% PEM: ≥ 90% DDT, Endrin breakdown must each be ≤ 20% (≤ 30% combined)
Semivolatiles by GC/MS	Initial	OLM03.1	5 levels RRF and RSD must meet method criteria
	Continuing	OLM03.1	Mid-level every 12 hours after tuning check %D and RRF must meet minimum criteria
	Ending	OLM03.1	Not Applicable
	Other	OLM03.1	DFTPP tuning at the beginning of every 12 hour shift
Volatiles by GC/MS	Initial	OLM03.1	5 levels RRF and RSD must meet method criteria
	Continuing	OLM03.1	Mid-level every 12 hours after tuning check %D and RRF must meet minimum criteria
	Ending	OLM03.1	Not Applicable
	Other	OLM03.1	BFB tuning at the beginning of every 12 hour shift

TABLE 8.6-1
Precision and Accuracy Measurements

Measurement	Definition
Accuracy	The degree of agreement of a measurement with an accepted reference or true value. The only true or known values in the laboratory are spiked samples. Expressed as laboratory control sample (LCS) percent recovery (% R): $$LCS\ \%Recovery = \frac{X}{t} x\ 100$$ where: X = observed concentration t = concentration of spike added Expressed as matrix spike/matrix spike duplicate (MS/MSD) sample percent recovery (% R): $$MS/MSD\ \%Recovery = \frac{X_s - X}{t} x\ 100$$ where: X_s = observed concentration in spiked sample X = observed concentration in unspiked sample t = concentration of spike added
Precision	The measure of analytical reproducibility of two values. Expressed as the relative percent difference (RPD) of two values. $$RPD = \left[\frac{\lvert X_1 - X_2 \rvert}{\left(\frac{X_1 + X_2}{2} \right)} \right] x\ 100$$ where: X_1 = first observed concentration X_2 = second observed concentration

TABLE 8.6-1
Precision and Accuracy Measurements
(Continued)

Measurement	Definition
Arithmetic mean	The average of a set of values. $\bar{x} = \frac{\sum_{i=1}^{n} x_i}{n}$ where: $\bar{x}$ = the mean x_i = the i^{th} data value n = number of data values
Standard Deviation	A measure of the random (probable) error associated with a single measurement within a data set. $s = \sqrt{\frac{\sum_{i=1}^{n}(x_i - \bar{x})^2}{n-1}}$ where: s = sample standard deviation $\bar{x}$ = the mean x_i = the i^{th} data value n = number of data values
Quality Control Chart	A graphical representation of analytical accuracy. Displays the arithmetic mean of a data set, the upper and lower warning limits and the upper and lower control limits.
ACCURACY	
Upper Control Limit (UCL)	$UCL = \bar{x} + 3s$
Upper Warning Limit (UWL)	$UWL = \bar{x} + 2s$
Lower Warning Limit (LWL)	$LWL = \bar{x} - 2s$
Lower Control Limit (LCL)	$LCL = \bar{x} - 3s$
PRECISION	
RPD	Zero to (mean RPD + 3s)

TABLE 8.11-1
Instrument Maintenance Schedule
Ion Chromatograph[1]

As Needed	Daily	Weekly	Monthly	Semi-annually
Clean micromembrane suppressor when decreases in sensitivity are observed.	Check plumbing/leaks.	Check pump heads for leaks.	Check all air and liquid lines for discoloration and crimping, if indicated.	Lubricate left hand piston.
Check fuses when power problems occur.	Check gases.	Check filter (inlet)	Check/change bed supports guard and analytical columns, if indicated.	Clean conductivity cell.
Reactivate or change column when peak shape and resolution deteriorate or when retention time shortening indicates that exchange sites have become deactivated.	Check pump pressure.			Check conductivity cell for calibration.
De-gas pump head when flow is erratic.	Check conductivity meter.			

TABLE 8.11-2
Instrument Maintenance Schedule
Total Organic Halide Analyzer[1]

Daily	As Needed
Check electrodes for damage, polish the electrodes.	Examine and clean or replace pyrolysis tube.
Replace dehydrating fluid and electrolyte fluid.	Clean titration cell.
Clean quartz boat.	Observe gas flow.
Observe check valves during use for backfeed.	Replace reference electrode fluid.
At end of each day of use, wash out absorption module, empty electrolyte and fill cell with DI water. Empty dehydrator tube	Change quartz wool.
Perform cell performance check.	Replace o-rings and seals.

TABLE 8.11-3
Instrument Maintenance Schedule
High Pressure Liquid Chromatograph[1]

Daily	As Needed
Check level of solution in reservoirs. If adding, verify that solvent is from the same source. If changing, rinse gas and delivery lines to prevent contamination of the new solvent.	Replace columns when peak shape and resolution indicate that chromatographic performance of column is below method requirements.
Check gas supply.	Oil autosampler slides when sample does not advance.
Flush with an appropriate solvent to remove all bubbles.	Rinse flow cell with 1N nitric acid if sensitivity low.
Pre-filter all samples.	Change pump seals when flow becomes inconsistent.
	Repack front end of column Backflush column.

TABLE 8.11-4
Instrument Maintenance Schedule
Flame Atomic Absorption Spectroscopy[1]

Daily	Monthly	As Needed
Verify proper safety precautions are working.	Clean all filters and fans.	Check drain receptacle.
Verify gas box operates properly and safely.	Change capillary tubing	Check background corrector for alignment.
Verify sensitivity using elements in UV/VIS spectrum.	Clean optical windows	Clean burner head.
		Clean nebulizer.
		Clean spray chamber.
		Check sample introduction O-rings.

TABLE 8.11-5
Instrument Maintenance Schedule
ICP & ICP/MS[1]

Daily	Monthly or As Needed	Semi-annually	Annually
Check gases Check that argon tank pressure is 50-60 psi and that a spare tank is available. Check aspiration tubing	Clean plasma torch assembly to remove accumulated deposits.	Change vacuum pump oil.	Notify manufacturer service engineer for scheduled preventive maintenance service.
Check vacuum pump gage. (<10 millitorr)	Clean nebulizer and drain chamber; keep free flowing to maintain optimum performance.	Replace coolant water filter. (may require more or less frequently depending on the quality of water)	
Check that cooling water supply system is full and drain bottle is not full. Also that drain tubing is clear, tight fitting and has few bends.	Clean filters on back of power unit to remove dust.		
Check that nebulizer is not clogged.	Replace when needed: peristaltic pump tubing sample capillary tubing autosampler sipper probe		
Check that capillary tubing is clean and in good condition.	Check yttrium position. Check O-rings Clean/lubricate pump rollers.		
Check that peristaltic pump windings are secure.			
Check that high voltage switch is on.			
Check that exhaust screens are clean.			
Check that torch, glassware, aerosol injector tube, bonnet are clean			

TABLE 8.11-6
Instrument Maintenance Schedule
Graphite Furnace Atomic Absorption[1]

Daily	Weekly	Monthly	Semi-annually	Annually
Check gas lines and gas supply.	Clean optical windows.	Check coolant level in cooling unit. Add coolant if error message appears.	Change graphite contacts	Notify manufacturer service engineer to clean optics.
Clean contact cylinders.				Check optics
Check tubes and platform; replace if corroded, faking, or if low absorbance results. Check autosampler tubing and alignment. Flush autosampler tubing				
PE4100ZL: clean fume extraction tip, replace fume extraction filter and H_2O trap.				
As needed, trim sampling capillary.				
Check drain lines and waste containers; empty as needed.				
Check acid rinse containers; fill as needed.				

TABLE 8.11-7
Instrument Maintenance Schedule
CVAS & CVAFS

Daily	As Needed	Annually
Change drying tube	Change pump tubing	Change Hg lamp.
Check pump tubing/drain tubing	Check/change Hg lamp	
Check gas pressure	Clean optical cell	
Check aperture reading Check tubing	Lubricate pump	

TABLE 8.11-8
Instrument Maintenance Schedule
Gas Chromatograph[1]

Daily	As Needed	Quarterly/Semi-annually/Annually
Check for sufficient supply of carrier and detector gases. Check for correct column flow and/or inlet pressures.	Replace front portion of column packing or break off front portion of capillary columns. Replace column if this fails to restore column performance or when column performance (e.g. peak tailing, poor resolution, high backgrounds, etc.) indicates it is required.	Quarterly ELCD: change-roughing resin, clean cell assembly. Quarterly FID: clean detector
Check temperatures of injectors and detectors. Verify temperature programs.	Change glass wool plug in injection port and/or replace injection port liner when front portion of column packing is changed or front portion of capillary column is removed.	Semi-annually ECD: perform wipe test.
Check inlets, septa. Replace septum Clean injector port		Annually ELCD: change finishing resin, clean solvent filter. Annually FID: Replace flame tip ECD: detector cleaning and re-foiling, every five years or whenever loss of sensitivity, or erratic response or failing resolution is observed.
Check baseline level.	Perform gas purity check (if high baseline indicates that impure carrier gas may be in use).	
Check reactor temperature of electrolytic conductivity detector.	Replace or repair flow controller if constant gas flow cannot be maintained.	
	Replace fuse.	
Inspect chromatogram to verify symmetrical peak shape and adequate resolution between closely eluting peaks.	Reactivate external carrier gas dryers.	
Clip column leader	Detectors: clean when baseline indicates contamination or when response is low. FID: clean/replace jet, replace ignitor. NPD: clean/replace collector assembly. PID: clean lamp window monthly or replace as needed, replace seals. ELCD: check solvent flow weekly, change reaction tube, replace solvent, change reaction gas, clean/replace Teflon® transfer line. ECD: follow manufacturers suggested maintenance schedule	
	Reactivate flow controller filter dryers when presence of moisture is suspected.	

TABLE 8.11-8
Instrument Maintenance Schedule
Gas Chromatograph[1]
(Continued)

Daily	As Needed	Quarterly/Semi-annually/Annually
GC (continued)	HP 7673 Autosampler: replace syringe, fill wash bottle, dispose of waste bottle contents.	
	Purge & trap devices: periodic leak checks quarterly, replace/condition traps (when poor response or disappearance of reactive or poorly trapped compounds), clean sample lines, valves (if they become contaminated), clean glassware. Clean sparger weekly. Check purge flow monthly. Bake trap as needed to correct for high background. Change trap annually, or as needed whenever loss of sensitivity, or erratic response or failing resolution is observed.	
	Purge & trap autosamplers: leak check system, clean sample lines, valves. PTA-30 autosampler also requires cleaning the syringes, frits, valves, and probe needles, adjustment of micro switches, replacement of Teflon® valve, and lubrication of components.	

TABLE 8.11-9
Instrument Maintenance Schedule
Mass Spectrometer[1]

Daily	Weekly	As Needed[2]	Quarterly	Semi-Annually	Annually
Check for sufficient gas supply. Check for correct column flow and/or inlet pressure.	Check mass calibration (PFTBA or FC-43)	Check level of oil in mechanical pumps and diffusion pump if vacuum is insufficient. Add oil if needed between service contract maintenance.	Check ion source and analyzer (clean, replace parts as needed)		Replace the exhaust filters on the mechanical rough pump every 1-2 years.
Check temperatures of injector, detector. Verify temperature programs.		Replace electron multiplier when the tuning voltage approaches the maximum and/or when sensitivity falls below required levels.	Check vacuum, relays, gas pressures and flows	Clean rods	
Check inlets, septa.		Clean Source, including all ceramics and lenses - the source cleaning is indicated by a variety of symptoms including inability of the analyst to tune the instrument to specifications, poor response, and high background contamination.	Change oil in the mechanical rough pump. Relubricate the turbomolecular pump-bearing wick.		
Check baseline level.		Repair/replace jet separator.			
Check values of lens voltages, electron multiplier, and relative abundance and mass assignments of the calibration compounds.		Replace filaments when both filaments burn out or performance indicates need for replacement.			

TABLE 8.11-10
Instrument Maintenance Schedule
TRAACS Auto Analyzer [1]

As Needed	Daily	Monthly	Semi-annually	Annually
Replaces air filter when progressive loss of air pressure is observed.	Check air pressure gauge (22 ± 2 psi)	Change all pump tubes (or after 200 hours of pumping time)	(or after 1000 hours of pumping time)	Lightly lubricate the Linear Sample Rails (use semi-fluid lubricant)
Replace air valve tubing when occlusion in tubing is observed	Use recommended washout procedure (at end of analysis operations)	Clean sample probe shaft	Replace pump platens	Replace colorimeter lamp (or after 2500 hours of use)

TABLE 8.11-11
Instrument Maintenance Schedule
Sonicator [1]

Daily	As Needed
Daily when used: Inspect probe tips for inconsistencies (etching/pitting).	Replace probe tip.
	Disassemble and clean sonicator probe tips.
	Tune sonicator assembly.

TABLE 8.11-12
Instrument Maintenance Schedule
Analytical/Top Loading Balances[1]

Daily	Annually
Check using Class S-verified weights once daily or before use Clean pan and weighing compartment	Manufacturer cleaning and calibration.

TABLE 8.11-13
Instrument Maintenance Schedule
Refrigerators/Walk-in Coolers[1]

Daily	As Needed
Temperatures checked and logged.	Refrigerant system and electronics serviced.

TABLE 8.11-14
Instrument Maintenance Schedule
Ovens[1]

Daily	As Needed
Temperatures checked and logged.	Electronics serviced.

TABLE 8.11-15
Instrument Maintenance Schedule
Specific Digital Ion Analyzer[1]

Daily	As Needed
Daily when used: Calibrate with check standards. Inspect electrode daily, clean as needed. Inspect electrode proper levels of filling solutions daily, fill as needed. Clean probe, each use.	Electronics serviced.

TABLE 8.11-16
Instrument Maintenance Schedule
Turbidimeter[1]

Daily	Monthly	As Needed
Daily when used: Adjust linearity on varying levels of NTU standards. Standardize with NTU standards. Inspect cells.	Clean instrument housing	Electronics serviced.

TABLE 8.11-17
Instrument Maintenance Schedule
Dissolved Oxygen Meter[1]

Daily	As Needed
Daily when used: Calibrate with check standards. Check probe membrane for deterioration Clean and replace membrane with HCl solution.	Electronics serviced.

TABLE 8.11-18
Instrument Maintenance Schedule
Conductance Meter[1]

Daily	As Needed
Daily when used: Check probe and cables. Standardize with KCl. Inspect conductivity cell	Electronics serviced.

TABLE 8.11-19
Instrument Maintenance Schedule
Chemical Oxygen Demand (COD) Reactor[1]

Daily	As Needed
Daily when used: Calibrate with check standards.	Electronics serviced.

TABLE 8.11-20
Instrument Maintenance Schedule
Spectrophotometer[1]

As Needed	Daily	Monthly	Annually
Dust the lamp and front of the front lens.	Check the zero %A adjustment.	Clean windows	Check instrument manual.
	Clean sample compartment		Perform wavelength calibration.
	Clean cuvettes		Replace lamp annually or when erratic response is observed.
			Clean and align optics.

TABLE 8.11-21
Instrument Maintenance Schedule
pH Meter[1]

As Needed	Daily
Clean electrode.	Inspect electrode. Verify electrodes are properly connected and filled.
Refill reference electrode.	Inspect electrode proper levels of filling solutions. Make sure electrode is stored in buffer.

TABLE 8.11-22
Instrument Maintenance Schedule
Fourier Transform Infrared Spectrometry (FTIR)[1]

Check desiccant every 3 months.
Check KBr window every 3 months.

TABLE 8.11-23
Instrument Maintenance Schedule
Total Organic Carbon Analyzer

Daily	As Needed	Weekly	Monthly	Semi-Annually
Check: Oxygen supply Persulfate supply Acid supply Carrier gas flow rate (~ 150 cc/min) IR millivolts for stability (after 30 min. warm-up) Reagent reservoirs	Check injection port septum after 50-200 runs. Tube end-fitting connections after 100 hours or use. Indicating drying tube. NDIR zero, after 100 hours of use. Sample pump, after 2000 hours for use. Digestion vessel/condensation chamber, after 2000 hours of use. Permeation tube, after 2000 hours of use. NDIR cell, after 2000 hours of use.	Check liquid-flow-rate-pump-tubing conditions on autosampler Check injection port septum	Clean digestion vessel Clean condenser column Do the leak test	Change pump tubing

Footnotes to Preventive Maintenance Tables

(1) Refer to manufacturer's instructions for each instrument to identify and perform maintenance operations.

TABLE 8.11-24
Instrument Maintenance Schedule
Digestion Block

Annually
Check temperature with NIST thermometer

TABLE 8.11-25
Instrument Maintenance Schedule
Flash Point Tester

Daily	As Needed
Check tubing. Clean sample cup each use.	Check thermometer against NIST thermometer, when used.
Check gas.	
Clean flash assembly	
Check stirrer	

TABLE 9.4-1
Proficiency Testing Programs

PT Sample Program Description	Analysis Performed	Frequency of Participation
Water Pollution Program Samples provided by Environmental Resource Associates, a NIST-approved PT Provider	Trace Metals, Minerals, Nutrients, Demand, PCBs in Water, PCBs in Transformer Oil, Pesticides, Volatile Halocarbons, Volatile Aromatics, Semivolatiles, Polynuclear Aromatic Hydrocarbons (HPLC) and Miscellaneous inorganics (e.g. TSS, Cyanide, Total Phenolics)	Semi-annual
Water Supply Program Samples provided by Environmental Resource Associates, a NIST-approved PT Provider	Trace Metals, Nitrate/Nitrite/Fluoride, Pesticides, Herbicides, Trihalomethanes, Volatile Organics, and Miscellaneous (TDS, Hardness, pH, Alkalinity, Sodium, Sulfate, Cyanide, Turbidity, and TOC)	Semi-annual
Hazardous Waste Program + UST and California Samples provided by Environmental Resource Associates, a NIST-approved PT Provider	Semivolatile Organics (BNA), Pesticides, Herbicides, Volatile Organics, Metals, Anions, PAH, TPH Gas and Diesel	Semi-annual
Army Corps of Engineers – MRD and US Navy	Anions, Herbicides, Organochlorine Pesticides, Polychlorinated Biphenyls, Semivolatile Organics, Volatile Organics, TAL Metals, Cyanide	18 months
STL Corporate Double Blind – samples provided by ASI	Volatile Organics, Metals, General Chemistry, Base/Neutral Acid Extractables, Project Management	Annually
OhioVoluntary Action Program – samples provided by APG	Based on Certification at the time of PT analysis – MSS, MSV, GCS, GCV, Metals, General Chemistry	Semiannually
New York DOH/CLP – samples provided by state	Based on Certification at the time of PT analysis – MSS, MSV, GCS, GCV, Metals, General Chemistry, Expanded Deliverable Package (CLP)	Semiannually
West Virginia – samples provided by APG (PETs)	Based on Certification at the time of PT analysis – MSS, SMV, GCS, GCV, Metals, and General Chemistry. Standard and "plus" concentration levels required	Annually

PT Sample Program Description	Analysis Performed	Frequency of Participation
Allied Signal	Volatile Organics, Semivolatile Organics, Metals, BOD, COD, TSS, TPH	Annually
STL North Canton Internal PEs – Single or Double Blinds	As needed	As a follow-up to unacceptable PTs from other programs or internal/external audits

Note: Various client and agency single and double blind PTs are introduced in the laboratory throughout the year.

Acronyms and Initialisms

A2LA	American Association for Laboratory Accreditation
AA	Atomic Absorption
ANSI	American National Standards Institute
AR/COC	Analysis Request/Chain-of-Custody
ASQC	American Society for Quality Control
ASTM	American Society for Testing and Materials
BFB	Bromofluorobenzene
BLK	Blank
BOD	Biochemical Oxygen Demand
CCC	Calibration Check Compound
CEO	Chief Executive Officer
CF	Calibration Factor
CFR	Code of Federal Regulations
CHP	Chemical Hygiene Plan
CLP	Contract Laboratory Program
CERCLA	Comprehensive Environmental Response, Compensation and Liability Act (Superfund)
COC	Chain-of-Custody
COD	Chemical Oxygen Demand
CRDL	Contract Required Detection Limit
CRQL	Contract Required Quantitation Limit
CSM	Customer Service Manager
CSRM	Certified Standard Reference Material
CST	Customer Service Team
CUR	Condition Upon Receipt
CV	Coefficient of Variation
CVAA	Cold Vapor Atomic Absorption (Spectroscopy)
DFTPP	Decafluorotriphenylphosphine
DOC	Dissolved Organic Carbon
DOE	Department of Energy
DOT	Department of Transportation
DQO	Data Quality Objective

Acronyms and Initialisms (continued)

EH&S	Environmental Health and Safety
EPA	(U. S.) Environmental Protection Agency
FAS	Field Analytical Services
FLAA	Flame Atomic Absorption (Spectroscopy)
FTIR	Fourier Transform Infrared (Spectrometry)
GC	Gas Chromatograph(y)
GC/MS	Gas Chromatography/Mass Spectrometry
GFAA	Graphite Furnace Atomic Absorption (Spectroscopy)
HDPE	High Density Polyethylene
HPLC	High Performance Liquid Chromatography
HRGC	High Resolution Gas Chromatography
HRMS	High Resolution Mass Spectrometry
ICAP	Inductively Coupled Argon Plasma (Spectroscopy)
ICAP/MS	Inductively Coupled Argon Plasma/Mass Spectrometry
ICS	Interference Check Sample
IDL	Instrument Detection Limit
IR	Infrared (Spectroscopy)
IS	Information Systems
IS	Internal Standard
ISO	International Organization for Standardization
IT	Information Technology
KRI	Key Result Indicator
LAN	Local Area Network
LCL	Lower Control Limit
LCS	Laboratory Control Sample
LCSD	Laboratory Control Sample Duplicate
LIMS	Laboratory Information Management System
LRGC	Low Resolution Gas Chromatography
LRMS	Low Resolution Mass Spectrometry
LWL	Lower Warning Limit
MBAS	Methylene Blue Active Substance

Acronyms and Initialisms (continued)

MDC	Minimum Detectable Concentration
MDL	Method Detection Limit
MS	Matrix Spike
MSA	Method of Standard Additions
MSD	Matrix Spike Duplicate
MSDS	Material Safety Data Sheet
NELAC	National Environmental Laboratory Accreditation Conference
NELAP	National Environmental Laboratory Accreditation Program
NCM	Nonconformance Memo
NIOSH	National Institute for Occupational Safety and Health
NIST	National Institute of Standards Technology
NMOC	Non-Methane Organic Compounds
NPDES	National Pollutant Discharge Elimination System
NRC	Nuclear Regulatory Commission
NRM	National Reference Material
PAH	Polynuclear Aromatic Hydrocarbons (or PNA)
PC	Personal Computer
PCB	Polychlorinated Biphenyls
PDS	Post Digestion Spike
PE	Performance Evaluation
PEM	Performance Evaluation Mixture
PM	Project Manager
PQL	Practical Quantitation Limit
PSRL	Project-Specific Reporting Limit
PUF	Polyurethane Foam
QA	Quality Assurance
QAMP	Quality Assurance Management Plan
QAPP	Quality Assurance Project Plan or Quality Assurance Program Plan
QAS	Quality Assurance Summary
QC	Quality Control
QS	Quality System

Acronyms and Initialisms (continued)

QuantIMS	STL North Canton Laboratory Information Management System
QRI	Quality-Related Item
RCRA	Resource Conservation and Recovery Act
RF	Response Factor
RFP	Request for Proposal
RFQ	Request for Quote
RL	Reporting Limit
RPD	Relative Percent Difference
RRF	Relative Response Factor
RSD	Relative Standard Deviation
RSO	Radiation Safety Officer
SDG	Sample Delivery Group
SOP	Standard Operating Procedure
SOW	Statement of Work
SPCC	System Performance Check Compounds
SPLP	Synthetic Precipitation Leaching Procedure
SRL	Standard Reporting Limit
SRM	Standard Reference Material
TCLP	Toxicity Characteristic Leaching Procedure
TIC	Tentatively Identified Compound
TKN	Total Kjeldahl Nitrogen
TOC	Total Organic Carbon
TOX	Total Organic Halides
UCL	Upper Control Limit
UPS	Uninterruptable Power Supply
USEPA	United States Environmental Protection Agency
UWL	Upper Warning Limit
VOA	Volatile Organic Analysis
VOST	Volatile Organic Sampling Train
WAN	Wide Area Network
WS	Water Supply
WP	Water Pollution

Glossary

acceptance limits
Data quality limits specified for analytical method performance.

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. Systematic errors affect accuracy. For chemical properties, accuracy is expressed either as a percent recovery (R) or as a percent bias (R - 100).

aliquot, aliquant
A measured portion of a sample taken for analysis.

analytical spike
A sample created by spiking target analytes into a prepared portion of a sample just prior to analysis. (Also see matrix spike.)

anomaly
See nonconformance.

areas needing improvement
Represent isolated instances of noncompliance or issues that are judged to have a less immediate impact on data quality. Laboratory management must correct the situation or otherwise ensure that the condition does not recur. This term replaces the previous term used "Observations."

arithmetic mean
The arithmetic mean ($\bar{x}$) is the average of a set of values. It is equal to the sum of the observed values divided by the number of observations. Also called "average".

Glossary (continued)

where: $\bar{x}$ = the mean
x_i = the i^{th} data value
n = number of data values

$$\bar{x} = \frac{\sum_{i=1}^{n} x_i}{n}$$

assessment
The evaluation process used to measure the performance or effectiveness of a system and its elements. Assessment is used as an all-inclusive term to denote any of the following: performance, systems, data and compliance audits, management systems reviews, peer reviews, inspections, or spot assessments.

associate
Employee.

audit
A planned and documented investigative evaluation of an item or process to determine its adequacy and effectiveness as well as compliance with established procedures, instructions, drawings, quality management plans, and other applicable documents.

benchmarking
A step-by-step method of improving performance by identifying and studying best practices and comparing them to industry practices.

bias
A systematic (consistent) error in test results. Bias is expressed as the difference between the population mean and the true or reference value, or as estimated from sample statistics, the difference between the sample average and the reference value.

Glossary (continued)

blind performance evaluation sample
A sample either submitted to the laboratory or prepared in the laboratory whereby the concentrations of parameters of concern are known by the preparer and not by the laboratory.

calibration
Establishment of a relationship between various calibration standards and the measurements of them obtained by a measurement system, or portions thereof. The levels of the calibration standard should bracket the range of levels at which actual measurements are to be made. Calibration is also the act of making a scheduled comparison of instrument performance against national standards for instruments which measure physical parameters such as mass, time, and temperature. This type of calibration is independent of use in specific analyses and projects.

calibration curve
The graphical relationship between the known values for a series of calibration standards and instrument responses.

calibration factor (CF)
The ratio of the instrument response of an analyte to the amount injected. CFs are used in external standard calibrations.

$$CF = \frac{Total\ Area\ of\ Peak}{Mass\ Injected}$$

calibration standard
A standard used to quantitate the relationship between the output of a sensor and a property to be measured. Calibration standards should be traceable to standard reference materials (provided by NIST, or other recognized standards agencies) or a primary standard.

Glossary (continued)

Certificate of Analysis
A STL® report format containing analytical results without supporting/backup information.

certified reference material
A reference material accompanied by a certificate issued by an organization certifying the contents and concentration(s) of the material. (See also standard reference material.)

chain-of-custody (COC)
A system of documentation demonstrating the physical custody and traceability of samples.

check standard analyses
A standard (often a midpoint standard) analyzed at a frequency specified in the method or in a SOP to verify the continuing calibration of the standard curve.

client
Any individual or organization for whom items or services are furnished or work is performed in response to defined requirements and expectations.

client sample
The material or collection media submitted to the laboratory for analysis. Field QC samples are considered client samples but laboratory QC samples are not counted as client samples when counting samples for QC batches.

coefficient of variation (relative standard deviation)
A measure of precision (relative dispersion). It is equal to the standard deviation (s) divided by the mean ($\bar{x}$) and multiplied by 100 to give a percentage value.

Glossary (continued)

$$CV\ (RSD) = \left(\frac{s}{\bar{x}}\right) x\ 100$$

collocated samples
Independent samples collected in such a manner that they are equally representative of the variable(s) of interest at a given point in space and time. The results will indicate sampling as well as analytical variability.

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 (i.e., SOPs) and a uniform set of units and calculations for analyzing and reporting environmental data.

completeness
Completeness is a measure of the percentage of measurements that are judged to be valid measurements. At a minimum, the objective for completeness of data is 90% for each constituent analyzed. It is usually expressed as a percentage:

$$\%\ Completeness = \frac{V}{n} x\ 100$$

where: V = number of measurements judged valid
n = total number of measurements

composite
A sample composed of two or more increments.

control chart
A graphical representation of analytical accuracy. Displays the arithmetic mean of a data set, the upper and lower warning limits and the upper and lower control limits.

Glossary (continued)

control table
A tabular presentation of test results with respect to time or sequence of measurement, together with limits within which the results are expected to lie when the analytical process is in a state of control.

controlled document
A document for which the distribution is known. Updates of the document are sent to the original recipients, unless the copy distributed is an uncontrolled copy.

corrective action
A measure taken to rectify conditions adverse to quality and, where necessary, to preclude their recurrence.

Glossary (continued)

correlation coefficient
The correlation coefficient (r) is a determination of how closely data "fits" a straight line. It is a number between -1 and 1 that indicates the degree of linear relationship between two sets of numbers. A correlation coefficient of +1 (usually calculated to three decimal places or 1.000) means the data falls exactly on a straight line with positive slope. A correlation coefficient of -1 (or -1.000) means the data falls exactly on a straight line with negative slope.

customer
See client.

data quality objective (DQO)
Data quality objectives (DQOs) 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 (EPA 1994). Typically, DQOs are identified during project scope and development of sampling and analysis plans. In this QA manual, however, we refer to only the analytical DQOs because laboratories generally do not have any authority over sample collection, shipment, or other field-related activities that may affect the data quality of the environmental sample before the sample is received in the laboratory. EPA has established six primary analytical DQOs for environmental studies: precision, accuracy, representativeness, completeness, comparability, and detectability.

The components of analytical variability (uncertainty) can be estimated when QA and QC samples of the right types and quantities are incorporated into measurement procedures at the analytical laboratory. STL® incorporates numerous QA and QC samples to obtain data for comparison with the analytical DQOs and to ensure that the measurement system is functioning properly. The QA and QC samples and their applications, described in Section 8.4 and are selected on the basis of method- or client-specific requirements. Field blanks, field duplicates, and performance evaluation (PE) samples are received from the client as unknown samples. Analytical laboratory QC samples for inorganic, organic, and radionuclide analyses may include calibration or instrument blanks, method blanks, background, duplicates, replicates, laboratory control samples (LCSs), calibration standards, matrix spikes (MSs), matrix spike duplicates (MSDs), surrogate spikes, and yield tracers.

data validation

Glossary (continued)

See validation - data.

data verification
See verification - data.

deficiency
See nonconformance or finding.

degrees of freedom
The number of independent deviations used in calculating an estimate of the standard deviation.

double blind performance evaluation sample
A sample that contains select parameters at defined levels. The levels are unknown to the laboratory. The laboratory is also unaware that the sample is a performance evaluation sample.

duplicate sample analyses
Different aliquots of the same sample are analyzed to evaluate the precision of an analysis.

error
The difference between an observed or measured value and its true value.

field blank
A blank that is prepared and handled in the field and analyzed in the same manner as its corresponding client samples.

field matrix spike
A sample created by spiking target analytes into a sample in the field at the point of sample acquisition.

finding
Noncompliant practices or policies which have significant adverse impact on data quality, technical defensibility, or regulatory acceptance of data. Findings require immediate attention by

Glossary (continued)

the laboratory management and must be resolved to comply with STL's® quality documents and laboratory-established procedures often called deficiencies by auditors.

geometric mean
The n^{th} root of the product of all values in a set of n values or the antilogarithm of the arithmetic mean of the logarithms of all the values of a set of n values. The geometric mean is generally used when the logarithms of a set of values are nearly normally (Gaussian) distributed, such as is the case of much population data.

initial calibration
Analysis of a series of analytical standards at different specified concentrations; used to define the linearity and dynamic range of the response of an instrument to the target compounds prior to the analysis of samples.

inspection
Examination or measurement of an item or activity to verify conformance to specific requirements.

instrument detection limit (IDL)
IDL is a calculated estimate of instrument detectability defined by the USEPA Contract Laboratory Program (CLP).

internal standard (IS)
A compound added to every standard, QC sample, client sample, or sample extract at a known concentration prior to analysis for the purpose of quantitation. For example, internal standards are used as the basis for quantitation of the target compounds by GC/MS.

linear regression
A statistical method for finding a straight line that best fits a set of two or more data points, thus providing a relationship between two or more variables.

matrix

Glossary (continued)

The component or substrate which contains the analyte(s) of interest. Examples of matrices are water, soil or sediment, and air. Matrix is not synonymous with phase (liquid or solid).

matrix effect
An interference in the measurement of analyte(s) in a sample that is caused by materials in the sample. Matrix effects may cause elevated reporting limits or may prevent the acquisition of acceptable results.

matrix spike (MS)
An aliquot of a matrix fortified (spiked) with known quantities of specific compounds and subjected to an entire analytical procedure in order to indicate the appropriateness of the method for a particular matrix. The percent recovery for the respective compound(s) is then calculated.

matrix spike duplicate (MSD)
A second aliquot of the same matrix as the matrix spike (above) that is spiked in order to determine the precision of the method.

may
Denotes permission but not a requirement.

mean
See arithmetic mean.

measurement
The process or operation of ascertaining the extent, degree, quantity, dimensions, or capability with respect to a standard.

median
The middle value of a set of data when the data set is ranked in increasing or decreasing order.

method
An assemblage of techniques.

method blank (MB)

Glossary (continued)

An analytical control consisting of all reagents, which may include internal standards and surrogate standards, that is carried through the entire analytical procedure. The method blank is used to define the level of laboratory background contamination. Examples of method blanks are a volume of deionized or distilled laboratory water for water samples, a purified solid matrix for soil/sediment samples, or a generated zero air.

method detection limit (MDL)
The minimum concentration of an analyte that, in a given matrix and with a specific method, can be identified, measured, and reported with 99% confidence that the analyte concentration is greater than zero. The MDL is operationally defined as:

$$MDL = st_{(n-1,\ \alpha=0.99)}$$

where:

s = the standard deviation of a number of measurements of a blind or sample matrix containing the analyte at a concentration near the lowest standard recommended in the method and

$t_{(n-1,\ \alpha=0.99)}$ = the student's value for a one-sided t-statistic appropriate for the number of samples used to determine (s), at the 99% confidence level and n-1 degrees of freedom.

modified method
A standard or reference method which has been changed to meet project or matrix requirements.

must
Denotes a requirement is mandatory and has to be met.

notable practices
Laboratory practices that increase effectiveness and quality and represent improvements with respect to conventional laboratory operations.

nonconformance

Glossary (continued)

An unplanned deviation from an established protocol or plan. The deviation may be the result of STL's® actions, then termed a deficiency. If the deviation is the result of events beyond the control of STL®, it is termed an anomaly.

operational calibration
Routinely performed as part of instrument usage, such as the development of a standard calibration curve. Operational calibration is generally performed for instrument systems.

outlier
A result excluded from the statistical calculations due to being deemed "suspicious" when applying the "Grubbs Test" (or equivalent).

parameter
A constant or coefficient that describes some characteristic of a population (e.g., standard deviation, mean, regression coefficients). Also, a chemical being measured, i.e., an analyte.

percent difference
When two independent measurements of the same characteristics are available, it is possible to use the percent difference instead of the coefficient of variation to measure precision.

$$\%D = \left| \frac{X_1 - X_2}{X_1} \right| x\ 100\%$$

where: $\%D$ = percent difference
X_1 = first value
X_2 = second value

percent recovery
A measure of accuracy determined from the comparison of a reported spike value to its true spike concentration.

$$\%R = \frac{observed\ conc. - sample\ conc.}{true\ spike\ conc.} x\ 100\%$$

Glossary (continued)

performance audit
See performance evaluation.

performance evaluation (PE)
A type of audit in which a known or characterized value is compared to the result obtained through the routine analysis of the sample in the laboratory to evaluate the proficiency of an analyst or laboratory.

periodic calibration
A calibration that is performed at prescribed intervals for equipment such as balances, thermometers, and balance weights. In general, they are performed on equipment that are distinct, singular purpose units, and are relatively stable in performance.

population
A generic term denoting any finite or infinite collection of individual things, objects, or events.

practical quantitation limit (PQL)
The lowest concentration a method can reliably achieve within limits of precision and accuracy and is derived from empirical, matrix-free method performance studies.

precision
Precision is an estimate of variability, that is, it is an estimate of agreement among individual measurements of the same physical or chemical property, under prescribed similar conditions. The precision of a measurement system is affected by random errors. Precision is expressed either as relative standard deviation (RSD) for replicate measurements greater than two or as relative percent difference (RPD) for duplicate measurements. Table 8.6-1 illustrates the formulae used to calculate units of precision (i.e., RSD and RPD).

preventive maintenance
An organized program within STL® laboratories of actions (such as equipment cleaning, lubricating, reconditioning, adjustment and/or testing) taken to maintain proper instrument and equipment performance and to prevent instruments and equipment from failing during use.

Glossary (continued)

primary standard
A material having a known, stable property that can be accurately measured or derived from established physical or chemical constants. It is readily reproducible and can be accepted (within stated limits) and used to establish the same value of another substance or item.

procedure
Detailed instructions to permit replication of a method. (See standard operating procedure.)

proficiency testing
A series of planned tests which will determine the ability of field technicians or laboratory analysts to perform routine analyses. The results from this testing may be used for comparison against established criteria or for relative comparisons among the data from a group of technicians or analysts.

project-specific reporting limit (PSRL)
See reporting limit.

protocol
Methodology specified in regulatory, authoritative, or contractual situations.

QC batch
The QC batch consists of a set of up to 20 field samples that behave similarly (i.e., same matrix) and are processed using the same procedures, reagents, and standards within the same time period.

QC check sample
A reference matrix containing known concentrations of parameters of interest. If prepared in the laboratory, it is made using stock standard solutions independent of those used for calibration. If the results of these parameters do not meet acceptance criteria, corrective actions are taken.

qualification (personnel)
The characteristics of abilities gained through education, training, or experience, as measured against established requirements, such as standards or tests, that qualify an individual to perform a required function.

Glossary (continued)

quality
The sum of features and properties/characteristics of a process, item, or service that bears on its ability to meet the stated needs of the user. STL® has defined quality as meeting the needs of our clients, both internal and external.

quality assurance (QA)
An integrated system of management activities involving planning, implementation, assessment, reporting, and quality improvement to ensure that a process, item, or service is of the type and quality needed and expected by the customer.

Quality Assurance Directive
QA directives are memos issued by the QA Director (or the QA Managers for their facility) to clarify policies, Procedures, and the QMP; or to give direction for an immediate action to ensure or maintain quality.

Quality Management Plan (QMP)
The Quality Management Plan for Environmental Analyses (QMP) is a formal document that describes quality systems in terms of organizational structure, functional responsibilities of management, and staff, and lines of authority. The QMP documents the QMS and describes both the organizational and project-specific principles, goals, controls, and tools of the QMS. The QMP provides the criteria and specifications for the generation of environmental analytical data.

Quality Assurance Project or Program Plan (QAPP)
A formal document describing in comprehensive detail the necessary QA, QC, and other technical activities that must be implemented to ensure the results of the work performed will satisfy the stated performance criteria.

quality control (QC)
The overall system of technical activities that measures the attributes and performance of a process, item, or service against defined standards to verify that it meets the stated requirements established by the client or by STL®.

quality improvement

Glossary (continued)

The process of improving the quality of operations. This process encourages worker recommendations for improvement of work processes and requires timely management evaluation and feedback or implementation.

quality management
That aspect of the overall management system of the organization that determines and implements the quality policy. Quality management includes strategic planning, allocation of resources, and other systematic activities (e.g., planning, implementation, and assessment) pertaining to the quality management system.

quality management system (QMS)
A structured and documented management system describing the policies, objectives, principles, organizational authority, responsibilities, and implementation plan of an organization for ensuring quality in its work processes, products, and services. The quality system provides the framework for planning, implementing, and assessing work performed by the organization and for carrying out required QA and QC.

random error
Variations of repeated measurements that are random in nature and individually not predictable.

range
The difference between the largest and smallest numbers in a set of numbers.

raw data
All documentation associated with the original recording of analytical results pertinent to a specific sample or set of samples. This may include laboratory worksheets, calculation forms, instrument-generated output, analyst notes, etc., from sample receipt through final reporting.

reagent water
Water in which an interferant is not observed at or above the minimum quantitation limit of the parameters of interest. The reagent water's purity and acceptability is verified by analysis with each set of samples.

Glossary (continued)

recovery
See percent recovery.

reference method
A method of known and demonstrated accuracy.

regression coefficients
The quantities describing the slope and intercept of a regression line.

relative error
An error expressed as a percentage of the true value or accepted reference value.

relative percent different (RPD)
Statistic for evaluating the precision of a replicate set. For replicate results:

$$RPD = \left[\frac{|X_1 - X_2|}{\left(\frac{X_1 + X_2}{2} \right)} \right] x\ 100$$

where: X_1 = first observed concentration
X_2 = second observed concentration

relative response factor (RRF)
A measure of the relative mass spectral response of a compound compared to its internal standard. RRFs are determined by analysis of standards and are used in the calculation of concentrations of analytes in samples. Because a RRF is the comparison of two responses, it is a unitless number. RRFs are determined by the following equation:

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

Glossary (continued)

where: A = area of the characteristic ion measured
C = concentration
IS = internal standard
x = analyte of interest

relative standard deviation (RSD)
See coefficient of variation.

reporting limit (RL)
One of two types of reporting limit conventions within STL®. The Reporting Limit (RL) is a uniform, STL® -wide reporting limit based on an evaluation of the PQLs at STL® laboratories and the expected method performance in routine water and soil matrices. Project Specific Reporting Limits (PSRLs) are reporting limits that are defined by project requirements.

representative sample
A sample taken to represent a lot or population as accurately and precisely as possible.

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 (concentration) is representative of the constituent concentration in the sample matrix. At each STL® laboratory, every effort must be made to analyze an aliquot that is representative of the original sample, and to ensure the homogeneity of the sample before subsampling.

reproducibility
The precision, usually expressed as a standard deviation, measuring the variability among results of measurements of the same sample at different laboratories.

response factor (RF)

Glossary (continued)

A factor derived from the calibration of a compound that is used in the quantitation calculation of sample analytes. A response factor may be derived from an external standard calibration (then called a Calibration Factor) or from an internal standard calibration (then called a Relative Response Factor).

secondary standard
A material having a property that is calibrated against a primary standard.

self assessment
Assessments of work conducted by individuals, groups, or organizations directly responsible for overseeing or performing the work.

shall
Denotes a requirement that is mandatory and has to be met.

should
Denotes a guideline or recommendation.

standard addition
The procedure of adding known increments of the analyte of interest to a sample to cause increases in detection response to subsequently establish, by extrapolation of the plotted responses, the level of the analyte of interest present in the original sample.

standard deviation
A measure of the dispersion about the mean of the elements in a population. The square root of the variance of a set of values:

$$s = \sqrt{\frac{\sum_{i=1}^{n}(x_i - \bar{x})^2}{n-1}}$$

where: s = standard deviation
Σ = sum of

Glossary (continued)

X = observed values
n = number of observations

standardization
The establishment of the value of a potential standard with respect to an established or known standard.

standard method
A method of known and demonstrated precision issued by an organization generally recognized as competent to do so.

standard operating procedure (SOP)
A written document that details an operation, analysis, or action, with prescribed techniques and steps, that is officially approved as the method for performing certain routine or repetitive tasks.

standard reference material (SRM)
A material produced in quantity, of which certain properties have been certified by the National Institute of Standards and Technology (NIST), formerly NBS, or other agencies to the extent possible to satisfy its intended use.

standard verification
Standard is checked by STL® or the vendor versus a known specification. See Section 8.5.4.3.

statistic
A constant or coefficient that describes some characteristic of a sample. Statistics are used to estimate parameters of populations.

stock solution
A concentrated solution of analyte(s) or reagent(s) prepared and verified by prescribed procedure(s), and used for preparing working standards or standard solutions.

Glossary (continued)

subsample
A portion taken from a sample. A laboratory sample may be a subsample of a gross sample; similarly, a test portion may be a subsample of a laboratory sample.

supplier
See vendor.

surrogate (surrogate standard)
Compounds, when required by a method, that are used added to every blank, sample, LCS, matrix spike, matrix spike duplicate, and standard. They are used to evaluate analytical efficiency by measuring recovery. Surrogates include brominated, fluorinated, or isotopically-labeled compounds that are not expected to be detected in environmental media.

systematic error
The condition of a consistent deviation of the results of a measurement process from the reference or known level.

systems audit or evaluation
A systematic on-site qualitative review of facilities, procedures, equipment, training, record keeping, data verification, and reporting aspects of a quality assurance system to arrive at a measure of the capability of the system. Within STL®, system audits or evaluations are performed on a periodic basis under the direction of the STL® Corporate Director of Quality Assurance.

technique
Physical or chemical principle for characterizing materials of chemical systems.

traceability of data
The entire documented chain of acquired data from the original acquisition effort through to the final tabulation, synthesis, reduction, and storage activities. The documentation will allow complete reconstruction of the data.

traceability of samples

Glossary (continued)

During all environmental monitoring field efforts, acquired samples will be assigned specific and unique identification numbers. These sample numbers shall be accompanied by documentation (chain-of-custody form) which clearly identifies all parameters associated with sample acquisition. All additional sample numbering systems applied to the sample must be clearly cross-referenced to the field sample number to provide for traceability of samples from acquisition to reporting of sample results.

traceability of standards
The ability of an analytical standard material used for calibration purposes to be traced to its source. The standards used by STL® must be traceable via written documentation to sources which produce or sell verified or certified standards, i.e., National Institute for Standards and Technology, or vendors preparing standards from those sources which they have certified.

validation - computer software
The process of establishing documented evidence which provides a high degree of assurance that a specific process will consistently produce a product meeting predetermined specifications and quality attributes. This process demonstrates and documents that the software performs correctly and meets all specified requirements.

validation - data
The process of a second party performing a systematic review of the raw and final data produced by a laboratory using predetermined criteria to ascertain the validity of the data with respect to the criteria (e.g., HAZWRAP data validation).

vendor
Any individual or organization furnishing items or services or performing work according to a procurement document. This is an all-inclusive term used in place of any of the following: supplier, seller, contractor, subcontractor, or consultant.

verification - computer software
The process of checking the accuracy of manually entered or automatically (electronically) calculated information.

verification - data

Glossary (continued)

The process of reviewing data to ensure that data reduction has been correctly performed and that analytical results to be reported correspond to the data acquired and processed.

ENVIRONMENTAL TESTING & CONSULTING, INC. – MEMPHIS LQM

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Quality Manual

Laboratory Management Partners, Inc.

For Use By:
A&L Analytical Laboratories, Inc.
Environmental Testing & Consulting, Inc.

Laboratory Management Partners, Inc.
2790 Whitten Road
Memphis, TN 38133
Telephone (voice) 901-213-2400
Facsimile. (901) 213-2440

1 Quality Mission Statement

The goal of Laboratory Management Partners, Inc. is to provide its clients with data of the highest quality, integrity, accuracy, precision and defensibility. To achieve this goal, the management of Laboratory Management Partners, Inc. commits its support to the establishment and full implementation of the policies and procedures described in this Quality Manual and in supporting documents. This Quality System applies to all personnel. Management recognizes that this Quality System is an essential element of our business for sustaining profitability with the dedicated support of our personnel and clients.

Scott McKee

President / CEO

1.1 Management Review and Authorization

The following individuals attest that the information provided in this QM to be true and an accurate representation of the policies and procedures of LMP, Inc. and its holdings.

President / CEO

Signature: _____ Date: _____

Name: Scott McKee (signature on file)

Senior Vice President / CFO

Signature: _____ Date: _____

Name: Chris Langford (signature on file)

Chairman / Executive Vice President

Signature: _____ Date: _____

Name: Nathan Pera, IV (signature on file)

Technical Director

Signature: _____ Date: _____

Name: Michael T. Kaufmann (signature on file)

Quality Assurance Officer

Signature: _____ Date: _____

Name: Dr. Richard Medina (signature on file)

1.2 Distribution Control

Distribution Copy No. _____

1.3 Amendments

None.

2 Table of Contents

Section Description ..... Section-Page

3 Introduction

This Quality Manual (QM) describes the Quality System established for use by Laboratory Management Partners, Inc (LMP, Inc) This document and associated Quality System documents are written in the declarative grammatical mood for ease of readability. The use of this grammatical mood declares the description of the laboratory's Quality System in the present tense. This Quality Manual and associated quality documents indicate the requirements from the most current promulgated version until it is subjected to revision and superceded by the updated revision with a new effective date. Occasionally, this document contains sentences in the imperative grammatical mood to provide emphasis to a critical requirement. The word 'shall', used as an auxiliary verb in these imperative sentences denotes a requirement for both the present and the future. The interpretation of the word 'shall' is not limited to only future commitment as it is in everyday usage. Likewise, the word 'must' denotes a requirement for both the present and the future. The auxiliary verbs 'should' or 'may', which are used sparingly in the text, allow some discretion in the execution of the stated activity but are used to delineate strongly recommended actions or as indicated by reference methods.

This QM provides employees, clients, subcontractors and accrediting agencies with information about how the Quality System operates within the laboratory. The Quality System includes Quality Assurance, Quality Control measures, and laboratory systems including feedback mechanisms for continuous improvement to meet client and/or regulatory needs.

Implementation of the QM is accomplished by documenting procedures, training personnel and reviewing operations for compliance and improvement. The Quality System procedures are contained within the QM and in auxiliary analytical and administrative Standard Operating Procedures (SOPs). The QM and SOPs are available to all laboratory staff as hard copies and in read-only electronic form. The provisions of the Quality System are binding on all temporary and permanent laboratory personnel. All laboratory personnel must adhere strictly to the QM and SOPs.

The QM is composed of sixteen (16) sections that provide overview descriptions of laboratory, policies, procedures, operations and the program defined by the laboratory for Quality Assurance and Quality Control activities. Additional sections are provided as Appendices. Related documentation includes, but does not limit to, the listing of analytical and administrative SOPs, QAQC forms, reference methods, instrument and supporting equipment, and laboratory personnel qualifications are available as separate laboratory records.

The QM describes the Quality System components of Laboratory Management Partners, Inc. to demonstrate competency in the operations for performing environmental analyses. The foundation for the analytical procedures are the methods published by the United States Environmental Protection Agency (EPA), US Army Corps of Engineers, ASTM, AOAC, NIOSH, and other procedures including those supplied by clients and state regulators. This Quality System is based on the current NELAC standards and other requirements of accrediting authorities.

The QM includes requirements and information for assessing competence and determining compliance by the laboratory to the Quality System. When more stringent standards or requirements are included in a mandated test method, by regulation, or specified in a project plan, the laboratory shall demonstrate these requirements through its procedural and documented processes

The QM is for the sole use by Laboratory Management Partners, Inc. for developing, implementing, and maintaining the Quality System. Accrediting authorities and regulators use the QM as a guide to monitor the Quality Systems of Laboratory Management Partners, Inc.

3.1 References

Solid Waste Manual, SW846 Update III, December 1996.

U.S. Army Corps of Engineers (USACE) Shell for Chemical Analytical Requirements, Engineering Manual 200-1-3, 1 Feb 01.

Standard Methods for the Examination of Water and Wastewater, 18th and 20th Edition.

Methods for Chemical Analysis of Water and Wastes, EPA –600/4-79-020, March 1983.

NELAC, Quality Systems, Revision 16, July 12, 2002

NELAC, Program Policy and Structure, Revision 155, July 12, 2002.

40 CFR Part 136 Appendix B.

EPA Guidance for Preparing Standard Operating Procedures (SOPs), EPA QA/G-6, EPA/240/B-01/004, March 2001

EPA 2185 – Good Automated Laboratory Practices (GALP)

ISO/IEC 17025, General Requirements for the Competence of Testing and Calibration Laboratories, First Edition, 1999-12-15.

A register of current promulgated reference documents and manuals are available for staff to determine the latest edition or version of the reference methods, regulations or national standards. The Quality Assurance (QA) Office maintains the register.

3.2 Definitions

Appendix A lists definitions as adopted by the laboratory. The definitions are derived from the standard approved in May 2001 by the National Environmental Laboratory Accreditation Conference (NELAC). The definitions are reviewed and updated, as necessary, after publication of updated versions of the NELAC-adopted Glossary.

4 Organization and Management

4.1 Legal Definition of Laboratory

This QM is applicable to all laboratory locations owned and/or managed by Laboratory Management Partners, Inc., a full service analytical laboratory located at 2790 Whitten Road; Memphis, Tennessee. The laboratory is a privately held corporation incorporated in the State of Tennessee. The owners of Laboratory Management Partners, Inc. of Memphis, Tennessee also own two other laboratories located in Atlantic, Iowa and Guadalajara, Mexico which are separate entities from the Memphis corporation and testing is limited to agricultural procedures. Satellite offices located in Little Rock, Arkansas and Paducah, Kentucky are also part of the corporation. These satellites facilitate sample couriering and regional sales only.

SMR Laboratories, Inc, a Tennessee Corporation doing business in Central City, KY is a wholly owned Corporation. This laboratory provides environmental sampling, delivery and testing services to the Western Kentucky area. Currently, the facility is not under the NELAC scope, but plans are in place to become NELAP accredited by July 2005.

Laboratory Management Partners, Inc. is the result of a joint venture of two established independent laboratories whose sum analytical experience total over 60 years. A&L Analytical Laboratories, Inc. and Environmental Testing and Consulting, Inc. joined forces in 2004. Combining these two laboratories into a single conglomerate laboratory allows for centralized analytical services, particularly in the mid-southern United States. Presently, LMP serves:

Consulting firms,
Engineering firms,
Waste management companies,
Industrial and wastewater treatment facilities,
Government and state agencies,
Private sector,
Universities and R&D organizations and
Other commercial businesses.

4.2 Organization

The laboratory operates a Quality System approach to management in order to produce data of known quality. The laboratory organization provides effective communication and lines of authority to produce analytical data meeting regulatory and client specifications. The organizational design provides open communication while ensuring that undue pressures and day-to-day operations do not compromise the integrity and authenticity of generated reportable data.

President, Scott McKee, is the Chief Executive Officer and reports directly to the Board of Directors. Scott McKee has twelve years experience as manager of an environmental laboratory. Mr. McKee is a graduate of Millsaps College with a B.S in Chemistry with over 15 years of experience in inorganic and organic environmental analysis with a focus on design and implementation of Lab Management Systems.

As President/CEO, Mr. McKee is responsible for the overall operations of LMP and its holdings and functions as the Laboratory Director. His primary function is to provide oversight and

assistance to Laboratory managers. Technical direction is provided on a regular basis with an emphasis on QA compliance, and efficiency. Systems and logistical planning are done on a regular basis to ensure company directives are followed and Quality guidelines are capable of being met. Integration of operational departments and assimilation of new acquisitions is also performed as needed.

Chairman of the Board, Nathan A. Pera, IV, is the Executive Vice President and has 21 years experience in laboratory operations. He has completed course work in Chemistry/Computer Science at Christian Brothers University and University of Memphis in Memphis, TN.

As Executive Vice President, Mr. Pera is responsible for the daily Environmental laboratory operations. His primary function is to ensure that the company is prepared to accept and execute projects for laboratory analysis. His project management responsibility begins as client liaison and extends through developing management plans that will ultimately present the client with valid data in an acceptable format Basic responsibilities include:

a) All functions relating to the operation of the Environmental Laboratory, Sales and Client Services Departments

b) Initiating project management plans for client specific projects. The plans include all project specific criteria such as detection limits, reporting formats and deliverables, turnaround times and methodology requirements. These Project Criteria are then relayed and implemented in the Laboratories affected, Client Services and Project managers. Mr. Pera ensures that all personnel involved with Project Management are aware of all pertinent information.

d) Daily interface with managers and department supervisors to ensure that each area is adequately equipped with personnel and instrumentation

e) Assisting the Technical Director and QA Officer in the final review of report packages. Mr. Pera acts as the initial laboratory project manager for new projects. As the Environmental Laboratory and Project Manager, Mr. Pera reports directly to the CEO.

Chief Financial Officer, Chris Langford, is the Senior Vice President and functions as the Agricultural Lab Manager and Administrative Manager. Mr. Langford has 12 years of Laboratory Management Systems design and coding experience He received his B.S. in Computer Science from Christian Brothers University in Memphis, TN in 1989. Mr Langford oversees the daily operations of the Agricultural Laboratory, Administrative personnel and Lab Support activities. Mr. Langford works with Agricultural Lab personnel on a daily basis to ensure that clients receive accurate results in a timely manner. All operational needs are determined and supplied to ensure that lab staff are completely equipped.

Mr. Langford also oversees all Administrative operations of LMP. This includes Personnel, AR/AP, Information Technology and Corporate Business.

Technical Director/Asst. Laboratory Director, Michael T. Kauffman has 21 years of extensive laboratory operations experience. He received his B.S in Chemistry from Christian Brothers University – Memphis, TN in 1983. Mr Kauffman is responsible for ensuring that all data presented fully meets the specific method requirements.

Asst. Technical Director, Jimmy Ferguson, has 5 years of experience performing a wide variety of laboratory procedures in both the Environmental area as well as Agricultural. He received his B.S. in Environmental Science from Delta State University in 1998

Quality Assurance Officer Dr Richard Medina has 14 years in QA/QC. He received his B.A. in Biology / Chemistry from Austin College in Sherman, TX and a Doctoral of Dental Surgery from

University of Tennessee in Memphis, TN in 1989. Dr. Medina is responsible for monitoring and implementing the Quality Assurance and Quality Control procedures in all sections of laboratory operations.

The QA Supervisor, Ginger Norman, has 6 years in QA/QC. She received her B.S. in Biology/Chemistry from Blue Mountain College in Blue Mountain, MS in 1996 and a Master of Arts in Organizational Management from University of Phoenix in 2000. Ms. Norman supports the Quality Assurance Officer and is responsible for quality system records. The QA Supervisor assists in technical data review, internal audits, maintains oversight of Corrective Action activities and assists the QA Officer in implementation and maintenance of all Quality System activities.

Environmental Project Manager, Randall H. Thomas, has 26 years of Environmental laboratory experience. He received his B.A. in Accounting from Memphis State University in Memphis, TN in 1985.

4.2.1 Operational Support Staff

Project Manager, Connie Bradberry, has 4 years laboratory operations experience. She received her B.S. in Biology from North Texas State University in Denton, TX in 1970 and her M.S. in Environmental Science from Florida Institute of Technology in Melbourne, FL in 1993. Ms. Bradberry has over twenty years experience in computer operations. The last six were in environmental laboratories in LIMS (Laboratory Information Management Systems) implementations and support and information systems management. In her role as Project Manager, Ms. Bradberry reports to the Environmental Laboratory Manager.

In Environmental Client Services, Marla Harshberger and Sally Herrmann are responsible for client services, the sample management area and providing information to the supervisors of the testing areas. The Environmental Client Services staff serves as the client advocate within the laboratory and are critical to coordination of laboratory activities with respect to client demands.

The Laboratory Supervisors are responsible for primary and secondary level data review, personnel training and implementation of the documented procedures defined by the laboratory. Laboratory supervisors provide input on technical and personnel needs to the Laboratory Managers. Laboratory supervisors report to the Laboratory /Technical Director.

Organic Laboratory Supervisor, Lisa Savage, has 15 years of laboratory experience. She received her B.S. in Biology from Mississippi Valley State University in 1988.

Inorganic Laboratory Supervisor, George Dunlap, has 14 years of laboratory experience. He attended Christian Brothers University in Memphis, TN and studied Chemistry.

Bioassay Laboratory Supervisor, Connie Cook, has 12 years of laboratory operations experience. She received her B.S. in Environmental Science from Delta State University in Cleveland, MS in 1990.

Organic Prep Laboratory Supervisor, Wanda Wallace, has 3 years of laboratory experience. She received her B.S. in Natural Sciences from LeMoyne Owen College in Memphis, TN in 1992.

Accounting personnel maintain records of pre-qualified suppliers and subcontractors as well as personnel payroll records.

Personnel job descriptions define the operational function duties and responsibilities for all staff members. Administration and laboratory personnel assignments include cross-functional training and work performance in multiple areas of operations Cross training ensures the availability of laboratory back up personnel during peak loads

During the absence of any staff member, assignment and duties of deputies are established. The manager or supervisor of the effected area authorizes the assignment. The naming of alternative personnel assures the continuing performance of critical tasks during the primary person's absence and ensures that lines of communication remain open for continued decision-making.

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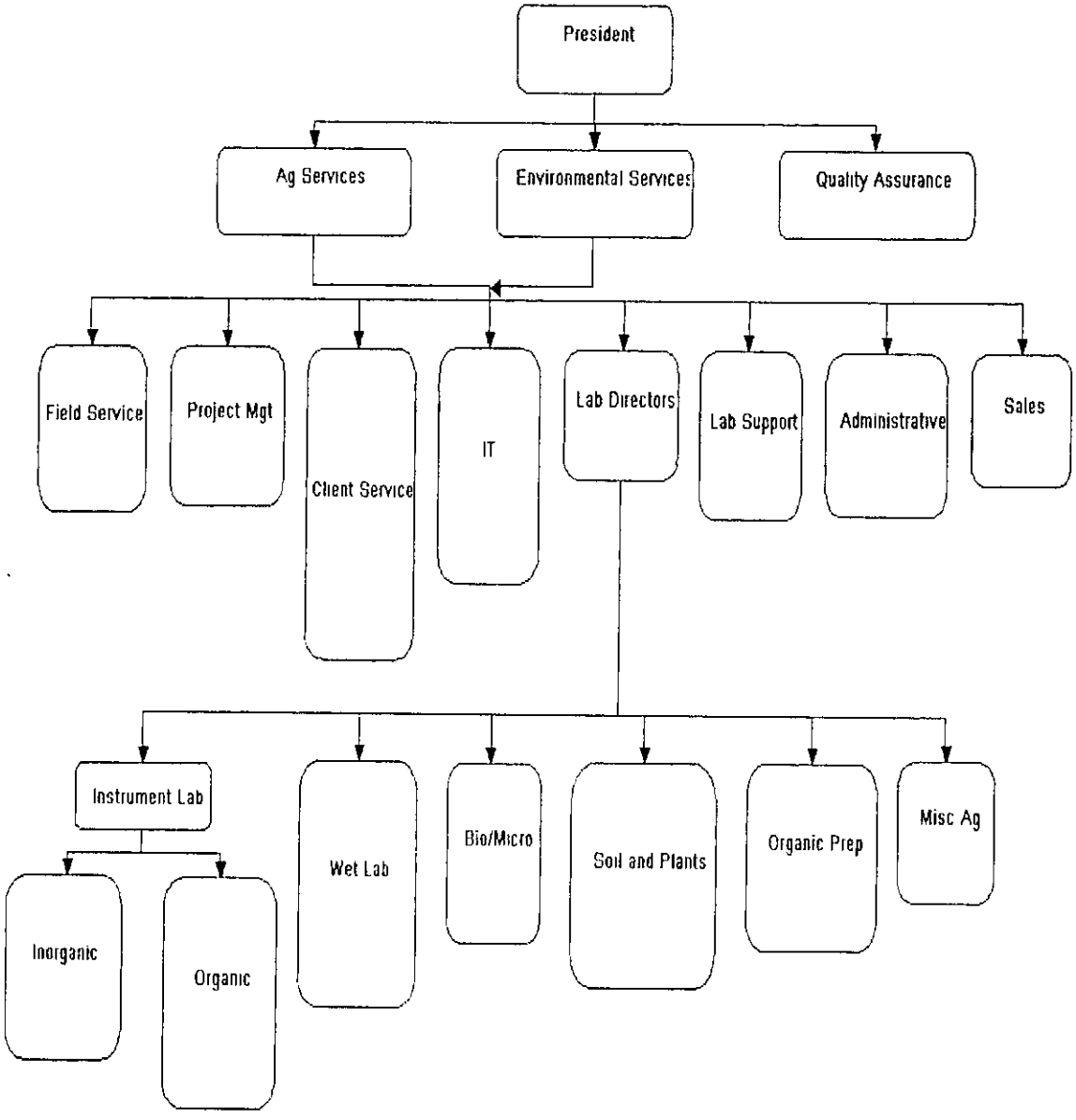
Organizational Chart



Laboratory Management Partners, Inc.

4.3 Business Practices

Laboratory Management Partners, Inc. maintains certifications and validations for the applicable programs as required. A complete listing of various certifications and validations from multiple accreditation programs are available upon request. Laboratory Management Partners, Inc. is open for operation Monday through Friday from 8:00 a.m. to 5.00 p.m (Normal Business Hours). Some areas of the laboratory are in operation around the clock. Sample delivery occurs during normal business hours unless arranged in advance. Management prepares and posts the annual holiday schedule for the year indicating closed operations.

Laboratory Management Partners, Inc.'s reputation depends on timely reporting of quality data. The standard turnaround time for samples from engineering and consulting firms is project specific and is indicated in the Quality Assurance Project Plan or contractual agreements. Standard turnaround that is not project specific is seven to ten business days from time of sample receipt, unless restricted by sample holding time. The time of sample receipt is verified upon meeting sample acceptance policy and is documented on the Chain of Custody. Laboratory Project Management staff must approve any special arrangements such as RUSH services, non-routine methods or lower than normal reporting limits. The basis for data quality depends on client, regulation or reference method performance criteria. Representiveness, accuracy, precision, completeness, sensitivity and comparability are expressions of method performance criteria.

All work is performed in the strictest confidence. New and any contract employees must review corporate confidentiality policy and protect client confidentiality and proprietary rights. The policy review occurs during orientation and ethics training It is the policy of the laboratory to release data only to client-authorized contacts. Personnel authorized in interacting with clients may only review project files and discuss data related to the project Personnel whose duties do not include routine client contact must obtain approval from management before discussing data with regulators or third parties approved by the client. Clients must provide written authorization to discuss any details of their analytical results or project data.

4.4 Laboratory Ethics Program

Laboratory Management Partners, Inc. is committed and dedicated to providing only the highest quality analytical data possible to its clients. Data produced, managed and reported must meet the requirements of its clients and comply with the letter and spirit of the various municipal, state and federal regulations and guidelines. Protocols and procedures are based primarily on EPA guidelines for the analysis of multimedia samples for a broad range of constituents. Laboratory Management Partners, Inc.'s Quality System encompasses the requirements for producing and reporting data of known and documented quality to its clients. It is understood that data is used by clients to make rational, confident, cost-effective decisions regarding assessment and resolution of their environmental compliance requirements and other management activities.

It is the policy of Laboratory Management Partners, Inc. to incorporate the highest standard of quality into all analytical programs by adhering to the following practices:

A. Laboratory Management Partners, Inc. must only offer environmental analyses for which it can consistently demonstrate compliance with high quality, traceable and scientifically defensible performance standards,

B Laboratory Management Partners, Inc.'s staff are committed to the practice of complete honesty in the production and reporting of data;

C Laboratory Management Partners, Inc 's staff who are aware of misrepresentation of facts regarding analytical data, or the unauthorized manipulation of data, are required to immediately inform the Quality Assurance Officer, Technical Director and/or President.

D. Laboratory Management Partners, Inc. operates under an *Open Door Policy* that enables every staff member to have free access to the corporate officers. This *Open Door Policy* is intended to foster two-way communication and encourage each staff member to carefully consider their duty and responsibility to report inappropriate data production and reporting practices to the corporate leadership. It is clearly understood that such information brought forth shall be treated confidentially, if so requested by the reporting staff member.

The *Ethical Conduct and Data Integrity Agreement* is signed at the time of hire. Furthermore, each staff member is required to review and sign this agreement each January. Such signature is a condition of continued employment at Laboratory Management Partners, Inc. Failure to comply with these requirements results in immediate discharge from Laboratory Management Partners, Inc. employment. This *Ethical Conduct and Data Integrity* Agreement supports the company's *Employment Agreement policy* and *Laboratory Ethics Program.*

5 Personnel

5.1 Laboratory Management Responsibilities

Management is responsible for communicating the requirements of the Quality System, client specifications and regulatory needs to all personnel Management is responsible for reviewing the client/project needs and providing the manpower and resources needed to complete the work. All personnel are responsible for complying with all QA/QC requirements that pertain to their function. Job descriptions detail the responsibilities of each position.

The Quality Assurance Office maintains job descriptions for all positions in the laboratory defining the level of qualifications, training, experience and laboratory skills. During initial training, management provides documented operations procedures, observes personnel performance, and evaluates personnel proficiency. Management documents laboratory staff's proficiency initially and on a continuing basis through use of method performance procedures and proficiency evaluation samples. Corrective Action for noncompliancy results in re-evaluation, retraining and re-testing until proficiency is established. Management requires successful proficiency demonstration before allowing independent production testing.

Management is responsible for verification of proper sample management and all aspects of data reporting. The communication of the operating practices of the laboratory is through the document control and acknowledgement process.

Management includes:

Technical Director:

Responsible for all technical operational activities of the laboratory. Plans and implements laboratory strategies and makes recommendations to the President. Gives final approval for all data and ensures the data is applicable to the clients needs. Provides technical service to all laboratory personnel and supports sales staff through technical expertise. Mr. Kauffman ensures that all analytical practices are technically sound and meet all Quality guidelines. Mr. Kauffman reports directly to the CEO. As Technical Director, Mr. Kauffman's responsibilities include the following areas:

a) Development, implementation and updating of laboratory standard operating procedures (SOPs) which includes establishment of Method QA/QC criteria

b) Ensuring that all technical aspects of methods are performed according to standard operating procedures

c) Personnel training and documentation to ensure that each analyst is capable of meeting the technical requirements as set forth in the SOPs

d) Implementation of project management plans as defined by the CEO. This includes direct client technical support when necessary

e) System Administrator for the HP NT ChemServer system

f) Technical Oversight for the NT Oracle database system

g) Assisting in the review of data generated by all Analytical Sections

Assistant Technical Director

Provides assistance to the Technical Director and carries out Technical Director's instructions. Provides technical assistance to all areas of the laboratory and performs data review. Mr. Ferguson is responsible for assisting the Technical Director in all aspects of Laboratory technical operations. As Assistant Technical Director, Mr. Ferguson's responsibilities include the following areas:

b) As needed, provide technical training to all laboratory personnel.

c) Assists Technical Director as required.

d) Investigates and responds to all Technical Corrective Actions.

e) Assists laboratory personnel when workload requires additional support.

f) Answers technical questions from internal staff as well as clients.

Laboratory Managers

Responsible for all operational and administrative business functions of the laboratories. Actively enforces company policy as required and informs the President of key issues. Authority is provided by the president to delegate all administrative duties as needed. Provides expertise to supervisory staff regarding economic, personnel, and operational decisions. Maintains a high level of contact with all staff to promote company philosophy.

Quality Assurance Officer:

Responsible for interacting and communicating certification requirements, implementing the quality manual and reporting to management the status of the Quality System. The QA Officer serves as the focal point for QA/QC and is responsible for the oversight and/or review of quality control data. The QA Officer has functions independent from laboratory operations for which he has quality assurance oversight and is able to evaluate data objectively and perform assessments without outside (e.g , managerial) influence He has an in-depth knowledge of analytical methodology and his main directive is to ensure that the laboratory's Quality Assurance System is generated, implemented, and maintained. Dr. Medina reports directly to the CEO. As Quality Assurance officer, Dr. Medina's areas of responsibility include:

a) Assist CEO/President with design of Quality Systems and make recommendations that will improve the effectiveness of the Quality Systems for LMP and its holdings.

b) Performance of internal Quality System audits. These include submitting Blind (System Audit) PE samples as well as the auditing of support documentation, personnel and facilities. Audit findings and conclusions are relayed to CEO/President for review and subsequent action.

c) Coordination of audits by clients and regulatory agencies and maintenance of applicable certifications and validations

d) Acts as liaison between the laboratory and certifying agencies

e) Review and approval of all Quality Criteria in Standard Operating Procedures

f) Assists the Environmental Lab Manager in providing an objective review of final report packages.

h) Review and verification of all Non-Compliance, Corrective Action and Sample Casualty Reports generated during the execution of analytical projects

i) Maintaining, updating, and archiving QC data and supporting documents

j) Implementation and maintenance of the laboratory's Waste Management, Chemical Hygiene and Safety Program

k) Responsible for maintenance of Quality Manual, SOP's and forms

l) Oversees QA Supervisor.

Quality Assurance Supervisor:

The QA Supervisor is responsible for quality system records and assists in data review and internal audits of the technical areas of the laboratory. The QA Supervisor assists in technical data review, internal audits and maintains oversight of Corrective Action activities, assists QA Officer in implementation and maintenance of all Quality System activities. The QA Supervisor is responsible for the maintenance of Quality System documents such as completed batch worksheets. The QA Supervisor reports to the QA Officer.

Environmental Project Manager:

As, Project Manager, Mr. Thomas' responsibilities include the following:

a) Delivery of reports to the client and ensuring that client reporting requirements are met. Processing of final reports including a completeness review that ensures all pertinent information from the Chain-of-Custody is included.

b) Interface with clients concerning price quotations and review of project invoices to ensure that the client is properly invoiced. Daily communication with sales staff to monitor quotations and prospective project status.

d) Coordinating field activities for clients that require sampling and/or courier services

e) Assists the Laboratory managers in Project implementation, oversight and delivery.

Laboratory Supervisors:

Responsible for data review, personnel training and implementation of the documented procedures defined by the laboratory. Laboratory supervisors provide input on technical and personnel needs to the Technical Director.

As Organic Laboratory Supervisor, Ms. Savage is responsible for the supervision and execution of daily activities. She reports directly to the Technical Director. Ms. Savage's areas of responsibilities include:

a) Supervising all activities within her section from instrument maintenance to execution of analytical projects and review of analytical data

b) Ensuring implementation of the QA/QC plan within this section

c) Providing technical support for analysts when questions or problems arise concerning sample preparation and/or analysis.

d) Reporting all non-conformance issues to the QA Officer for initiation of corrective action

e) Training of new analysts.

As Supervisor of the Inorganic Lab, Mr. Dunlap is responsible for analysis and supervision of all sample analysis for wet chemistry methods. Mr. Dunlap is responsible for the day-to-day execution of the Inorganic Lab procedures:

a) Supervising all daily activities within his section

b) Supervising the implementation of the QA/QC plan within this section

c) Providing technical support for analysts when questions or problems arise concerning sample preparations and/or analyses

d) Reporting all non-conformance issues to the QA Officer for initiation of corrective action

e) Training of new analysts

As Bioassay Laboratory Supervisor, Ms. Cook is responsible for the supervision and execution of daily activities. She reports directly to the Executive Vice President. Ms. Cook's areas of responsibility include:

a) Supervises all daily activities from instrument maintenance to execution of analytical projects and review of analytical data

b) Supervises the implementation of the QA/QC plan

c) Provides technical support for analysts when questions or problems arise concerning sample preparation and/or analysis

d) Report all non-conformance issues to the QA Officer for initiation of corrective action

e) Training of new analysts

As Organic Prep Laboratory Supervisor, Ms. Wallace is responsible for all organic extractions and supervision of all sample extraction personnel in support of the organic analytical sections. Ms. Wallace is responsible for the day-to-day organic prep lab activities with direct over-site by the Environmental Laboratory Manager She reports directly to the Technical Director. Ms. Wallace's areas of responsibility include:

a) Supervises all daily activities within the organic prep lab.

b) Provides technical support for questions or problems arising from sample preparations

c) Reporting of all non-conformances to the QA Officer for initiation of corrective action and follow-up

d) Training of new analysts

Ensures the accuracy and integrity of all test results. Oversees assigned analysts and laboratory personnel in the performance of analytical testing, ensuring quality control policies and that expected turnaround times are met.

Environmental Client Services Coordinator:

Responsible for client services, sample management and directing information between the client and supervisors of the laboratory. Coordinates sample reception, log-in and logistical activities of the laboratory. Acts as primary customer point of contact. Ensures that testing and other services are performed in a timely manner by providing the Laboratory Supervisor and Technical Director input and recommendations concerning client issues. Supervises clerical support staff and assists sales staff in effective customer service. Responsibilities include oversight and direction of sample receiving, project implementation and handling client phone calls. The Environmental Client services staff report to the Environmental Laboratory Director

Information Systems Manager:

Responsible for the proper operation of all software applications including maintenance of existing applications, installation and maintenance of computer hardware, daily maintenance and administration of the network, workstations, and all servers. Communicates daily with all laboratory staff to ensure the individual components of all computer systems are functioning properly and efficiently.

As a LIMS administrator, Ms. Bradberry is responsible for the day-to-day operation, maintenance and training for the Omega LIMS. Ms. Bradberry's responsibilities include:

a) Instrument data capture

b) Client database maintenance

c) Generation of management reports

d) Generation of Electronic Data Deliverables (EDD)

e) Creation and design of final report formats including client specific custom formats

f) Translating of project specifications (e g. target analyte list, detection limits, QC Package) into the LIMS to ensure samples are logged properly and that all project Data Quality Objectives are addressed

g) Review of projects after login to ensure that project criteria have been input and initiated. Create project specific checklists that track project progress and helps to ensure that project requirements will be met on time.

h) Performs QA Forms/Package data validation prior to the release of data to final reporting

i) Performs project level review of final report to ensure client requests have been met

5.2 Laboratory Staff Requirements

Recruitment is the responsibility of the Technical Director with input from other personnel as required. The Technical Director defines the minimal levels of qualifications, experience and skills for each position to ensure personnel have adequate skills and competence for the job function.

Job descriptions detail the necessary requirements for each job and includes position title, minimum educational requirements, skills, responsibilities and reporting relationships and any supervisory responsibility

Orientation training for all new employees includes review of the laboratory business practices, employment specifications, Ethics Policy, Quality Manual, Safety Manual, and all SOPs required for the job function.

Managers ensure the training of new employees and review the continuing training for current employees. Training may include on-site and off-site programs presented by staff members, regulators, contractors, equipment manufacturers, and institutions of higher learning

Training of new personnel to any job assignment takes place on-site. An individual performs the technical procedure without supervision after documentation of acceptable proficiency. The training file contains the information on the current training status.

Off-site training takes place on an as-needed basis. Recommendations and suggestions about educational programs come from all levels of staff. The Technical Director approves off-site training. It is the employee's responsibility to present a copy of any certificates or attendance information. The information is added to the individual's training record.

5.3 Training

The Quality Manual and supporting documents are available to all employees. Cross training, supervisory training and other related training takes place on a scheduled and as needed basis. Training ensures the communication and understanding of all personnel in the laboratory-documented procedures and practices.

On-the-job training includes demonstration of skills during job performance, annual demonstration of proficiency, and annual review of SOPs. Safety and health training takes place on an annual basis with careful introduction to new principles. Personnel have access to the Safety Manual and Material Safety Data Sheets. On-site training includes side-by-side hands-on training, formal classroom type instruction on the SOP or a meeting to discuss procedural changes or to address questions related to laboratory operations

Training is an on-going opportunity to evaluate the laboratory operations. In all cases, an Initial Training Form (ITF) is signed and dated by the trainer and the trainee(s) and documents training. For Test Method SOPs, the laboratory completes a Demonstration of Capability (DOC) Form. The QA Officer and the Technical Director, upon successful completion, sign the DOC Form. This form references the documentation of performance of the analyst.

Performing four replicate samples and demonstrating acceptable precision and accuracy must document initial performance of any test method that allows spiking. Acceptable performance is defined as within precision and accuracy of the reference method or laboratory-generated limits when reference method limits are not available Ongoing performance demonstration must be conducted once per year by performing any one of the following: acceptable performance of a blind sample; another demonstration of capability, successful analysis of a blind performance sample on a similar test method using the same technology; analysis of at least 4 consecutive lab control samples with acceptable levels of precision and accuracy; or if one of the above cannot be performed, the analysis of authentic samples that have been an analyzed by another trained analyst with statistically indistinguishable results.

The DOC is completed prior to using any test method, and at any time there is a substantial change in instrument type, personnel or test method. In general, this demonstration does not test the performance of the method in real world samples, but in the applicable and available clean matrix (a sample of a matrix in which no target analytes or interferences are present at concentrations that impact the results of a specific test method). However, before any results are reported using a method, actual sample spike results are used to meet this standard where

available. In addition, for analytes, which do not lend themselves to spiking, e.g., TS, the demonstration of capability is performed using replicates of real world samples.

The DOC certification statement shall be used to document the completion of each demonstration of capability. A copy of the certification statement shall be retained in the personnel records of each employee and SOP file This certification form must be completed each time a demonstration of capability study is completed.

5.4 Training Records

The QA Supervisor is responsible for maintaining training records. Training forms, certificates and other records of training are located in the employee's training file.

The QA Officer or designee notifies appropriate personnel when a revision is complete for the controlled version of any document. Laboratory staff must acknowledge receipt of the change and agree to implement the change as of the effective date. The training records include the documented acknowledgements. The laboratory supervisor of the area determines when a change is significant to require training

Resumes and job descriptions are included in the training record files. The Technical Director and Laboratory Supervisors review the job descriptions, resumes and training records at least once every two years to ensure up-to-date information on the job descriptions and resumes. The Technical Director and Laboratory Supervisor and the individual update the resume on an as needed basis. Technical information in the training records is audited for completeness as part of the internal audit process of the laboratory.

6 Quality System

6.1 Establishment

The Quality Mission Statement presents the policy and objectives for Laboratory Management Partners, Inc. The Quality Manual provides the framework for the processes and operations to accomplish the Quality Mission. The Quality Manual and controlled supporting documents detail the management-authorized operations for achieving the objectives of the company. It is the policy of Laboratory Management Partners, Inc. that all activities occurring within the laboratory, whether analytical or administrative, be validated against the quality objectives.

The laboratory operates a Quality System approach to management in order to produce data of known quality that meet regulatory requirements and guidelines. Laboratory Management Partners, Inc. is a full service laboratory designed to provide its clients with accurate, precise and defensible data. Laboratory Management Partners, Inc. employs chemists and analysts with the highest training, ethics and caliber in the field of analytical chemistry. Combining an experienced staff with state of the art instrumentation, documented procedures and enhanced automation ensures data of known and documented quality.

6.2 Quality Manual

The QA Officer is responsible for the management and distribution of the Quality Manual. The Quality Manual is reviewed by management minimally once per year. The QA Officer is responsible for monitoring and implementing the Quality Assurance and Quality Control procedures in all sections of operation. Implementation of major changes in the Quality System occurs after revision to the Quality Manual, other supporting documents and authorization by management. Based on input from laboratory operations, a Document Revision Form shall be submitted to indicate revision to the QM based on current operations. The Quality Manual is also reviewed to ensure that laboratory practices meet applicable city, state, federal, and national independent accreditation regulators.

The signatures found on the authorization page in Section 1.1 of this manual signify management review and approval of the Quality Manual. The Signature section is kept current and reflects any organizational changes affecting the authorizing positions. Updates to this manual occur at any time throughout the year. The revision number and date of promulgation indicate the most current version of the QM, which has undergone management review and approval.

Document control procedures apply to the Quality Manual. Distributions of controlled copies of the manual are made to laboratory personnel, authorized clients and certifying agencies. Persons or organizations outside of Laboratory Management Partners, Inc. may receive uncontrolled copies. These copies are distinctly marked as "Uncontrolled Copies". A distribution list is maintained for all controlled copies of the Quality Manual. All parties listed on the controlled distribution list receive document updates. Copies marked as uncontrolled copies are not subject to updates.

6.3 Internal and External Audits

Internal and external audits review and examine the operations performed in the laboratory. Internal audits are self-reviews and external audits are reviews by external organizations to evaluate the ability of the laboratory to meet regulatory or project requirements. These audits are performed to provide an objective evaluation of compliance with established requirements, methods and procedures.

The QA Officer schedules internal audits of each operational area. The internal audit is designed to ensure that the laboratory and its personnel are in compliance with the Quality Manual, laboratory Standard Operating Procedures, and regulatory agency requirements by reviewing the analytical and administrative processes and implementation of the documented Quality System. The audit may include operations from sample receipt to sample disposal.

The purpose of the internal system audit is:

Verification that adequate written instructions are available for use;

Analytical practices performed in the laboratory are consistent with SOP's;

The quality control practices are applied correctly during production;

Corrective actions are applied as necessary;

Deviations from approved protocols are occurring only with proper authorization and documentation;

Reported data is correct and accurate for reporting;

SOP's, quality records, analytical records are maintained properly; and

Personnel training and records are satisfactory and current

Before a scheduled audit, the assigned auditor reviews checklists or the SOPs specific to the area The checklist may be from an external source or prepared by the auditor. The checklist includes all references to the documented Quality System or referenced requirements document. Audit findings are presented to the Chief Executive Officer as a Management Report of Internal Audit Summary Report, along with proposed Corrective Action and follow-up audit and subsequent reports as required. Once any corrective action and follow-up procedures have been completed, the entire audit package will be filed by the QAO. The Internal Audit Summary Report will be completed following any internal audit. The Internal Audit Summary Report records any deficiencies or discrepancies found during the audit.

Technical personnel are responsible for the inspection and monitoring of in-process and final data The Quality Assurance Office and personnel independent of those having direct responsibility for the work may perform the audit of the Quality System and processes.

Representatives sent by clients and government or accrediting agencies often perform external audits These audits are most often announced inspections. The QA Officer accompanies the external audit team through the laboratory. All laboratory personnel and applicable data will be made available to the assessor and the audit team during the audit. The auditors receive a brief overview of company objectives, activities, and facilities. Interviews with essential supervisory staff and technical staff are arranged, along with retrieval of any documentation pertinent to the objective of the audit. Auditors usually provide a report on their findings shortly after the audit. The QA Officer receives the audit report and completes Corrective Action Report Forms in response to any cited deficiencies. During the on-site audit, the audit team may come into possession of information claimed as Confidential Business Information (CBI). The EPA regulations for handling confidential business information are detailed in *Title 40, Code of Federal Regulations, Part 2, Subpart B.* Subpart B defines a business confidentiality claim as "a claim or allegation that business information is entitled to confidential treatment for reasons of business confidentiality or a request for a determination that such information is entitled to such treatment."

Where the findings of an audit cast doubt on the correctness or validity of the laboratory's calibrations or test results, the laboratory shall take immediate corrective action and shall immediately notify, in writing, any client whose data was affected.

6.4 Audit Review

Management reviews internal and external audit reports to evaluate system effectiveness. Tracking of the audit findings occurs through the corrective action process. The QAO and Supervisor shall agree upon corrective action for noted deficiencies, along with the timetable for implementation. The QA Supervisor tracks the time line and informs the QA Officer and Technical Director of any outstanding audit findings corrective actions.

6.5 Laboratory Performance Testing Program

Laboratory Management Partners, Inc. participates in various inter-laboratory performance testing programs required by clients and certifying agencies. The performance audits provide information on laboratory performance from analytical data generated. Performance evaluation or proficiency testing samples received by the laboratory are handled following routine laboratory procedures. Laboratory personnel analyze these samples using techniques utilized as with real life samples.

Proficiency evaluation samples are unpacked and undergo the sample receipt policy procedures. Reporting requirements and deviations to routine practices are noted as required for any project. Proficiency samples are analyzed minimally twice per year per matrix and analyte to meet the NELAC and other accrediting authority requirements

Analysts demonstrate proficiency by analyzing an external proficiency test sample or an internally prepared blind test sample. The results of performance audits serve several purposes. Aside from monitoring lab proficiency, the QA Officer uses performance audits for evaluating analyst ongoing proficiency, laboratory performance in a specified area to facilitate laboratory improvement efforts and to provide corrective action to an accrediting agency on a previous unacceptable performance audit.

6.6 Corrective Actions

The Corrective Action (CA) Program at Laboratory Management Partners, Inc. utilizes various reporting formats conducive to specified laboratory operations to document the investigative and remediation processes inherent to the corrective action program. The mechanism for recording, reviewing and acting upon all quality issues is self-evident to the reporting formats utilized at the laboratory. This process ensures continuous self-improvement of company performance by an active corrective action program to prevent the recurrence of quality issues and improve the Quality System.

Laboratory personnel document any deviations or departures from the documented quality manual, standard operation procedures, method or client specifications or recommended improvements to the Quality System on the various corrective action report forms. Management and supervisory staff review all corrective action reports and approves the corrective action recommendation for implementation. The completed CA date for implementation is specified in each corrective action report. The QA Officer and QA Supervisor monitor the corrective action process and reviews the implementation to evaluate the effectiveness of the corrective action implemented. The Closure CA date is assigned after correction is deemed effective.

Corrective Action Reports are tracked for closure date and category. On a timely basis, reports to management include the listing of open Corrective Action Reports. The QA Supervisor records the forms and monitors their completeness. The QA Officer and Technical Director verify actions are complete and acceptable.

6.7 Managerial Review

Management review occurs on a periodic basis as part of the strategic planning process. Documentation of the management report is the responsibility of the Quality Assurance team indicating corrective action procedures derived from the managerial review. The focus of the quality management review identifies the types of corrective action, closure status of correction action reports, audit progress, assessments by external bodies, the results of inter-laboratory comparisons or proficiency tests, any changes in volume and type of work undertaken, feedback from clients and other quality assurance actions. Meetings include discussion and progress on Quality System initiatives since the last meeting. Minimally, on an annual basis, the QA team generates a Management Review Summary Report indicating all corrective action procedures and remediation actions undertaken for the major Quality System components.

6.7.1 Reports to Management

In support of Laboratory Quality System Management Review process reports are generated reassessing the effectiveness and suitability of the Quality System implemented. The review encompasses a composite of the various quality objectives implemented at the laboratory. These quality assessments will include reports from managerial and supervisory personnel.

The various types of feedback mentioned above determine the form and function of the managerial review of Quality Systems being generated A report may be generated for any one department indicating a review of quality systems. All reviews and assessments will address the CEO, the Technical Director, the QAO, other administrators, managers, supervisors, and/or a combination of the above. Support information for the reports may take the form of memos, e-mails, letters, and/or specialty reports such as internal audits, performance testing, corrective action, sample casualty reports, noncompliance-corrective action, or the like.

Each report identifies:

Quality System Report to Management Identification Number
Laboratory Section
Identification of the Quality System affected
Reason for Quality Systems Assessment
Outcome of Investigation/Assessment
Persons involved in the report
Corrective Action or Assessment Directive
Areas affected by Managerial Report
Special notes or conditions
Notification to Clients affected if required

6.8 Essential Quality Control Procedures

To ensure the generation of quality data under the system of analytical quality objectives, the laboratory establishes and maintains essential quality control procedures in the analytical scheme of data generation. The following sections list the essential quality control elements and requirements for routine assessment of analytical data as required by reference methods or project requirements. These sections provide standard criteria for defining, implementing, evaluating and reporting of these elements to be used by the laboratory. However, these requirements may be superceded by specific method and/or project requirements. Further, Laboratory SOPs specify the quality control requirements for assuring precision, accuracy, representiveness, comparability, completion and sensitivity of each test method.

The following essential quality control procedures apply according to the requirements of the test method.

6.8.1 Batch

The basic unit for application of laboratory quality control is the batch. Samples shall be prepared, analyzed, and reported in batches and be traceable to their respective batches. Each batch shall be uniquely identified within the laboratory. Field QC samples (i.e., trip blanks, rinsates, etc.) shall not knowingly be used for batch QC purposes. Samples are grouped together by method and similar matrix A batch is defined as samples prepared together using the same process and reagents and prepared over a limited continuous time period. The following batch sizes apply:

Wastewater Methods Batch Size: 20 field sample maximum or 12 hour shift

SW-846 Methods Batch Size: 20 field sample maximum or 12 hour shift

Each batch is assigned a unique batch number that will allow the analysis of any sample to be traced back to the original preparation. In addition, QC samples (e.g. Blank, Laboratory Control, MS/MSD) are assigned using the batch number in order to link the environmental samples with the appropriate QC samples. QC samples are assigned based on specific method requirements and is detailed within the analytical method SOP.

Analytical Batch (Sequence):

The analytical batch sequence or instrument run sequence is defined as samples that are analyzed together within the same time period or in continuous time periods on one instrument. Analytical sequences are bracketed by the appropriate continuing calibration verification standards and other QC samples as defined by the analytical method. Each sequence contains the requisite number and type of calibration standards, QC samples, and regular analytical samples as defined by the reference method. These requirements are defined in the method SOPs and summarized in part in the following sections.

6.8.2 Laboratory Blank (LB)

Purpose:

Laboratory blanks are analyzed to assess background interference or contamination that exists in the analytical system that might lead to the reporting of elevated concentration levels or false positive data. The LB is carried through the complete sample preparation, concentration, cleanup, and determinative procedures where applicable.

Frequency:

At least one laboratory blank is required for each preparation batch or analytical batch where equivalent. Refer to method SOPs for laboratory blank requirements.

Composition:

A laboratory blank is an analyte and interference-free matrix to which all reagents are added in the same volumes or proportions as used in sample analysis.

a) Organics - Analyte-Free Reagent Water / Sodium Sulfate

b) Metals - Analyte-Free Reagent Water / Sea sand

c) Inorganics - Analyte-Free Reagent Water / Sea Sand

Evaluation Criteria and Corrective Action:

While the goal is to have no detectable analytes, each laboratory blank must be critically evaluated as to the nature of the contamination or interference and the effect on the analysis of each sample within the batch. The source of any contamination shall be investigated and measures taken to minimize or eliminate the problem. The LB is considered acceptable if it meets one or more of the following requirements dictated by the method or project:

Less than the MQL

Less than the MDL – This criterion is used when data is specifically evaluated against the MDL (e.g Risk Based Action Levels).

Less than the RL – This criterion is used when RLs are specified in a project specific statement of work (SOW)..

- Less than 10% of the sample result for the same analyte.

If the laboratory blank results do not meet the acceptance criteria above, then the laboratory shall take corrective action to locate and reduce the source of the contamination. If feasible when the holding time is not exceeded, the lab shall re-extract and reanalyze any samples associated with the contaminated laboratory blank. Any samples associated with a contaminated laboratory blank shall be reprocessed for analysis or the results reported with the appropriate data qualifying code (i.e., "B" flag).

If an analyte is found only in the laboratory blank, but not in any batch samples, no further corrective action may be necessary. Steps shall be taken to find/reduce/eliminate the source of this contamination in the laboratory blank. A case narrative shall be processed to indicate the situation.

Subtraction of laboratory blank results from associated samples is not permitted unless expressly allowed in the reference method. Laboratory blanks and/or solvent blanks may also be used to check for contamination by carryover from a highly -concentrated sample into subsequent samples.

Reporting Criteria:

Analytes are evaluated to the MDL for all samples and laboratory blanks.

Results for an analyte above the MDL, but below the MQL, may be reported as estimated values (i.e. "J" flag).

Results for an analyte identified in a sample and the laboratory blank and the concentration is less than 10 times the laboratory blank, the result is reported with a "B" flag. This indicates that the concentration of the analyte in the sample may be due to an interferent introduced in the laboratory.

Results for an analyte identified in a sample above the MDL but below the MQL that is also found in the associated laboratory blank will be reported as "not detected" at the MQL. Results are not reported flagged "JB" Results for analytes considered common laboratory contaminants are not reported below the MQL for samples. No "J" flag is reported for the following analytes: Methylene Chloride, Bis-2(ethylhexyl)phthalate, Zinc, Aluminum, Calcium or Sodium.

There are instances where no contamination was present in the associated blank, but qualification of the sample(s) data is deemed necessary. Contamination introduced through dilution water is one example. Instances of this type contamination can be detected when contaminants are found in the diluted sample result, but are absent in the undiluted sample result. An explanation is provided in the case narrative

6.8.3 Laboratory Control Sample (LCS)

Purpose:

The LCS is used to evaluate the performance of the entire analytical system, including all preparation and analytical steps. Recoveries of the LCS are compared to established recovery acceptance criteria and, if found to be outside of these criteria, indicates that the analytical system is "out of control". LCSs are not performed for methods for which spiking solutions are not typically available.

Frequency:

A LCS is required for each preparation batch or analytical batch where equivalent. A laboratory control sample duplicate (LCSD) is required when a matrix spike/matrix spike duplicate cannot be performed. Whenever an LCS/LCSD pair is analyzed with a MS/MSD pair, the first LCS result is used when the MS/MSD pair criteria are acceptable. When the MS/MSD pair criteria fail and the LCS/LCSD pair pass comparable criteria, the LCS/LCSD data may be used to demonstrate matrix interference and the sample data may be reported with appropriate narratives.

Composition:

The LCS is similar in composition to the laboratory blank. An analyte and interference-free matrix to which all reagents are added in the same volumes or proportions as used in sample preparation. The LCS is spiked with all single-component target analytes before it is carried through the preparation, cleanup, and determinative procedures.

Organics Analyte-Free Reagent Water / Sodium Sulfate

Metals Analyte-Free Reagent Water

Inorganics Analyte-Free Reagent Water

When multi-component analytes are the only targets (e.g method 8082 or project specific pesticide for Chlordane or Toxaphene) the LCS must be spiked with at least one of the multi-component analytes. Method 8082 for PCBs requires that the LCS contain at least one PCB (e.g., 1242/1260 mixture). The LCS contains all target analytes of interest that are reported under a particular method. The LCS is generally performed near the middle of the procedure's analytical range. The recommended concentration of the LCS is detailed within the analytical SOP.

Evaluation Criteria and Corrective Action:

The results of the individual batch LCS are calculated in percent recovery. Where a LCSD is performed, the relative percent difference (RPD) is also evaluated.

The LCS is evaluated by comparing the percent recovery/RPD for all of the target analytes to the quality objectives as determined by Method Performance Criteria found in this manual. Control limits are established for each analyte/analytical method/prep method performed by LMP, Inc. Refer to method performance procedures for details on how control limits are established. For an LCS/LCSD to be acceptable:

Recoveries for all target analytes must be within acceptance criteria.

Recoveries are high and no target analytes are identified in associated samples (requires reference in Level II, III, IV case narrative).

RPDs for all analytes must be within acceptance criteria.

RPDs for an analyte is high, however, recoveries are within acceptance criteria (requires reference in Level II, III, IV case narrative).

Note. When samples are not subjected to a separate preparatory procedure (i.e., low-level GC or GC/MS analyses) the CCV may be used as the LCS, provided the CCV acceptance limits are used for evaluation

The effect of any LCS QC failure on the associated samples must be evaluated. Regardless of this assessment, steps shall be taken to find the source of the problem and correct it.

Typically, the LCS is reanalyzed for the failed analytes only. If the second analysis fails, then the entire batch (QC samples and field samples) would be re-prepared and reanalyzed for the failed analytes only. If sufficient sample is not available for re-preparation and reanalysis or if the corrective action is ineffective, the sample results reported within that batch shall be flagged accordingly, and a discussion of the impact included within the case narrative.

The case narrative shall discuss the corrective action taken and any other information that will assist in the evaluation of the impact of the QC failure on the data quality objectives.

Method SOPs will contain additional guidance for compounds that are considered marginal, problem or non-standard analytes (e.g. benzidine method 8270C, tetryl method 8330, Antimony method 6010B). The issue of Sporadic Marginal Failures (SMF – USCOE projects only) is addressed in the Technical Guidance Memo Generating Control Limits for Precision & Accuracy.

6.8.4 Matrix Spike Sample (MS)

Purpose:

The Matrix Spike (MS) and Matrix Spike Duplicate (MSD) are QC samples that indicate the effect of the sample matrix on the precision and accuracy of the results generated using the selected method. The information from these QC samples is project/sample/matrix specific and would not normally be used to determine the validity of the entire batch. MSs are not performed for methods for which spiking solutions are not available.

Frequency:

A MS/MSD is required for most preparation batches or analytical batch where equivalent A MS and MS Duplicate (MSD) are used to assess precision and accuracy.

Composition:

The MS/MSD are performed on a field sample contained in a batch. The MS/MSD contains all target analytes that are reported under a particular method. The MS is generally performed at the same concentration as the LCS and is spiked with all single-component target analytes before it is carried through the preparation, cleanup, and determinative procedures.

When multi-component analytes are the only targets (e.g. method 8082 or project specific pesticide for chlordane) the MS/MSD must be spiked with at least one of the multi-component analytes. Method 8082 for PCBs requires that the MS/MSD contain at least one PCB (e.g., 1242/1260 mixture).

The MS/MSD contains all target analytes that are reported under a particular method and is generally performed at the mid-range concentration of the initial calibration curve. The concentration of the MS/MSD is detailed within the analytical SOP. In situations where enough sample has not been provided to perform a MS/MSD, a LCS/LCSD is substituted.

Evaluation Criteria and Corrective Action:

The results of the individual batch MS are calculated in percent recovery. Where a MSD is performed, the relative percent difference (RPD) is also evaluated.

The MS/MSD is evaluated by comparing the percent recovery/RPD for all of the target analytes to the recovery measurement quality objectives as determined according to Method Performance Criteria of this manual. Control limits are established for each analyte/analytical method/prep method performed by LMP, Inc. Refer to method performance procedures for details on how control limits are established For an MS/MSD to be acceptable:

Recoveries for all target analytes should be within acceptance criteria

Recoveries are high and no target analytes are identified in associated samples (requires reference in Level II, III, IV case narrative)

RPDs for all analytes should be within acceptance criteria

RPDs for an analyte is high, however, recoveries are within acceptance criteria (requires reference in Level II, III, IV case narrative)

The LCS/LCSD % recovery and RPD for the failed analyte (MS/MSD) must be within acceptance limits MS/MSD samples indicate the effect of the sample matrix on the precision and accuracy of the results generated using the selected method. The information from these QC samples is sample/matrix specific and is not used to determine the validity of the entire batch.

The effect of any MS/MSD QC failure on the associated samples must be evaluated. If necessary, corrective action is performed with the following order of priority·

Perform specific corrective action as listed in analytical SOP (e.g. post digestion spike or dilution test for metals).

Compare against project-specified MS acceptance range (if available). If the recovery is outside the LCS range but within the project-specified range, this indicates a matrix affect that is within tolerable limits as defined by the project. No corrective action is required.

Analyte recovery that fails in the MS/MSD must be within QC limits in the associated LCS/LCSD. This indicates that the system is in control and that the failure is likely due to the sample matrix interference. An example of recovery failure due to matrix effects is high analyte concentrations in the sample.

Re-extract/Re-analyze – The availability of additional sample and holding times must be taken into account. Samples re-extracted outside of holding time may be useful in some instances to verify high levels of target analytes. Re-extraction should occur only if method specified corrective action (e.g. cleanup, reduced sample size) is expected to significantly reduce or eliminate the matrix interference.

Provide narrative to client explaining the outlier and its effect on the sample data. The narrative is provided regardless of the corrective action taken. Review the laboratory blank for contamination and laboratory control sample recoveries Organics should also review surrogate recoveries for the MB and LCS(s). If these are all within limits, this indicates that the system is in control, that the data provided meets method requirements and that the problem may be due to the sample matrix. Further evaluation may be required to provide the client with information regarding potential matrix problems with their sample(s).

Note: Problems with LCS or MB indicate a system problem, which makes it difficult to point to a sample matrix as the cause of problems with matrix spikes. The entire batch must be reviewed to decide the appropriate corrective action.

Always evaluate the specific affect the recoveries will have on the data. (Matrix spike data will only affect the project samples from which the MS sample was selected.) No further action is required for samples in the batch, which are not directly associated with the project. MS data may be provided for information purposes only for non-project samples.

Some typical review scenarios are provided below:

If MS recoveries are high and no target analytes are identified in the sample, no further action is required Any general trend in this direction should be investigated and corrected.

If MS recoveries are low and no target analytes are identified, this indicates that the ability to detect target analytes at or near the detection limit may have been affected. Review surrogate recoveries to verify matrix affect (project samples only).

If MS recoveries are low and target analytes are identified, this indicates that the result may be biased low. This will directly impact data that is at or near a regulatory limit. Review surrogate recoveries to verify matrix affect. A case narrative is provided for project samples only to discuss the impact on the data.

Some samples may require dilution in order to bring one or more target analytes within the calibration range or to overcome significant interferences with some analytes. This may result in the dilution of the MS responses to the point that the recoveries cannot be measured. Data is reported from the lowest dilution that yields usable data. If the MS recoveries are available from a less-diluted or undiluted aliquot of the sample or sample extract, those recoveries may be used to demonstrate that the MS was within the QC limits, and no further action is required. Either report results from all data (e.g. report dilutions) or provide narrative

Levels of non-target analytes (e.g gasoline, diesel, oil...) may interfere with MS recoveries. If review of the raw data indicates obvious matrix interference, document the interference on the appropriate form (e.g SAM result forms, Inorganic bench sheets, Metals result forms). Provide a narrative to the client.

Review surrogate recoveries for all samples in the same QC Batch (Form 2). Look for trends that would indicate a system problem (e.g. calibration, solution...). Recovery problems across the entire batch would point towards a system problem and would require that all samples in the batch be reextracted/ re-analyzed or report the data with the proper qualifiers and narrative. However, if all samples in a batch are from a single project and the MB/LCS recoveries are within QC limits, this could be indicative of a project-specific interference. The case narrative shall discuss the corrective action taken and any other information that will assist in the evaluation of the impact of the QC failure on the data quality objectives.

Method SOPs will contain additional guidance for compounds that are considered marginal, problem or non-standard analytes (e.g. benzidine method 8270C, tetryl method 8330, Antimony method 6010B).

The issue of Sporadic Marginal Failures (SMF – USCOE projects only) is addressed accordingly.

Reporting Criteria:

MS/MSD recovery, RPD and evaluation criteria are reported for Level II, III and IV data packages. Any QC issues/failures associated with the MS/MSD will be flagged on the recovery reports and detailed in the case narrative as part of the standard reporting process.

In all situations, regardless of the data package level, where MS/MSD issues/failures have an impact on the analytical data, a case narrative is generated to explain the impact on the data The

case narrative shall discuss the corrective action taken and any other information that will assist in the evaluation of the impact of the QC failure on the data quality objectives

6.8.5 Matrix Duplicates (DUP)

Purpose:

Matrix duplicates are defined as replicate aliquots of the same sample taken through the entire analytical procedure. The results from this analysis indicate the precision of the results for the specific sample using the selected method. The matrix duplicate provides a usable measure of precision only when target analytes are found in the sample chosen for duplication.

Frequency:

Duplicates are performed, when required by the method, for every preparation batch.

Composition:

Matrix duplicates are performed on replicate aliquots of actual samples. The composition is usually not known.

Evaluation Criteria and Corrective Action:

The results from matrix duplicates are primarily designed to assess the precision of analytical results in a given matrix and are expressed as relative percent difference (RPD).

The matrix duplicate is evaluated by comparing the RPD for all of the target analytes to the recovery measurement quality objectives as determined according to Method Performance Criteria of this manual. Control limits are established for each applicable analyte/analytical method/prep method performed by LMP, Inc. Refer to static procedures for assessing method performance on how control limits are established. When no RPD are available or exist, the default RPD acceptance criteria are 20%.

Corrective action for RPD outliers is specified in the appropriate analytical SOP. In general, corrective action includes:

- Review calculations to ensure that no error has been made
- Re-analyze the samples to verify the RPD. If re-analysis shows RPD within range, then report from reanalysis

Reporting Criteria:

RPD and evaluation criteria are reported for Level II, III and IV data packages. Any QC issues/failures associated with the duplicate will be flagged on the RPD report and detailed in the case narrative as part of the standard reporting process.

In all situations, regardless of the data package level, where the duplicate issues/failures have an impact on the analytical data, a case narrative is generated to explain the impact on the data. The case narrative shall discuss the corrective action taken and any other information that will assist in the evaluation of the impact of the QC failure on the data quality objectives.

6.9 Additional Essential Quality Control Procedures

Laboratory Management Partners, Inc. utilizes additional data quality control procedures to assess quality control for testing.

Surrogates are used most often in organic chromatography test methods and are chosen to reflect the chemistry of the targeted components of the method. Added prior to sample preparation/extraction, they provide a measure of recovery for every sample matrix. Except where the matrix precludes its use or when not available, surrogate compounds must be added to all samples, standards, and blanks for all appropriate organic test methods. Surrogate compounds are chosen to represent the chemistry of the target analytes in the method. They are often specified by the mandated method and are deliberately chosen for being unlikely to occur naturally. Often this is accomplished by using deuterated analogs of select compounds. The results are compared to the acceptance criteria as published in the mandated test method, until internal criteria has been established. Where there are no established criteria, the laboratory determines internal criteria and documents the method used to establish the limits. Surrogates outside the acceptance criteria must be evaluated for the effect on the individual sample results. The appropriate corrective action is guided by the data quality objectives or other site-specific requirements. Results reported from analyses with surrogate recoveries outside the acceptance criteria shall include appropriate data qualifiers. Other examples of spiking procedures used by the laboratory to provide quality control for testing as required by the reference methods and test protocols are described in the laboratory's technical method SOPs.

6.9.1 Surrogate Standard (Applies to Organics Only)

Purpose:

Surrogates are analyzed to assess the ability of the method to successfully recover these specific non-target analytes from an actual matrix. Surrogates are organic compounds that are similar to the analytes of interest in chemical behavior but are not normally found in environmental samples. Specific surrogates used are identified within the analytical SOPs.

Frequency:

Surrogate compounds are spiked into all field samples and accompanying QC samples requiring GC, GC/MS or HPLC analyses prior to any sample manipulation for every sample batch extracted/analyzed.

Composition:

Surrogates are added to all samples, field and QC, for each batch before they are carried through the preparation, cleanup, and determinative procedures. The amount of surrogate to be added and specific surrogate compounds are detailed in the analytical SOP.

Evaluation Criteria and Corrective Action:

Surrogates are used in much the same way that matrix spikes are used, but cannot replace the function of the MS. The results of the surrogates are evaluated, in conjunction with other QC information, to determine the effect of the matrix on the bias of the individual sample determinations.

The results of the surrogates are calculated in percent recovery. Surrogates are evaluated by comparing the percent recovery to the recovery measurement quality objectives. Control limits are established for each surrogate/analytical method/prep method performed by LMP, Inc.

Surrogates outside the acceptance criteria must be evaluated for the effect indicated for the individual sample results. Corrective action includes the review of calculations, re-analysis of the sample or reextraction/ re-analysis of the sample.

If surrogate recoveries are high and no target analytes are identified, no further action is required.

If the re-analysis or the re-extraction/re-analysis recovery is within QC limits, the results will be reported from the re-analysis and no further corrective action is required.

If the surrogate failure us due to obvious high levels of target/non-target analytes based on review of the chromatogram, no re-analysis or re-extraction/re-analysis is required unless the specified corrective action (e.g. cleanup, reduced sample size) will significantly reduce or eliminate the matrix interference. However, based on action limits, re-analysis at a lower sample size may not be an option. A case narrative is required to detail the effects on the sample.

Reporting Criteria:

By default, surrogate recoveries are reported for all organic methods for all reporting levels.

Surrogate recovery outside acceptance criteria must include the appropriate data qualifier.

A case narrative is required when surrogate recovery outside acceptance criteria is deemed to have an impact on the sample results.

6.9.2 Dilution Test (DT – Applies to Metals Only)

Purpose:

A dilution test (serial dilution) is performed to confirm the absence of chemical (positive or negative) interferences operating on any element to distort the accuracy of the reported value.

Frequency:

It is recommended that whenever a new or unusual sample matrix is encountered, a dilution test be performed on at least one sample per digestion batch. The sample used is generally the sample selected for the matrix spike/matrix spike duplicate. A dilution test is performed as corrective action when any failure is noted for the MS/MSD recoveries. Dilution tests are performed according to the following.

Performed when there is a new or unusual matrix with high level of an analyte(s)

Performed when MS/MSD failure is due to a high level of the analyte present relative to the spike amount

A 1:5 dilution test may be performed for an analyte to evaluate matrix interference if the analyte concentration in the original (undiluted) sample is at least 50 times the MDL

A 1:5 dilution test may be performed for an analyte to evaluate matrix interference if the analyte concentration is minimally a factor of 10 above the instrumental detection limit after dilution

USACE projects require that a dilution test be performed once per batch

Composition:

Dilution tests are performed on the digestates of actual samples.

Evaluation Criteria and Corrective Action:

A matrix effect for a particular analyte is suspected if the RPD between the undiluted and diluted result is greater than 10 percent. If a matrix effect is identified for an analyte, all associated

samples within the batch must be analyzed by the method of standard addition (MSA) for the failing analyte(s).

Reporting Criteria:

Evaluation criteria are reported for Level II, III and IV data packages.

6.9.3 Post Digestion Spike (PDS - Applies to Metals only)

Frequency:

It is recommended that whenever a new or unusual sample matrix is encountered, a post digestion spike (PDS) be performed on at least one sample per digestion batch. The sample used is generally the sample selected for the matrix spike/matrix spike duplicate. A PDS is performed as corrective action when any failure is noted for the MS/MSD recoveries. PDS tests are performed according to the following:

When there is a new or unusual matrix.

When there is a MS/MSD failure.

If the result of the sample is less than 25 times the detection limit.

Composition:

Past digestion spikes are performed on the digestates of actual samples. The digestate is spiked with a known amount of the failing analyte(s).

Evaluation Criteria and Corrective Action:

The recovery must be within 75% to 125% of the expected value. If the recovery fails, all samples in the associated batch must be analyzed by the method of standard additions (MSA) for the failing analyte(s).

Reporting Criteria:

Evaluation criteria are reported for Level II, III and IV data packages.

6.9.4 Method of Standard Additions (MSA - Applies to metals only)

Frequency:

If the result of the Dilution Test or Post Digestion Spike fails evaluation criteria, all associated samples for batch must be analyzed by the method standard addition for the failing analyte(s).

Composition:

Known amounts of standard are added to one or more aliquots of the processed sample solution.

Evaluation Criteria and Corrective Action:

When the method of standard additions is used, standards are added at one or more levels to portions of a prepared sample This technique compensates for enhancement or depression of an analyte signal by a matrix It will not correct for additive interferences, such as contamination, interelement interferences, or baseline shifts. This technique is valid in the linear range when the

interference effect is constant over the range, the added analyte responds the same as the endogenous analyte, and the signal is corrected for additive interferences. The simplest version of this technique is the single addition method.

Reporting Criteria:

Final results are reported from the MSA analysis. The use of MSA is noted in the case narrative for Level II, III and IV data packages.

6.9.5 Method Detection Limits

The laboratory follows the procedure found in 40CFR Part 136 Part B to determine the MDL for each matrix type for all test components for which spiking solutions are available. This determination is performed on an annual basis. Additionally, the laboratory determines the MDL whenever there is a significant change in equipment or substantive revision of the technical protocols for preparation and analysis of samples by this test method. The relative significance and substantiveness of any changes to equipment or protocols that require re-determination of the MDLs shall be decided, and documented in QA files, by the QA Officer in conjunction with technical assistance from the laboratory Technical Director. All processing steps of the analytical method, including any routine preparation steps, are included in the determination of the MDL. The MDLs measured by the laboratory are on file for review in the technical SOP files. All supporting documentation used in the determination of the laboratory MDLs is maintained in the laboratory QA files.

The laboratory determined MDL must be less than the reporting limit (RL). The terms reporting limit, method quantitation limit (MQL) and detection limit (DL) are used interchangeably. The RLs are calculated as three to ten times the laboratory measured MDLs but this relationship varies dependent on dilution of sample aliquots, matrix interferences, moisture adjustments (in solid samples), or client/method-specified requirements. The reporting limits for each method are found on the Method Detection Limit Calculation Form.

Refer to LMP's Determination of MDL/MQL/RL SOP.

6.10 Method Performance

Laboratory Management Partners, Inc. has established and maintains a quality system based on the required elements of EPA methodologies, the National Environmental Laboratory Accreditation Conference (NELAC), the International Standard General Requirements for the Competence of Testing and Calibration Laboratories (ISO/IEC 17025) and the US Army Corps of Engineers Shell for Analytical Chemistry Requirements (EM 200-1-3 Appendix I).

LMP's policy on Method Performance combines analytical assessment indicators that are designed to ensure that quality data is continuously produced during analysis. These data quality indicators are known as precision, accuracy, representativeness, comparability, completeness and sensitivity.

Precision:

Precision refers to the distribution of a set of reported values about the mean, or the closeness of agreement between individual test results obtained under prescribed conditions. Precision reflects the random error and may be affected by systematic error. In order to assess the effect these variables have on the total precision of data, both field and laboratory replicates should be acquired. Laboratory precision is determined on the basis of replicate analysis, usually duplicate or matrix spike duplicate samples. To determine the precision of a given analytical method

without the effect of a matrix, a duplicate laboratory control sample is used. The statistical measure of precision is expressed as Relative Percent Difference (RPD).

Relative Percent Difference (% RPD)

$$\frac{R_1 - R_2}{(R_1 + R_2)/2} * 100 = \% \; RPD$$

where. R_1 = Larger of two observed values
R_2 = Smaller of two observed values

And where applicable, Percent Difference is calculated as:

% Difference (%D)

$$\frac{X - \overline{X}}{\overline{X}} * 100 = \% \; D$$

where: $\overline{X}$ = Average of all values
X = Result of measurement

Accuracy

Accuracy is the measure of the closeness of an observed value to the "true" value (e.g., theoretical or reference value, or population mean). Accuracy is defined as the degree of bias in a measurement system. Accuracy is determined using laboratory control samples and matrix spikes. The statistical measure of accuracy precision is expressed as percent recovery (%R). Accuracy is the measure of the closeness of an observed value to the "true" value.

% Recovery (R)

$$\frac{Xs - Xu}{K} * 100 = \% \; R$$

where: Xs = measured value of the spiked sample
Xu = measured value of the unspiked sample
K = known amount of the spike in the sample

% Recovery (LCS)

$$\frac{MV}{TV} * 100 = \% \; R_{LCS}$$

where: MV = Measured Value
TV = True Value

% Recovery (MS or MSD)

$$\frac{MV - SV}{TV} * 100 = \%\ R_{MS}$$

where: MV = Measured Value in MS or MSD
TV = True Value
SV = Amount found in sample

Representativeness

Representativeness is a parameter that is concerned primarily with the proper design of the sampling program or sub-sampling of a given sample. There is no measurement other then precision measurement of the field and laboratory duplicate samples. Representativeness refers to the degree to which sample data accurately and precisely describe the characteristics of a population of samples, parameter variations at a sampling point, or environmental condition. The representativeness criterion is best satisfied in the laboratory by making certain that all sub-samples taken from a given sample are representative of the sample as a whole. Representativeness can be assessed by a review of the precision obtained from the field and laboratory duplicate samples. In this way, they provide both precision and representativeness information. Applicability of representativeness in assessing a contaminant population is improved by using a larger number of samples.

Comparability

Comparability is a qualitative objective of the data, expressing the confidence with which one data set can be compared with another Calculating RPD assesses the measurement of comparability between data sets. Comparability is a qualitative objective of the data, expressing the confidence with which one data set can be compared with another. Sample data should be comparable for similar samples and sample conditions. This goal is achieved through the use of standard techniques to collect representative samples, consistent application of analytical method protocols, and reporting analytical results with appropriate units. Comparability is unknown unless precision and bias are provided.

Completeness

Completeness goals, if defined for individual sampling and analytical protocols, are normally combined to ascertain the expectations of the project as a whole. Completeness is the percentage of measurements that are judged to be usable (i.e., which meet project-specific requirements) compared to the total number of measurements planned.

Overall level of completeness must be addressed as part of the project DQOs. It is important that critical samples are identified and appropriate QC maintained to ensure that valid data is obtained in order to ensure the type, quantity and quality of data necessary to complete the project. The desired level of completeness is dependent on the project-specific DQOs. This information and should be conveyed to this laboratory in the Scope of Work or Sampling and Analysis Plan. The level of completeness must be established and data quality requirements defined in order to meet the intended use of the data (usability).

In the event that more than one data user is requesting the same data, the most stringent data user requirements are applied to ensure the suitability (validity) of the data by all requesting parties. This information is then used to decide the most appropriate analytical strategy to generate the required data. Realistic completeness goals (i.e., 85-90%) are determined based upon the size and complexity of the project. Percent completeness is calculated as

Completeness = $N / P * 100$

where: N = Number of measurements

P = Number of measurements planned

Sensitivity

This term is broadly used to describe prescribed project method detection/quantitation/reporting limits established to meet the project-specific data quality objectives. Several limits have been established to describe project sensitivity requirements such as MDL, SQL, MQL and RL.

Method Detection Limit (MDL)

The MDL is defined as the minimum concentration of an analyte that can be determined with 99 percent confidence that the true value is greater than zero. The laboratory determines MDLs using the procedures and protocols presented in *40 CFR Part 136 Appendix B*.

MDL (See 40CFR Part 136 for details)

$$\left[\sqrt{\frac{\sum_{i=1}^{n} x_i^2 - \left(\sum_{i=1}^{n} x_i \right)^2 / n}{n - 1}} \right] * t_{0.99} = MDL$$

where: MDL = The method detection limit

X = Result of each measurement

n = Number of values

t(n-1,1 = .99) = The students' t value appropriate for a 99% confidence level and a standard deviation estimate with n-1 degrees of freedom. (See Students t Test Table)

Sample Quantitation Limit (SQL)

The SQL is defined as the MDL adjusted for sample-specific action such as dilution or use of non-nominal sample sizes.

Method Quantitation Limit (MQL)

The MQL is the lowest concentration of an analyte that can be reliably achieved within specified limits of precision and accuracy during routine laboratory operating conditions. The MQL is defined as the lowest concentration calibration standard that is analyzed during an initial calibration.

Reporting Limit (RL)

The RL is a threshold value below which LMP, Inc. reports a non-detect (ND). It is based upon project specific concentrations of concern or regulatory action levels but can be no lower than the MDL

Reporting Limit (RL)

Lowest calibration standard

At least 3 times the calculated MDL

The laboratory report indicates this value as the MQL

Statistical Procedures for Assessing Method Performance

In order to assess method performance for accuracy and precision, procedures are in place for the generation of limits. These control limits are based on statistical computations using typically 20-30 data points from historical data accumulated. The use of the average mean, X and standard deviation, is from this population is used for generating the following control limits, Upper Control, Upper Warning, Lower Warning and Lower Control.

Average ($\overline{X}$)

$$\frac{\sum_{i=1}^{n} X_i}{n} = \overline{X}$$

where: $\overline{X}$ = Average of all values
X = Result of each measurement
n = Number of values

Standard Deviation of the sample (S_x) – expressed in sample concentration units

$$\sqrt{\frac{\sum (X - \overline{X})^2}{n - 1}} = S_x$$

where: $\overline{X}$ = Average of all values
X = Result of each measurement
n = Number of values

Control Limits

Upper Control Limit: $\overline{X} + 3 * S_x = UCL$

Lower Control Limit: $\overline{X} - 3 * S_x = LCL$

Warning Limits

Upper Warning Limit: $\overline{X} + 2 * S_x = UWL$

Lower Warning Limit: $\overline{X} - 2 * S_x = LWL$

S_x = Standard Deviation

Normally to assess accuracy the use of % R values are used while for precision the use of RPDs are used to generate the standard deviation as noted above

Before acceptance and use of any method, satisfactory initial demonstration of method performance is required. In all cases, appropriate forms are completed and retained by the

laboratory and made available upon request. All associated supporting data necessary to reproduce the analytical results are retained Initial demonstration of method performance is completed each time there is a significant change in instrument type, personnel or method. Initial demonstration of method performance acceptance criteria is defined in the SOP.

6.10.1 Measurement of Method Performance/Control Limits

Control limits for all analytes are established based on the following hierarchy:

Project Specific Control Limits

In some instances, the LCS, MS/MSD acceptance criteria are detailed within the specific project statement of work (SOW). These limits will supercede the laboratory defined control limits and be used to validate data quality and usability.

Method Specific Control Limits

While certain methods require the use of specific control limits (e.g. ICP method 6010B), LMP, Inc. will use historical data to generate in-house limits that will be used to monitor overall laboratory performance for routine comparison against the method requirements.

In some instances, the LCS, MS/MSD acceptance criteria are detailed within the published method. These limits will supercede the laboratory defined control limits and be used to validate data quality and usability. (i.e. Method 624)

In-House Statistically Generated Limits.

These limits will be statistically calculated from four (4) replicate laboratory control samples (Recent DOC studies may be used.) when enough data points are not available.

Where enough data points exist (e.g. using 20 or more data points), QC limits are generated from laboratory QC samples for each analyte. Refer to Method Performance Procedures for details on generating in-house QC limits.

Interim Default Limits.

Interim default limits may be established in-lieu of in-house generated limits in instances where not enough data points exist. Default limits must be established prior to the analysis of samples. Whenever interim limits are established, there must be scientific validity to the range used The following criteria is used:

Demonstration of Capability (DOC)

Interim control limits may be statistically calculated from the four (4) replicate laboratory control samples used in the DOC study. These limits remain in effect until in-house limits can be generated.

US Army Corps of Engineers Shell Document.

Document EM 200-1-3 February 1, 2001 contains criteria for the evaluation of LCSs for specific lists of target analytes for the following methods:

a) VOCs Method 8021B

b) Pesticides Method 8081A

c) PCBs Method 8082

d) VOCs Method 8260B ✓

e) SVOCs Method 8270C ✓

f) Explosives Method 8330

Department of Defense QSM Document.

DoD Quality Systems Manual – Version 2 Draft. This document contains control limits for specific lists of target analytes for the following methods:

a) VOCs Method 8260B

b) SVOCs Method 8270C

c) Herbicides Method 8151A

d) PAHs Method 8310

e) Explosives Method 8330

f) Pesticides Method 8081A

g) PCBs Method 8082

h) Metals Method 6010B/7470A/7471A

Method/Program Recommendations.

For example, *USPEPA Test Methods for Evaluating Solid Waste Physical/Chemical Methods (SW-846), 3rd Edition, Section 8.5.4, Method 8000B,* recommends the following: "Many methods may not contain recommended acceptance criteria for LCS results. The laboratory should use 70 - 130% as interim acceptance criteria for recoveries of spiked analytes, until in-house LCS limits are developed."

Technical Knowledge

Technical knowledge of the method and predicted performance are the responsibility of the laboratory. In some instances, the method recommendation may not be appropriate for all target analytes. For example, SW-846 recommends a 70-130% evaluation criteria. However, for the acid compounds for method 8270C, recoveries will usually fall in the 30-110% range.

6.11 Automated Data Capture and Reduction

Data Review and Validation

Data reduction procedures, whether performed by an instrument or manually, follow methodologies outlined within the laboratory SOP/analytical method. Automated procedures are verified as required by EPA's guidance on GALP (EPA 2185) where all software is tested with a sample set of data to verify its correct operation via accurate capture, processing, manipulation, transfer, recording, and reporting of data.

All analytical data captured by laboratory instruments are reviewed prior to report release to assure the validity of the reported data. This internal data evaluation process covers the areas of data generation, reduction, and ultimately three levels of documented review. For each level, the review process is documented using an appropriate checklist or worksheet that is signed and dated by the reviewer.

The analyst who generates the analytical data has the prime responsibility for the correctness and completeness of the data. Each step of this review process involves evaluation of data quality based on both the results of the QC data and the professional judgment of those conducting the review. This application of technical knowledge and experience to data evaluation is essential in ensuring that data of known quality is generated consistently. All data generated and reduced follows in-house protocols.

For GC, GC/MS, HPLC instruments data manipulation and reduction, the following procedures are undertaken:

Chromatography data requiring manual integration of peak areas or heights must follow defined protocol. The analyst must perform manual integration when software does not properly integrate or identify the peak. Manual integration must not occur for the purpose of achieving acceptable quality control or calibration. The analyst and reviewer sign and date the hardcopy of all manual integration. The analyst notes the rationale for performing the manual integration on the hardcopy printout and ensures the "TIC" marks from the software represent the integration area used for reporting the results. The analyst must minimize and avoid manual integration whenever possible Additionally, the establishment of the proper integration parameters in the software reduces the number of manual integration occurrences. The documentation for all manual integrations shall clearly show the before and after picture of the circumstance for needing to perform the manual integration. (i.e The unaltered, raw data file must remain intact.) A program is in place describing the manual integration procedure.

The SOP for each test presents the formula used for data reduction for the individual method. These SOPs present the procedures to be used for calculating and documenting procedures such as linear regression, absolute retention times, relative retention time windows, and reporting of the results from second column confirmations.

Data Qualifiers

The laboratory adds data qualifiers during the data generation/ review process. These qualifiers are applied when data quality objectives are not met or affected. All flags used are defined completely within the final data report packages.

The following data qualifiers are currently in use:

- Q Surrogate Recovery outside QC Limits
- J Estimated Value. Presence of the compound was confirmed but less than the reported reporting limit. (i e. SQL, MQL, RL)
- E Concentration exceeds the established method calibration range but is within the working range of the instrument.
- B Analyte detected in the associated Laboratory blank.
- U Reported result was unconfirmed. Refer to Case Narrative.
- C Result reported from GC/MS confirmation analysis.
- M Result reported represents a minimum value. Refer to Case Narrative.
- NC Result reported from Primary Column Result did not confirm or were not analyzed by secondary column.
- * QC Data (Percent Recovery/RPD for a particular analyte was outside QC Limits)

6.12 Laboratory Records, Management, and Document Control

Laboratory Management Partners, Inc. has a record system that produces unequivocal, accurate records, which document all laboratory activities from sample receipt to sample disposal. All required records are retained for the period required by the prevailing accrediting authority. The system retains records longer than the minimum retention time upon the request of authorized clients, agencies or regulation.

Laboratory Management Partners, Inc. shall retain at the minimum the following records:

1. Analytical worksheets, batch worksheets, supporting documents, and data output and quantitiation records;
2. Calculation steps including dilutions and non-nominal sample size to assist in data reduction to a reportable value;
3. Copies of all final reports;
4. Archived SOPs and supporting documents;
5. Correspondence relating to laboratory activities for a specific project;
6. All corrective action reports, audits and audit responses;
7. Proficiency test results and raw data;
8. Data review, assessment, and validation processes.

In addition, Laboratory Management Partners, Inc. maintains records of:

1. Personnel qualifications, experience and training,
2. Initial and continuing demonstration of proficiency for each analyst;
3. A log of names, initials and signatures for all individuals who are responsible for signing or initialing any laboratory records.

6.12.1 Record System and Design

The recordkeeping system allows reconstruction of all laboratory processes that produce the analytical data for the sample.

a) The records include the names of personnel involved in sampling, preparation, calibration or analysis.

b) All information relating to laboratory facilities equipment, analytical methods, and activities such as sample receipt, preparation, or data verification are documented.

c) The recordkeeping system facilitate retrieval of all working files and archived records for inspection and verification purposes.

d) All generated data, except those generated by automated data collection systems, are recorded directly, promptly and legibly in permanent ink.

e) All changes to records are signed or initialed and dated by responsible staff. The reason for the change/correction is clearly indicated. Entries in records are not obliterated by methods such as erasures, correction fluids, or scratch outs All corrections to record-keeping errors are made by one line marked through the error.

f) Data entry is minimized by electronic data transfer and ensuring the number of manual data transcriptions is reduced.

6.12.2 Records Management and Storage

The management of laboratory records is a vital and integral part of a Quality System. Not only do these documents provide a record of results, they document traceability of laboratory activities related to a specific analytical function. The essential laboratory logbooks and records follow Document Control Procedure. Authorized documents or logbooks will be used at this facility. Documents and logbooks are regulated through the Document Tracking Process.

Procedures and systems are in place to handle management, storage and archival of information generated by LMP, Inc.

LMP, Inc. retains records of all original observations (including those pertaining to calibration and test equipment), calculations and derived data, calibration records, raw data and a copy of the final report package for a minimum of five (5) years as specified by NELAC, or as specified by project requirements, if longer periods are defined. This includes all information necessary for the historical reconstruction of data.

a) The laboratory maintains all hardware and software necessary for reconstruction of data.

b) Records, which are stored only on electronic media, remain supported by the hardware and software necessary for their retrieval

c) Records that are stored or generated by computers have hard copy or write-protected backup copies or image file copy.

d) Laboratory Management Partners, Inc. has established a record management system, for control of all laboratory notebooks.

e) Access to archived information is carefully controlled and is limited to authorized personnel. These records are protected against fire, theft, loss, environmental deterioration, vermin, and in the case of electronic records, electronic or magnetic sources

f) In the event that Laboratory Management Partners, Inc. transfers ownership or goes out of business, the laboratory must ensure that the records are maintained or transferred according to the client's instructions.

6.12.3 Record Archival

Accumulation of boxed archived records are maintained in a secure environment Archival of administration and laboratory records to the record storage area occurs to ensure traceability and data security. The Quality Assurance Office maintains record system indices and labels the box with the contents, date and laboratory area. The Quality Assurance Office assigns and records into a permanent index records of box number and box contents. Boxes are stored on site and off-site for the record retention period identified.

In support of LMP's Quality System procedures and polices are in place detailing information directly related to EPA's recommendation and requirements for protecting, storing and archiving the integrity of data generated at this facility. All raw data, logbooks, documents, correspondences and other documents relating to interpretation and evaluation of data collected, analyzed, processed, or maintained on the automated data collection system(s) are retained as required by NELAC or by other accreditation authorities. Its application supports those requirements outlined in *EPA's Good Automated Laboratory Practices 2185, Section 7.12* and other referenced documents. The scope for maintaining and archiving allows for the following:

a) Systematic methods of creating and indexing reference documents

b) Generating methods of traceability at various levels of data generation

c) Accounting system for storing and archiving records

d) System of regenerating and/or retrieving data both electronically and physically

e) Archiving data for specified time

f) Method to retrieve data used for, or in support of, legal and non-legal issues

Data destruction is subcontracted to a licensed document shredding company.

7 Physical Facilities – Accommodation and Environment

Laboratory Management Partners, Inc. maintains a secure testing facility that accommodates the proper performance for the type, range, and volume of analytical services it provides. This laboratory facility encompasses a total area of 18,000 square feet. The floor plan is available upon request.

The laboratory functional areas include:

Administration and offices,
Sample Receipt and handling,
Lab Support – Shipping,
Microbiological and Bioassay,
Agricultural lab,
Classical analytical chemistry,
Inorganic Lab – Preparation and Instrumentation,
Inorganic Instrumentation Lab – ICP, GFAA and Cold Vapor,
Organic sample preparation
Volatile organic analysis (GC and GC/MS),
Semi volatile analysis (GC, GC/MS and HPLC),
Waste Management and Disposal,
Miscellaneous mechanical and storage areas

7.1 Environment

Each laboratory section is provided with effective separation of any incompatible testing activities (e.g. separation of organic extractions from the volatile analytical section). Lighting, noise, humidity, heating, ventilation and air conditioning, and energy sources satisfy the needs of the testing performed in the permanent facilities. The laboratory building design ensures regulated temperature control for analytical equipment This ensures stability of voltage, temperature, and other pertinent environmental conditions. Air-handling systems minimize airborne contaminants that jeopardize sample integrity or analytical performance.

The analytical instrumentation is in separate rooms from laboratory activities that involve the use of large quantities of organic solvents or inorganic acids. A separate room provides the facilities for the microbiological and bioassay testing.

Standards and other materials requiring sub-zero storage temperatures are placed in freezers at temperatures of –10 to –20C and separated from samples or potential contaminating materials. Refrigerators provide cooling temperature for samples requiring the appropriate range of 4 +/-2C. Sample storage areas for volatiles are separated from other samples and monitored for any effects due to cross contamination.

7.2 Laboratory Work Areas

The monitoring of environmental conditions and general housekeeping is maintained to avoid any influence on the testing activities performed and to provide assured and continued safe environment. The separations of analytical sections are vitally important such as in the microbiological and bioassay departments. Additional requirements are in support of Laboratory

Safety Practices. Good housekeeping is the responsibility of all personnel. Each person is responsible for assuring clean and unrestricted work areas.

7.3 Security

Laboratory Management Partners, Inc. provides a secure environment for its employees, guests, clients, samples and analytical data. Security procedures require that all exterior doors remain locked unless manned internally. Access to the laboratory is limited to employees and contractors. All visitors are required to sign in and out using the Visitors Log and must be accompanied by a laboratory employee at all times within the appropriate areas.

All doors are locked after hours and require a key for entry. The security alarm continuously monitors for smoke and fire related heat When the alarm is activated, the appropriate emergency response departments are notified. The local emergency departments have the emergency contact list for the laboratory. Likewise, the laboratory posts emergency numbers in each area

There are high-resolution security cameras placed strategically within the facility. The cameras continuously monitor their respective areas with digital recording at all times.

8 Instrument Equipment

8.1 Instrumentation

LMP, Inc. maintains a full complement of state-of-the-art instrumentation for the proper performance of analytical services. All instrumentation and equipment is selected to meet method specific sensitivity requirements. All laboratory equipment is purchased through an approved vendor. LMP, Inc. maintains a purchase order system that allows the use of approved vendors only. This system tracks the ordering, receipt and approval of all equipment and supplies utilized by the laboratory.

The installation and verification process of new instrumentation occurs according to the following schedule:

a) Receipt of equipment and assignment of unique instrument ID

b) Installation by a vendor approved service engineer

c) General operation verification

d) Sensitivity Check

e) Initial Calibration

f) Method Detection Limit Study

g) Demonstration of Capability Study

The laboratory is furnished with all of the items of equipment required for the correct performance of the tests, which it conducts. A listing of the major equipment used for testing is available upon request. The equipment list details the unique identification number and a summary of the maintenance information. The unique identification number is listed on the equipment, in the maintenance record book and in the calibration records.

8.2 List of Laboratory Equipment

8.2.1 Organic Instrumentation

Volatiles – GC and GC/MS

Semi Volatiles – GC and GC/MS

GC Systems – GC/FID

Organochlorine/Pesticides/PCBs/Herbicides – GC/ECD

Nitroaromatics/Nitroamines - HPLC/UV

Miscellaneous Analysis – HPLC/UV/DA/Fluorescence

8.2.2 Metals Instrumentation

Metals by ICP, Trace ICP, GFAA, and CVAA

8.2.3 Inorganic Instrumentation

Total Organic Carbon Analyzers

Automated Cyanide Preparation System

Automated Oil and Grease Solvent Extraction and Solvent Trap, and Recovery System

LECO Nitrogen Analyzer

Ion Chromotography

8.2.4 Additional Instrumentation

Spectronic Spectrophotometer

Glas-Col 3-D 8 position Floor Shaker

Nitrogen Rapid-Vap Concentrators

Turbidimeter

Sonicators

Penski Martin apparatus for ignitability

8.3 Support Equipment

The laboratory performs analyses using state of the art equipment. In addition to the major equipment, the most common equipment used in the laboratory are: thermometers, balances, autopipets, water baths, hot plates, autoclaves, pH meters, conductivity meters, 8-position floor shakers, Geiger Counter and a variety of labware. The SOP lists the calibration and verification requirements for all laboratory equipment used in measurements.

Laboratory reagent water is purified using ion exchange resin bed which is monitored daily with digital in-line meters. The reagent water resistivity normally is greater than 17 megohms.

Reference materials include: Class I and II weights, NIST-traceable thermometers and reference standards. Logbooks record the reference materials used for calibration and verification. The quality control staff maintains any certificates received with the reference materials. Laboratory personnel record in the standards logbook the reference standards date received, unique identification number, and expiration date. Each laboratory area, records the unique identifier on the reference standard certificate which is filed in the QA office.

8.4 Maintenance

The laboratory has a proactive instrument and equipment maintenance program to minimize downtime in analytical work. The laboratory maintains service contracts for major equipment, which include routine preventative maintenance visits by the service provider. Personnel perform manufacturer-specified maintenance on a routine basis to ensure equipment operates at peak performance.

Routine maintenance procedures are detailed within the applicable standard operating procedure (SOP) is available. All instrument preventative and corrective maintenance are recorded in the maintenance logbook assigned to the equipment. After instrument maintenance or repair, the instruments must successfully calibrate following the method SOP. Laboratory personnel are trained in routine maintenance procedures for all major instrumentation and must demonstrate quality control performance before sample analysis.

The laboratory maintains a stock of replacement parts and consumables for analytical equipment. Instrumentation contingency plans are available for use in case of major equipment failure. In the event of equipment maintenance problems or failures, equipment is taken out of service.

Maintenance logbooks are kept for all major laboratory instrumentation and equipment. These logbooks document manufacturer-recommended maintenance procedures, specific cleaning

procedures, comments on calibration and replacement of worn or damaged parts and any work by outside contractors. When repairs are necessary, the equipment is taken out of service, repairs performed by either trained staff or trained service engineers, and an evaluation of the impact on previous calibrations or tests performed are recorded in the appropriate logbook with analyst's initials and date. If an instrument is down for maintenance, a complete record of all corrective actions taken to reinstate the instrument back into operational service is recorded including reference to the new calibration and quality control checks. Maintenance contracts are maintained on all major analytical instruments. Work conducted by service providers is noted in the logbook and reference is made to the service report. Service reports are also kept on file.

Minimally, the maintenance records shall include:

Equipment name;

Manufacturer's name, type identification, serial number or other unique identification;

Date received, date put into service, condition when received;

Current location;

Details of past maintenance and future scheduling of maintenance;

A history of any damage, malfunction, modification or repair;

Dates and results of calibration or verification;

Records of maintenance performed by an outside contractor.

9 Measurement Traceability and Calibration

9.1 General Requirements

All measuring operations and testing equipment having an effect on the accuracy and validity of tests. These operations and equipment are calibrated and/or verified before put into service and on a continuing basis. The results are recorded in the instrument- or equipment-specific logbook. The laboratory has a program for the calibration and verification of its measuring and test equipment. The program includes all major instrumentation and support equipment such as balances, thermometers and quality control standards. This QM describes the calibration program including frequency and acceptance criteria

9.2 Reference Standards and Reagents Program

Laboratory Management Partners, Inc. maintains a Solution Validation Program for the reagents and standards used. The Reagent and Standard Solutions Validation Program assure that all solutions and reagents purchased are traceable and accurate. Standards are purchased as certified solutions or made in the laboratory from neat materials. All reagents, standards and solvents used within the laboratory must be traceable to the original source. All chemicals and reagents are stored in appropriate storage areas. All flammable stock solvents are stored in OSHA and NFPA approved cabinets Acids are stored in acid cabinets.

Upon receipt, chemicals, reagents and neat standards are logged into an automated system that allows traceability of chemicals, standards and reagents, and manufacturer's lot number by assigning an unique identification number. On the Certificates of Analysis, the unique identification number assigned for that standard or reagent is documented Certificates of Analysis are maintained in the Quality Assurance Department. Upon depletion of the chemical, the container is removed from the inventory and the inventory is updated.

Once the chemicals/reagents or standards are properly logged in, they are affixed with the lab-generated label by the automated system indicating the name of chemical, standard or reagent, unique laboratory identification number, expiration date, and date received. The open date must be completed when the analyst physically opens standard or reagent. The chemicals are stored in the chemical stockroom until use in the preparation of reagents or standards. Chemicals used directly in the laboratory are assigned a reagent number before use in the laboratory area.

When the secondary standards are to be generated, the analyst shall locate in the appropriate automated system the name of the secondary standard to be generated. This database generates a programmed formula, which describes the specific reagents and standards to be used, and the quantity of the standards or reagents and the preparation schedule. Upon the determination of quantity required, the analyst shall generate a bench sheet that summarizes aforementioned formula.

Secondary standards are generated and maintained within the specific area of use. The analyst shall record preparation of standards in a standards logbook, which is maintained. Each secondary standard is identified as to test method, quantities and identification of reagents and standards used in preparing the standard solution. The documents shall include the analyst's initials, date received, date opened, and expiration date. When required, standards are verified using a second source or lot number different from that calibration standard.

The purchase, receipt and storage of consumable materials used for the technical operations of the laboratory include the following;

a) The laboratory retains records of manufacturer's statement of purity, of the origin, purity and traceability of all chemical and physical standards.

b) Original reagent and standard containers are labeled with the date opened and the expiration date. When the manufacturer does not specify the day of the month in the expiration date, the container expiration date defaults to the last day of the month.

c) Detailed records are maintained on reagent and standards preparation. These records indicate traceability to purchased stocks or neat reagents. The following are also recorded for purchased and prepared standards as applicable: The manufacturer, lot number/bench sheet unique identifier, chemical name, mfg expiration date, purity or concentration, reagents used for preparation including source, lot number and expiration date, person recording or preparing the standard/reagent and the date of preparation or receipt of stock.

d) All prepared reagents and standards are uniquely identified and the contents are clearly identified with expiration date, concentration and preparer's initials.

9.3 Traceability of Calibration

The program of calibration and /or verification and validation of equipment is such that measurements are traceable to national standards, where available. Likewise, calibration standards are traceable to national standards, provide information of purity, and associated uncertainty of measurement and/or a statement of compliance with identified metrological specifications. The laboratory maintains a permanent file of all such certifications.

9.4 General Calibration Procedures

Essential to the Quality System, calibration procedures are in place that ensures measurements of traceability for each phase of the analytical system Such calibrations are required in instrument calibrations and the use of calibration standards such as initial and continuing calibrations. Minimally, each calibration record is dated and labeled with method, instrument, analysis date, analyst(s), analyte name, concentration and response The data processing system shall compute the calibration curve for each analyte. The curve may be derived from linear or nonlinear procedures. The curves generated are traceable to a database file and the filename shall be recorded on the appropriate batch worksheet. This is also true for manually prepared curves. Criteria for acceptance of a calibration curve is established and documented.

The following instrumentation shall undergo the following general calibration procedures.

a) Purge & Trap Gas Chromatography/Mass Spectrometry (GC/MS)

b) Purge & Trap Gas Chromatography (GC)

c) Automated Direct Injection GC/MS

d) Automated Direct Injection GC

e) High Pressure Liquid Chromatography (HPLC)

9 4.1 General Instrumentation Calibration Procedures

The following instrument calibration procedures are used in the laboratory's daily analytical procedures where applicable:

a) Initial Calibration, ICAL

The calibration of instruments is required to ensure that the analytical system is operating correctly and functioning at the proper precision, bias (accuracy), and sensitivity. The

frequencies of calibration and calibration verification are presented in the following sections and are based upon the various analytical methods and industry standards.

An instrument is considered calibrated when an instrumental response can be related to the concentration of an analyte. This relationship can be depicted graphically and is termed a calibration curve.

ICALs are based on a requisite number of standards identified within the individual method for each target analyte The MQL shall be established at the low standard for each target analyte See individual method SOPs for details on concentrations, number of standards used, and evaluation of the ICAL.

b) Initial Calibration Verification, ICV

The ICAL is verified as accurate with a standard purchased or prepared from an independent source. This procedure involves the analysis of a standard containing all of the target analytes (usually at the middle level of the curve) Refer to individual method SOPs for details on the concentration and evaluation of the ICV

c) Continuing Calibration Verification, CCV

The initial calibration is verified on each day of testing through the analysis of a continuing calibration standard. The concentration and frequency of use of the continuing calibration standard is performed in compliance with the requirements of the specific method. The relative response factors for all analytes of interest are calculated and verified against the initial calibration mean relative response factors. The percent difference (%D) for each analyte is calculated and must be less than the acceptance criteria stated in the method.

This standard is analyzed to determine whether the analytical system is working properly and/or if a new ICAL is required. Refer to individual method SOPs for details on the frequency and evaluation of CCVs. Calibration Verification verifies compliance with the initial calibration curve, but does not overwrite the response factors used for the quantitation, nor allows re-sloping of the calibration curve.

An acceptable continuing calibration run must have measured percent differences for the analytes within method-specified ranges. If any criteria for an acceptable calibration are not met, either instrument maintenance must be performed until the continuing calibration analysis meets all criteria or a new initial calibration is established before any samples can be analyzed. No samples are analyzed unless the acceptance criteria are met for the initial and continuing calibration.

d) Initial Calibration Blank, ICB and CCB, Continuing Calibration Blank

These QC samples are required for inorganic metals analyses to verify the system is free of contamination.

The found concentrations for each target analyte in the ICB/CCB must be less than or equal to the MDL.

9.5 Instrument Calibration

Laboratory Management Partners, Inc. performs the following instrument calibration procedures Instrument calibration is discussed in SW 846 method 8000 and in individual reference SOPs.

Calibration is defined as the determination of response versus concentration of an analyte. This relationship is referred to as a calibration curve. Initial calibration curves are established utilizing a prescribed number of standards for each target analyte. The initial calibration curve is established as specified in the individual methods, using (a minimum of) five standards for all single-component target analytes and surrogates, and at least three standards for multiple component target analytes (e.g., Toxaphene, Chlordane, and PCBs).

Linearity is determined using linear regression analysis for each target analyte by calculating the correlation coefficient (r) or the squared correlation coefficient (r^2). In the generation of the calibration curve, the origin is not used as a calibration point nor is force through the origin. Individual SOPs indicate minimum r or r^2 values.

Alternatively for chromatographic methods, the average calibration factor (CF) or response factors (RF) are calculated for each target analyte. Linearity is be evaluated by calculating the percent relative standard deviation (%RSD) of the CFs/RFs from the initial calibration standards for each target analyte. The individual reference SOPs indicate minimum acceptance criteria for %RSD.

Linearity is presumed if the correlation coefficient (r) is >= 0.995 or the coefficient of determination (r2) is >=0.99. An RSD criteria for linearity is specified in the applicable method Analysis will not proceed until the method specific initial calibration evaluation criteria are met. Exception is made with TX1005 where r^2 is greater than or equal to 0.995

Note: SW-846 has incorporated an allowance to evaluate the mean of the RSD values for all target analytes in the calibration if this average value is less than the method acceptance criterion The option for use of the mean of the RSD values for validation of initial calibrations is currently optionally applied.

9.5.1 GC/MS Methods

GC/MS methods have additional specific evaluations that must be performed to validate the initial calibration:

a) System Performance Check Compounds (SPCCs). These compounds are designated within the analytical method (e.g. 8260B, 8270C.) and are used to verify compound stability and to check for degradation caused by contaminated lines or active sites in the system. The SPCCs are evaluated for minimum response factors as specified in the method. Analysis of samples is not to proceed unless this criterion is met.

b) Calibration Check Compounds (CCCs). The purpose of the CCCs are to evaluate the calibration from the standpoint of the integrity of the system. High variability for these compounds is indicative of system leaks or reactive sites on the column. The CCCs are evaluated for %RSD maximums as specified in the method Analysis of samples is not to proceed unless this criterion is met.

9.5.2 GC and HPLC Methods

GC and HPLC initial calibrations are performed

a) Multi-Component Pesticides. For each multi-component pesticide (e.g. Chlordane and Toxaphene), a mid-level standard is analyzed each sequence to aid in pattern recognition. If a multi-component pesticide is identified in a sample, the system is re-calibrated for that pesticide with a minimum of three (3) standards. The extract is re-analyzed with the new calibration. Calibrations for the multicomponent pesticides are

based on three (3) to five (5) major characteristic peaks or area sum in the case of Toxaphene.

b) PCBs – Initial calibrations are maintained for Aroclors 1260 and 1242 (unless otherwise specified, the LCS and MS/MSD will be spiked with a mixture of 1260/1242). Mid-level standards of the other Aroclors are analyzed each analytical batch to aid in pattern recognition. If another Aroclor is identified in a sample, the system is calibrated for that Aroclor with a minimum of three (3) standards. The extract is re-analyzed with the new calibration. Calibrations for the Aroclors are based on three to five major characteristic peaks.

For multiple component pesticides, PCBs or hydrocarbons the quantitation consists of the average of selected peaks or the integration of the area defined by a reference standard The SOP details the integration criteria for each compound.

Internal standard calibration or external standard calibration is utilized for analysis by GC. The method-specified number of calibration standards is used. Each solution is analyzed once and the analyte relative response factors or calibration factors are calculated. The mean relative response factor for each analyte is then obtained by using the expression in the formula listed in the SOP. Integrated areas are used for these expressions. Retention time windows for peak responses are calculated in compliance with the method requirements.

The initial calibration is verified as accurate by the analysis of an independent calibration verification standard. This standard is prepared independently of the ICAL from a second source or different lot number. The ICV standard must meet method continuing calibration verification (CCV) criteria in order to validate the initial calibration.

CVs are analyzed to determine if the analytical system is operating properly and to validate that the current initial calibration remains valid. The CV is typically the analysis of a single, mid-level standard at method specified intervals. Typically, a calibration verification (CV) is analyzed at the beginning of the sequence, continuing CVs are dispersed throughout the sequence and at the end of the sequence.

The calibration verification process does not overwrite the original response factors from the initial calibration. Calibration verification is used for all organic analytical methods.

Initial CVs are evaluated based on method specific criteria. If CV does not meet the evaluation criteria requirements specified by the method, a second CV is analyzed. If the CV remains unacceptable, corrective action must be taken (e.g. instrument maintenance). The CV must meet method evaluation criteria prior to the analysis of samples or a new initial calibration must be performed.

Note: SW-846 has incorporated an allowance to evaluate the mean of the percent difference or percent drift values for all target analytes in the calibration verification if this average value is less than the method acceptance criterion. Currently, the use of the mean evaluation is permitted for all GC and HPLC method CCVs only. However, use of the mean evaluation for the CV (initial calibration verification) requires supervisor approval.

GC/MS methods have additional specific evaluations that must be performed to validate the CV:

a) Evaluate the CCCs and SPCCs in the CV according to method specific evaluation criteria.

b) Evaluate the responses and retention times of the internal standards in the CV according to method specific evaluation criteria.

Method performance is monitored by the introduction of various internal quality control checks and samples. These checks allow the evaluation of data on a method, matrix and sample basis.

In general, each extraction batch requires, at a minimum, a laboratory blank (LB), laboratory control sample (LCS) and a matrix spike/matrix spike duplicate (MSD).

a) Batch QC evaluation is based on the analysis of laboratory control sample(s) and laboratory blank(s).

b) Matrix QC evaluation is based on the fortification of an environmental sample with known amounts of target analytes (matrix spike, matrix spike duplicate).

c) Sample evaluation is performed with the assistance of surrogate recoveries.

9.5.3 Additional Organic Calibration Procedures

Retention Time Windows

Retention time windows are crucial to the identification of target analytes. Absolute retention times are used for compound identification in all GC and HPLC methods that do not employ internal standard calibration. Retention time windows are established to compensate for minor shifts in absolute retention times as a result of sample loading and normal chromatographic variability The width of the retention time window is established to minimize the occurrence of both false positive and false negative results. Tight retention time windows may result in false negatives and/or may cause unnecessary reanalysis of samples when surrogates or spiked compounds are erroneously not identified. Overly wide retention time windows may result in false positive results that cannot be confirmed upon further analysis.

LMP, Inc. establishes the majority of retention time windows based on the default standard deviation criteria of 0.01 minutes listed in *Method 8000B Section 7.6.3 of SW-846.* RTWs are established and monitored using the following procedure:

a) Make three injections of all single component standard mixtures, including surrogate standards and multi-component analytes (such as PCBs) over the course of a 72-hour period. Typically, working standards analyzed the previous week are used to generate the retention time window report. This report is automatically generated by the Target system.

b) The mean and standard deviation of the absolute retention times for each single component analyte and surrogate are calculated.

c) For multi-component analytes (e.g. chlordane, PCBs), three to five major peaks are chosen. The mean and standard deviation of those peaks are calculated.

d) According to *Section 7.6.3 of SW-846 method 8000B*, "If the standard deviation of the retention times for a target compound is 0.000(i.e., no difference between the absolute retention times), then the laboratory must either collect data from additional injections of standards or use a default standard deviation of 0.01 minutes."

e) The width of the retention time window for each analyte, surrogate, and major constituent in multicomponent analytes is defined as ± 3 times the standard deviation of the mean absolute retention time established during the RTW study. If the default standard deviation of 0.01 minutes is employed, the width of the window will be 0.03 minutes.

Instrument Performance Checks

Several methods outline additional QC procedures to verify the instrumentation is in good working condition. These QC samples must be analyzed and meet method-specified acceptable limits prior to commencing sample analyses.

Mass Spectrometer Tuning – GC/MS Methods

The GC/MS is hardware tuned before performing the initial and continuing calibrations. Results must meet the peak ratio specifications of the analytical methods. Volatiles analyses use bromofluorobenzene (BFB) and Semivolatiles analyses use decafluorotriphenylphosphine (DFTPP) for instrument tuning.

Prior to the analyses of any standard or samples, the MS standard mass spectral abundance criteria must be evaluated. BFB (4-Bromofluorobenzene) tune standard is analyzed for GC/MS volatiles (e.g. method 8260B) and DFTPP (Decafluorotriphenylphosphine) for GC/MS semi-volatiles (e.g. method 8270C). The tune standard must be analyzed at the beginning of the analytical shift/sequence and every 12 hours of continuous analysis. The 12-hour clock starts at the time of injection of the tune standard. Tune standards evaluation reports are generated by the Target system. Analysis will not proceed until tune standard meets method specific acceptance criteria

Injection Port Inertness - GC/MS Semi-Volatiles

To verify column condition and injection port inertness, the DFTPP tune standard also contains 50ng of 4'4'DDT, Benzidine and Pentachlorophenol. Analysis will not proceed until tune standard meets method specific acceptance criteria.

a) Injection Port Inertness

The injection port inertness of the GC portion of the GC/MS is evaluated by the %breakdown of 4,4'- DDT This procedure is done to verify acceptable instrument performance, regardless of whether DDT is a target analyte The %breakdown of 4,4'-DDT to 4,4'-DDE and 4,4'-DDD must not exceed method specific acceptance criteria in order to proceed.

b) Column Performance Check

The condition of the GC column is evaluated by the tailing of benzidine and pentachlorophenol (PCP). Benzidine and pentachlorophenol must be present at their normal responses, with no visible peak tailing, as demonstrated by the peak tailing factors. The acceptance criteria for the peak tailing factor for benzidine and pentachlorophenol is specified in the applicable method. Breakdown and tailing factor reports are generated by the Target system.

Injection Port Inertness Check – GC Pesticides

The inertness of the GC system must be checked prior to beginning the analytical sequence. A mid-range standard of 4,4'-DDT and Endrin is analyzed and monitored for breakdown. If the breakdown for either compound exceeds the specified criteria, corrective action must be performed. Analysis will not proceed until the breakdown standard meets method specific acceptance criteria.

Additional quality control surveillances are part of the GC/MS analysis. These include internal standards, surrogates, laboratory blanks, instrument blanks, laboratory control samples, matrix spikes and matrix spike duplicates The frequency and control criteria are defined in the laboratory SOP.

9.5.4 Analytical Methods – Metals Laboratory

Analyses for metallic analytes utilize the following instrumentation.

a) ICP – Inductively coupled plasma-atomic-emission spectroscopy

b) GFAA – Graphite furnace atomic absorption spectrophotometer

c) CVAA – Cold vapor atomic absorption spectrophotometer

Calibration Procedures

Initial Calibration (ICAL)

For metals analyses, an initial calibration must be performed at the beginning of each analytical shift, and when a CCV fails or significant instrument maintenance is performed In general, linearity is acceptable only if the linear regression coefficient r is >= 0.995 or if the squared correlation coefficient r_2 is >=0.99. If r < 0.995 or r_2 < 0.99, take corrective action and recalibrate.

ICP

ICP initial calibration is performed with a high-level standard and an initial calibration blank (ICB). The concentration of the single standard establishes the linear calibration range and the linear dynamic range of the method. To ensure accuracy of concentrations at the MQL, a low-level check (LLC) standard is prepared from the primary source standard and analyzed after initial calibration. Results for the LLC must meet method specific acceptance criteria.

GFAA/CVAA

GFAA initial calibration is performed using a minimum of three (3) standards and a calibration blank. CVAA initial calibration requires a minimum of five (5) standards and a calibration blank. The low standard is at or below the MQL For GFAA, standards are analyzed in duplicate. The RPD between duplicate injections for all standards must meet method specific acceptance criteria.

Linearity is acceptable only if the linear regression coefficient r is >= 0.995 or r_2 is >=0.99. If the calibration criterion does not meet any of the above requirements, corrective action is required prior to the analysis of any samples.

Initial Calibration Verification (ICV)

ICP

The initial calibration is verified as accurate prior to the continuation of the analytical sequence. The verification is performed based on the following:

a) Read-Back Analysis

Standards used for the initial calibration are re-analyzed as samples. The read-back results must meet method specific acceptance criteria to proceed. Corrective action is taken when this criterion is not met.

b) Initial Calibration Blank (ICB)

The ICB is analyzed as a sample. All analytes must be below the MQL unless otherwise specified in the analytical SOP.

c) Independent Calibration Verification Standard (ICV)

The ICV is a standard prepared independently (e.g. second source) of the initial calibration standards

The ICV must meet method specific acceptance criteria.

d) Interference Check Sample (ICS)

Standards ICS-A (majors) and ICS-A/B (majors/minors) are analyzed. The results must meet method specific acceptance criteria.

e) Low Level Check (LLC)

The LLC standard is analyzed and must meet method specific acceptance criteria.

GFAA/CVAA

The initial calibration is validated based on the analysis of the Independent Calibration Verification Standard (ICVS) The ICVS is a standard prepared independently (e.g. second source) of the initial calibration standards. The ICVS must meet method specific acceptance criteria.

Continuing Calibration Verification (CCV)

ICP

To verify the initial calibration throughout the analytical sequence, a series of checks are performed every 10th sample and at the end of the sequence.

a) Continuing Calibration Blank (CCB) Analyze after 10 production/QC samples and at the end of the sequence. All results must be less than the MQL unless otherwise specified in the analytical SOP

b) CCV standards Analyze after 10 production/QC samples and at the end of the sequence and must meet method specific acceptance criteria.

c) ICS-A and ICS-A/B. Analyze at the beginning and end of the sequence and meet method specific acceptance criteria.

GFAA/CVAA

To verify the initial calibration throughout the analytical sequence, a series of checks are performed after 10 production/QC samples and at the end of the sequence.

a) Continuing Calibration Blank (CCB) Analyze after 10 production/QC samples and at the end of the sequence. All results must be less than the MQL unless otherwise specified in the analytical SOP.

b) CCV standards. Analyze every 10th and at the end of the sequence and must meet method specific acceptance criteria.

Quality Control Procedures

Quality control procedures (e.g. sample batching, batch QC requirements) are detailed in Section 5.10 of this document. In general, each sample batch requires the digestion and analysis of a laboratory blank (LB), laboratory control sample (LCS) and a matrix spike/matrix spike duplicate (MS/MSD).

Method performance is monitored by the introduction of various internal quality control checks and samples. These checks allow the evaluation of data on a method and matrix basis. Method QC evaluation is based on the analysis of laboratory control samples and laboratory blanks. Matrix QC evaluation is based on the analysis of matrix spikes, matrix spike duplicates).

Refer to method SOPs for details on quality control procedures and their evaluation.

MS/MSD Failures – ICP Corrective Action

When MS/MSD recoveries are flagged as outside QC limits, the following corrective action is required:

Dilution Test (DT)

This test is generally performed when analytes are present at high levels in the parent sample when compared to the spike levels of the target analytes. If the result of the parent sample is at least 25 times the detection limit, then the digestate is diluted 1:5. The diluted and undiluted results of the failing analyte(s) must agree within 10%. If this test fails, all samples in the associated batch must be analyzed by the method of standard additions (MSA) for the failing analyte(s).

Post Digestion Spike (PDS)

If the results of the sample are less than 25 times the detection limit, then the parent sample digestate is spiked with a known amount of the failing analyte(s). The recovery must be within 75% to 125% of the expected value. If the recovery fails, all samples in the associated batch must be analyzed by the method of standard additions (MSA).

MS/MSD Failures – GFAA/CVAA Corrective Action

Dilution Test (DT)

This test is generally performed when analytes are present at high levels in the parent sample when compared to the spike levels of the target analytes. If the result of the sample is at least 25 times the detection limit, then the parent sample digestate is diluted 1:5. The diluted and undiluted results of the failing analyte(s) must agree within 10%. If this test fails, all samples in the associated batch must be analyzed by the method of standard additions (MSA) for the failing analyte(s).

Post Digestion Spike (PDS)

If the results of the sample are less than 25 times the detection limit, then the parent sample digestate is spiked with a known amount of the failing analyte(s). The recovery must be within 85% to 115% of the expected value. If the recovery fails, all samples in the associated batch must be analyzed by the method of standard additions (MSA) for the failed analyte(s).

Method of Standard Additions (MSA)

The standard addition technique involves adding known amounts of standard to one or more aliquots of the processed sample solution This technique compensates for a sample constituent that enhances or depresses the analyte signal, thus producing a different slope from that of the calibration standards.

Preliminary Method Set-Up for ICP

a) Linear Dynamic Range.

The upper limit of the linear dynamic range for the ICP is determined for each analyte wavelength used in order to determine an appropriate concentration for the high calibration standard. This number is the upper limit of the linear range. The linear dynamic range is checked initially, whenever there is a significant change in instrumental hardware or operating conditions or annually.

LMP, Inc. has established the upper limit for the ICP analytes as the concentration of the highest calibration standard used to perform the initial calibration. These values are well under the manufacture's recommendation of upper dynamic range for each analyte. Any sample results above these calibration values are diluted to bring the concentration of the analyte below the highest initial calibration concentration. This ensures that no sample result is reported that is above the initial calibration range of the instrument

b) Interference Check Standard (ICS)

Spectral interferences can be caused by background emission interference from several sources (e.g. recombination phenomena, stray light or overlap). These factors are compensated for by the use of Interelement Spectral Correction Factors (IECs). IECs must be verified prior to the analysis of samples using ICS-A and ICS-A/B QC solutions.

9.5.5 Analytical Methods – Inorganic Laboratory

Instrumentation/Techniques

The inorganic laboratory employs a variety of wet chemistry techniques. Inorganic analyses generally refer to wet chemistry methods which fall into the following categories:

a) Colorimetric - UV/VIS Analysis

b) Titration

c) Gravimetric

d) Ion-specific Electrode

e) Instrumental

1) Ion Chromatography

2) Total Organic Carbon

Total organic carbon calibration is obtained by analyzing a set of five or more initial calibration solutions. The concentrations must bracket the expected concentration range of the samples analyzed. Procedures for verifying the calibration curve are method specific The calibration curve is verified at least every 20 samples.

f) Spectrophotometric

Analytical worksheets have been developed for each method. The worksheet is method specific and is designed to record all necessary information, QA/QC requirements and sample results for each analytical batch. Data reduction procedures are presented within the analytical SOP. Sample preparation is generally included within the analytical method SOP.

Calibration

Initial Calibration (ICAL):

Analytical systems for wet chemistry methods are calibrated to define the working range by use of a series of standard solutions. A minimum of three (3) to five (5) standards is typically used depending on the specific method requirements. The concentrations and responses are used to generate a calibration curve using linear regression. The correlation of coefficient, r must be >= 0.995 or the coefficient of determination r^2 must be >=0.99 to validate the curve. Calibration curves are generated and documented.

Initial Calibration Verification (ICV).

When feasible, a second source is utilized to verify the ICAL. The ICV is analyzed and evaluated using the calibration verification criteria. Refer to individual SOPs for evaluation criteria. Also, instrumental read backof initial calibration solutions may be applicable.

Continuing Calibration Verification (CCV):

Prior to the analysis of samples and periodically throughout the analytical sequence, the calibration must be verified to ensure that the system is performing properly. Typically, CCVs are analyzed at the beginning, every 10 to 20 samples and at the end of an analytical sequence. Evaluation criteria are specified within the analytical SOP.

9.5.6 Analytical Methods – Bioassay

The Bioassay Laboratory is a self-contained lab maintained at a constant temperature with a regulated light cycle and dedicated heating and air conditioning systems. The laboratory is designed to meet criteria as outlined in EPA document EPA/600/4-91/002. The bioassay department maintains certifications in several states including Arkansas, Mississippi, and Tennessee and participates in the annual DMR-QA study as part of EPA Certification.

This facility routinely performs testing required by NPDES permits for both industrial and municipal clients. This testing includes both Acute and Short Term Chronic testing in freshwater for Ceriodaphnia dubia and Pimphales promelas (fathead minnow) and acute testing for Daphnia pulex. In addition to regulatory testing, the Bioassay laboratory performs a variety of chemical product screen tests.

Bioassay procedures differ significantly from typical environmental analytical requirements. This department requires its own Laboratory Quality Management Plan and is therefore not specifically addressed here.

9.6 Calibration of Laboratory Support Equipment

9.6.1 Thermometers

Thermometers are used in temperature-dependent equipment where the method or procedure requires the use of such. These include the monitoring of refrigerators, freezers, oven, incubators and water baths.

On a daily basis, the QAO or his designate checks and records the temperatures of working thermometers used at this laboratory. In aiding the monitoring of temperatures, thermometer bulbs are placed in sand for ovens and water or glycerin for water baths, freezers, refrigerators and incubators.

Laboratory thermometers are checked routinely for accuracy against certified, NIST traceable thermometers. These calibrations are performed annually for mercury or alcohol in glass thermometers, semi-annually for infrared thermometers and quarterly for digital thermometers. The temperature difference between the working thermometer and the NIST-Traceable thermometer shall be known as the correction factor. Correction factors derived from the calibrations, which are applied to temperature readings where applicable. The analyst records the corrected temperature for all observations.

NIST traceable thermometers are calibrated by a certified agency and re-certified annually Records of thermometer calibrations are retained in one logbook in the QA Supervisor's office. Working thermometers are identified according to location and serial number. All thermometers are tagged with the ID number, correction factor to be applied and the expiration of the calibration check.

Thermometers are not used past the calibration expiration date or if the thermometer is not reading properly Thermometers are replaced when a change occurs in a thermometer due to alcohol separation, breakage, damage or expired calibration.

9.6.2 Balances and Top Loaders

This laboratory routinely uses analytical balances and toploaders to weigh sample aliquots and reagents/standards.

This equipment is calibrated daily using pre-calibrated weights. Calibration checks are performed for each day of use, for each balance. The weights used cover the range of the intended use for each balance/toploader The calibration consists of a minimum of four weights, which bracket the weight to be measured. The Balance Record Form lists the acceptance criteria and performance criteria for the various balances used in the laboratory. The actual weight for each weight used is recorded on the Daily Balance Calibration Logsheet. The acceptance criterion for analytical balances is +/- 0.1% and +/- 1% for toploaders. Calibration weight measurements must meet the acceptance criteria listed on the record form

Each balance is serviced and calibrated by a professional at least annually. The accuracy of the calibration weights used by Laboratory Management Partners, Inc. are verified on a yearly basis by an accredited calibration service. Certificates of Calibration are issued and are filed with the Office of Quality Assurance Balances are labeled with the balance number, date of service and the expiration date for the annual service check. The balance number used for any measurements requiring traceability is recorded with measurement data. Balances are not used past the expiration date or when the weight check is not within acceptable criteria.

9.6.3 Automatic Pipettes

Where applicable, variable pipettes are used to spike samples, make dilutions and generate standards Where quantitative volume transfers are made, the delivery volumes are checked gravimetrically. Delivery volumes for the automatic pipettes are checked and recorded gravimetrically before use. The daily verification is performed at the volume of use. The daily check must be within the criteria stated in the pipette calibration logbook. The automatic pipette serial number is recorded in the data logbook for the measurement made with the auto pipette.

Where variable pipettes are used, calibration procedures are utilized daily. Each area maintains a variable pipette logbook to document multiple (3) weightings based on the specific density of water at 20°C. The specific density of water is determined to be 0.9982 g/ml. The established acceptance criterion is that accuracy must be within 3%.

9.6.4 pH Meters

pH meters are calibrated prior to each day of use. The meter is calibrated following the procedure for pH analysis. The records of the calibration are recorded in an instrument logbook or in the raw data for the analysis being performed. Three buffer solutions that bracket the measurement range for the analysis are used for calibration. An independent buffer is used weekly to verify meter stability. Buffer solutions used for calibration are traceable. Standard buffer solutions are not retained or re-used The lot number of the buffer solutions is recorded in the data record to ensure traceability of the measurement.

9.6.5 Conductivity Meters

Conductivity meters are calibrated daily using two NIST traceable solutions. The calibration standards are used to verify instrument performance. The acceptance criteria are defined in the

test SOP If unacceptable performance is found, the cell is cleaned and rechecked. The cell is not used until satisfactory performance is achieved.

9.6.6 Autoclave

The date, contents, sterilization time and temperature, total cycle time and analyst's initials are recorded each time the autoclave is used. Maintenance is conducted annually and recorded. Routine maintenance includes cleaning the autoclave seal to ensure freedom of caramelized media and cleaning drain screens to remove any debris buildup. For the efficient operation of the unit, overcrowding is avoided. A maximum temperature-registering thermometer is used during each cycle to ensure proper temperature is reached and not exceeded. Autoclave timing mechanisms are checked quarterly with a stopwatch to verify timing controls.

9.6.7 Spectrophotometer

LMP, Inc. utilizes a spectrophotometer for various inorganic methods. In order to verify that the spectrophotometer readings are accurate, a calibration procedure is used. This calibration involves the use of a wavelength calibration check. This check requires the generation of a standard, Cobalt Chloride, upon which multiple reading are taken at 505, 510, 515 and 520 nm. The instrument is in proper calibration when maximum absorbance (minimum transmittance) occurs between 505 and 515 nm. The specific absorbance values are unimportant.

9.6.8 Turbidimeter

Turbidity meter is used for various inorganic methods. In order to verify that the spectrophotometer readings are accurate, a calibration procedure is used. Multiple primary standards are used for verifying that the turbidimeter is reading correctly. These primary standards are various concentrations of Formazin. The acceptance criteria are +/- 5 percent of the concentration of the primary standard(s).

9.6.9 Volumetric Glassware

LMP, Inc. utilizes volumetric glassware appropriate for the applicable method. All glassware is routinely inspected for damage All glassware found to be structurally compromised is removed from service and replaced. All calibration certificates are maintained and filed in the respective department utilizing the device(s). In instances where a certificate is not available, the QAO shall conduct a Volumetric Glassware/Device Calibration Check. LMP employs *ASTM E 54-01* and *ASTM E 969-95* procedures for the verification of volumetric devices.

10 Test Methods and Standard Operating Procedures

10.1 Test Methods

The laboratory uses appropriate methods and procedures for all tests and related administrative activities. The method and procedures are consistent with the accuracy required, and with any standard specification relevant to the calibrations or tests concerned. When the use of mandated methods for a sample matrix is required, only those methods are used. Where methods are employed that are not required (e.g. a Performance Based Measurement System), the methods are fully documented and validated and are available to the client and applicable recipients. Refer to Appendix for Certified Analytical Methods.

10.2 Methods Documentation

Methodology documentation is a vital component in the QS. This document allows for an analytical pattern to exist that allows reproducibility in analytical procedures. Test method SOPs describe the sample analysis procedures, quality control frequencies and acceptance criteria. EPA accepted methods, nationally recognized methods or client-specified methods are the basis for performance criteria, instrument conditions and the steps of the procedure. The method performance requirements of the published methods are followed unless otherwise specified by the client

The reference methods define the operating conditions. In many of the reference methods a range or general guidance on the operating conditions are defined Documented modifications to the operating conditions clarify the reference methods or improve the quality of the results. In all cases where the method modifications are adopted, the performance criteria from the reference method must be met. Where such modifications occur, the laboratory shall obtain approval from their accrediting authority in writing Modifications to the operating conditions are stated in the SOP. Changes in the operating conditions are documented in the appropriate document revision form. A revision to a SOP takes place, when a change in the operating condition improves performance and/or as required by regulation.

10.3 Standard Operating Procedures (SOPs)

Laboratory Management Partners, Inc. maintains SOPs that accurately reflect all phases of current laboratory activities such as assessing data integrity, corrective actions, handling customer complaints, sample receipt and storage, purchasing of all materials, and all test methods. These documents include reference to equipment manuals provided by the manufacturer, internally referenced documents, and published promulgated methods.

Controlled copies of all SOPs are accessible to all personnel in either electronic or hard copy. The SOPs are organized in a standard format with the signatures of the approving authorities. Each SOP clearly indicates the effective date of the document and the revision number.

10.3.1 Laboratory Analytical Methods SOP(s)

Each laboratory area maintains a Laboratory Methods Manual containing SOPs utilized in this area. Manuals consist of copies of method and administration SOPs prepared by the laboratory Each test method includes or references where applicable:

1) identification of the test method;

2) applicable matrix or matrices;

3) method detection limit;

4) scope and application;

5) summary of method;

6) definitions;

7) interferences;

8) safety;

9) equipment, maintenance, and supplies

10) reagents and standards

11) sample collection, preservation, shipment and storage;

12) quality control;

13) calibration and standardization;

14) procedure;

15) calculations;

16) method performance;

17) pollution prevention;

18) data assessment and acceptance criteria for quality control measurements;

19) corrective actions for out-of-control data;

20) contingencies for handling out-of-control or unacceptable data;

21) waste management;

22) references; and

23) any tables, diagrams, flowcharts and validation data.

In cases where modifications to the published method have been made by the laboratory or where the referenced method is ambiguous or provides insufficient detail, these changes or clarifications are clearly described in the SOP.

10.3.2 Laboratory Administrative SOP(s)

Administrative SOPs are generated in support of analytical and non-analytical operating procedures. These SOPs maintain the framework and directives of all administrative duties required by the accrediting authorities. Both management and support staff undergo the appropriate training and such verification of compliance is documented on a Document Acknowledgement Form.

Administrative SOPs must include at least the following headings. Additional headings are added based on procedural requirements.

1) scope and application

2) definitions

3) procedure

4) corrective action

5) references

10.4 Computers and Electronic Data Related Requirements

Computers or automated equipment are used for the capture, processing, manipulation, recording, reporting, storage or retrieval of test data. The laboratory ensures that computer software is documented and adequate. The goals of the software development methodology, existing system validations and the change control system are to ensure that:

the software systems perform the required functions accurately,

the users understand how to use the system, and

the automated system can demonstrate data traceability and validity even after changes have been implemented (i.e. original data is captured).

The computer systems used at Laboratory Management Partners, Inc. are purchased from reputable dealers. A coordinated effort is made with the supplier to assure the computer operations meet the laboratory requirements for data integrity. Laboratory Management Partners, Inc. has a formal validation program of its computer systems. The validation program is a comprehensive program to ensure data transmitted, reported or manipulated by electronic means is correct and free of errors. The validation and verification approach is separated into three areas.

1. New software is developed and validated using test data Records of validation include the test data report, date and initials. Where formulas are part of the program, documentation includes manual verification of the final calculated values. New software includes the development of macros for spreadsheets and other tools using commercial software packages.
2. Request for changes to software are documented on the Corrective Action Report. Changes are identified through flaws in existing documentation or the need to improve system processes. Final implementation of the change is documented on the Nonconformance Action Form. The tracking and timelines of making the change is readily available This process also provides the complete documentation of all software and electronic data reporting problems.
3. Verification of system integrity is through routine maintenance, protection from unauthorized access and electronic verification programs. Routine maintenance including system backups are performed on a scheduled basis. The backup process, password and access protections are defined in the computer specific standard operating procedures. Electronic verification is used to assure the commercially purchased software is performing at its original specifications. This includes virus checking of all network operations on an ongoing basis Documentation of all verification and maintenance operations is retained in the equipment logbook for the computer network.

Where computers, automated equipment, or microprocessors, are used for the capture, processing, manipulation, recording, storage or retrieval of test data, LMP, Inc. ensures that:

a) Computer software is tested and documented to be adequate for use (e.g., internal audits, personnel training, focus point of QA and QC)

b) Procedures are established and implemented for protecting the integrity of data; such procedures include, but are not limited to, integrity of data entry or capture (e.g. instrument interface), data storage and archival (e.g. tape backup and off-sight storage), data transmission, data processing and virus detection

c) Computer and automated equipment are maintained to ensure proper functioning and provided with the environmental and operating conditions necessary to maintain the integrity of calibration and test data

d) Appropriate procedures are implemented for the maintenance of security of data including, but not limited to, the prevention of unauthorized access to, and the unauthorized amendment of, computer records

Data generated either electronically or physically is stored and archived using several methods of traceability. All raw data, documentation, and records generated are archived in a manner that is orderly and facilitates retrieval. Conditions of storage minimize potential deterioration of documents or magnetic media in accordance with the requirements for the retention period Maintenance of this media includes electronic computer backup of all in-house servers, scanning of final reports and raw data onto discs and storage of physical raw data. LMP, daily removes its electronic backup tapes from the laboratory. Likewise, final reports and raw data are scanned and the disc backup is stored at the bank. Raw data is archived and stored at an off-site location. LMP executes all possible methods to ensure the security of data. These procedures allow for reconstruction of data in case of a catastrophe at the laboratory.

11 Sample Handling Protocols

LMP, Inc. supports a sample management program designed to provide proper sample custody. A system of checks and balances are implemented to ensure that samples are received, handled, and maintained.

The sample management personnel check for proper sample labeling, preservation and handling at the time of arrival at the laboratory. Table 1 in Appendices specifies laboratory requirements for the proper sample preservation, containers, and holding times. Additional client specific criteria are documented on the Cooler Receipt Form and/or Project Information Form. Sample management staff records all observations and immediately notifies the Environmental Services Manager of any discrepancies or questions arising during sample receipt. Records of the sample condition are documented on the Cooler Receipt Form and/or Chain of Custody. In the event of noncompliance, the Environmental Services Manager generates a Noncompliance Report and contacts the client for instructions and documents the directive requested by the client.

Clients or courier service deliver samples to the laboratory during normal business hours. Sample receiving occurs in the sample management area.

11.1 Sample Receipt and Acceptance Policy

A sample is physical evidence collected from a facility or site Sample Login procedures detail sample control and the maintaining of sample integrity from receipt at the laboratory until disposal.

Samples arrive by common courier, e.g. UPS, Federal Express, Greyhound, Air Borne, or special courier or are delivered directly by the client All samples are routed through the Login area of the laboratory.

Samples arriving at the laboratory must be properly identified, logged in and assigned appropriate analyses Sample Chain of Custody procedure must be maintained during the receiving process. The COC document is designed to provide documentation that samples were received by and maintained. The COC is designed to pass pertinent information to the laboratory to ensure that samples are analyzed for the appropriate methods. The COC minimally contains client's name, contact information, client sample ID, date/time sampled, matrix, analytical testing requested, signature and date/time of relinquishment to the laboratory and signature and date/time of receipt by the laboratory.

The Login Coordinator (LC) will inspect the samples and record discrepancies encountered on the COC or Cooler Receipt Form or Noncompliance Report. The following items are inspected:

1) Condition of shipping container/cooler

2) Temperature of shipment

3) Condition and number of sample containers

4) Condition (including presence or absence) of custody seals on shipping containers

5) Sample container information

6) Test analysis requested

7) Sample chemical preservation

8) Chain of Custody correlation with sample received

11.1.1 Preservation and Container Verification

If temperature preservation is a required for the analyses requested, the cooler temperature must be monitored using a calibrated electronic or infrared thermometer. The temperature is taken by using the following procedures:

Temperature Blank

A container filled with DI water is provided specifically for the monitoring of temperature..

Note: LMP, Inc. recommends the use of a temperature blank. Sample kits provided by LMP, Inc. will contain a temperature blank.

Direct Monitoring

Direct insertion of the temperature probe in an area adjacent to the samples. (This method) generally applies to samples arriving preserved with Blue Ice type ice packs. LMP, Inc. does not recommend the use of Blue Ice

Samples arriving on crushed ice (not melted) are assumed to be at 4°C and is checked if a temperature blank is provided. Under no circumstances will the temperature probe be inserted into a sample.

Exception: The State of Arkansas requires that the temperature of all Bioassay samples be taken directly from the sample being analyzed.

Chemical Preservation

Sample labels are reviewed for proper preservation indication. Sample pH is verified at this time as an indication of proper chemical preservation unless prohibited by reference method

Containers

Proper container types are verified. Refer to Appendix Table 1 for approved containers and preservations

When samples are received outside the temperature preservation requirements of 4°C ± 2°C, or have been improperly preserved upon collection, or samples in improper improper containers, corrective action is initiated.

Holding Time Verification

a) The COC is reviewed to compare method holding times to sample dates. If samples are approaching holding times, the LC will issue RUSH requests via the current e-mail system to each laboratory to ensure that the samples are extracted/analyzed within method holding times.

b) If upon arrival to the laboratory a sample is found to be reaching its specified holding time and extraction/analysis may not be performed in time to meet this requirement, the client is contacted and a directive shall be taken. All correspondences and instructions are noted in a case narrative or corrective action.

c) Samples arriving outside of method specified holding times indicate a Noncompliance Report. Any directions by the client are documented.

d) Holding times are evaluated based on the following:

1) Holding times begin on the day of sample collection.

2) Holding times are measured in days, unless specified differently (e.g. hours) within the analytical method.

3) Samples that require a preparation step are said to be within holding time when the preparation is initiated within holding time.

4) Sample analyses are said to be within holding time when the analysis (introduction into the instrument), including dilutions, is initiated within holding time.

COC Verification

The Login Coordinator (LC) signs COC forms, as well as indicate date and time of sample receipt. Sample labels are compared to those listed on the COC. Any discrepancy will require the initiation of corrective action.

The analyses requested on the COC shall be verified by to the following:

a) Client specific job requirements

b) State specific analytical requirements

c) Method specific analytical requirements

If any discrepancy is identified, or if the LC has any doubt as to the analyses requested, clarification is requested from the client and documented on a corrective action or the client shall forward a revised chain of custody.

Noncompliance/Corrective Action/Client Contact

Login corrective action is designed to identify problems or potential problems that may result in noncompliant analytical data The overall objective is to ensure that the client is provided with information that will allow evaluation as to the affect on the quality of the associated analytical data. Projects require a Cooler Receipt Form (CRF), which details observances when the cooler arrives at the laboratory. If generated, the CRF accompanies the final report.

LMP, Inc. utilizes Laboratory Project Managers (LPM) and Client Service Representatives (CSR). The LC shall inform the LPM or the CSR of any noncompliance and shall direct them to contact the client. However, the LC has the authority to contact the client directly should time be of the essence. When a potentially non-compliant situation arises or questions arise as to the requests made on the COC, the login process does not proceed without notifying the client.

a) When contact is made with the client, the LC, LPM or CSR records their conversation in the customer log. At this time, the situation is discussed and options presented to the client.

b) Changes or modifications resulting from this conversation to the original COC request must be done so in writing (fax or e-mail). Likewise, changes made to the original COC are initialed by the LC. These changes are also recorded in the LIMS.

c) At the discretion of the LC, LPM or CSR, receipt of the client's written request may be required prior to the commencement of work.

d) If the client is being notified of a non-compliance situation, they will be given the option to re-sample and re-submit the work. The client may also choose to proceed with the analysis with the understanding that a Non-Compliance Report will accompany the final report.

It is possible for samples or sample containers to be lost, damaged, or determined to be unsuitable, for whatever reason, after initial receipt at Laboratory Management Partners, Inc. Should this happen, the event is recorded on a Sample Casualty Report Form by the observer. The problem is brought to the attention of the Environmental Services Manager who reports it to the client. Plans for disposition of the affected samples are agreed upon with the client and recorded in the Sample Casualty Report

11.1 2 Sample Login

Once verification procedures have been completed and any necessary corrective action implemented, the samples are logged into the current Laboratory Information Management System (LIMS). Samples received by LMP, Inc. are assigned an unique identifier.

11.2 Sample Tracking and Storage

The entire building at LMP, Inc. is maintained as a secure area. Once login is complete, samples are distributed to the proper storage area. All refrigerated storage area temperatures are monitored daily by the QAO or a designate.

Samples are stored in the central walk-in refrigerator until preparation or analysis. Samples are separated by department (e.g., inorganic, organic) and date arrived.

Samples for same day testing (e.g. chlorine, fecal coliform, BOD) are analyzed on the same day that they arrive. These samples are transferred directly to the inorganic laboratory for immediate processing

Samples designated for VOC analyses are transferred directly to the VOC laboratory refrigerator regardless of any other analyses being performed. Samples leave the volatile laboratory only after the VOC analyses are complete.

All samples for metals analyses are transferred to the metal's digestion area. Samples for metals analyses do not require refrigerated storage (Chromium VI requires refrigeration and is treated as an inorganic same day test.)

Samples for Microbiology and Bioassay testing are stored in a refrigerator within the Microbiology laboratory.

Samples classified as RUSH analyses are transferred directly to the appropriate laboratory for immediate processing.

Extracts from organic extractions are stored in a dedicated refrigerator. Samples are removed from the storage areas as needed by each department. Extracts are transferred for analysis directly to organic instrument laboratory as needed.

LMP, Inc. maintains records documenting all phases of sample handling from sample receipt to final analysis. NELAC specifies two levels of sample handling: sample tracking and legal chain of custody protocols, which are used for evidentiary or legal purposes.

LMP, Inc 's document control system allows historical reconstruction of all laboratory activities that produced the analytical data. The history of the sample can be readily tracked through the documentation.

Laboratory Management Partners, Inc. supplies laboratory pure water for field QC blanks. Water used for volatile organics must be free of volatile compounds below the method detection limit.

The quality of the laboratory water is monitored for resistivity once per day. Additional water quality criteria are monitored based on client specific requests.

Sample management personnel remove samples to the sample storage area after analysis and when data is correct and complete. Sample coolers are removed to a designated storage area for recycling. Samples are stored in the designated process storage areas until testing is complete. Sample removal from the process storage occurs after mailing of the final report. The sample management staff places the samples in the archive storage area for thirty days after report release. Upon written client request samples are held for up to six months in an uncontrolled area. Requests for controlled sample storage or extended sample storage periods must be arranged at the time of contractual commitment. Based on EPA's specifications, samples are properly disposed or returned to the client.

11.3 Sample Kits

Sample containers provided by Laboratory Management Partners, Inc. include labels, preservatives and a blank Chain of Custody Form. The sampling supplies sent to the client are documented on the Sampling Kit Form and provide for traceability back to the original certificate associated with the container lots Chain of Custody Forms accompanies all samples received by laboratory personnel The Chain of Custody Form indicates the sample origin and arrival at the laboratory and identifies the analyses requested.

EPA methods require that samples be collected in specific type containers. Some methods require additional chemical and/or temperature preservation to ensure that sample integrity is maintained from sampling through arrival at the laboratory.

Based on project requirements, LMP, Inc. will provide sample kits designed for each specific sampling event. At a minimum, sample kits will contain the following items:

a) Pre-preserved containers are color-coded and types of preservations are indicated.

b) Temperature Blank

c) Trip Blank (by request)

d) Coolers

e) Chain-of-Custody (COC) documents

f) Sample Labels

g) Sampling Instructions

h) Sample Kit Request Form to summarize the provided containers and appropriate analysis.

i) Shipping/Packing Materials

Sample kits are assembled and tracked by the shipping department. Vendor's analytical certificates for bottles that are provided by LMP, Inc. are maintained. Reagent Reference Numbers of chemicals used as preservatives are recorded.

11.4 Sample Aliquots

In the scheme of the sampling process, an aliquot of a submitted sample may be required in any part of analytical phase. The laboratory uses documented and appropriate procedures and techniques to obtain representative sub-samples. Sample aliquots removed for analysis are homogenized and representative portions removed from the sample container. Evidence of sub sampling is documented and recorded on the test method worksheets.

11.5 Waste Minimization and Disposal

11.5.1 Waste Management

The Laboratory's Waste Management Plan has established a "cradle to grave" system for the disposal of waste, whether hazardous or non-hazardous, generated from the daily process activities at this facility.

LMP, Inc. actively practices the following waste management and pollution control procedures:

a) Reducing the quantity of waste generated

b) Reducing the amount of sample material used for analysis/preparation

c) Offer to an approved TSDF for incinerating, fuel blending, or land filling

d) Separating hazardous waste from non-hazardous waste in order to better provide a proper route of disposal.

11.5.2 EPA Identification Number

As LMP is a small quantity generator, SQG, and has obtained an EPA Identification number as mandated by 40CFR 263.11. Without this EPA ID number, this facility would be barred from storing, transporting, or offering for transportation, treating and disposing of any hazardous waste. Likewise, as a generator, we are barred from offering any hazardous waste to any transporter or TSD facility that does not have an EPA ID number Presently, the QAO is responsible for ensuring that the waste management plan is followed and the applicable RCRA regulations are fulfilled. The laboratory is responsible for assuring that all LMP employees are trained in the laboratory requirements for waste management and that all procedures are performed in accordance with the rules for safely handling lab materials.

11.5.3 Lab Packing

LMP's overall procedure for waste management is the lab packing of waste sample retains. Because of the various wastes that are generated, LMP has established waste profiles. These profiles have been verified by the TSDF and are active which is discussed in the Waste Management Program.

11.5.4 Documentation Required for Disposal

For the proper disposal of waste, EPA requires several documents These documents and their relevance are summarized in the following sections:

Uniform Waste Manifest Form

US EPA Form 8700-22A is required of generators, transporters of hazardous waste and owners/operators of hazardous waste treatment, storage and disposal facilities for both interstate and intrastate transportation.

Land Ban Forms

The Land Disposal Restriction/Land Ban Forms A/B is notification of restrictions of land disposal for certain hazardous waste. Both the Generator and TSDF are affected by such restrictions.

Placarding and Labeling

To transport hazardous waste off-site, this facility must ensure proper packaging, labeling, marking and placarding of the packaged waste (40 CFR 262.30-.33) Labeling, marking and placarding of the packaged waste is done to inform the shipping crew,

firefighter and emergency responders about the characteristics and dangers associated with the waste being transported. This information is vital in case of an emergency situation. No waste leaves LMP without the proper labeling. For this purpose, the following labels and placards are required:

a) Hazardous Waste Identification Label

b) Domestic Label /Placard

c) PCB Caution Waste Notification Label (PCB waste only)

Records, Reporting and Recordkeeping

LMP maintains records for all its waste management activities in order to demonstrate cradle to grave disposal. The documents used at this facility indicate the process of traceability. The following procedure and associated logbooks are used in the lab-packing activities utilized at this facility:

a) Lab Packing Logbooks- Per Profile

b) Manifested Drum Inventory Log

c) Manifest Logs to record individual shipments:

1) Manifest Summary Sheets

2) Uniform Manifest Forms

3) Land Ban Forms A and B

4) Certificate of Disposals

These records allow the step-by-step process that traces individual samples/solvent waste to a specific drum These drums in turn are manifested and disposed by an approved TSDF.

12 Analytical Data Review and Validation

12.1 Level 1 - Analyst Review

Each analyst reviews the quality of his/her work based on a set of guidelines established in each method SOP or in this guidance. This review, at a minimum, covers the following:

a) Sample preparation information is correct and complete.

b) Analysis information is correct and complete.

c) The appropriate SOPs have been followed.

d) Analytical results are correct and complete.

e) Raw data, including all manual integrations, have been correctly interpreted and flagged.

f) QC samples are within established control limits.

g) Any special sample preparation and analytical requirements have been met.

h) Data transfers were verified.

i) Documentation is complete (e.g., all anomalies in the preparation and analysis have been

j) Review shall be documented by using a checklist or worksheet and by the signature of the reviewer and date.

12.2 Level 2 – Supervisor/Peer Review

Level 2 reviews are performed by a supervisor, another analyst, or data review specialist who has documentation that supports demonstration of performance for all areas for which he/she provides review.

The function of this review is to provide an independent, complete peer review of the analytical batch data package. This review shall also be conducted according to a set of guidelines established in each method SOP or in this guidance. This review is structured to ensure the following:

a) All appropriate laboratory SOPs have been referenced (checklist or worksheet)

b) Calibration data are scientifically sound, appropriate to the method, and completely documented

c) QC samples are within established guidelines

d) Qualitative identification of sample components is correct

e) Quantitative results, including calculations and any associated flags, are correct

f) Raw data, including manual integrations, have been correctly interpreted and flagged

g) Documentation is complete and correct (e.g., anomalies in the preparation and analysis have been documented, nonconformance forms are complete, holding times are documented, etc.)

h) Analytical data is ready for transfer to the LIMS

12.3 Level 3 – Project Manager Review

Level 3 reviews are performed by the laboratory project manager or designate. This review provides a total overview of the data package, including sample receipt, to ensure its consistency and compliance with project-specific requirements. Level 3 reviews are documented with the signature of the reviewer and date.

12.4 QA Review

QA review is performed by the QA Officer or designate. This review is not part of the normal production data review process. The QA Officer typically reviews at least 10 percent of the data produced by the laboratory using the procedures as outlined in the Level 3 data reviews. Additional technical details may be reviewed in this QA review, similar to Levels 1 and 2, along with a total package review, i.e , correlation of results from differing but related chemical parameters. Typically, the QA Officer selects the data packages reviewed at random. Nonconformance and/or corrective action reports would be required for any errors noted.

13 Laboratory Report Format and Contents

The Process Planning and Control Procedure details the recording and reporting of data as required by the client and in accordance with relevant environmental regulations. The results of each test or series of tests carried out by the laboratory shall be reported accurately, clearly, unambiguously and objectively. The results are reported in a test report and include all the information necessary for the interpretation of the test results and all information required by the method used. Clients specify the report delivery and deliverables required for the work submitted. Report delivery includes standard turnaround and rush turnaround. Clients specify the delivery address or multiple addresses and method of delivery such as U.S. Mail, facsimile or electronic at the start of the project. Laboratory Management Partners, Inc. provides data deliverables in hardcopy or electronic format. At the start of any project, the electronic deliverable formats required must be received before sample arrival.

The final report data package contains the required and relevant information to demonstrate that the project DQOs have been fulfilled.

Refer to Appendix Table 2 for a list of LMP's data packages.

All reported data packages are retained by the laboratory for a minimum of five (5) years, or as dictated by project requirements (if longer than five years).

Reporting packages are available for routine regulatory reporting requirements. Regulatory reporting packages include only the information requested by the regulatory agency. In additional to regulatory report packages, LMP, Inc. prepares a standard report format. The standard report format includes:

1) a title, "Report of Analysis";

2) name and address of laboratory, and location where the test was carried out if different from the address of the laboratory and phone number with name of contact person for questions;

3) unique report number (a serial number) and of each page, and the total number of pages;

4) name and address of client and project name, if applicable;

5) description and unambiguous identification of the tested sample including the client identification code;

6) identification of test results derived from any sample that did not meet sample acceptance requirements such as improper container, holding time, or temperature are flagged;

7) date of receipt of sample, date and time of sample collection, date(s) of performance of test, and time of sample preparation and/or analysis;

8) identification of the test method used, or unambiguous description of any non-standard method used;

9) if the laboratory collected the sample, reference to sampling procedure;

10) any deviations from (such as failed quality control), additions to or exclusions from the test method (such as environmental conditions), and any non-standard conditions that have an affected the quality of results, and including the use and definitions of data qualifiers are flagged,

11) measurements, examinations and derived results, supported by tables, graphs, sketches and photographs as appropriate, and any failures identified; identify whether data are calculated on a dry weight or wet weight basis; identify the reporting units;

12) Data qualifiers are added by the laboratory during the data generation/ review process. These qualifiers are applied when data quality objectives are not met. All flags used are defined completely within the final data report packages.

13) a statement of the estimated uncertainty of the test result, is not required for chemical analysis at the present time; The laboratory currently monitors performance based on the LCS recovery limits,

14) a signature and title, or an equivalent electronic identification of the person(s) accepting responsibility for the content of the certificate or report, and date of issue;

16) a statement to the effect that the results relate only to the items tested or to the sample as received by the laboratory;

16) a statement that the certificate or report shall not be reproduced except in full, without the written approval of the laboratory,

17) clear identification of all test data provided by outside sources, such as subcontracted laboratories, clients, etc;

18) clear identification of any numerical results with values outside of quantitation limits;

19) The laboratory shall provide certification that the tests results meet NELAC standards or appropriate regulation. Modifications to regulatory requirements are documented.

After issuance of the report, the report shall remain unchanged. Amendments to an analytical report after issue shall be made in the form of amended document.

Results transmitted by facsimile or other electronic means include a statement of confidentiality.

The laboratory notifies the client in writing of any circumstance that casts doubt on the validity of the results. The amended or modified report lists the change, reason for the change, affected page numbers, date of the amendment and authorized signature.

14 Outside Support Services and Supplies

When Laboratory Management Partners, Inc. purchases outside services and supplies in support of tests, the laboratory uses only those outside services and supplies that are of adequate quality to maintain confidence in the tests.

The laboratory reviews suppliers and services for quality of the materials and supplies. Where no independent assurance of the quality of outside support services or supplies are available, the laboratory ensures that purchased equipment, materials, and services comply with specifications by evaluating method performance before routine use.

The laboratory checks shipments upon receipt as complying with purchase specifications. The use of purchased equipment and consumables is only after the evaluation and compliance to the specifications is complete. The laboratory supervisors purchase supplies and materials based on the quality specified in the laboratory technical SOPs. The laboratory purchasing procedure defines the process for documenting purchases and receiving materials and supplies.

Purchases from suppliers must be approved by the General Manager. The laboratory maintains records of all suppliers and subcontractors from whom it obtains support services or supplies required for tests

14.1 Subcontracting

Analytical tests not routinely performed by LMP, Inc. are subcontracted. In such cases, the sample(s) are sent to a laboratory that meets client and regulatory requirements. LMP, Inc 's policy is to advise the client of intention to subcontract any portion of the testing to another party. The laboratory approves testing and sampling subcontractors by review of current state, national or other external parties' certifications or approvals The laboratory maintains subcontractor current certification or approval documents. These records are kept to indicate current approval for the subcontracted work. Subcontracting laboratories must maintain validations, certifications or approvals commiserate with the type or work being sub-contracted (e.g., NELAC, USACE). For the parameters accredited by NELAC, the laboratory must use a NELAC accredited laboratory, unless the parameters are clearly denoted as not meeting NELAC requirements and the client specifies the subcontractor in writing.

The process for sample handling when subcontracting samples must include the use of a Chain of Custody Form. The client must be notified of subcontracted work and approval is in writing before releasing samples to the subcontractor.

The Environmental Services Coordinator and Technical Director review the subcontractor documents for completeness and meeting the specifications defined for the project. Documented report reviews and any requested corrections are written on the Corrective Action Report Form.

The laboratory performing the subcontracted analysis is clearly indicated in the final report. Additionally, the laboratory shall be clearly identified as either NELAC or non-NELAC accredited.

15 Client Relations

15.1 Client Services

Majority of the client services occur from personnel in the administration and laboratory services areas. Client Service department acts as the liaison between the laboratory and client. The client service goal is to ensure that the client's expectations are met on a consistent basis. The client service department acts as the client's representative within the laboratory to ensure that adequate priority is assigned to each project. Client service/marketing involves inquiries into services offered, technical consulting, placing orders, receiving orders, providing updates on the status of orders and completing orders. Client service representatives (CSRs) offer the client a single source within the laboratory for the following types of activities or requests:

a) Current status on in-house samples/projects

b) Support for questions and inquiries relating to analytical results

c) Support for questions related to Quality Control issues

d) Verification of analytical regulatory requirements (e.g UST, wastewater)

e) Request for sample kits

f) Customer complaints resolution

g) Sample tracking and custody procedures

h) Sample containers – containers, preservation and holding time

i) Field QC sample requirements

j) General sampling procedures

k) Focal point for issuing directives regarding the project

Personnel interacting with clients must document and review client specific project requirements. Personnel must document client interactions following the appropriate laboratory procedures in the LIMS automated system. Each person must communicate deviations, modifications and client requests to the Technical Director. The CSR operates under the premise that client's needs come first. CSR's work closely with Project Managers and report directly to the CEO.

The reference method applied for sample analysis is usually based on the regulatory program. The Technical Director and/or Environmental Services Manager may assist the client with method selection when the client specifies the regulatory program. The Technical Director recommends methods for regulatory programs. In all cases, recommendation of methods is based on client-defined method performance criteria as long as the client specifications are more stringent than the reference method criteria. The Environmental Services Manager handles the process for inquiry receipt and actions taken of inquiries, processing sales orders and process for requesting sample containers.

15.2 Project Management

The laboratory management reviews requests for new work during staff meetings. The Technical Director addresses all capacity and capability issues. Where conflicts in workload arise, client notification is immediate. The Project Information Form contains the documentation of all project information. Cooperation between laboratory and client services staff allows direct communication

and scheduling with the client. Management arranges scheduling and coordination between departmental areas.

The Project Manager documents all clients' complaints or concerns regarding data quality or laboratory operations. The Corrective Action Report records complaints and laboratory correction action with the client. The process uses the same form and process as the corrective action process. Customer service representatives log all customer complaints in LIMS Customer Log screen. The customer log program in LIMS records customer requests, concerns, and complaints. In the event that a complaint or concern is filed, a systemic approach is conducted in order to identify, investigate and ultimately rectify when applicable. Because the nature of this facility is in producing analytical data, the majority of complaints or concerns are directly or indirectly related to client analytical results. This may include any associated quality control or quality assurance issues. In the scope of this the following procedure is undertaken in order to address any complaint:

Identification of complaint;

Nature of complaint;

Investigate procedures for retrieval of data;

Investigate procedures for data reviewed materials;

Investigate procedures for review process; and

Investigate report attached to a corrective action.

16 Safety Program

Laboratory Management Partners, Inc. is totally committed to employee safety and loss control. It is this company's intention that:

a) All employees work under the safest conditions possible with a proactive attitude toward safety

b) A workplace and equipment, which are free from recognized hazards, are provided

c) Information, training, and supervision are offered to enable employees to perform their jobs safely

The Laboratory Safety Plan states basic safety rules and procedures that are to be followed by all company employees While this plan will help the employee recognize and avoid obvious hazards, it cannot possibly cover all situations. When in doubt, consult the supervisor or Safety Officer for guidance LMP, Inc. endeavors to comply with safety regulations implemented by federal, state, and local agencies. It is company policy that every employee and all property be protected from controllable hazards. This laboratory believes that accidents can be avoided using good training methods, common sense, and personal initiative. Therefore, each employee is responsible for complying with all safety regulations

16.1 Chemical Hygiene Program

The working and storage environments are maintained in a safe and appropriate manner. The Chemical Hygiene Plan details the requirements for working and handling chemicals and reagents. This plan assists the employees in the safe use of chemicals and reagents. Material Safety Data Sheets are available to employees and authorized safety officers for review.

17 Appendix A – Acronyms and Definitions

17.1 Acronyms

AE	Automatic Entry
ASQ	American Society for Quality
ARAR	Applicable or Relevant and Appropriate Requirements
CBI	Confidential Business Information
CCV	Continuing Calibration Verification
CERCLA	Comprehensive Environmental Response, Compensation, and Liability Act
CFR	Code of Federal Regulations
CLP	Contract Laboratory Program
COC	Chain of Custody
CRDL	Contract Required Detection Limit
CRQL	Contract Required Quantitation Limit
CRM	Certified Reference Material
CSR	Client Service Representives
CV	Coefficient of Variation
CVAA	Cold Vapor Atomic Absorption
CWA	Clean Water Act
DQA	Data Quality Assessment
DQO	Data Quality Objective
EDL	Environmental Detection Limit
EPA	Environmental Protection Agency
GC	Gas Chromatograph
GC-MS	Gas Chromatography - Mass Spectrometer (interfaced together)
GPC	Gel Permeation Chromatography
GFAA	Graphite Furnace Atomic Absorption (Spectroscopy)
HPLC	High Performance Liquid Chromatography
IC	Ion Chromotography
ICB	Initial Calibration Blank
ICP	Inductively Coupled Plasma (Atomic Emission Spectroscopy)
ICP/MS	Inductively Coupled Plasma/Mass Spectrometry
ICV	Initial Calibration Verification
IR	Installation Restoration
ISO	International Standards Organization
LB	Laboratory Blank
LC	Liquid Chromatograph
LCS	Laboratory Control Sample
LCSD	Laboratory Control Sample Duplicate
LD	Analytical Detection Limit
LIMS	Laboratory Information Management System
MDL	Method Detection Limit
MQL	Method Quantitation Limit
MRL	Method Reporting Limit
MS	Matrix Spike
MSD	Matrix Spike Duplicate
NELAC	National Environmental Laboratory Accreditation Conference
NIST	National Institute of Standards and Technology
OSHA	Occupational Safety and Health Administration
PDS	Post Digestion Spike

PQL	Practical Quantitation Limit
PT	Proficiency Testing
PBMS	Performance Based Measurement System
QA	Quality Assurance
QMS	Quality Management System
QC	Quality Control
R	Recovery
RCRA	Resource Conservation and Recovery Act
RL	Reporting Limit
RSD	Relative Standard Deviation
S	Standard Deviation
SAP	Sampling and Analysis Plan
SARA	Superfund Amendments and Reauthorization Act
SDWA	Safe Drinking Water Act
SOP	Standard Operating Procedure
SOW	Statement of Work
SQL	Sample Quantitation Limit
TCLP	Toxicity Characteristic Leaching Procedure
TIC	Tentatively Identified Compounds
TSDF	Treatment Storage and Disposal Facility
USCOE	U. S. Army Corps of Engineers
USDA	U. S. Department of Agriculture
UST	Underground Storage Tanks
WP	Water Pollution
WS	Water Supply

17.2 Definitions of Terms

The definitions presented here have been compiled using the following reference materials: definitions developed by the National Environmental Laboratory Accreditation Conference (NELAC- May 25, 2001). The source of each definition is noted. When more than one source is indicated the original source document is the first reference presented.

Terms may have more than one definition due to the multiple documents used for project planning. Each project should define the term as used for the site specific project.

Acceptance Criteria: specified limits placed on characteristics of an item, process, or service defined in requirement documents. (ASQC) (NELAC)

Accreditation: the process by which an agency or organization evaluates and recognizes a laboratory as meeting certain predetermined qualifications or standards, thereby accrediting the laboratory. In the context of the National Environmental Laboratory Accreditation Program (NELAP), this process is a voluntary one. (NELAC)

Accrediting Authority: the Territorial, State, or federal agency having responsibility and accountability for environmental laboratory accreditation and which grants accreditation (NELAC)[1.5.2.3]

Accrediting Authority Review Board (AARB): five voting members from Federal and State Accrediting Authorities and one non-voting member from USEPA, appointed by the NELAP Director, in consultation with the NELAC Board of Directors, for the purposes stated in 1.6.3.e. (NELAC) [1.6.3.]

Accuracy: the degree of agreement between an observed value and an accepted reference value. Accuracy includes a combination of random error (precision) and systematic error (bias) components which are due to sampling and analytical operations, a data quality indicator. (QAMS)

Assessor Body: the organization that actually executes the accreditation process, i e., receives and reviews accreditation applications, reviews QA documents, reviews proficiency testing results, performs on-site assessments, etc., whether EPA, the State, or contracted private party.(NELAC)

Analyst: the designated individual who performs the "hands-on" analytical methods and associated techniques and who is the one responsible for applying required laboratory practices and other pertinent quality controls to meet the required level of quality. (NELAC)

Applicant Laboratory or **Applicant:** the laboratory or organization applying for NELAP accreditation. (NELAC)

Assessment: the evaluation process used to measure or establish the performance, effectiveness, and conformance of an organization and/or its systems to defined criteria (to the standards and requirements of NELAC). (NELAC)

Assessment Criteria: the measures established by NELAC and applied in establishing the extent to which an applicant is in conformance with NELAC requirements. (NELAC)

Assessment Team: the group of people authorized to perform the on-site inspection and proficiency testing data evaluation required to establish whether an applicant meets the criteria for NELAP accreditation. (NELAC)

Assessor: one who performs on-site assessments of accrediting authorities and laboratories' capability and capacity for meeting NELAC requirements by examining the records and other physical evidence for each one of the tests for which accreditation has been requested. (NELAC)

Audit: a systematic evaluation to determine the conformance to quantitative *and qualitative* specifications of some operational function or activity. (EPA-QAD)

Batch: environmental samples that are prepared and/or analyzed together with the same process and personnel, using the same lot(s) of reagents. A **preparation batch** is composed of one to 20 environmental samples of the same NELAC-defined matrix, meeting the above mentioned criteria and with a maximum time between the start of processing of the first and last sample in the batch to be 24 hours. An **analytical batch** is composed of prepared environmental samples (extracts, digestates or concentrates), which are analyzed together as a group. An analytical batch can include prepared samples originating from various environmental matrices and can exceed 20 samples. (NELAC Quality Systems Committee)

Blank: a sample that has not been exposed to the analyzed sample stream in order to monitor contamination during sampling, transport, storage or analysis. The blank is subjected to the usual analytical and measurement process to establish a zero baseline or background value and is sometimes used to adjust or correct routine analytical results. Blanks include:

> **Equipment Blank:** a sample of analyte-free media which has been used to rinse common sampling equipment to check effectiveness of decontamination procedures. (NELAC)
>
> **Field Blank:** blank prepared in the field by filling a clean container with pure de-ionized water and appropriate preservative, if any, for the specific sampling activity being undertaken. (EPA-OSWER)
>
> **Instrument Blank:** a clean sample (e.g , distilled water) processed through the instrumental steps of the measurement process; used to determine instrument contamination. (EPA-QAD)
>
> **Laboratory Blank:** a sample of a matrix similar to the batch of associated samples (when available) that is free from the analytes of interest and is processed simultaneously with and under the same conditions as samples through all steps of the analytical procedures, and in which no target analytes or interferences are present at concentrations that impact the analytical results for sample analyses. (NELAC)
>
> **Reagent Blank:** (method reagent blank)· a sample consisting of reagent(s), without the target analyte or sample matrix, introduced into the analytical procedure at the appropriate point and carried through all subsequent steps to determine the contribution of the reagents and of the involved analytical steps. (QAMS)

Blind Sample: a sub-sample for analysis with a composition known to the submitter. The analyst/laboratory may know the identity of the sample but not its composition. It is used to test the analyst's or laboratory's proficiency in the execution of the measurement process. (NELAC)

Calibration: to determine, by measurement or comparison with a standard, the correct value of each scale reading on a meter, instrument, or other device. The levels of the applied calibration standard should bracket the range of planned or expected sample measurements. (NELAC)

Calibration Curve: the graphical relationship between the known values, such as concentrations, of a series of calibration standards and their instrument response. (NELAC)

Calibration Method: a defined technical procedure for performing a calibration. (NELAC)

Calibration Standard: a substance or reference material used to calibrate an instrument. (QAMS)

Certified Reference Material (CRM): a reference material one or more of whose property values are certified by a technically valid procedure, accompanied by or traceable to a certificate or other documentation which is issued by a certifying body. (ISO Guide 30 - 2.2)

Chain of Custody Form: record that documents the possession of the samples from the time of collection to receipt in the laboratory. This record generally includes: the number and types of containers; the mode of collection; the collector; time of collection; preservation; and requested analyses. (NELAC)

Clean Air Act: the enabling legislation in 42 U.S.C. 7401 *et seq.*, Public Law 91-604, 84 Stat. 1676 Pub. L. 95-95, 91 Stat., 685 and Pub. L. 95-190, 91 Stat., 1399, as amended, empowering EPA to promulgate air quality standards, monitor and to enforce them. (NELAC)

Comprehensive Environmental Response, Compensation and Liability Act (CERCLA/Superfund): the enabling legislation in 42 U.S.C. 9601-9675 *et seq.*, as amended by the Superfund Amendments and Reauthorization Act of 1986 (SARA), 42 U.S.C. 9601*et seq.*, to eliminate the health and environmental threats posed by hazardous waste sites. (NELAC)

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. NELAC and its representatives agree to safeguarding identified CBI and to maintain all information identified as such in full confidentiality.

Confirmation: verification of the identity of a component through the use of an approach with a different scientific principle from the original method. These may include, but are not limited to:

Second column confirmation

Alternate wavelength

Derivatization

Mass spectral interpretation

Alternative detectors or

Additional cleanup procedures. (NELAC)

Conformance: an affirmative indication or judgement that a product or service has met the requirements of the relevant specifications, contract, or regulation; also the state of meeting the requirements. (ANSI/ASQC E4-1994) (NELAC)

Contributor: a participant in NELAC who is not a Voting Member. Contributors include representatives of laboratories, manufacturers, industry, business, consumers, academia, laboratory associations, laboratory accreditation associations, counties, municipalities, and other political subdivisions, other federal officials not engaged in environmental activities, and other persons who are interested in the objectives and activities of NELAC. (NELAC)[Art III, Const]

Corrective Action: the action taken to eliminate the causes of an existing nonconformity, defect or other undesirable situation in order to prevent recurrence. (ISO 8402)

Data Audit: a qualitative and quantitative evaluation of the documentation and procedures associated with environmental measurements to verify that the resulting data are of acceptable quality (i.e., that they meet specified acceptance criteria). (NELAC)

Data Reduction: the process of transforming raw data by arithmetic or statistical calculations, standard curves, concentration factors, etc., and collation into a more useable form. (EPA-QAD)

Deficiency: an unauthorized deviation from acceptable procedures or practices, or a defect in an item. (ASQC)

Delegate: any environmental official of the States or the Federal government not sitting in the House of Representatives, who is eligible to vote in the House of Delegates. (NELAC)

Demonstration of Capability: a procedure to establish the ability of the analyst to generate acceptable accuracy. (NELAC)

Denial: to refuse to accredit in total or in part a laboratory applying for initial accreditation or resubmission of initial application. (NELAC)[4.4.1]

Detection Limit: the lowest concentration or amount of the target analyte that can be identified, measured, and reported with confidence that the analyte concentration is not a false positive value. See Method Detection Limit. (NELAC)

Document Control: the act of ensuring that documents (and revisions thereto) are proposed, reviewed for accuracy, approved for release by authorized personnel, distributed properly and controlled to ensure use of the correct version at the location where the prescribed activity is performed. (ASQC)

Environmental Laboratory Advisory Board (ELAB): a Federal Advisory Committee, with members appointed by EPA and composed of a balance of non-state, non-federal representatives, from the environmental laboratory community, and chaired by an ELAB member. (NELAC)[1.6.2]

Environmental Monitoring Management Council (EMMC): an EPA Committee consisting of EPA managers and scientists, organized into a Policy Council, a Steering Group, *ad hoc* Panels, and work groups addressing specific objectives, established to address EPA-wide monitoring issues. (NELAC)

Federal Insecticide, Fungicide and Rodenticide Act (FIFRA): the enabling legislation under 7 U.S.C. 135 *et seq.*, as amended, that empowers the EPA to register insecticides, fungicides, and rodenticides. (NELAC)

Federal Water Pollution Control Act (Clean Water Act, CWA): the enabling legislation under 33 U.S.C. 1251 *et seq.*, Public Law 92-50086 Stat. 816, that empowers EPA to set discharge limitations, write discharge permits, monitor, and bring enforcement action for non-compliance. (NELAC)

Field of Accreditation: (previously Field of Testing) NELAC's approach to accrediting laboratories by matrix, technology/method and analyte/analyte group. Laboratories requesting accreditation for a matrix-technology/method –analyte/analyte group combination or for an updated/improved method are required to submit only that portion of the accreditation process not previously addressed (see NELAC, section 1.8 ff). (NELAC)

Field of Proficiency Testing: NELAC's approach to offering proficiency testing by matrix, technology, and analyte/analyte group.

Finding: an assessment conclusion that identifies a condition having a significant effect on an item or activity. An assessment finding is normally a deficiency and is normally accompanied by specific examples of the observed condition (NELAC)

Governmental Laboratory: as used in these standards, a laboratory owned by a Federal, state, or tribal government; includes government-owned contractor-operated laboratories. (NELAC).

Holding Times (Maximum Allowable Holding Times): the maximum times that samples may be held prior to analysis and still be considered valid or not compromised. (40 CFR Part 136)

Inspection: an activity such as measuring, examining, testing, or gauging one or more characteristics of an entity and comparing the results with specified requirements in order to establish whether conformance is achieved for each characteristic. (ANSI/ASQC E4-1994)

Interim Accreditation: temporary accreditation status for a laboratory that has met all accreditation criteria except for a pending on-site assessment which has been delayed for reasons beyond the control of the laboratory. (NELAC)

Internal Standard: a known amount of standard added to a test portion of a sample as a reference for evaluating and controlling the precision and bias of the applied analytical method. (NELAC)

Laboratory: a body that calibrates and/or tests. (ISO 25)

Laboratory Control Sample (however named, such as laboratory fortified blank, spiked blank, or QC check sample): a sample matrix, free from the analytes of interest, spiked with verified known amounts of analytes or a material containing known and verified amounts of analytes. It is generally used to establish intra-laboratory or analyst specific precision and bias or to assess the performance of all or a portion of the measurement system. (NELAC)

Laboratory Duplicate: aliquots of a sample taken from the same container under laboratory conditions and processed and analyzed independently. (NELAC)

Legal Chain of Custody Protocols: procedures employed to record the possession of samples from the time of sampling until analysis and are performed at the special request of the client. These protocols include the use of a Chain of Custody Form that documents the collection, transport, and receipt of compliance samples by the laboratory. **In addition, these protocols document all handling of the samples within the laboratory.** (NELAC)

Manager (however named): the individual designated as being responsible for the overall operation, all personnel, and the physical plant of the environmental laboratory. A supervisor may report to the manager. In some cases, the supervisor and the manager may be the same individual. (NELAC)

Matrix: the substrate of a test sample.

Field of Accreditation Matrix: these matrix definitions shall be used when accrediting a laboratory (see Field of Accreditation).

Drinking Water: any aqueous sample that has been designated a potable or potential potable water source

Non-Potable Water: any aqueous sample excluded from the definition of Drinking Water matrix. Includes surface water, groundwater, effluents, water treatment chemicals, and TCLP or other extracts.

Solid and Chemical Materials: Includes soils, sediments, sludges, products and by-products or an industrial process that results in a matrix not previously defined.

Biological Tissue: any sample of a biological origin such as fish tissue, shellfish, or plant material. Such samples shall be grouped according to origin.

Air and Emissions: whole gas or vapor samples including those contained in flexible or rigid wall containers and the extracted concentrated analytes of interest from a gas or vapor that are collected with a sorbent tube, impinger solution, filter, or other device (NELAC)

Quality System Matrix: These matrix definitions are an expansion of the field of Accreditation matrices and shall be used for purposes of batch and quality control requirements (see Appendix D of Chapter 5). These matrix distinctions shall be used:

Aqueous: any aqueous sample excluded from the definition of Drinking Water matrix of Saline/Esturarine source. Includes surface water, ground water effluents, and TCLP or other extracts.

Drinking Water: any aqueous sample that has been designated a potable or potential potable water source

Saline/Estuarine: any aqueous sample from an ocean or estuary, or other salt-water source such as the Great Salt Lake.

Non-aqueous Liquid: any organic liquid with <15% settleable solids.

Biological Tissue: any sample of a biological origin such as fish tissue, shellfish, or plant material. Such samples shall be grouped according to origin.

Solids: includes soils, sediments, sludges and other matrices with >15% settleable solids.

Chemical Waste: a product or by-product of an industrial process that results in a matrix not previously defined

Air and Emissions: whole gas or vapor samples including those contained in flexible or rigid wall containers and the extracted concentrated analytes of interest from a gas or vapor that are collected with a sorbant tube, impinger solution, filter, or other device. (NELAC)

Matrix Spike (spiked sample or fortified sample): a sample prepared by adding a known mass of target analyte to a specified amount of matrix sample for which an independent estimate of Target analyte concentration is available. Matrix spikes are used, for example, to determine the effect of the matrix on a method's recovery efficiency. (QAMS)

Matrix Spike Duplicate (spiked sample or fortified sample duplicate): a second replicate matrix spike prepared in the laboratory and analyzed to obtain a measure of the precision of the recovery for each analyte. (QAMS)

May: denotes permitted action, but not required action (NELAC)

Method Detection Limit: the minimum concentration of a substance (an analyte) that can be measured and reported with 99% confidence that the analyte concentration is greater than zero and is determined from analysis of a sample in a given matrix containing the analyte. (40 CFR Part 136, Appendix B)

Must: denotes a requirement that must be met. (Random House College Dictionary)

National Accreditation Database: the publicly accessible database listing the accreditation status of all laboratories participating in NELAP. (NELAC)

National Institute of Standards and Technology (NIST): an agency of the US Department of Commerce's Technology Administration that is working with EPA, States, NELAC, and other public and commercial entities to establish a system under which private sector companies and interested States can be accredited by NIST to provide NIST-traceable proficiency testing (PT) to those laboratories testing drinking water and wastewater. (NIST)

National Environmental Laboratory Accreditation Conference (NELAC): a voluntary organization of State and Federal environmental officials and interest groups purposed primarily to establish mutually acceptable standards for accrediting environmental laboratories. A subset of NELAP. (NELAC)

National Environmental Laboratory Accreditation Program (NELAP): the overall National Environmental Laboratory Accreditation Program of which NELAC is a part. (NELAC)

National Voluntary Laboratory Accreditation Program (NVLAP): a program administered by NIST that is used by providers of proficiency testing to gain accreditation for all compounds/matrices for which NVLAP accreditation is available, and for which the provider intends to provide NELAP PT samples. (NELAC)

Negative Control: measures taken to ensure that a test, its components, or the environment do not cause undesired effects, or produce incorrect test results. (NELAC)

NELAC Standards: the plan of procedures for consistently evaluating and documenting the ability of laboratories performing environmental measurements to meet nationally defined standards established by the National Environmental Laboratory Accreditation Conference. (NELAC)

NELAP Recognition: the determination by the NELAP Director that an accrediting authority meets the requirements of the NELAP and is authorized to grant NELAP accreditation to laboratories. (NELAC)

Non-Governmental Laboratory: any laboratory not meeting the definition of the governmental laboratory. (NELAC)

Performance Audit: the routine comparison of independently obtained *qualitative and* quantitative measurement system data with routinely obtained data in order to evaluate the proficiency of an analyst or laboratory. (NELAC)

Performance Based Measurement System (PBMS): a set of processes wherein the data quality needs, mandates or limitations of a program or project are specified and serve as criteria for selecting appropriate test methods to meet those needs in a cost-effective manner. (NELAC)

Positive Control: measures taken to ensure that a test and/or its components are working properly and producing correct or expected results from positive test subjects. (NELAC)

Precision: the degree to which a set of observations or measurements of the same property, obtained under similar conditions, conform to themselves, a data quality indicator. Precision is usually expressed as standard deviation, variance or range, in either absolute or relative terms. (NELAC)

Preservation: refrigeration and/or reagents added at the time of sample collection (or later) to maintain the chemical and/or biological integrity of the sample. (NELAC)

Primary Accrediting Authority: the agency or department designated at the Territory, State or Federal level as the recognized authority with responsibility and accountability for granting NELAC accreditation for a specified field of testing. (NELAC)[1.5.2.3]

Proficiency Testing: a means of evaluating a laboratory's performance under controlled conditions relative to a given set of criteria through analysis of unknown samples provided by an external source. (NELAC)[2.1]

Proficiency Testing Oversight Body/Proficiency Testing Provider Accreditor (PTOB/PTPA): an organization with technical expertise, administrative capacity and financial resources sufficient to implement and operate a national program of PT provider evaluation and oversight that meets the responsibilities and requirements established by NELAC standards. (NELAC)

Proficiency Testing Program: the aggregate of providing rigorously controlled and standardized environmental samples to a laboratory for analysis, reporting of results, statistical evaluation of the results and the collective demographics and results summary of all participating laboratories. (NELAC)

Proficiency Testing Study Provider: any person, private party, or government entity that meets stringent criteria to produce and distribute NELAC PT samples, evaluate study results against published performance criteria and report the results to the laboratories, primary accrediting authorities, PTOB/PTPA, and NELAP (NELAC)

Proficiency Test Sample (PT): a sample, the composition of which is unknown to the analyst and is provided to test whether the analyst/laboratory can produce analytical results within specified acceptance criteria. (QAMS)

Protocol: a detailed written procedure for field and/or laboratory operation (e g., sampling, analysis), which must be strictly followed. (EPA-QAD)

Quality Assurance: 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. (QAMS)

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. (EPA-QAD)

Quality Control: the overall system of technical activities whose purpose is to measure and control the quality of a product or service so that it meets the needs of users (QAMS) (NELAC)

Quality Control Sample: an uncontaminated sample matrix spiked with known amounts of analytes from a source independent from the calibration standards. It is generally used to establish intra-laboratory or analyst specific precision and bias or to assess the performance of all or a portion of the measurement system (EPA-QAD)

Quality Manual: a document stating the management policies, objectives, principles, organizational structure and authority, responsibilities, accountability, and implementation of an agency, organization, or laboratory, to ensure the quality of its product and the utility of its product to its users. (NELAC)

Quality System: a structured and documented management system describing the policies, objectives, principles, organizational authority, responsibilities, accountability, and implementation plan of an organization for ensuring quality in its work processes, products (items), and services. The quality system provides the framework for planning, implementing, and assessing work performed by the organization and for carrying out required QA and QC (ANSI/ASQC E-41994)

Quantitation Limits: levels, concentrations, or quantities of a target variable (e.g., target analyte) that can be reported at a specified degree of confidence. (NELAC)

Range: the difference between the minimum and the maximum of a set of values. (EPA-QAD)

Raw Data: any original factual information from a measurement activity or study recorded in a laboratory notebook, worksheets, records, memoranda, notes, or exact copies thereof 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 media, including dictated observations, and recorded data from automated instruments. If exact copies of raw data have been prepared (e.g., tapes which have been transcribed verbatim, data and verified accurate by signature), the exact copy or exact transcript may be submitted (EPA-QAD)

Recognition: previously known as reciprocity. The mutual agreement of two or more parties (i.e., States) to accept each other's findings regarding the ability of environmental testing laboratories in meeting NELAC standards. (NELAC) [1.5.3]

Reference Material: a material or substance one or more properties of which are sufficiently well established to be used for the calibration of an apparatus, the assessment of a measurement method, or for assigning values to materials. (ISO Guide 30-2.1)

Reference Method: a method of known and documented accuracy and precision issued by an organization recognized as competent to do so. (NELAC)

Reference Standard: a standard, generally of the highest metrological quality available at a given location, from which measurements made at that location are derived. (VIM-6.08)

Reference Toxicant: the toxicant used in performing toxicity tests to indicate the sensitivity of a test organism and to demonstrate the laboratory's ability to perform the test correctly and obtain consistent results (see Chapter 5, Appendix D, section 2.1f). (NELAC)

Replicate Analyses: the measurements of the variable of interest performed identically on two or more sub-samples of the same sample within a short time interval. (NELAC)

Requirement: denotes a mandatory specification; often designated by the term "shall" (NELAC)

Resource Conservation and Recovery Act (RCRA): the enabling legislation under 42 USC 321 *et seq.* (1976), that gives EPA the authority to control hazardous waste from the "cradle-to-grave", including its generation, transportation, treatment, storage, and disposal. (NELAC)

Revocation: the total or partial withdrawal of a laboratory's accreditation by the accrediting authority. (NELAC)[4.4.3]

Safe Drinking Water Act (SDWA): the enabling legislation, 42 USC 300f *et seq.* (1974), (Public Law 93-523), that requires the EPA to protect the quality of drinking water in the U S. by setting maximum allowable contaminant levels, monitoring, and enforcing violations. (NELAC)

Sample Tracking: procedures employed to record the possession of the samples from the time of sampling until analysis, reporting, and archiving. These procedures include the use of a Chain of Custody Form that documents the collection, transport, and receipt of compliance samples to the laboratory. In addition, access to the laboratory is limited and controlled to protect the integrity of the samples. (NELAC)

Secondary Accrediting Authority: the Territorial, State or federal agency that grants NELAC accreditation to laboratories, based upon their accreditation by a NELAP-recognized Primary

Accrediting Authority See also **Reciprocity** and **Primary Accrediting Authority**. (NELAC)[1.5.2.3]

Selectivity: (Analytical chemistry) the capability of a test method or instrument to respond to a target substance or constituent in the presence of non-target substances. (EPA-QAD)

Sensitivity: the capability of a method or instrument to discriminate between measurement responses representing different levels (e.g., concentrations) of a variable of interest. (NELAC)

Shall: denotes a requirement that is mandatory whenever the criterion for conformance with the specification requires that there be no deviation. This does not prohibit the use of alternative approaches or methods for implementing the specification so long as the requirement is fulfilled. (ANSI)

Should: denotes a guideline or recommendation whenever noncompliance with the specification is permissible. (ANSI)

Spike: a known mass of target analyte added to a blank sample or sub-sample; used to determine recovery efficiency or for other quality control purposes. (NELAC)

Standard: the document describing the elements of laboratory accreditation that has been developed and established within the consensus principles of NELAC and meets the approval requirements of NELAC procedures and policies. (ASQC)

Standard Operating Procedures (SOPs): 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 (QAMS)

Standardized Reference Material (SRM): a certified reference material produced by the U.S. National Institute of Standards and Technology or other equivalent organization and characterized for absolute content, independent of analytical method. (EPA-QAD)

Statistical Minimum Significant Difference (SMSD): the minimum difference between the control and a test concentration that is statistically significant; a measure of test sensitivity or power. The power of a test depends in part on the number of replicates per concentration; the significance level selected, e.g., 0 05, and the type of statistical analysis. If the variability remains constant, the sensitivity of the test increases as the number of replicates is increased. (NELAC)

Supervisor (however named): the individual(s) designated as being responsible for a particular area or category of scientific analysis. This responsibility includes direct day-to-day supervision of technical employees, supply and instrument adequacy and upkeep, quality assurance/ quality control duties and ascertaining that technical employees have the required balance of education, training and experience to perform the required analyses (NELAC)

Surrogate: a substance with properties that mimic the analyte of interest. It is unlikely to be found in environment samples and is added to them for quality control purposes. (QAMS)

Suspension: temporary removal of a laboratory's accreditation for a defined period of time, which shall not exceed six months, to allow the laboratory time to correct deficiencies or area of non-compliance with the NELAC standards. (NELAC)[4.4.2]

Technical Director: individual(s) who has overall responsibility for the technical operation of the environmental testing laboratory. (NELAC)

Test: a technical operation that consists of the determination of one or more characteristics or performance of a given product, material, equipment, organism, physical phenomenon, process

or service according to a specified procedure. The result of a test is normally recorded in a document sometimes called a test report or a test certificate. (ISO/IEC Guide 2-12.1, amended)

Test Method: an adoption of a scientific technique for a specific measurement problem, as documented in a laboratory SOP or published by a recognized authority. (NELAC)

Testing Laboratory: a laboratory that performs tests. (ISO/IEC Guide 2-12.4)

Test Sensitivity/Power: the minimum significant difference (MSD) between the control and test concentration that is statistically significant. It is dependent on the number of replicates per concentration, the selected significance level, and the type of statistical analysis (see Chapter 5, Appendix D, section 2.4.a). (NELAC)

Tolerance Chart: a chart in which the plotted quality control data is assessed via a tolerance level (e.g. +/- 10% of a mean) based on the precision level judged acceptable to meet overall quality/data use requirements instead of a statistical acceptance criteria (e.g. +/- 3 sigma) (applies to radiobioassay laboratories). (ANSI)

Toxic Substances Control Act (TSCA): the enabling legislation in 15 USC 2601 *et seq.*, (1976), that provides for testing, regulating, and screening all chemicals produced or imported into the United States for possible toxic effects prior to commercial manufacture. (NELAC)

Traceability: the property of a result of a measurement whereby it can be related to appropriate standards, generally international or national standards, through an unbroken chain of comparisons. (VIM-6 12)

United States Environmental Protection Agency (EPA): the federal governmental agency with responsibility for protecting public health and safeguarding and improving the natural environment (i.e., the air, water, and land) upon which human life depends. (US-EPA)

Validation: the process of substantiating specified performance criteria. (EPA-QAD)

Verification: confirmation by examination and provision of evidence that specified requirements have been met. (NELAC)

> NOTE: In connection with the management of measuring equipment, verification provides a means for checking that the deviations between values indicated by a measuring instrument and corresponding known values of a measured quantity are consistently smaller than the maximum allowable error defined in a standard, regulation or specification peculiar to the management of the measuring equipment.
>
> The result of verification leads to a decision either to restore in service, to perform adjustment, to repair, to downgrade, or to declare obsolete. In all cases, it is required that a written trace of the verification performed shall be kept on the measuring instrument's individual record.

Voting Member: officials in the employ of the Government of the United States, and the States, the Territories, the Possessions of the United States, or the District of Columbia and who are actively engaged in environmental regulatory programs or accreditation of environmental laboratories. (NELAC)

Work Cell: a well-defined group of analysts that together perform the method analysis. The members of the group and their specific functions within the work cell must be fully documented. (NELAC)

Sources:

40CFR Part 136

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Webster's New World Dictionary of the American Language

18 Appendix B – Table 1 Sample Preservation, Containers, and Holding Time Table

Sample preservation, containers, and holding time requirements for samples for the test parameters performed by the laboratory are listed in the following table.

Sample Preservation, Containers, and Holding Time Table

Parameter	**Matrix A = Aqueous S = Solids**	**Container P= Polyethylene G= Glass**	**Preservation**	**Maximum Holding Time If preservation conditions are met**
Ammonia	A	P	H2SO4	28 days
Biochemical Oxygen Demand	A	P, G	Cool, 4°C	48 hours
Chemical Oxygen Demand	A	P, G	Cool, 4°C, H2SO4 pH < 2	28 days
Cyanide	A	P, G	Cool, 4°C, NaOH pH > 12 0.6 g ascorbic acid (Note 1)	14 days
Nitrate	A	P, G	Cool, 4°C	48 hours
Nitrate-Nitrite	A	P, G	Cool, 4°C, H2SO4 pH < 2	28 days
Boron	A	P, PFTE, Quartz	HNO3 pH < 2	6 months
Chromium - +6	A	P, G	Cool, 4°C	24 hours
Mercury	A	P, G	Cool, 4°C HNO3 pH < 2	28 days
Mercury	S	P, G	Cool, 4°C	28 days
Metals (except boron, mercury, Chromium +6)	A	P, G	HNO3 pH < 2	6 months
Metals (except boron, mercury, Chromium +6)	S	P, G	Cool, 4°C	6 months
Phenols	A	G	Cool, 4°C H2SO4 pH < 2	28 days
Total Solids (Residue)	A	P, G	Cool, 4°C	7 days
Total Dissolved Solids (Residue)	A	P, G	Cool, 4°C	7 days
Total Suspended Solids (Residue)	A	P, G	Cool, 4°C	7 days
Total Volatile Solids (Residue)	A	P, G	Cool, 4°C	7 days
Total Phosphorus-ICP analysis	A	P, G	Cool, 4°C HNO3 pH < 2	28 days
Oil & Grease	A	G	Cool, 4°C HCl pH < 2	28 days
Total Organic Carbon	A	P, G	Cool, 4°C H2SO4 pH < 2	28 days
Diesel Range Organics (DRO)	A	G, Teflon-lined cap	Cool, 4°C 0 008% Na2S2O3 (Note 1)	7 days extraction, 40 days after extraction

Parameter	**Matrix A = Aqueous S = Solids**	**Container P= Polyethylene G= Glass**	**Preservation**	**Maximum Holding Time If preservation conditions are met**
Diesel Range Organics (DRO)	S	G, Teflon-lined cap	Cool, 4°C	14 days extraction, 40 days after extraction
Gasoline Range Organics (GRO)	A	G, Teflon-lined septum	Cool, 4°C	14 days
Gasoline Range Organics (GRO)	S	G, Teflon-lined cap	Cool, 4°C	14 days
Volatile Organic Compounds (VOC)	A	G, Teflon-lined septum	Cool, 4°C HCl pH < 2 10% sodium thiosulfate (Note 1)	14 days
Volatile Organic Compounds (VOC)	S	G, Teflon-lined cap	Cool, 4°C	14 days
Semivolatiles Organic Compounds (SVOC)	A	G, Teflon-lined cap	Cool, 4°C 0.008% Na2S2O3 (Note 1)	7 days extraction, 40 days after extraction
Semivolatiles Organic Compounds (SVOC)	S	G, Teflon-lined cap	Cool, 4°C	14 days extraction, 40 days after extraction
Pesticides/PCBs	A	G, Teflon-lined cap	Cool, 4°C 0 008% Na2S2O3 (Note 1)	7 days extraction, 40 days after extraction
Pesticides/PCBs	S	G, Teflon-lined cap	Cool, 4°C	14 days extraction, 40 days after extraction
Herbicides	A	G, Teflon	Cool	7 days
Herbicides	S	G, Teflon	Cool	14 days
Nitroaromatics and Nitroamines	A	G, Teflon	Cool	7 days
Nitroaromatics and Nitroamines	S	G, Teflon	Cool	14 days
Polyaromatic Hydrocarbons	A	G, Teflon	Cool	14 days
Polyaromatic Hydrocarbons	S	G, Teflon	Cool	14 days

(Note 1) The use of a dechlorinating agent is only required when residual chlorine is present in the sample.

19 Appendix C – Table 2 Data Packages

Data Packages

QC Level I – Results Only	
QC Report	**QC Form or Equivalent**
Case Narrative – When problems are encountered.	CN Summary
Non-Conformance Report, when applicable	NCR
Sample Casualty Report, when applicable	SCR
Surrogate Recoveries (Organics Only)	LMP Report Format
Sample Results	LMP Report Format
QC Level II QA/QC – Minimum QC	
Report	**QC Form or Equivalent**
Level I requirements	
Surrogate Summary Report	Form 2
Laboratory Blank Summary	Form 4
Laboratory Blank Results	Form 1 or equivalent
Laboratory Control/Laboratory Control Sample Dup	Form 3 or equivalent
Matrix Spike/Matrix Spike Duplicate	Form 3 or equivalent
Post Digestion Spike Recovery (Metals only)	LPM Format
Dilution Test RPD (Metals only)	LMP Format
Post Digestion Spike Recovery (Metals only)	LMP Format
Dilution Table (Inorganics and Metals only)	LMP Format
QC Level III QA/QC	
Report	**QC Form or Equivalent**
Level II requirements	
Instrument Tune Report (Semi-volatile GC/MS only)	Form 5
Initial Calibration	Form 6
Calibration Verification (Narrative Only)	Form 7, or equivalent
Internal Standard Summary	Form 8
Sample TIC Summary Reports (GC/MS only)	Form 1 TIC
QC Level IV QA/QC	
QC Report	**QC Form or Equivalent**
Level III requirements	
Raw Data	Worksheets, Prep Logs, Instrument Printouts, Chromatograms, etc.

20 Appendix D – Table 3 Analytical Methods – Program Specific

Table 3 - Analytical Methods - Program Specific

		Certified by/Programs					
SW 846	Analytical Method	NELAC	USCOE	CWA-WP	SDWA-WS	RCRA	KYUST
	6010B	X	X			X	X
	7000A		X				
	7060A						
	7041						
	7091						
	7191						
	7196A	X	X			X	X
	7131A						
	7211						
	7421						
	7470A	X	X			X	X
	7471A	X	X			X	X
	7481						
	7740						
	7761						
	7481						
	8015B	X	X	X		X	X
	8021B	X	X	X		X	X
	8081A	X	X	X		X	X
	8082	X	X	X		X	
	8121	X					X
	8151A	X	X	X		X	X
	8260B	X	X	X		X	X
	8270C	X	X	X		X	X
	8310	X	X	X		X	
	8330	X				X	X
	9010B	X	X			X	X
	9012A		X				
	9030B	X				X	X
	9038						
	9040B	X				X	X
	9045C	X				X	X
	9050A						
	9056	X				X	X
	9060		X				
	9065	X	X			X	
	9066		X				
	9071B						
	9095	X				X	X
	7 3 3	X			X		
	7.3 4	X			X		

Table 3 - Analytical Methods - Program Specific (continued)

		Certified by/Programs					
EPA	Analytical Method	NELAC	USCOE	CWA-WP	SDWA-WS	RCRA	KYUST
	10029						
	1010	X				X	
	120.1			X			
	130.2	X		X			
	150.1	X		X	X		
	160.1	X		X	X		
	160.2	X		X			
	160.3	X		X			
	160.4	X		X			
	160.5	X		X			
	1664	X	X	X			
	180.1	X		X	X		
	200.7	X		X	X		
	200.9				X		
	204.2						
	206.2						
	210.2						
	213.2						
	2150-B						
	218.2						
	220.2						
	239.2						
	245.1	X		X	X		
	246.2						
	270.2						
	272.2						
	279.2						
	300.0	X	X	X	X		
	305.1	X		X			
	310.1	X		X			
	314.0						
	325.3						
	330.5						
	335.1	X		X			
	335.2	X		X			
	340.2						
	350.3	X		X			
	351.4	X		X			
	353.3						
	360.1						
	365.2			X			
	375.4						
	376.2	X		X			
	377.1	X		X			

Table 3 - Analytical Methods - Program Specific (continued)

		Certified by/Programs					
EPA	Analytical Method	NELAC	USCOE	CWA-WP	SDWA-WS	RCRA	KYUST
	405.1			X			
	410.2						
	410.4						
	415.1	X		X	X		
	420.1	X		X			
	425.1	X		X			
TKN-NH_3	Calc	X		X			
	508				X		
	515				X		
	524				X		
	525				X		
	602						
	608	X		X			
	624	X		X			
	625	X		X			

		Certified by/Programs					
SW 846	Prep Method	NELAC	USCOE	CWA-WP	SDWA-WS	RCRA	KYUST
	1311	X				X	
	1312	X				X	
	3005A						
	3051						
	3510C			X			
	3535A			X			
	3550B			X			
	5030B			X			
	5035			X			

Table 3 - Analytical Methods - Program Specific (continued)

Standard Methods 18th ED.	Analytical Method	Certified by/Programs					
		NELAC	USCOE	CWA-WP	SDWA-WS	RCRA	KYUST
	2120-B						
	2310B	X		X			
	2320B	X		X	X		
	2340C	X		X			
	2710-C						
	3500-D						
	4500-B			X			
	4500-NH3-B	X		X			
	4500-C				X		
	4500-D						
	4500-E			X			
	4500-NH3-F	X		X			
	4500-G						
	5210B	X					
	5210C						
	5220C	X					
	5220C/D			X			
	5220D	X					
	5310B	X		X			
	5540C	X		X			
	9222B						
	9222D			X			

MISC.	Analytical	Certified by/Programs					
		NELAC	USCOE	CWA-WP	SDWA-WS	RCRA	KYUST
Wisconsin	SW-141						
Tennessee	8015M						
Tennessee	TN GRO						
Texas	TX 1005						
Temperature					X		
Langlier Index					X		
Aggressive Index					X		
Acute Toxicity					X		
Chronic 1002.0					X		
Chronic 1000.0					X		

APPENDIX B

SUMMARY OF CALIBRATION AND QUALITY CONTROL PROCEDURES

APPENDIX B
SUMMARY OF CALIBRATION AND INTERNAL QUALITY CONTROL PROCEDURES

ANALYTICAL METHOD	APPLICABLE PARAMETER	QUALITY CONTROL CHECK	MINIMUM FREQUENCY	ACCEPTANCE CRITERIA	CORRECTIVE ACTION
SW8260B	Volatile Organics	FIELD QC			
		Trip Blank	1 for each batch of samples shipped to laboratory	No analytes detected at > PQL	1) Review lab QC data to determine if there is a laboratory problem. 2) If same compounds are found in field samples at similar concentrations, qualify the data. OR 3) Resample the batch
		Ambient Blank	Collected when samples are collected downwind of possible volatile sources	No analytes detected at > PQL	1) Review lab QC data to determine if there is a laboratory problem. 2) If same compounds are found in field samples at similar concentrations, qualify the data OR 3) Resample the batch
		Duplicate	1 for every 10 field samples	RPD <30% for water and soil samples	1) Review lab QC data to determine if they are in control. 2) Qualify data. Use data to evaluate whether proper collection procedures were followed. 3) Determine further corrective action.
		Rinsate	1 per day per sampling team per matrix if using non-dedicated equipment	No analytes detected at > PQL	Qualify data.
		CALIBRATION			
		Check of mass spectral ion intensities using BFB	Initially, prior to calibration, and once per every 12-hour shift	Established criteria in Table 4 of SW-846 8260B	1) Retune instrument 2) Repeat BFB analysis.
		Multi-point calibration (minimum five points)	Initial calibration prior to sample analysis	1) Avg. RF ≥ 0.30 (RF ≥0.10 for bromoform, chloromethane and 1,1-dichloroethane) for SPCCs. 2) % RSD ≤ 30% for CCCs and ≤ 15% average for remaining compounds. 3) If % RSD for any compound other than CCC > 15%, a regression fit may be used for calibration. Acceptance criterion for first order regression is correlation coefficient ≥ 0.995. For second or third order regression, 6 (for 2nd order) and 7 (for 3rd order) data points shall be used and the acceptance criteria is COD ≥ 0.990.	1) Evaluate system and take corrective action. 2) Repeat calibration. 3) Qualify data if corrective action was unsuccessful or not performed.

APPENDIX B
SUMMARY OF CALIBRATION AND INTERNAL QUALITY CONTROL PROCEDURES

ANALYTICAL METHOD	APPLICABLE PARAMETER	QUALITY CONTROL CHECK	MINIMUM FREQUENCY	ACCEPTANCE CRITERIA	CORRECTIVE ACTION
SW8260B (Continued)	Volatile Organics (Continued)	Second source initial calibration verification (ICV)	One each time a five-point calibration is performed	Compounds within ± 20% expected value	1) Correct problem. 2) Recalibrate. 3) Qualify the data if the corrective action was unsuccessful or was not performed.
		Continuing calibration verification (CCV)	Once per each 12 hours, prior to sample analysis (criteria for these checks must be met prior to sample analysis)	1) RF ≥ 0.30 (≥ 0.10 for bromoform, chloromethane and 1,1-dichloroethane) SPCCs , CCCs <30% difference 2) Expected Value within ± 15% for compounds when using average RFs or ± 15% drift when using linear regression or quadratic equation	1) Evaluate system and take corrective action. 2) Rerun calibration check 3) If still out, prepare new calibration curve for any analyte not meeting criteria. 4) Reinject any samples analyzed after criteria were exceeded. 5) Qualify the data.
		Demonstrate ability to generate acceptable accuracy and precision using four replicate analyses of a second source QC check sample	Once per analyst	Analyte-specific limits as per laboratory historical limits	1) Recalculate results 2) Locate and fix the source of the problem. 3) Rerun demonstration for those analytes that did not meet criteria.
		MDL Study	Once per year, upon any major system change, or quarterly MDL check.	Method detection limits established as described in 40 CFR Part 136, App B shall not exceed	Method detection limits that exceed established criteria shall be submitted to the USACE for approval prior to the analysis of any project samples
		QC ELEMENT			
		Internal Standards (IS) - Retention Time (RT) and area response checked from daily calibration check	Immediately after or during data acquisition of sample spike, standard, and reagent blank	RT ± 30 seconds and EICP area within -50% to +100% of ***the mid-point standard in the initial calibration*** for each IS compound	1) Inspect MS and GC for malfunctions. 2) Take appropriate corrective actions. 3) Reanalyze samples analyzed while system was malfunctioning 4) If sample exceeds criteria, reanalyze sample If still out, report both analyses and document corrective action.
		Matrix spike (MS) and matrix spike duplicate (MSD)	1 pair per every 20 samples	% R = 70% - 130% for water or soil Up to 5 failures allowed at 60% - 140% if full list of 68 analytes is run. RPD = ± 30 % for water, with no RPD above ± 40 %	1) Qualify the data.
		Laboratory Control Sample (LCS)	One per analytical batch	**Water:** %R = 80% - 120% **Soil** % R = 75% - 125% Up to 5 failures at %R = 60% - 140% if full list of 68 analytes is run.	1) The analytical batch must be reprocessed. 2) Reprep and analyze LCS and affected samples 3) Qualify the data if corrective action was unsuccessful or was not performed.

APPENDIX B
SUMMARY OF CALIBRATION AND INTERNAL QUALITY CONTROL PROCEDURES

ANALYTICAL METHOD	APPLICABLE PARAMETER	QUALITY CONTROL CHECK	MINIMUM FREQUENCY	ACCEPTANCE CRITERIA	CORRECTIVE ACTION
SW8260B (Continued)	Volatile Organics (Continued)	Surrogate standards	Every sample, spike, standard, and reagent blank	**Interference free matrices.** Water: %R = 80% - 120% Soil: %R = 75% - 125% **Project Sample Matrices:** % R = 70% - 130%	1) Recalculate result, and reanalyze sample if still out. 2) Re-extract and reanalyze sample, if still out. 3) Report both analyses and document in report that steps 1 and 2 were performed. 4) Qualify the data.
		Method blank	One per analytical batch	No analytes detected at ≥ PQL	1) Investigate source of contamination. 2) Take and document appropriate corrective action 3) Reanalyze all samples processed with a contaminated blank. 4) Qualify the data if the corrective action was not successful or was not performed.

Notes:
BFB = 4-Bromofluorobenzene
CCC = Calibration Check Compounds
COD = Coefficient of Determination
COI = Compound/Analyte of Interest
EICP = Extracted Ion Current Profile
PQL = Practical Quantitation Limit
RF = Response Factor
RPD = Relative Percent Difference
% RSD= Percent Relative Standard Deviation
SPCC = System Performance Check Compounds
% R = Percent Recovery

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APPENDIX B
SUMMARY OF CALIBRATION AND INTERNAL QUALITY CONTROL PROCEDURES

ANALYTICAL METHOD	APPLICABLE PARAMETER	QUALITY CONTROL CHECK	MINIMUM FREQUENCY	ACCEPTANCE CRITERIA	CORRECTIVE ACTION
SW8270C	Semi-Volatile Organics	FIELD QC			
		Duplicate	1 for every 10 field samples	RPD ≤ 50% for water samples	1) Review lab QC data to determine if they are in control 2) Qualify data. Use data to evaluate whether proper collection procedures were followed 3) Determine further corrective action
		Rinsate	1 per day per sampling team per matrix if using non-dedicated equipment	No analytes detected at > PQL	Qualify data if sample result is less than five times rinsate result Non-detect field sample data need not be qualified
		CALIBRATION:			
		Check of mass spectral ion intensities using DFTPP	Initially, prior to calibration, once per every 12-hour shift	Established criteria in Table 3 of SW-846 8270C	1) Retune instrument 2) Repeat DFTPP analysis.
		Multi-point calibration (minimum five points)	Initial calibration prior to sample analysis	1) Average RF ≥ 0 050 for SPCCs 2) % RSD for CCCs ≤ 30% and ≤ 15% average for remaining compounds 3) If % RSD for any compound other than a CCC > 15%, regression fit may be used for the calibration curve for that analyte. Acceptance criterion for first order regression is correlation coefficient ≥ 0.995. For a second or third order regression, 6 (for 2nd order) and 7 (for 3rd order) data points shall be used and the acceptance criteria is COD ≥ 0 990.	1) Evaluate system and take corrective action. 2) Repeat calibration. 3) Qualify the data if the corrective action was unsuccessful or not performed.
		Second source initial calibration verification (ICV)	Once each time a five-point calibration is performed	Compounds within ± 30% of expected value	1) Correct problem 2) Repeat initial calibration. 3) Qualify data if corrective action was not successful or not performed.
		Continuing calibration verification (CCV)	Once per each 12 hours, prior to sample analysis (criteria for these checks must be met prior to sample analysis)	1) SPCCs average RF ≥ 0.05, CCCs < 20% difference 2) Expected value within ± 20% difference of compounds when using average RFs and ± 20% drift when using linear regression or a quadratic equation	1) Evaluate system and take corrective action 2) Rerun calibration check. 3) If still out, prepare new calibration curve for any analyte not meeting criteria 4) Reinject any samples analyzed after criteria were exceeded.

APPENDIX B
SUMMARY OF CALIBRATION AND INTERNAL QUALITY CONTROL PROCEDURES

ANALYTICAL METHOD	APPLICABLE PARAMETER	QUALITY CONTROL CHECK	MINIMUM FREQUENCY	ACCEPTANCE CRITERIA	CORRECTIVE ACTION
SW8270C (Continued)	Semi-Volatile Organics (continued)	Demonstrate ability to generate acceptable accuracy and precision using four replicate analyses of a QC check sample	Once per analyst	Analyte-specific limits as per laboratory historical limits	1) Recalculate results 2) Locate and fix the source of the problem. 3) Rerun demonstration for those analytes that did not meet criteria.
		MDL Study	Once per year, upon any major system change, or quarterly MDL check.	Method detection limits established as described in 40 CFR Part 136, App B shall not exceed	Method detection limits that exceed established criteria shall be submitted to the USACE for approval prior to the analysis of any project samples.
		QC ELEMENT:			
		Internal Standards (IS) - Retention Time (RT) and area response checked from daily calibration check	Immediately after or during data acquisition of sample, spike, standard, and reagent blank	RT ± 30 seconds and EICP within -50% to +100% of the mid-point standard in the initial calibration for each IS compound	1) Inspect MS and GC for malfunctions. 2) Take appropriate corrective actions. 3) Reanalyze samples analyzed while system was malfunctioning. 4) If sample exceeds criteria, reanalyze sample. If still out, report both analyses and document corrective action
		Matrix spike (MS) and matrix spike duplicate (MSD)	1 pair per every 20 samples	% R = 45% - 135% Up to 5 failures are allowed at 15% - 150% for water and 20% - 150% for soil if 75 compounds are analyzed. RPD = 50% for water and 60% for soil with none > 69% for water and soil	Qualify the data.
		Laboratory Control Sample (LCS)	One per analytical/extraction batch.	**Water:** % R = 60 – 120% - stable cmpds. % R = 20 – 150% - poor performers %R = 45 – 135% - all others **Soil:** % R = 60 – 120% - stable cmpds. % R = 30 – 150% - poor performers %R = 45 – 135% - all others Up to 5 failures allowed within the range of 15-150% for water and 25 – 150% for soils.	1) The analytical batch must be reprocessed. 2) Reprep and analyze LCS and affected samples. 3) Qualify the data if the corrective action was unsuccessful or not performed.
		Surrogate standards	Every sample, spike, standard, and reagent blank	**INTERFERENCE FREE MATRIX** **Water:** B/N cmpds. %R = 60 – 120% Acid cmpds % R = 45-135% with up to one failure in each at 15-150% **Soil:** B/N cmpds. %R = 60 – 120% Acid cmpds %R = 45-135% with up to one failure in each at 20 – 150 %	1) Recalculate result and reanalyze sample, if still out 2) Re-extract and reanalyze sample 3 If still out, report both analyses and document in report that steps 1 and 3 were performed. 4) Qualify the data.

APPENDIX B
SUMMARY OF CALIBRATION AND INTERNAL QUALITY CONTROL PROCEDURES

ANALYTICAL METHOD	APPLICABLE PARAMETER	QUALITY CONTROL CHECK	MINIMUM FREQUENCY	ACCEPTANCE CRITERIA	CORRECTIVE ACTION
SW8270C (Continued)	Semi-Volatile Organics (continued)	Surrogate Standards (continued)		**PROJECT SAMPLE MATRIX** **Water:** B/N cmpds %R = 45 – 135% Acid cmpds %R = 35 – 140% with up to one failure in each at 15 – 150% **Soil:** B/N cmpds. %R = 45 – 135% Acid cmpds. %R = 35 – 130% with up to one failure in each at 20-150%	
		Method blank	One per analytical/extraction batch	No analytes detected at ≥ PQL	1) Investigate source of contamination. 2) Take appropriate corrective action and document it. 3) Re-extract and reanalyze all samples processed with a contaminated blank. 4) Qualify the data if the corrective action was unsuccessful or not performed.

Notes:
CCC = Calibration Check Compounds
COD = Coefficient of Determination
COI = Compound/Analyte of Interest
DFTPP = Decafluorotriphenylphosphine
EICP = Extracted Ion Current Profile
MDL = Method Detection Limit
PQL = Practical Quantitation Limit
RPD = Relative Percent Difference
%RSD = Percent Relative Standard Deviation
SIM = Selective Ion Monitoring
SPCC = System Performance Check Compounds

APPENDIX B
SUMMARY OF CALIBRATION AND INTERNAL QUALITY CONTROL PROCEDURES

ANALYTICAL METHOD	APPLICABLE PARAMETER	QUALITY CONTROL CHECK	MINIMUM FREQUENCY	ACCEPTANCE CRITERIA	CORRECTIVE ACTION
SW8081A	Organochlorine Pesticides	FIELD QC:			
		Duplicate	1 for every 10 field samples (5% wipe samples)	RPD ≤ 50% for water or soil	1) Review lab QC data to determine if they are in control. 2) Qualify data Use data to evaluate whether proper collection procedures were followed. 3) Determine further corrective action.
		Rinsate	1 per day per sampling team per matrix	No analytes detected at > PQL	Qualify data if sample result is < 5x rinsate result. Non-detect field sample data need not be qualified.
		Field Blanks	(Wipe samples only)	No analyses detected at > PQL	Qualify data
		CALIBRATION			
		Multi-point calibration (minimum five points) for each pesticide analyte[a]	Initially and as required	1) % RSD ≤ 20% for RFs or correlation coefficient ≥ 0.995 2) Use calibration curve if % RSD > 20%, correlation coefficient must be ≥ 0.995 for first order regression. If using second or third order regression, 6 (for 2nd order) and 7 (for 3rd order) data points shall be used and acceptance criteria is COD ≥ 0.990	1) Evaluate system and take corrective action 2) Recalibrate system. 3) Qualify the data if the corrective action was unsuccessful or not performed.
		Second source initial calibration verification (ICV) for each pesticide analyte[a]	Once each time five-point calibration is performed	Compounds within ± 15% of expected value with up to 2 failures allowed at 70 - 130% if a full list of 21 compounds is analyzed.	1) Correct problem. 2) Repeat initial calibration. 3) Qualify data if the corrective action was not successful or was not performed.
		Retention time window calculated for each pesticide analyte[a]	Each calibration verification	Each analyte retention time (RT) must be within ± 3 times standard deviation of RT from 72-hour study [a][c]	1) Evaluate system. 2) Reanalyze affected samples
		Breakdown check (Endrin and DDT-pesticide analysis only)	Daily before sample analysis	Degradation ≤ 15% (for either Endrin and DDT)	1) Clean injection port 2) Repeat breakdown check 3) Qualify the data.

APPENDIX B
SUMMARY OF CALIBRATION AND INTERNAL QUALITY CONTROL PROCEDURES

ANALYTICAL METHOD	APPLICABLE PARAMETER	QUALITY CONTROL CHECK	MINIMUM FREQUENCY	ACCEPTANCE CRITERIA	CORRECTIVE ACTION
SW8081A (Continued)	Organochlorine Pesticides (Continued)	Initial calibration verification (ICV) for each pesticide analyte	Daily, before sample analysis	Compounds within ± 15% of expected value with up to 2 failures allowed at 70 - 130% if a full list of 21 compounds is analyzed.	1) Evaluate system. 2) Repeat calibration standard. 3) Recalibrate system. 4) Reanalyze affected samples. 5) Qualify the data
		Continuing calibration verification (CCV) for each pesticide analyte	Every 10 samples for single pesticide analytes and every 20 samples for multi-component analytes, and at the end of the analytical sequence	Compounds within ± 15% D from multi-point calibration, or average of all %D ≤15% with no analyze > 30%	1) Evaluate system. 2) Repeat calibration check standard. 3) Recalibrate system. 4) Reanalyze affected samples. 5) Qualify the data.
		Demonstrate ability to generate acceptable accuracy and precision using four replicate analyses of a QC check sample	Once per analyst	Analyte-specific limits as per laboratory historical limits	1) Recalculate results. 2) Locate and fix the source of the problem 3) Rerun demonstration for those analytes that did not meet criteria.
		MDL Study	Once per year, upon any major system change, or quarterly MDL check	Method detection limits established as described in 40 CFR Part 136, App. B shall not exceed	MDLs that exceed established criteria shall be submitted to the USACE for approval prior to the analysis of any project samples
		QC ELEMENT			
		Method blank	1 per analytical batch	No analytes detected at ≥PQL	1) Investigate source of contamination. 2) Take and document appropriate corrective action 3) Reanalyze all samples processed with a contaminated blank. 4) Qualify the data if the corrective action was unsuccessful or was not performed
		Matrix spike(MS)/matrix spike duplicate(MSD) [b]	1 pair per 20 samples	%R = 40 – 140% for water and soil. If a full list of 21 compounds are analyzed, the 2 failures are allowed at 30 – 150% RPD ≤ 50% with none above 60%.	Qualify the data
		Surrogate standards	Every sample, spiked sample, standard, and method blank	Interference Free Matrices: %R = 50 – 130% Project Sample Matrices %R = 40 – 140%	1) If both surrogates are out, check calculation. 2) Re-extract and reanalyze affected samples 3) Assess impact on data 4) Qualify the data

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SUMMARY OF CALIBRATION AND INTERNAL QUALITY CONTROL PROCEDURES

ANALYTICAL METHOD	APPLICABLE PARAMETER	QUALITY CONTROL CHECK	MINIMUM FREQUENCY	ACCEPTANCE CRITERIA	CORRECTIVE ACTION
SW8081A (Continued)	Organochlorine Pesticides (Continued)	Laboratory Control Sample (LCS) (b)	1 per analytical batch	% R = 50 – 130% for water and soil. Up to 2 failures are allowed at %R = 30 – 150% if a full list of 21 compounds is analyzed.	1) Reprocess the analytical batch. 2) Reprep and analyze LCS and affected samples. 3) Qualify the data if the corrective action was unsuccessful or not performed.
		Confirmation (excluding toxaphene and chlordane)	100% for all positive results ≥ the PQL	Same as primary analysis, confirmation result must be within 40% of primary result to be considered. If comparison is greater than 40%, the data is considered estimated.	Same as primary analysis

Notes:
(a) Since the identification of multi-component compounds (i.e., toxaphene, chlordane) is based upon pattern recognition, RT window criteria do not apply
(b) Does not include toxaphene or technical chlordane.
(c) If calculated window is too restrictive, a default SD of ± 0 03 minutes will be used
CF = Calibration Factor
COI = Compound/Analyte of Interest
MDL = Method Detection Limit
PQL = Practical Quantitation Limit
RF = Response Factor
RPD = Relative Percent Difference
%RSD= Percent Relative Standard Deviation
SD = Standard Deviation

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SUMMARY OF CALIBRATION AND INTERNAL QUALITY CONTROL PROCEDURES

ANALYTICAL METHOD	APPLICABLE PARAMETER	QUALITY CONTROL CHECK	MINIMUM FREQUENCY	ACCEPTANCE CRITERIA	CORRECTIVE ACTION
SW8082	PCBs	FIELD QC			
		Duplicate	1 for every 10 field samples (5% wipe samples)	RPD ≤ 50% for water or soil	1) Review lab QC data to determine if they are in control. 2) Qualify data Use data to evaluate whether proper collection procedures were followed. 3) Determine further corrective action
		Rinsate	1 per day per sampling team per matrix if using non-dedicated equipment.	No analytes detected at > PQL	Qualify data if sample result is < 5x rinsate result Non-detect field sample data need not be qualified.
		Field Blanks	(Wipe samples only)	No analyses detected at > PQL	Qualify data
		CALIBRATION.			
		Multi-point calibration (minimum five points) for each PCB	Initially and as required	1) % RSD ≤ 20% for RFs or correlation coefficient ≥ 0.995 2) Use calibration curve if % RSD > 20%; correlation coefficient must be ≥ 0 995 for first order regression If using second or third order regression, 6 (for 2nd order) or 7 (for 3rd order) data points shall be used and acceptance criteria is COD ≥ 0 990	1) Evaluate system and take corrective action 2) Recalibrate system. 3) Qualify the data if the corrective action was unsuccessful or not performed
		Second source initial calibration verification (ICV)	Once each time a five-point calibration is performed	Compounds within ± 15% of expected value	1) Evaluate system. 2) Repeat calibration standard 3) Recalibrate system 4) Reanalyze affected samples
		Retention time window calculated[1]	Each initial and calibration verification	Retention time (RT) must be within ± 3 times standard deviation of RT from 72-hour study[(a)]	1) Evaluate system. 2) Reanalyze affected samples.
		Continuing calibration verification (CCV)	Every 20 samples and at the end of the analytical sequence	Compounds within ± 15% difference from multi-point calibration	1) Evaluate system 2) Repeat calibration check standard. 3) Recalibrate system 4) Reanalyze affected samples 5) Qualify the data
		Demonstrate ability to generate acceptable accuracy and precision using four replicate analyses of a QC check sample	Once per analyst	Analyte-specific limits as per laboratory historical limits	1) Recalculate the results. 2) Locate and fix source of the problem. 3) Rerun demonstration for those analytes that did not meet criteria.

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SUMMARY OF CALIBRATION AND INTERNAL QUALITY CONTROL PROCEDURES

ANALYTICAL METHOD	APPLICABLE PARAMETER	QUALITY CONTROL CHECK	MINIMUM FREQUENCY	ACCEPTANCE CRITERIA	CORRECTIVE ACTION
SW8082 (Continued)	PCBs (Continued)	MDL Study	Once per year, upon any major system change, or quarterly MDL check	Method detection limits established as described in 40 CFR Part 136, App B shall not exceed	MDLs that exceed established criteria shall be submitted to the USACE for approval prior to the analysis of any project samples.
		QC ELEMENT:			
		Method blank	1 per analytical batch	No analytes detected at ≥PQL	1) Investigate source of contamination. 2) Take and document appropriate corrective action 3) Reanalyze all samples processed with a contaminated blank 4) Qualify the data if the corrective action was unsuccessful or was not performed.
		Matrix spike(MS)/matrix spike duplicate(MSD)	1 pair per 20 samples	%R = 40 – 140% for water or soil %RPD < 50	Qualify the data
		Surrogate standards	Every sample, spiked sample, standard, and method blank	Interference Free Matrices %R = 50 – 130% Project Sample Matrices. %R = 40 – 140%	1) If both surrogates are out, check calculation. 2) Re-extract and reanalyze affected samples. 3) Assess impact on data. 4) Qualify the data
		Laboratory Control Sample (LCS)	1 per analytical batch	% R = 50 – 130%	1) Reprocess the analytical batch 2) Reprep and analyze LCS and affected samples 3) Qualify the data if the corrective action was unsuccessful or not performed.

Notes:

(a) If the RT is too restrictive or zero, a default SD of ± 0.03 minutes will be used
CF = Calibration Factor
COD = Coefficient of Determination
COI = Compound/Analyte of Interest
MDL = Method Detection Limit
PQL = Practical Quantitation Limit
RF = Response Factor
RPD = Relative Percent Difference
%RSD= Percent Relative Standard Deviation
RT = Retention Time
SD = Standard Deviation

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SUMMARY OF CALIBRATION AND INTERNAL QUALITY CONTROL PROCEDURES

ANALYTICAL METHOD	APPLICABLE PARAMETER	QUALITY CONTROL CHECK	MINIMUM FREQUENCY	ACCEPTANCE CRITERIA	CORRECTIVE ACTION
SW8151A	Chlorinated Phenoxy Acid Herbicides	FIELD QC.			
		Duplicate	1 for every 10 field samples	% RPD ≤ 30% for water % RPD ≤ 50% for soil	1) Review lab QC data to determine if they are in control. 2) Qualify data. Use data to evaluate whether proper collection procedures were followed. 3) Determine further corrective action.
		Rinsate	1 per day per sampling team	No analytes detected at > PQL	Qualify data if sample result is < 5x rinsate result. Non-detect field sample data need not be qualified.
		CALIBRATION.			
		Multi-point calibration (minimum five points)	Initially and as required	1) % RSD ≤ 20% for CFs or RFs or correlation coefficient ≥ 0.995 2) Use calibration curve if % RSD > 20%, correlation coefficient must be ≥ 0.995 for first order regression. If using second or third regression 6 (for 2nd order) and 7 (for 3rd order) data points shall be used and acceptance criteria is COD ≥ 0 990.	1) Evaluate system 2) Recalibrate system. 3) Qualify the data if the corrective action was unsuccessful or not performed.
		Second source calibration verification	Once each time a five-point calibration is performed	Compounds within ± 15% of expected value; Dalapon, dichloroprop, and dinoseb ± 35%	1) Correct problem. 2) Repeat initial calibration. 3) Qualify data if the corrective action was not successful or was not performed.
		Retention time window calculated for each analyte.	Each calibration verification	Each analyte retention time (RT) must be within ±3 times the standard deviation of RT from 72-hour study. [a]	1) Evaluate system 2) Reanalyze affected samples
		Initial calibration verification (ICV)	Daily, prior to analysis	Compounds within ± 15% of expected response from multi-point calibration	1) Evaluate system 2) Reanalyze standard. 3) Recalibrate if appropriate 4) Reanalyze affected samples 5) Qualify the data.
		Continuing calibration verification (CCV)	After every 10 samples and at the end of analytical sequence	Compounds within ± 15% difference from multi-point calibration	1) Evaluate system 2) Assess impact on data. 3) Recalibrate if required. 4) Reanalyze standard and samples if required. 5) Qualify the data.

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ANALYTICAL METHOD	APPLICABLE PARAMETER	QUALITY CONTROL CHECK	MINIMUM FREQUENCY	ACCEPTANCE CRITERIA	CORRECTIVE ACTION
SW8151A (Continued)	Chlorinated Phenoxy Acid Herbicides (Continued)	Demonstrate ability to generate acceptable accuracy and precision using four replicate analyses of a QC check sample	Once per analyst	Analyte-specific limits as per laboratory historical limits	1) Recalculate results 2) Locate and fix the source of the problem. 3) Rerun demonstration for those analytes that did not meet criteria.
		MDL Study QC ELEMENT:	Once per year, upon any major system change, or quarterly MDL check	Method detection limits established as described in 40 CFR Part 136, App B shall not exceed	MDLs that exceed established criteria shall be submitted to the USACE for approval prior to the analysis of any project samples
		Method blank	1 per analytical batch	No analytes detected at ≥ PQL	1) Investigate source of contamination. 2) Take and document appropriate corrective action 3) Reanalyze all samples processed with a contaminated blank. 4) Qualify the data if the corrective action was unsuccessful or was not performed
		Matrix spike (MS)/matrix spike duplicate(MSD)	1 pair per 20 samples		Qualify the data.
		Surrogate Standards	Every sample, standard, spiked sample, and method blank		1) Re-extract and reanalyze affected samples. 2) Assess impact on data. 3) Qualify the data.
		Laboratory Control Sample (LCS)	1 per analytical batch		1) Reprocess the analytical batch 2) Reprep and analyze LCS and affected samples. 3) Qualify the data if the corrective action was unsuccessful or not performed.
		Confirmation	100% for all positive results ≥ the PQL	Same as primary analysis, confirmation results must be within 40% of primary results to be considered confirmed. If comparison is greater than 40%, results are considered estimated.	Same as primary analysis

Notes:
[a] = If calculated window is too restrictive or zero, the SD of ± 0.03 minutes will be used
CF = Calibration Factor
COI = Compound/Analyte of Interest
MDL = Method Detection Limit
PQL = Practical Quantitation Limit
RF = Response Factor
RPD = Relative Percent Difference
% RSD= Percent Relative Standard Deviation

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ANALYTICAL METHOD	APPLICABLE PARAMETER	QUALITY CONTROL CHECK	MINIMUM FREQUENCY	ACCEPTANCE CRITERIA	CORRECTIVE ACTION
RSK 175	Methane, Ethane, Ethene, and Carbon Dioxide	FIELD QC:			
		Trip Blank	1 for each batch of samples shipped to laboratory	No analytes detected > PQL	1) Review lab QC data to determine if there is a laboratory problem. 2) If same compounds are found in field samples at similar concentrations, consult with the senior chemist to determine need to resample entire batch 3) Qualify the data.
		Ambient Blank	Collected when samples are collected downwind of possible volatile sources.	No analytes detected > PQL	1) Review lab QC data to determine if there is a laboratory problem 2) If same compounds are found in field samples at similar concentrations, consult with the senior chemist to determine need to resample entire batch 3) Qualify the data.
		Duplicate	1 for every 10 field samples	% RPD = ± 20 %	1) Review lab QC data to determine if they are in control. 2) If not in control, flag data. 3) Use data to evaluate whether proper collection procedures were followed. 4) Determine further corrective action.
		Rinsate	1 per day per sampling team if using non-dedicated sampling equipment	No analytes detected at > PQL	Qualify data if sample result is <5x rinsate result. Non-detect field sample data need not be qualified.
		CALIBRATION			
		Five-point calibration	Initial calibration prior to sample analysis	1) Correlation Coefficient (r) ≥ 0 995 or % RSD ≤ 25 2) Mean %RSD of all analytes ≤ 25 %	1) Check GC system. 2) Repeat calibration as needed to meet criteria.
		Demonstrate ability to generate acceptable accuracy and precision by analyses of a QC check sample	Daily	± 25% difference of the true value	1) Recalculate results. 2) Locate and fix the source of the problem. 3) Rerun demonstration for those analytes that did not meet criteria

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ANALYTICAL METHOD	APPLICABLE PARAMETER	QUALITY CONTROL CHECK	MINIMUM FREQUENCY	ACCEPTANCE CRITERIA	CORRECTIVE ACTION
RSK 175 (Continued)	Methane, Ethane, Ethene, and Carbon Dioxide (Continued)	Continuing Calibration Verification	Daily	± 25% difference of the true value	1) Evaluate system and take corrective action. 2) Rerun calibration check. 3) Prepare new calibration curve for any analyte not meeting criteria. 4) Reinject any samples analyzed after criteria were exceeded. 5) Qualify the data.
		MDL Study	Once per year, upon any major system change, or quarterly MDL study	Method detection limits established as described in 40 CFR Part 136, App B shall not exceed	MDLs that exceed established criteria shall be submitted to the USACE for approval prior to the analysis of any project samples.
		QC ELEMENT			
		Method blank	One per analytical batch	No analytes detected at ≥ PQL	1) Investigate source of contamination 2) Take and document appropriate corrective action 3) Reanalyze all samples processed with a contaminated blank. 4) Qualify the data
		Laboratory Control Sample (LCS)	One per analytical batch	80 % or spiked analytes must recover within laboratory control limits in both the LCS and its duplicate	1) Check calibration 2) Rerun LCS and affected samples 3) Discuss in case narrative. 4) Qualify the data.

Notes:
LCS = Laboratory Control Sample
MDL = Method Detection Limit
PQL = Practical Quantitation Limit
RPD = Relative Percent Difference

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SUMMARY OF CALIBRATION AND INTERNAL QUALITY CONTROL PROCEDURES

ANALYTICAL METHOD	APPLICABLE PARAMETER	QUALITY CONTROL CHECK	MINIMUM FREQUENCY	ACCEPTANCE CRITERIA	CORRECTIVE ACTION
SW6010B	ICP Metals	FIELD QC.			
		Duplicate	1 for every 10 field samples	For water or soil - RPD < 25%	1) Review lab QC data to determine if they are in control 2) Qualify data. Use data to evaluate whether proper collection procedures were followed. 3) Determine further corrective action
		Rinsate	1 per day per sampling team per matrix if using non-dedicated equipment	No analytes detected > PQL	Qualify data if sample result is < 5x rinsate result Non-detect field sample data need not be qualified.
		CALIBRATION:			
		Initial multi-point calibration (minimum blank and one standard) [a]	Daily, prior to analyses	Correlation coefficient (r) ≥ 0 995	Recalibrate system.
		Lowest calibration standard	Before beginning a sample run	Analytes must be ± 20% of expected value	1) Locate and correct problem. 2) Recalibrate system. 3) If corrective action was unsuccessful or was not performed, qualify the data
		Initial calibration verification (ICV) (independent, approximate mid-range standard)	After calibration and high standard	Analytes ± 10% of expected response	1) Locate and correct problem. 2) Recalibrate system. 3) If corrective action was unsuccessful or was not performed, qualify the data.
		Initial calibration blank (ICB)	After initial calibration verification (ICV)	No analytes detected ≥ PQL	1) Rerun blank 2) Clean system. 3) Reanalyze previous 10 samples 4) If corrective action was unsuccessful or was not performed, qualify the data
		Continuing calibration blank (CCB)	After each continuing calibration verification (CCV)	No analytes detected > PQL	1) Rerun blank. 2) Clean system 3) Reanalyze previous 10 samples. 4) If corrective action was unsuccessful or was not performed, qualify the data

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ANALYTICAL METHOD	APPLICABLE PARAMETER	QUALITY CONTROL CHECK	MINIMUM FREQUENCY	ACCEPTANCE CRITERIA	CORRECTIVE ACTION
SW6010B (Continued)	ICP Metals (continued)	ICP interference check sample (ICS)	At the beginning of the daily run, after 8 hours and/or at the end of run	ICS - A All non-spiked analytes <2 x the PQL ICS - AB 80-120% of true value for USEPA check standard elements	1) Verify calibration 2) Verify IECs and update as necessary 3) Recalibrate system. 4) If corrective action was unsuccessful or was not performed, qualify the data.
		Continuing calibration verification (CCV)	After every 10 samples and at the end of the run sequence	± 10% of expected response	1) Reanalyze CCV. 2) Recalibrate system. 3) Reanalyze affected samples. 4) If corrective action was unsuccessful or was not performed, qualify the data.
		MDL Study	Once per year, upon any major system change, or quarterly MDL check	Method detection limits established as described in 40 CFR Part 136, App. B shall not exceed	MDLs that exceed established criteria shall be submitted to the USACE for approval prior to the analysis of any project samples.
		Demonstrate ability to generate acceptable accuracy and precision using four replicate analyses of a QC check sample	Once per analyst	Analyte-specific limits as per laboratory historical limits	1) Recalculate results. 2) Locate and fix the source of the problem. 3) Rerun demonstration for those analytes that did not meet criteria.
		QC ELEMENT			
		Method blank	1 per analytical batch	No analytes detected ≥ PQL	1) Investigate source of contamination. 2) Take and document appropriate corrective action. 3) Reanalyze all samples processed with a contaminated blank. 4) Qualify the data if the corrective action was unsuccessful or was not performed.
		Matrix Spike (MS) / Matrix Spike Duplicate (MSD)	1 pair per 20 samples per matrix	% R = 75 – 125% for water or soil, unless the amount present in the parent sample is ≥ 4x the spike amount. % RPD ≤ 25	Qualify the data
		Laboratory Control Sample (LCS)	1 per analytical batch	% R = 80 – 120 % for water or soil, with up to 2 failures allowed if more than 15 metals are reported at up to % R = 60 – 140%	1) Reanalyze LCS. 2) If still out, correct problem. 3) Redigest and reanalyze LCS and affected samples 4) If corrective action was unsuccessful or was not performed, qualify the data.

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ANALYTICAL METHOD	APPLICABLE PARAMETER	QUALITY CONTROL CHECK	MINIMUM FREQUENCY	ACCEPTANCE CRITERIA	CORRECTIVE ACTION
SW6010B (Continued)	ICP Metals (continued)	Serial Dilution	Each new sample matrix	1:4 Dilution must agree within ± 10% of original determination	1) Perform post digestion spike addition. 2) Qualify the data.
		Post Digestion Spike	When serial dilution fails	Recovery within 75-125% of expected results	1) Correct problem. 2) Reanalyze post digestion spike addition. 3) Qualify the data.
		Method of Standard Additions	As needed for samples with suspected or confirmed matrix effects	Correlation Coefficient (r) > 0 995	1) Correct problem 2) Reanalyze method of standard additions. 3) Qualify the data.

Notes:
COI = Compound/Analyte of Interest
ICP = Inductively Coupled Plasma
IEC = Interelement check
MDL = Method Detection Limit
PQL = Practical Quantitation Limit
RPD = Relative Percent Difference

(a) If instrumentation does not allow for a multi-point calibrations, then a minimum of a blank and one standard is allowed

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ANALYTICAL METHOD	APPLICABLE PARAMETER	QUALITY CONTROL CHECK	MINIMUM FREQUENCY	ACCEPTANCE CRITERIA	CORRECTIVE ACTION
SW7470A/ SW7471A	Mercury	FIELD QC:			
		Duplicate	1 for every 10 field samples	Water or soil- RPD < 25%	Review lab QC data to determine if they are in control. If not in control, flag data. Use data to evaluate whether proper collection procedures were followed. If not, determine further corrective action.
		Rinsate	1 per day per sampling team if using non-dedicated equipment	No analytes detected at ≥ PQL	Qualify data if sample result is < 5x rinsate result. Non-detect field sample data need not be qualified.
		CALIBRATION:			
		Initial multi-point calibration (minimum five standards and a blank)	Daily, prior to analyses	Correlation coefficient of ≥ 0.995	Recalibrate.
		Second source initial calibration verification (ICV)	After initial calibration before analysis of samples.	Analyte within ± 10% of expected value	1) Correct problem Rerun ICV 2) Repeat initial calibration. 3) Qualify data if the corrective action was not successful or was not performed.
		Initial calibration blank (ICB)	After ICV before analysis of samples	No analytes detected at ≥ PQL	1) Rerun blank. 2) If analyte detected at or above PQL, clean system 3) Reanalyze affected samples 4) If corrective action was unsuccessful or was not performed, qualify the data
		Continuing calibration verification (CCV)	After every 10 samples and at the end of the run sequence	± 20% expected response	1) Reanalyze CCV. 2) Recalibrate system. 3) Reanalyze affected samples 4) If corrective action was unsuccessful or was not performed, qualify the data
		Continuing calibration blank (CCB)	After each continuing calibration verification	No analytes detected at ≥ PQL	1) Rerun blank. 2) If analyte detected at or above PQL, clean system. 3) Reanalyze affected samples. 4) If corrective action was unsuccessful or was not performed, qualify the data
		MDL Study	Once per year, upon any major system change, or quarterly MDL check.	Method detection limits established as described in 40 CFR Part 136, App B shall not exceed	MDLs that exceed established criteria shall be submitted to the Air Force for approval prior to the analysis of any project samples.

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APPENDIX B
SUMMARY OF CALIBRATION AND INTERNAL QUALITY CONTROL PROCEDURES

ANALYTICAL METHOD	APPLICABLE PARAMETER	QUALITY CONTROL CHECK	MINIMUM FREQUENCY	ACCEPTANCE CRITERIA	CORRECTIVE ACTION
SW7470A/ SW7471A (Continued)	Mercury (Continued)	Demonstrate ability to generate acceptable accuracy and precision using four replicate analyses of a QC check sample	Once per analyst	Analyte-specific limits as per laboratory historical limits	1) Recalculate results. 2) Locate and fix the source of the problem. 3) Rerun demonstration for those analytes that did not meet criteria.
		QC ELEMENT			
		Method blank	1 per analytical batch	No analytes detected ≥ PQL	1) Investigate source of contamination 2) Take and document appropriate corrective action 3) Redigest and reanalyze all samples processed with a contaminated blank. 4) Qualify the data if the corrective action was unsuccessful or was not performed
		Matrix Spike (MS)/Matrix Spike Duplicate (MSD)	1 pair per 20 samples per matrix	% R = 80 - 120% for water or soil RPD ≤ 20%	Qualify the data
		Laboratory Control Sample (LCS)	1 per analytical batch`	% R = 80 – 120%	1) Reanalyze LCS. 2) If still out, correct problem. 3) Reprep and reanalyze LCS and affected samples. 4) Qualify the data if the corrective action was unsuccessful or not performed
		Serial dilution	Each new sample matrix	Dilution result must be ± 10% of the undiluted sample result	1) Perform recovery test 2) Qualify the data.
		Post Digestion Spike	When serial dilution fails	Recovery within 85-115% of expected results	1) Perform method of standard additions 2) Qualify the data.
		Method of standard additions	As needed for samples with suspected or confirmed matrix effects	Correlation coefficient (r) ≥ 0 995	Qualify the data

Notes:
COI = Compound/Analyte of Interest
MDL = Method Detection Limit
PQL = Practical Quantitation Limit
RPD = Relative Percent Difference

APPENDIX B
SUMMARY OF CALIBRATION AND INTERNAL QUALITY CONTROL PROCEDURES

ANALYTICAL METHOD	APPLICABLE PARAMETER	QUALITY CONTROL CHECK	MINIMUM FREQUENCY	ACCEPTANCE CRITERIA	CORRECTIVE ACTION
SW9060M/ Walkley Black	Total and Dissolved Organic Carbon	FIELD QC:			
		Duplicate	1 for every 10 field samples	Water - RPD ≤ 20% Soil - RPD ≤ 50%	1) Review lab QC data to determine if they are in control 2) Qualify data. Use data to evaluate whether proper collection procedures were followed. 3) Determine further corrective action.
		Rinsate	1 per day per sampling team if using non-dedicated equipment	No analytes detected at > PQL	Qualify data if sample result is < 5x rinsate result. Non-detect field sample data need not be qualified.
		Trip Blank	1 for each batch of samples shipped to laboratory	No analytes detected at > PQL	1) Review lab QC data to determine if there is a laboratory problem. 2) If same compounds are found in field samples at similar concentrations, consult with the senior chemist to determine the need to resample entire batch. 3) Qualify the data.
		CALIBRATION:			
		Multi-point calibration (minimum 3 standards and a blank)	Initially and as required	Correlation coefficient (r) ≥ 0.995	1) Evaluate system 2) Recalibrate system 3) Qualify the data if the corrective action was unsuccessful or not performed.
		Initial Calibration Verification (ICV)	Immediately following initial calibration before analysis of samples	Within ± 10 % of expected value	1) Evaluate system. 2) Reanalyze ICV. 3) Qualify the data if the corrective action was unsuccessful or not performed.
		Continuing calibration verification (CCV)	Every 10 samples	± 10% of expected value	1) Evaluate system 2) Reanalyze CCV. 3) If corrective action was unsuccessful or was not performed, qualify the data.
		Calibration blank	Initially & after each continuing calibration verification	TOC not detected at ≥ PQL	1) Clean system. 2) Recalibrate system 3) Reanalyze blank and affected samples 4) If corrective action was unsuccessful or was not performed, qualify the data

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APPENDIX B
SUMMARY OF CALIBRATION AND INTERNAL QUALITY CONTROL PROCEDURES

ANALYTICAL METHOD	APPLICABLE PARAMETER	QUALITY CONTROL CHECK	MINIMUM FREQUENCY	ACCEPTANCE CRITERIA	CORRECTIVE ACTION
		MDL Study	Once per year, upon any major system change, or quarterly MDL check.	Method detection limits established as described in 40 CFR Part 136, App B shall not exceed	MDLs that exceed established criteria shall be submitted to the USACE for approval prior to the analysis of any project samples.
SW9060M / Walkley Black (Continued)	Total and Dissolved Organic Carbon (continued)	Demonstrate ability to generate acceptable accuracy and precision using four replicate analyses of a QC check sample	Once per analyst	Analyte-specific limits as per laboratory historical limits	1) Recalculate results. 2) Locate and fix the source of the problem. 3) Rerun demonstration for those analytes that did not meet criteria
		QC ELEMENT			
		Method blank	1 per analytical batch	TOC not detected at ≥ PQL	1) Investigate source of contamination. 2) Take and document appropriate corrective action 3) Reanalyze all samples processed with a contaminated blank. 4) Qualify the data if the corrective action was unsuccessful or was not performed.
		Matrix spike(MS)/matrix spike duplicate (MSD)	1 pair per every 20 samples	% R and RPD within laboratory limits for water or soil if sample result is ≤ 4x the spike amount.	Qualify the data
		Laboratory Control Sample (LCS)	1 per analytical batch	% R within laboratory limits	1) Check calculations 2) Reanalyze LCS and affected samples 3) Assess impact on data. 4) Qualify the data if the corrective action was unsuccessful or not performed

Notes:
MDL = Method Detection Limit
PQL = Practical Quantitation Limit
RPD = Relative Percent Difference
TOC = Total Organic Carbon

APPENDIX B
SUMMARY OF CALIBRATION AND INTERNAL QUALITY CONTROL PROCEDURES

ANALYTICAL METHOD	APPLICABLE PARAMETER	QUALITY CONTROL CHECK	MINIMUM FREQUENCY	ACCEPTANCE CRITERIA	CORRECTIVE ACTION
MCAWW 300.0	Bromide, Chloride, Nitrate, Nitrite, Sulfate	FIELD QC:			
		Duplicate	1 for every 10 field samples	RPD ≤ 20% for water or soil	1) Review lab QC data to determine if they are in control. 2) Qualify data Use data to evaluate whether proper collection procedures were followed. 3) Determine further corrective action.
		Rinsate	1 per day per sampling team if using non-dedicated equipment	No analytes detected at > PQL	Qualify data if sample result is <5x rinsate result. Nondetect field sample data need not be qualified
		CALIBRATION.			
		Multi-point calibration (minimum five points)	Initially prior to sample analysis and as required	Correlation coefficient (r) ≥ 0.995	1) Evaluate system 2) Recalibrate system. 3) Qualify the data if the corrective action was unsuccessful or not performed
		Second source calibration verification	Once each time a five-point calibration is performed	Analytes within ± 10% of expected value	1) Correct problem. 2) Repeat initial calibration. 3) Qualify data if the corrective action was not successful or was not performed.
		Initial calibration verification (ICV)	Daily, prior to sample analysis or when eluent is changed.	± 10% of expected value	1) Recalibrate system 2) Reanalyze affected samples 3) If corrective action was unsuccessful or was not performed, qualify the data.
		Continuing calibration verification (CCV)	After every 10 samples and at the end of the run sequence	± 10 % of expected value	1) Reanalyze CCV. 2) Recalibrate system. 3) Reanalyze affected samples. 4) If corrective action was unsuccessful or was not performed, qualify the data
		Retention time (RT) window calculated for each analyte	RT windows determined for each IC column and whenever a new column is installed	Each analyte RT is compared to ± 3 standard derivations (SD) of RT from initial calibration. If calculated window is too restrictive, use the SD of similar analyte in the same RT range.	1) Evaluate system 2) Reanalyze affected samples.

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APPENDIX B
SUMMARY OF CALIBRATION AND INTERNAL QUALITY CONTROL PROCEDURES

ANALYTICAL METHOD	APPLICABLE PARAMETER	QUALITY CONTROL CHECK	MINIMUM FREQUENCY	ACCEPTANCE CRITERIA	CORRECTIVE ACTION
MCAWW 300.0 (Continued)	Bromide, Chloride, Nitrate, Nitrite, Sulfate (continued)	MDL Study	Once per year, upon any major system change, or quarterly MDL check.	Method detection limits established as described in 40 CFR Part 136, App. B shall not exceed	Method detection limits that exceed established criteria shall be submitted to the USACE for approval prior to the analysis of any project samples.
		Demonstrate ability to generate acceptable accuracy and precision using four replicate analyses of a QC check sample	Once per analyst	Analyte-specific limits as per laboratory historical limits	1) Recalculate results. 2) Locate and fix the source of the problem. 3) Rerun demonstration for those analytes that did not meet criteria
		QC ELEMENT:			
		Method blank	1 per analytical batch	No analyte ≥ PQL	1) Investigate source of contamination. 2) Take and document appropriate corrective action 3) Re-analyze all samples processed with a contaminated blank 4) Qualify the data if the corrective action was unsuccessful or was not performed
		Matrix spike (MS)/matrix spike duplicate (MSD)	1 pair per every 20 samples per matrix	% R and RPD must be within the laboratory limits	Qualify the data.
		Laboratory Control Sample (LCS)	1 per analytical batch	% R must be within the laboratory limits	1) Check calculations 2) Reprep and reanalyze LCS and affected samples. 3) Qualify the data if the corrective action was unsuccessful or not performed.

Notes:
COI = Compound/Analyte of Interest
MDL = Method Detection Limit
PQL = Practical Quantitation Limit
RPD = Relative Percent Difference

APPENDIX B
SUMMARY OF CALIBRATION AND INTERNAL QUALITY CONTROL PROCEDURES

ANALYTICAL METHOD	APPLICABLE PARAMETER	QUALITY CONTROL CHECK	MINIMUM FREQUENCY	ACCEPTANCE CRITERIA	CORRECTIVE ACTION
MCAWW 310 1	Alkalinity	FIELD QC			
		Duplicate	1 for every 10 field samples	RPD ≤ 20% for water or soil	1) Review lab QC data to determine if they are in control. 2) Qualify data. Use data to evaluate whether proper collection procedures were followed. 3) Determine further corrective action
		Rinsate	1 per day per sampling team if using non-dedicated equipment	No analytes detected at > PQL	Qualify data if sample result is <5x rinsate result. Nondetect field sample data need not be qualified.
		CALIBRATION.			
		Minimum two point calibration (typically pH 4 and 7)	Initially prior to sample analysis and as required	Correlation coefficient (r) ≥ 0.995 if more than two standards are analyzed.	1) Evaluate system 2) Recalibrate system 4) Qualify the data if the corrective action was unsuccessful or not performed.
		Calibration check standard, at pH = 10	Once each time a calibration is performed	pH within ± 10% of expected value	1) Reanalyze standard. 2) Repeat initial calibration. 4) Qualify data if the corrective action was not successful or was not performed.
		Initial calibration verification (ICV)	Daily, prior to sample analysis or when eluent is changed.	± 10% of expected value	1) Reanalyze ICV 2) Recalibrate system. 3) Reanalyze affected samples 4) If corrective action was unsuccessful or was not performed, qualify the data.
		Continuing calibration verification (CCV)	After every 10 samples and at the end of the run sequence	± 10 % of expected value	1) Reanalyze CCV 2) Recalibrate system. 3) Reanalyze affected samples. 4) If corrective action was unsuccessful or was not performed, qualify the data.
		Titrant standardization	As needed, before the analysis of samples	Samples analyzed before expiration of standardization.	1) Restandardize titrant 2) Reanalyze affected samples.
		MDL Study	Once per year, upon any major system change, or quarterly MDL check	Method detection limits established as described in 40 CFR Part 136, App B shall not exceed	Method detection limits that exceed established criteria shall be submitted to the USACE for approval prior to the analysis of any project samples.

APPENDIX B
SUMMARY OF CALIBRATION AND INTERNAL QUALITY CONTROL PROCEDURES

ANALYTICAL METHOD	APPLICABLE PARAMETER	QUALITY CONTROL CHECK	MINIMUM FREQUENCY	ACCEPTANCE CRITERIA	CORRECTIVE ACTION
MCAWW 310.1	Alkalinity	Demonstrate ability to generate acceptable accuracy and precision using four replicate analyses of a QC check sample	Once per analyst	Analyte-specific limits as per laboratory historical limits	1) Recalculate results. 2) Locate and fix the source of problem. 3) Rerun demonstration for those analytes that did not meet criteria.
		QC ELEMENT			
		Method blank	1 per analytical batch	Alkalinity ≤ PQL	1) Investigate source of contamination 2) Take and document appropriate corrective action. 3) Re-analyze all samples processed with a contaminated blank 4) Qualify the data if the corrective action was unsuccessful or was not performed
		Matrix spike(MS)/matrix spike duplicate(MSD)	1 pair per every 20 samples per matrix	% R within laboratory limits of 80 – 120% RPD ≤ 20 %	Qualify the data.
		Laboratory Control Sample (LCS)	1 per analytical batch	% R within laboratory limits of 90 – 127 % for water and soil	1) Check calculations. 2) Reprep and reanalyze LCS and affected samples. 3) Qualify the data if the corrective action was unsuccessful or not performed.

Notes:
COI = Compound/Analyte of Interest
MDL = Method Detection Limit
PQL = Practical Quantitation Limit
RPD = Relative Percent Difference

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SUMMARY OF CALIBRATION AND INTERNAL QUALITY CONTROL PROCEDURES

ANALYTICAL METHOD	APPLICABLE PARAMETER	QUALITY CONTROL CHECK	MINIMUM FREQUENCY	ACCEPTANCE CRITERIA	CORRECTIVE ACTION
MCAWW 376 1	Sulfide	FIELD QC:			
		Duplicate	1 for every 10 field samples	RPD ≤ 20% for water or soil	1) Review lab QC data to determine if they are in control. 2) Qualify data Use data to evaluate whether proper collection procedures were followed. 3) Determine further corrective action.
		Rinsate	1 per day per sampling team if using non-dedicated equipment	No analytes detected at > PQL	Qualify data if sample result is <5x rinsate result. Nondetect field sample data need not be qualified.
		CALIBRATION			
		Titrant standardization	As needed, before the analysis of samples	Samples analyzed before expiration of standardization.	1) Restandardize titrant 2) Reanalyze affected samples
		MDL Study	Once per year, upon any major system change, or quarterly MDL check	Method detection limits established as described in 40 CFR Part 136, App. B shall not exceed	Method detection limits that exceed established criteria shall be submitted to the USACE for approval prior to the analysis of any project samples.
		Demonstrate ability to generate acceptable accuracy and precision using four replicate analyses of a QC check sample	Once per analyst	Analyte-specific limits as per laboratory historical limits	1) Recalculate results. 2) Locate and fix the source of problem. 3) Rerun demonstration for those analytes that did not meet criteria.
		QC ELEMENT:			
		Method blank	1 per analytical batch	Sulfide ≤ PQL	1) Investigate source of contamination. 2) Take and document appropriate corrective action. 3) Re-analyze all samples processed with a contaminated blank. 4) Qualify the data if the corrective action was unsuccessful or was not performed.

APPENDIX B
SUMMARY OF CALIBRATION AND INTERNAL QUALITY CONTROL PROCEDURES

ANALYTICAL METHOD	APPLICABLE PARAMETER	QUALITY CONTROL CHECK	MINIMUM FREQUENCY	ACCEPTANCE CRITERIA	CORRECTIVE ACTION
MCAWW 376.1 (Continued)	Sulfide (continued)	Matrix spike (MS)/matrix spike duplicate (MSD)	1 pair per every 20 samples per matrix	% R and RPD within laboratory limits	Qualify the data.
		Laboratory Control Sample (LCS)	1 per analytical batch	% R within laboratory limits	1) Check calculations. 2) Reprep and reanalyze LCS and affected samples. 3) Qualify the data if the corrective action was unsuccessful or not performed.

Notes:
COI = Compound/Analyte of Interest
MDL = Method Detection Limit
PQL = Practical Quantitation Limit
RPD = Relative Percent Difference

APPENDIX B
SUMMARY OF CALIBRATION AND INTERNAL QUALITY CONTROL PROCEDURES

ANALYTICAL METHOD	APPLICABLE PARAMETER	QUALITY CONTROL CHECK	MINIMUM FREQUENCY	ACCEPTANCE CRITERIA	CORRECTIVE ACTION
SM 5560	Volatile Fatty Acids	FIELD QC:			
		Duplicate	1 for every 10 field samples	RPD ≤ 20% for water or soil	1) Review lab QC data to determine if they are in control 2) Qualify data. Use data to evaluate whether proper collection procedures were followed. 3) Determine further corrective action.
		Rinsate	1 per day per sampling team if using non-dedicated equipment	No analytes detected at > PQL	Qualify data if sample result is <5x rinsate result. Nondetect field sample data need not be qualified
		CALIBRATION:	Calibration will be assumed to be within criteria unless otherwise specified by the laboratory		
		QC ELEMENT:			
		Method blank	1 per analytical batch	Volatile Fatty Acids ≤ PQL	1) Investigate source of contamination. 2) Take and document appropriate corrective action 3) Re-analyze all samples processed with a contaminated blank. 4) Qualify the data if the corrective action was unsuccessful or was not performed
		Matrix spike (MS)/matrix spike duplicate (MSD)	1 pair per every 20 samples per matrix	% R and RPD within laboratory limits	Qualify the data.
		Laboratory Control Sample (LCS)	1 per analytical batch	% R within laboratory limits	1) Check calculations. 2) Reprep and reanalyze LCS and affected samples. 3) Qualify the data if the corrective action was unsuccessful or was not performed.

Notes:
MDL = Method Detection Limit
PQL = Practical Quantitation Limit
RPD = Relative Percent Difference

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APPENDIX C

SEVERN TRENT LABORATORIES – STANDARD OPERATING PROCEDURES

ENVIRONMENTAL TESTING & CONSULTING, INC. – STANDARD OPERATING PROCEDURES

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Implementation Date: 06/05/01

SOP No. CORP-MS-0002NC
Revision No. 2.3
Revision Date: 05/23/01
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STL STANDARD OPERATING PROCEDURE

TITLE: DETERMINATION OF VOLATILE ORGANICS BY GC/MS BASED ON METHOD 8260B, 8260A, AND 624

(SUPERSEDES: REVISION 2.2, DATED 11/28/00)

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Corporate Technology and/or Corporate Quality Assurance Date

Proprietary Information Statement:

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SEVERN TRENT LABORATORIES – STANDARD OPERATING PROCEDURES

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1.0 SCOPE AND APPLICATION

1.1. This method is applicable to the determination of Volatile Organic Compounds in waters, wastewater, soils, sludges and other solid matrices. Standard analytes are listed in Tables 5 and 6.

1.2. This SOP is applicable to method 8260B. It may also be used for analysis following method 8260A. Appendix A presents modifications to the procedures in the main SOP that are necessary for analysis of wastewater by method 624. The associated LIMS method codes are QK (8260B), DN (624), and MZ (8260A). Ohio VAP projects are distinguished by Program Code 2J. The following Prep Codes are used: 15 (5 mL purge), 25 (25 mL purge), 4B (Methanol preservation, EnCore™), 4D (Sodium Bisulfate preservation, EnCore™), 4P (Frozen, EnCore™), and 73 (5030A Methanol Prep).

1.3. This method can be used to quantify most volatile organic compounds that have boiling points below 200°C and are insoluble or slightly soluble in water. Volatile water soluble compounds can be included in this analytical technique; however, for more soluble compounds, quantitation limits are approximately ten times higher because of poor purging efficiency.

1.4. The method is based upon a purge and trap, gas chromatograph/mass spectrometric (GC/MS) procedure. The approximate working range is 5 to 200 µg/L for 5 mL waters, 1 to 40 µg/L for 25 mL purge waters, 5 to 200 µg/kg for low-level soils, and 250 to 25,000 µg/kg for medium-level soils. Reporting limits are listed in Tables 1 and 3.

1.5. Method performance is monitored through the use of surrogate compounds, matrix spike/matrix spike duplicates, and laboratory control spike samples.

2. SUMMARY OF METHOD

2.1. Volatile compounds are introduced into the gas chromatograph by the purge and trap method. The components are separated via the chromatograph and detected using a mass spectrometer, which is used to provide both qualitative and quantitative information.

2.2. Aqueous samples are purged directly. Generally, soils are preserved by extracting the volatile analytes into methanol. If especially low detection limits are required, soil samples may be preserved with sodium bisulfate and purged directly.

2.3. In the purge and trap process, an inert gas is bubbled through the solution at ambient temperature or at 40°C (40°C required for low level soils) and the volatile components are efficiently transferred from the aqueous phase to the vapor phase. The vapor is swept through a sorbant

column where the volatile components are trapped. After purging is completed, the sorbant column (trap) is heated and backflushed with inert gas to desorb the components onto a gas chromatographic column. The gas chromatographic column is then heated to elute the components which are detected with a mass spectrometer.

2.4. Qualitative identifications are confirmed by analyzing standards under the same conditions used for samples and comparing the resultant mass spectra and GC retention times. Each identified component is quantified by relating the MS response for an appropriate selected ion produced by that compound to the MS response for another ion produced by an internal standard.

3. DEFINITIONS

3.1. Batch

The batch is a set of up to 20 samples of the same matrix processed using the same procedures and reagents within the same time period. Using this method, each BFB analysis will normally start a new batch. Batches for medium level soils are defined at the sample preparation stage and may be analyzed on multiple instruments over multiple days, although reasonable effort should be made to keep the samples together.

3.1.1. The Quality Control batch must contain a matrix spike/spike duplicate (MS/MSD), a Laboratory Control Sample (LCS), and a method blank. In some cases, at client request, the MS/MSD may be replaced with a matrix spike and sample duplicate. Refer to the STL QC Program document (QA-003) for further details of the batch definition.

3.2. Method Blank

3.2.1. A method blank consisting of all reagents added to the samples must be analyzed with each batch of samples. The method blank is used to identify any background interference or contamination of the analytical system which may lead to the reporting of elevated concentration levels or false positive data.

3.3. Laboratory Control Sample (LCS)

3.3.1. Laboratory Control Samples are well characterized, laboratory generated samples used to monitor the laboratory's day-to-day performance of routine analytical methods. The LCS, spiked with a group of target compounds representative of the method analytes, is used to monitor the accuracy of the analytical process, independent of matrix effects. Ongoing monitoring of the LCS results provides evidence that the laboratory is performing the method within accepted QC guidelines for accuracy and precision.

3.4. Surrogates

3.4.1. Surrogates are organic compounds which are similar to the target analyte(s) in chemical composition and behavior in the analytical process, but which are not normally found in environmental samples. Each sample, blank, LCS, and MS/MSD is spiked with surrogate standards. Surrogate spike recoveries must be evaluated by determining whether the concentration (measured as percent recovery) falls within the required recovery limits.

3.5. Matrix Spike/Matrix Spike Duplicate (MS/MSD)

3.5.1. A matrix spike is an environmental sample to which known concentrations of target analytes have been added. A matrix spike duplicate is a second aliquot of the same sample which is prepared and analyzed along with the sample and matrix spike. Matrix spikes and duplicates are used to evaluate accuracy and precision in the actual sample matrix.

3.6. Calibration Check Compound (CCC)

3.6.1. CCCs are a representative group of compounds which are used to evaluate initial calibrations and continuing calibrations. Relative percent difference for the initial calibration and % drift for the continuing calibration response factors are calculated and compared to the specified method criteria.

3.7. System Performance Check Compounds (SPCC)

SPCCs are compounds which are sensitive to system performance problems and are used to evaluate system performance and sensitivity. A response factor from the continuing calibration is calculated for the SPCC compounds and compared to the specified method criteria.

4. INTERFERENCES

4.1. Method interferences may be caused by contaminants in solvents, reagents, glassware, and other processing apparatus that lead to discrete artifacts. All of these materials must be routinely demonstrated to be free from interferences under conditions of the analysis by running laboratory method blanks as described in the Quality Control section. The use of ultra high purity gases, pre-purged purified reagent water, and approved lots of purge and trap grade methanol will greatly reduce introduction of contaminants. In extreme cases the purging vessels may be pre-purged to isolate the instrument from laboratory air contaminated by solvents used in other parts of the laboratory.

4.2. Samples can be contaminated by diffusion of volatile organics (particularly methylene chloride and fluorocarbons) into the sample through the septum seal during shipment and storage. A field blank prepared from reagent water and carried through the sampling and handling protocol can serve as a check on such contamination.

4.3. Matrix interferences may be caused by non-target contaminants that are coextracted from the sample. The extent of matrix interferences will vary considerably from source to source depending upon the nature and diversity of the site being sampled.

4.4. Cross-contamination can occur whenever high-level and low-level samples are analyzed sequentially or in the same purge position on an autosampler. Whenever an unusually concentrated sample is analyzed, it should be followed by one or more blanks to check for cross-contamination. The purge and trap system may require extensive bake-out and cleaning after a high-level sample.

4.5. Some samples may foam when purged due to surfactants present in the sample. When this kind of sample is encountered an antifoaming agent (e.g., J.T. Baker's Antifoam B silicone emulsion) can be used. A blank spiked with this agent must be analyzed with the sample because of the non-target interferences associated with the agent.

5. SAFETY

5.1. Procedures shall be carried out in a manner that protects the health and safety of all STL associates.

5.2. The Chemical Hygiene Plan (CHP) gives details about the specific health and safety practices which are to be followed in the laboratory area. Personnel must receive training in the CHP, including the written Hazard Communication plan, prior to working in the laboratory. Consult the CHP, the STL Health and Safety Policies and Procedures Manual, and available Material Safety Data Sheets (MSDS) prior to using the chemicals in the method.

5.3. Consult the STL Health and Safety Policies and Procedures Manual for information on Personal Protective Equipment. Eye protection that protects against splash and a laboratory coat must be worn in the lab. Appropriate gloves must be worn while samples, standards, solvents, and reagents are being handled. Disposable gloves that have been contaminated will be removed and discarded; other gloves will be cleaned immediately. Disposable gloves shall not be reused.

5.4. The health and safety hazards of many of the chemicals used in this procedure have not been fully defined, therefore each chemical compound should be treated as a potential health hazard. Additional health and safety information can be obtained from the MSDS files maintained in the laboratory. The following specific hazards are known:

5.4.1. Chemicals that have been classified as carcinogens, or potential carcinogens, under OSHA include: Acrylonitrile, benzene, carbon tetrachloride, chloroform, 1,2-dibromo-3-chloropropane, 1,4-dichlorobenzene, and vinyl chloride.

5.4.2. Chemicals known to be flammable are: **Methanol**.

5.5. 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 will be kept closed unless transfers are being made.

5.6. The preparation of standards and reagents will be conducted in a fume hood with the sash closed as far as the operations will permit.

5.7. All work must be stopped in the event of a known or potential compromise to the health and safety of a STL associate. The situation must be reported **immediately** to a laboratory supervisor.

5.8. Laboratory personnel assigned to perform hazardous waste disposal procedures must have a working knowledge of the established procedures and practices outlined in the STL Health and Safety Manual. These employees must have training on the hazardous waste disposal practices initially upon assignment of these tasks, followed by an annual refresher training.

6. EQUIPMENT AND SUPPLIES

6.1. Microsyringes: 10 µL and larger, 0.006 inch ID needle.

6.2. Syringe: 5 or 25 mL glass with luerlok tip, if applicable to the purging device.

6.3. Balance: Analytical, capable of accurately weighing 0.0001 g, and a top-loading balance capable of weighing 0.1 g

6.4. Glassware:

6.4.1. Vials: 20 mL with screw caps and Teflon liners.

6.4.2. Volumetric flasks: 10 mL and 100 mL, class A with ground-glass stoppers.

6.5. Spatula: Stainless steel.

6.6. Disposable pipets: Pasteur.

6.7. pH paper: Wide range.

6.8. Gases:

6.8.1. Helium: Ultra high purity, gr. 5, 99.999%.

6.8.2. Nitrogen: Ultra high purity, from cylinders of gas generators, may be used as an alternative to helium for purge gas.

6.8.3. Compressed air: Used for instrument pneumatics.

6.8.4. Liquid nitrogen: Used for cryogenic cooling if necessary.

6.9. Purge and Trap Device: The purge and trap device consists of the sample purger, the trap, and the desorber.

6.9.1. Sample Purger: The recommended purging chamber is designed to accept 5 mL samples with a water column at least 3 cm deep. The purge gas must pass through the water column as finely divided bubbles, each with a diameter of less than 3 mm at the origin. The purge gas must be introduced no more than 5 mm from the base of the water column. Alternative sample purge devices may be used provided equivalent performance is demonstrated. Low level soils are purged directly from a VOA vial.

6.9.2. Trap: A variety of traps may be used, depending on the target analytes required. For most purposes the Vocarb 3000 trap is suitable. Other traps, such as Vocarb 4000, or Tenax / Silica gel / Charcoal may be used if the Quality Control criteria are met.

6.9.3. Desorber: The desorber should be capable of rapidly heating the trap to 180°C. Many such devices are commercially available.

6.9.4. Sample Heater: A heater capable of maintaining the purge device at 40°C is necessary for low level soil analysis.

6.10. Gas Chromatograph/Mass Spectrometer System:

6.10.1. Gas Chromatograph: The gas chromatograph (GC) system must be capable of temperature programming.

6.10.2. Gas Chromatographic Columns: Capillary columns are used. Some typical columns are listed below:

6.10.2.1. Column 1: 105m x 0.53 ID Rtx-624 with 3 µm film thickness.

6.10.2.2. Column 2: 75 m x 0.53 ID DB-624 widebore with 3 µm film thickness.

6.10.2.3. Mass Spectrometer: The mass spectrometer must be capable of scanning 35-300 AMU every two seconds or less, using 70 volts electron energy in the electron impact mode and capable of producing a mass spectrum that meets the required criteria when 50 ng of 4-Bromofluorobenzene (BFB) are injected onto the gas chromatograph column inlet.

6.10.3. GC/MS interface: In general glass jet separators are used but any interface (including direct introduction to the mass spectrometer) that achieves all acceptance criteria may be used.

6.10.4. Data System: A computer system that allows the continuous acquisition and storage on machine readable media of all mass spectra obtained throughout the duration of the chromatographic program. The computer must have software that allows searching any GC/MS data file for ions of a specified mass and plotting such ion abundances versus time or scan number. This type of plot is defined as an Extracted Ion Current Profile (EICP). Software must also be available that allows integrating the abundances in any EICP between the specified time or scan-number limits. Also, for the non-target compounds, software must be available that allows for the comparison of sample spectra against reference library spectra. The most recent release of the NIST/EPA mass spectral library should be used as

the reference library. The computer system must also be capable of backing up data for long-term off-line storage.

6.10.5. Cryogenic Cooling: Some columns require the use of liquid nitrogen to achieve the subambient temperature required for the proper separation of the gases.

7. REAGENTS AND STANDARDS

7.1. Reagents

7.1.1. Methanol: Purge and Trap Grade, High Purity

7.1.2. Reagent Water: High purity water that meets the requirements for a method blank when analyzed. (See section 9.4) Reagent water may be purchased as commercial distilled water and prepared by purging with an inert gas overnight. Other methods of preparing reagent water are acceptable.

7.2. Standards

7.2.1. Calibration Standard

7.2.1.1. Stock Solutions: Stock solutions may be purchased as certified solutions from commercial sources or prepared from pure standard materials as appropriate. These standards are prepared in methanol and stored in Teflon-sealed screw-cap bottles with minimal headspace at -10° to -20°C.

7.2.1.2. Working standards: A working solution containing the compounds of interest prepared from the stock solution(s) in methanol. These standards are stored in the freezer or as recommended by the manufacturer. Working standards are monitored by comparison to the initial calibration curve. If any of the calibration check compounds drift in response from the initial calibration by more than 20% then corrective action is necessary. This may include steps such as instrument maintenance, preparing a new calibration verification standard or tuning the instrument. If the corrective actions do not correct the problem then a new initial calibration must be performed.

7.2.1.3. Aqueous Calibration Standards are prepared in reagent water using the secondary dilution standards. These aqueous standards must be prepared daily.

7.2.1.4. If stock or secondary dilution standards are purchased in sealed ampoules they may be used up to the manufacturers expiration date.

7.2.2. Internal Standards: Internal standards are added to all samples, standards, and blank analyses. Refer to Table 7 for internal standard components.

7.2.3. Surrogate Standards: Refer to Table 8 for surrogate standard components and spiking levels.

7.2.4. Laboratory Control Sample Spiking Solutions: Refer to Table 9 for LCS components and spiking levels.

7.2.5. Matrix Spiking Solutions: The matrix spike contains the same components as the LCS. Refer to Table 9.

7.2.6. Tuning Standard: A standard is made up that will deliver 50 ng on column upon injection. A recommended concentration of 25 ng/µL of 4-Bromofluorobenzene in methanol is prepared as described in Sections 7.2.1.1 and 7.2.1.2.

8. SAMPLE COLLECTION, PRESERVATION AND STORAGE

8.1. Holding times for all volatile analysis are 14 days from sample collection.

8.2. Water samples are normally preserved at pH $\leq$ 2 with 1:1 hydrochloric acid. If residual chlorine is present, 2 drops of 10% sodium thiosulfate are added.

8.3. Solid samples are field preserved with sodium bisulfate solution for low level analysis, or with methanol for medium level analysis. Soil samples can also be taken using the EnCore™ sampler and preserved in the lab within 48 hours of sampling. At specific client request, unpreserved soil samples may be accepted.

8.4. There are several methods of sampling soil. The recommended method, which provides the minimum of field difficulties, is to take an EnCore™ sample. (The 5 g or 25 g sampler can be used, depending on client preference). Following shipment back to the lab the soil is preserved in methanol. This is the medium level procedure. If very low detection limits are needed (< 50 µg/kg for most analytes) then it will be necessary to use two additional 5 g EnCore™ samplers or to use field preservation.

8.5. Sample collection for medium level analysis using EnCore™ samplers.

8.5.1. Ship one 5 g (or 25 g) EnCore™ sampler per field sample position.

8.5.2. An additional bottle must be shipped for percent moisture determination.

8.5.3. When the samples are returned to the lab, extrude the (nominal) 5g (or 25 g) sample into a tared VOA vial containing 5 mL methanol (25 mL methanol for the 25 g sampler). Obtain the weight of the soil added to the vial and note on the label.

8.5.4. Add the correct amount of surrogate spiking mixture. (Add 25 µL of 2500 µg/mL solution for a nominal 25 g sample, 5 µL for a nominal 5 g sample.) Refer to Section 17.8 for Michigan project criteria.

8.5.5. Add the correct amount of matrix spiking solution to the matrix spike and matrix spike duplicate samples. (Add 500 µL of 50 µg/mL solution for a nominal 25 g sample, 100 µL for a nominal 5 g sample.) Reduce the volume of methanol added to ensure the final volume is 25 mL for nominal 25 g sample or 5 mL methanol for a nominal 5 g sample. Refer to Section 17.8 for Michigan project criteria.

8.5.6. Prepare an LCS for each batch by adding the correct amount of matrix spiking solution to clean methanol. (50 µL of spike to 25 mL methanol or 10 µL spike to 5 mL methanol). Refer to Section 17.8 for Michigan project criteria.

8.5.7. Shake the samples for two minutes to distribute the methanol throughout the soil.

8.5.8. Allow to settle, then remove a portion of methanol and store in a clean Teflon capped vial at 4 ±2°C until analysis.

8.6. Sample collection for medium level analysis using field methanol preservation

8.6.1. Prepare a 2 oz sample container by adding 25 mL purge and trap grade methanol. (If a 5 g sample is to be used, add 5 mL methanol to a 2 oz container or VOA vial).

8.6.2. Seal the bottle and attach a label.

8.6.3. Weigh the bottle to the nearest 0.01g and note the weight on the label.

8.6.4. Ship with appropriate sampling instructions.

8.6.5. Each sample will require an additional bottle with no preservative for percent moisture determination.

8.6.6. At client request, the methanol addition and weighing may also be performed in the field.

8.6.7. When the samples are returned to the lab, obtain the weight of the soil added to the vial and note on the label.

8.6.8. Add the correct amount of surrogate spiking mixture. (Add 25 µL of 2500 µg/mL solution for a nominal 25 g sample, 5 µL for a nominal 5 g sample.) Refer to Section 17.8 for Michigan project criteria.

8.6.9. Add the correct amount of matrix spiking solution to the matrix spike and matrix spike duplicate samples. (Add 25 µL of 50 µg/mL solution for a nominal 25 g sample, 100 µL for a nominal 5 g sample.) Reduce the volume of methanol added to ensure the final volume is 25 mL for nominal 25 g sample or 5 mL methanol for a nominal 5 g sample. Refer to Section 17.8 for Michigan project criteria.

8.6.10. Prepare an LCS for each batch by adding the correct amount of matrix spiking solution to clean methanol. (500 µL of spike to 25 mL methanol or 100 µL spike to 5 mL methanol). Refer to Section 17.8 for Michigan project criteria.

8.6.11. Shake the samples for two minutes to distribute the methanol throughout the soil.

8.6.12. Allow to settle, then remove a portion of methanol and store in a clean Teflon capped vial at 4+2°C until analysis.

8.7. Low level procedure

8.7.1. If low detection limits are required (typically < 50 µg/kg) sodium bisulfate preservation must be used. However, it is also necessary to take a sample for the medium level (field methanol preserved or using the EnCore™ sampler) procedure, in case the concentration of analytes in the soil is above the calibration range of the low level procedure.

8.7.2. A purge and trap autosampler capable of sampling from a sealed vial is required for analysis of samples collected using this method. (Varian Archon or O.I. 4552).

8.7.3. The soil sample is taken using a 5g EnCore™ sampling device and returned to the lab. It is recommended that two EnCore™ samplers be used for each field sample position, to allow for any reruns than may be necessary. A separate sample for % moisture determination is also necessary.

8.7.4. Prepare VOA vials by adding a magnetic stir bar, approximately 1 g of sodium bisulfate and 5 mL of reagent water.

8.7.5. Seal and label the vial. It is strongly recommended that the vial is labeled with an indelible marker rather than a paper label, since paper labels may cause the autosampler to bind and malfunction. The label absolutely must not cover the neck of the vial or the autosampler will malfunction.

8.7.6. Weigh the vial to the nearest 0.1g and note the weight on the label.

8.7.7. Extrude the soil sample from the EnCore™ sampler into the prepared VOA vial. Reweigh the vial to obtain the weight of soil and note on the label.

Note: Soils containing carbonates may effervesce when added to the sodium bisulfate solution. If this is the case at a specific site, add 5 mL of water instead, and freeze at <-10°C within 48 hours, analyzed within 12 days after preserving with water, and stored at a 45 degree angle in the freezer.

Note: Freezing is not allowed for Ohio VAP soil samples.

8.7.9. Alternatively the sodium bisulfate preservation may be performed in the field. This is not recommended because of the many problems that can occur in the field setting. Ship at least two vials per sample. The field samplers must determine the weight of soil sampled. Each sample will require an additional bottle with no preservative for percent moisture determination, and an additional bottle preserved with methanol for the medium level procedure. Depending on the type of soil it may also be necessary to ship vials with no or extra preservative.

8.8. Unpreserved soils

8.8.1. At specific client request unpreserved soils packed into glass jars or brass tubes may be accepted and subsampled in the lab. This is the old procedure based on method 5030A and method 8260A. It is no longer included in SW846 and is likely to generate results that are biased low, possibly be more than an order of magnitude.

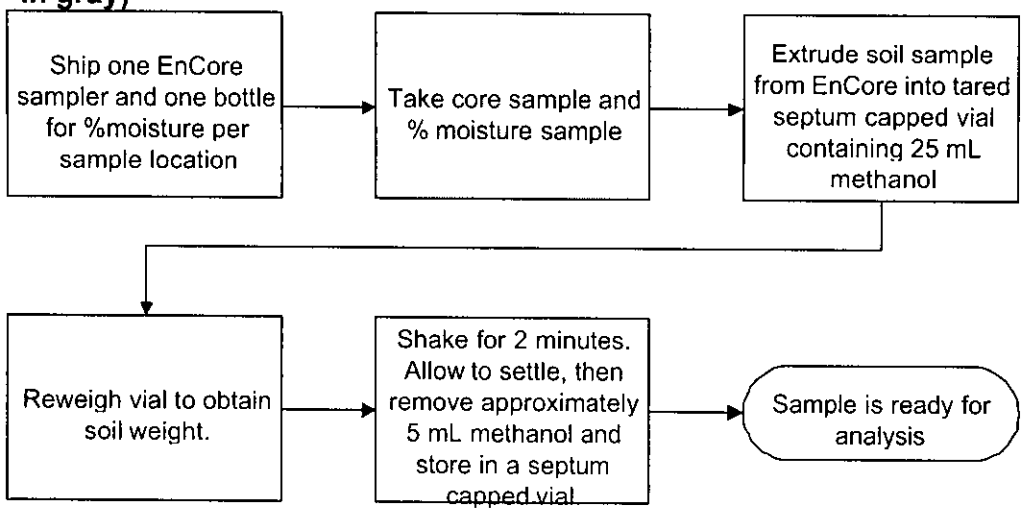
8.9. Aqueous samples are stored in glass containers with Teflon lined septa at 4°C +/- 2°C, with minimum headspace.

8.10. Medium level solid extracts are aliquoted into 2 - 5 mL glass vials with Teflon lined caps and stored at 4°C +/- 2°C. The extracts are stored with minimum headspace.

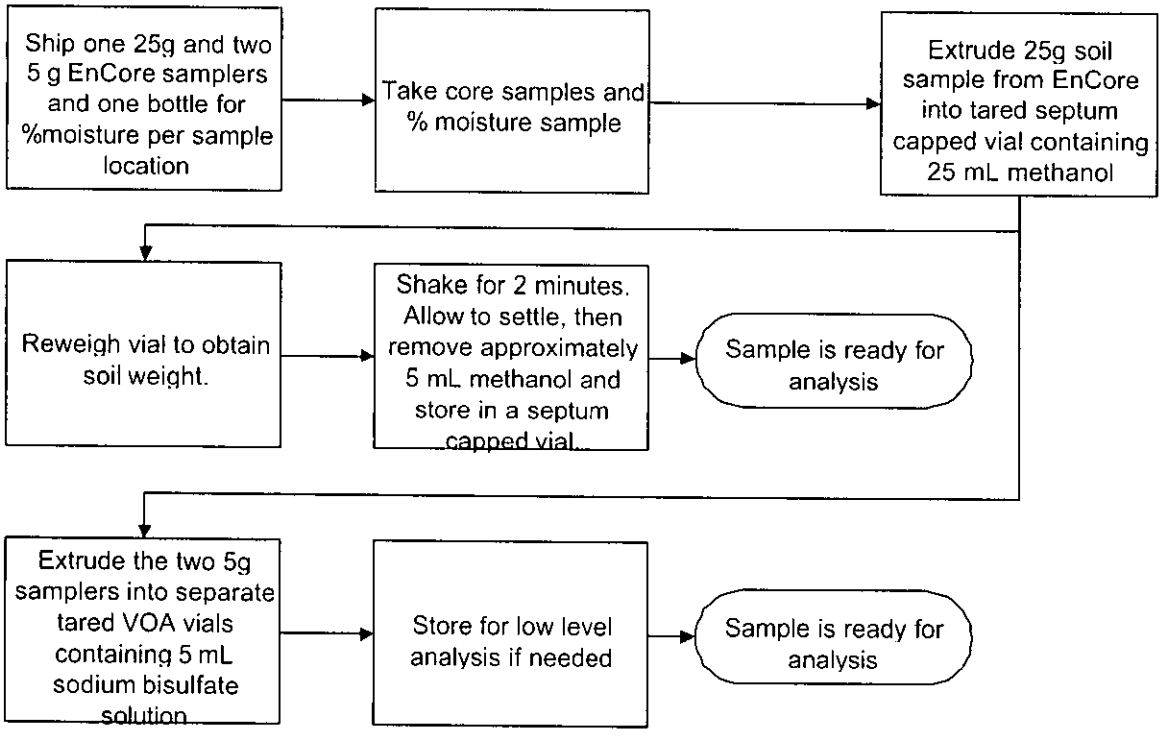
8.11. The maximum holding time is 14 days from sampling until the sample is analyzed. (Samples that are found to be unpreserved still have a 14 day holding time. However they should be analyzed as soon as possible. The lack of preservation should be addressed in the case narrative). Maximum holding time for the EnCore™ sampler (before the sample is added to methanol or sodium bisulfate) is 48 hours.

8.12. A holding blank is stored with the samples. This is analyzed and replaced if any of the trip blanks show any contamination. Otherwise it is replaced every 14 days.

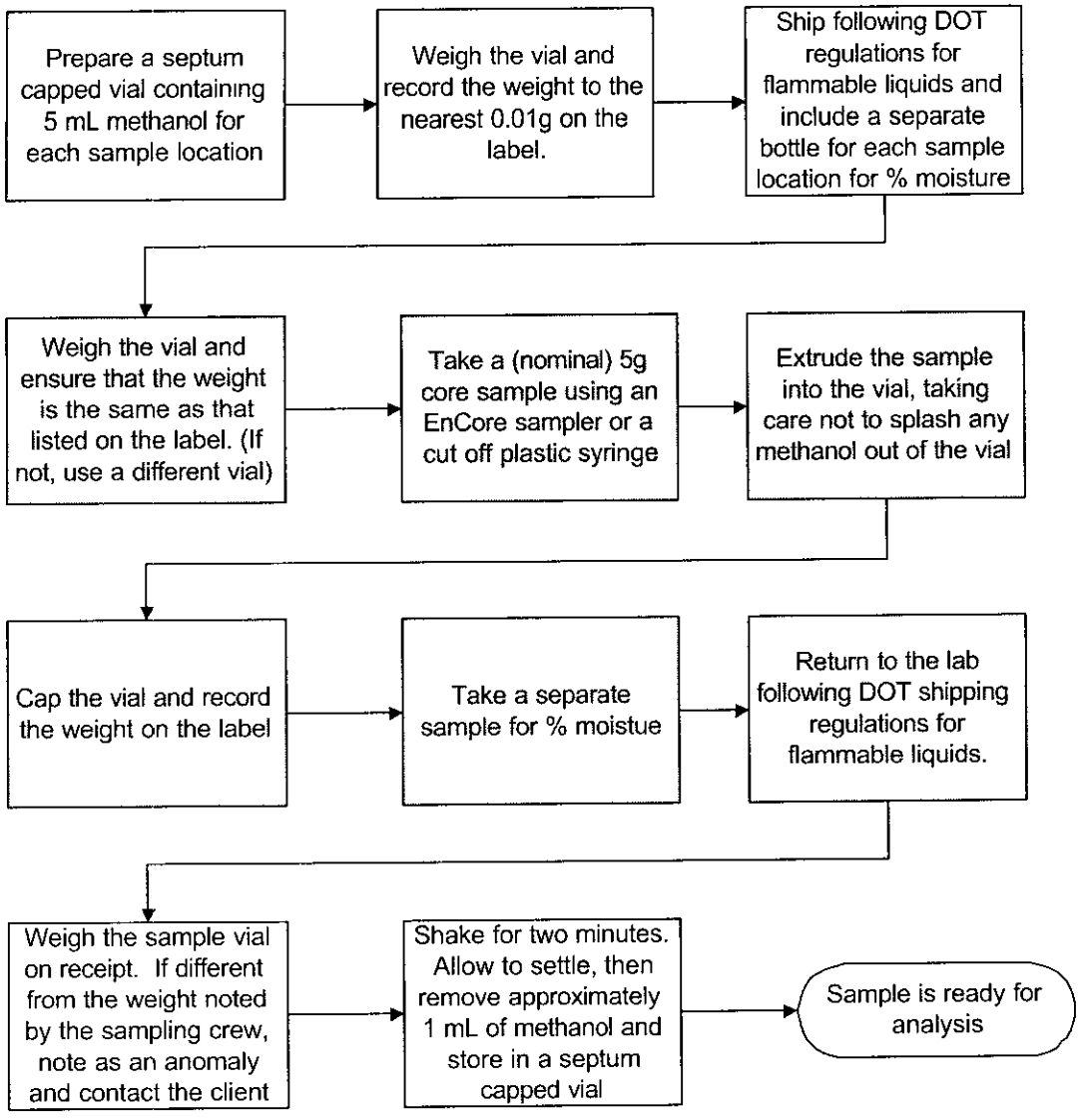
EnCore procedure when low level is not required (field steps in gray)



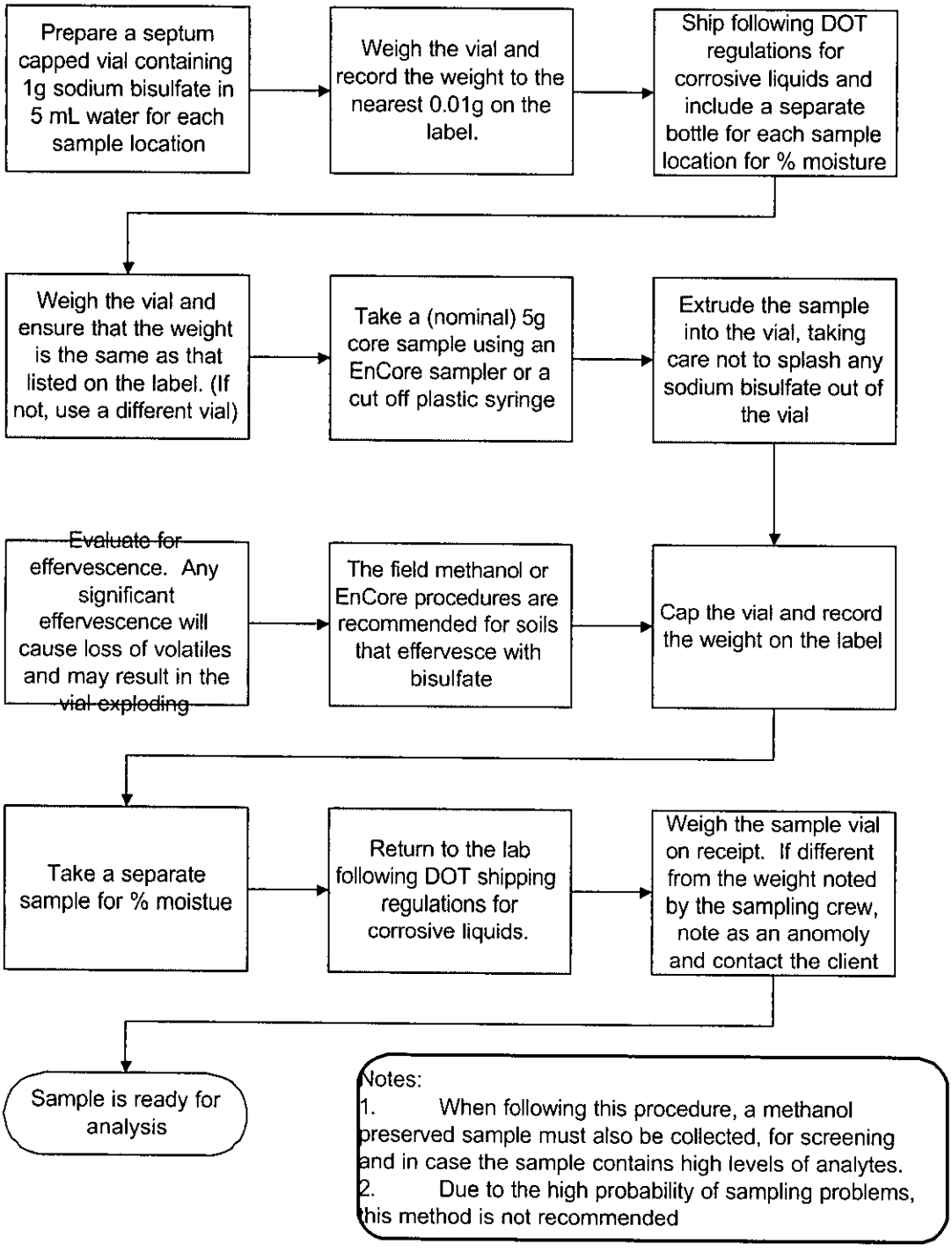
EnCore procedure when low level is required



Field methanol extraction procedure (field steps in gray)



Field bisulfate preservation procedure (field steps in gray)



9. QUALITY CONTROL

9.1. Initial Demonstration of Capability

9.1.1. For the standard analyte list, the initial demonstration described in Section 13 and method detection limit (MDL) studies must be acceptable before analysis of samples may begin. MDLs should be analyzed for low and medium soils and aqueous samples.

9.1.2. For non-standard analytes, a MDL study must be performed and calibration curve generated before analyzing any samples, unless lesser requirements are previously agreed to with the client. In any event, the minimum initial demonstration required is analysis of a standard at the reporting limit and a single point calibration.

9.2. Control Limits

In-house historical control limits must be determined for surrogates, matrix spikes, and laboratory control samples (LCS). These limits must be determined at least annually. The recovery limits are mean recovery +/- 3 standard deviations for surrogates, matrix spikes and LCS. Precision limits for matrix spikes / matrix spike duplicates are 0 to mean relative percent difference + 3 standard deviations.

9.2.1. All surrogate, LCS, and MS recoveries (except for dilutions) must be entered into QuantIMS (when available) or other database so that accurate historical control limits can be generated. For tests without a separate extraction, surrogates and matrix spikes will be reported for all dilutions.

9.2.2. Refer to the QC Program document (QA-003) for further details of control limits.

9.3. Surrogates

Every sample, blank, and QC sample is spiked with surrogates. Surrogate recoveries in samples, blanks, and QC samples must be assessed to ensure that recoveries are within established limits. The compounds included in the surrogate spiking solutions are listed in Table 8. If any surrogates are outside limits, the following corrective actions must take place (except for dilutions):

- Check all calculations for error.
- Ensure that instrument performance is acceptable.

- Recalculate the data and/or reanalyze if either of the above checks reveal a problem.

- Reprepare and reanalyze the sample or flag the data as "Estimated Concentration" if neither of the above resolves the problem.

The decision to reanalyze or flag the data should be made in consultation with the client. It is only necessary to reprepare/reanalyze a sample once to demonstrate that poor surrogate recovery is due to matrix effect, unless the analyst believes that the repeated out of control results are not due to matrix effect.

9.3.1. If the surrogates are out of control for the sample, matrix spike, and matrix spike duplicate, then matrix effect has been demonstrated for that sample and reprepparation is not necessary. If the sample is out of control and the MS and/or MSD is in control, then reanalysis or flagging of the data is required.

9.3.2. Refer to the STL QC Program document (QA-003) for further details of the corrective actions.

9.4. Method Blanks

9.4.1. For each batch of samples, analyze a method blank. The method blank is analyzed after the calibration standards, normally before any samples. For low-level volatiles, the method blank consists of reagent water. For medium-level volatiles, the method blank consists of 25.0 mL of methanol. Surrogates are added and the method blank is carried through the entire analytical procedure. The method blank must not contain any analyte of interest at or above the reporting limit (except common laboratory contaminants, see below) or at or above 5% of the measured concentration of that analyte in the associated samples, whichever is higher.

- If the analyte is a common laboratory contaminant (methylene chloride, acetone, 2-butanone) the data may be reported with qualifiers if the concentration of the analyte is less than five times the reporting limit. Such action must be taken in consultation with the client.

- Reanalysis of samples associated with an unacceptable method blank is required when reportable concentrations are determined in the samples.

- If there is no target analyte greater than the RL in the samples associated with an unacceptable method blank, the data may be reported with qualifiers. Such action should be done in consultation with the client.

9.4.2. The method blank must have acceptable surrogate recoveries. If surrogate recoveries are not acceptable, the data must be evaluated to determine if the method blank has served the

purpose of demonstrating that the analysis is free of contamination. If surrogate recoveries are low and there are reportable analytes in the associated samples re-extraction of the blank and affected samples will normally be required. Consultation with the client should take place.

9.4.3. If reanalysis of the batch is not possible due to limited sample volume or other constraints, the method blank is reported, all associated samples are flagged with a "B," and appropriate comments may be made in a narrative to provide further documentation.

9.4.4. Refer to the STL QC Program document (QA-003) for further details of the corrective actions.

9.5. Laboratory Control Samples (LCS)

9.5.1. For each batch of samples, analyze a LCS. The LCS is analyzed after the calibration standard, and normally before any samples. The LCS contains a representative subset of the analytes of interest (See Table 9), and must contain the same analytes as the matrix spike. If any analyte or surrogate is outside established control limits, the system is out of control and corrective action must occur. Corrective action will normally be repreparation and reanalysis of the batch.

- If the batch is not re-extracted and reanalyzed, the reasons for accepting the batch must be clearly presented in the project records and the report. (Examples of acceptable reasons for not reanalyzing might be that the matrix spike and matrix spike duplicate are acceptable, and sample surrogate recoveries are good, demonstrating that the problem was confined to the LCS.)

- If re-extraction and reanalysis of the batch is not possible due to limited sample volume or other constraints, the LCS is reported, all associated samples are flagged, and appropriate comments are made in a narrative to provide further documentation.

9.5.2. Refer to the STL QC Program document (QA-003) for further details of the corrective action.

9.5.3. If full analyte spike lists are used at client request, it will be necessary to allow a percentage of the components to be outside control limits as this would be expected statistically. These requirements should be negotiated with the client. Refer to Section 17.5 for Ohio VAP specific analytes.

9.6. Matrix Spikes

9.6.1. For each QC batch, analyze a matrix spike and matrix spike duplicate. Spiking compounds and levels are given in Table 9. Compare the percent recovery and relative percent difference

(RPD) to that in the laboratory specific historically generated limits. See Section 17.5 for Ohio VAP specific analytes.

- If any individual recovery or RPD falls outside the acceptable range, corrective action must occur. The initial corrective action will be to check the recovery of that analyte in the Laboratory Control Sample (LCS). Generally, if the recovery of the analyte in the LCS is within limits, then the laboratory operation is in control and analysis may proceed. The reasons for accepting the batch must be documented.

- If the recovery for any component is outside QC limits for both the matrix spike/ spike duplicate and the LCS, the laboratory is out of control and corrective action must be taken. Corrective action will normally include reanalysis of the batch.

- If a MS/MSD is not possible due to limited sample, then a LCS duplicate should be analyzed. RPD of the LCS and LCSD are compared to the matrix spike limits.

- The matrix spike/duplicate must be analyzed at the same dilution as the unspiked sample, even if the matrix spike compounds will be diluted out.

9.7. Nonconformance and Corrective Action

9.7.1. Any deviations from QC procedures must be documented as a nonconformance, with applicable cause and corrective action approved by the facility QA Manager.

9.8. Quality Assurance Summaries

Certain clients may require specific project or program QC which may supersede these method requirements. Quality Assurance Summaries should be developed to address these requirements.

9.9. STL QC Program

Further details of QC and corrective action guidelines are presented in the STL QC Program document (QA-003). Refer to this document if in doubt regarding corrective actions.

10. CALIBRATION AND STANDARDIZATION

10.1. Summary

10.1.1. Prior to the analysis of samples and blanks, each GC/MS system must be tuned and calibrated. Hardware tuning is checked through the analysis of the 4-Bromofluorobenzene (BFB) to establish that a given GC/MS system meets the standard mass spectral abundance criteria. The

GC/MS system must be calibrated initially at a minimum of five concentrations (analyzed under the same BFB tune), to determine the linearity of the response utilizing target calibration standards. Once the system has been calibrated, the calibration must be verified each twelve hour time period for each GC/MS system.

10.2.1. General

Electron Energy:	70 volts (nominal)
Mass Range:	35–300 AMU
Scan Time:	to give at least 5 scans/peak, but not to exceed 2 second/scan
Injector Temperature:	200–250°C
Source Temperature:	According to manufacturer's specifications
Transfer Line	Temperature: 250–300°C
Purge Flow:	40 mL/minute
Carrier Gas	Flow: 15 mL/minute
Make-up Gas Flow:	25–30 mL/minute

10.2.2. Gas chromatograph suggested temperature program

10.2.2.1. BFB Analysis

Isothermal:	170°C

10.2.2.2. Sample Analysis

Initial Temperature:	40°C
Initial Hold Time:	4 minutes
Temperature Program:	8°C/minute
Final Temperature:	184°C
Second Temperature	Program: 40°C/minute
Final Temperature:	240°C
Final Hold Time:	2.6 minutes

10.3. Instrument Tuning

10.3.1. Each GC/MS system must be hardware-tuned to meet the abundance criteria listed in Table 10 for a maximum of a 50 ng injection or purging of BFB. Analysis must not begin until these criteria are met. These criteria must be met for each twelve-hour time period. The twelve-hour time period begins at the moment of injection of BFB.

10.4. Initial Calibration

10.4.1. A series of five initial calibration standards is prepared and analyzed for the target compounds and each surrogate compound. Six standards must be used for a quadratic least squares calibration. Suggested calibration levels for a 5 mL purge are: 5, 20, 50, 100, and 200 µg/L. Certain analytes are prepared at higher concentrations due to poor purge performance. Suggested calibration levels for a 25 mL purge are 1, 5, 10, 20, and 40 µg/L. Again, some analytes are prepared at higher levels. Tables 2 and 4 list the calibration levels for each analyte. Other calibration levels and purge volumes may be used depending on the capabilities of the specific instrument. (For example, adequate sensitivity can be obtained on the Agilent 5973 instruments to use a 5 mL purge volume to reach the same reporting limits that once required a 25 mL purge. The calibration levels will still be the same 1, 5, 10, 20, 40µg/L.) However, the same purge volume must be used for calibration and sample analysis, and the low level standard must be at or below the reporting limit.

10.4.2. It may be necessary to analyze more than one set of calibration standards to encompass all of the analytes required for same tests. For example, the Appendix IX list requires the Primary standard (Table 5) and the Appendix IX standard (Table 6). If acceptable analytical performance can be obtained the primary and appendix IX standards may be analyzed together.

10.4.3. Internal standard calibration is used. The internal standards are listed in Table 7. Target compounds should reference the nearest internal standard. Each calibration standard is analyzed and the response factor (RF) for each compound is calculated using the area response of the characteristic ions against the concentration for each compound and internal standard. See equation 1, Section 12, for calculation of response factor.

10.4.4. The % RSD of the calibration check compounds (CCC) must be less than 30%. Refer to Table 12 for the CCCs.

10.4.4.1. If none of the CCCs are required analytes, project specific calibration specifications must be agreed with the client.

10.4.5. The average RF must be calculated for each compound. A system performance check is made prior to using the calibration curve. The five system performance check compounds (SPCC) are checked for a minimum average response factor. Refer to Table 11 for the SPCC compounds and required minimum response factors.

10.4.6. If the average of all the %RSDs in the calibration is $\leq$ 15%, then all analytes may use average response factor for calibration.

10.4.6.1. If the software in use is capable of routinely reporting curve coefficients for data validation purposes, and the necessary calibration reports can be generated, then the analyst should evaluate analytes with %RSD > 15% for calibration on a curve. If it appears that substantially better accuracy would be obtained using quantitation from a curve then the appropriate curve should be used for quantitation. If Relative Standard Error (RSE) is used to evaluate the curve it must be better than 15%. Otherwise the correlation coefficient (coefficient of determination for non-linear curves) must be $\geq$ 0.990.

10.4.6.2. If the average of all the %RSDs in the calibration is > 15% then calibration on a curve must be used for all analytes with %RSD > 15%. Linear or quadratic curve fits may be used. The analyst should consider instrument maintenance to improve the linearity of response. If Relative Standard Error (RSE) is used to evaluate the curve it must be better than 15%. If the % RSD is > 15%, the analyst may drop the low or high in the ICAL , as long as a minimum of 5 points are maintained and the quantitation range is adjusted accordingly. Otherwise the correlation coefficient, (coefficient of determination, r^2 for non-linear curves) must be $\geq$ 0.990. If the correlation coefficient is < 0.990, then any hit for these compounds must be flagged as estimated.

10.4.6.3. Refer to Section 17.5 for specific Ohio VAP criteria.

10.4.7. Weighting of data points

In a linear or quadratic calibration fit, the points at the lower end of the calibration curve have less weight in determining the curve generated than points at the high concentration end of the curve. However, in environmental analysis, accuracy at the low end of the curve is very important. For this reason it is preferable to increase the weighting of the lower concentration points. 1/Concentration^2 weighting (often called 1/X^2 weighting) will improve accuracy at the low end of the curve and should be used if the data system has this capability.

10.4.8. If time remains in the 12-hour period initiated by the BFB injection before the initial calibration, samples may be analyzed. Otherwise, proceed to continuing calibration.

10.4.9. The calibration standards for the initial 5-point calibration for low level soils that are not preserved in sodium bisulfate (i.e. are preserved by freezing, or not preserved) must be heated to 40°C for purging. Using this calibration curve for water samples is acceptable as long as all calibration, QC, and samples are also heated to 40°C. A separate five point calibration must be prepared for analysis of low level soils that are preserved with sodium bisulfate. Low level soils analysis requires the use of a closed vial autosampler such as the Varian Archon, O.I. 4552 or Tekmar Precept. Each standard for analysis of sodium

bisulfate preserved samples is prepared by spiking the methanolic standard solution through the septum of a VOA vial containing 5 mL of water and 1 g sodium bisulfate. The standards are heated to 40°C for purging. All low-level soil samples, standards, and blanks must also be heated to 40°C for purging. Medium soil extracts should be analyzed using the water (unheated or optionally heated) calibration curve as long as all calibration standards, samples, and QC samples are purged at the same temperature.

10.4.10. Non-standard analytes are sometimes requested. For these analytes, it is acceptable to analyze a single standard at the reporting limit with each continuing calibration rather than a five point initial calibration. If the analyte is detected in any of the samples, a five point initial calibration must be generated and the sample(s) reanalyzed for quantitation. However, if the analyte is not detected, the non-detect may be reported and no further action is necessary.

Note: This procedure is may not be used for Ohio VAP samples.

10.5. Continuing Calibration: The initial calibration must be verified every twelve hours.

10.5.1. Continuing calibration begins with analysis of BFB as described in Section 10.3. If the system tune is acceptable, the continuing calibration standard(s) are analyzed. The level 3 calibration standard is used as the continuing calibration.

10.5.2. The RF data from the standards are compared with the average RF from the initial five-point calibration to determine the percent drift of the CCC compounds. The calculation is given in equation 4, Section 12.3.4.

10.5.3. The % drift of the CCCs must be $\leq$ 20% for the continuing calibration to be valid. The SPCCs are also monitored. The SPCCs must meet the criteria described in Table 11. In addition, the % drift of all analytes must be $\leq$ 50% with allowance for up to six target analytes to have % drift > 50%.

10.5.3.1. If none of the CCCs are required analytes, project specific calibration specifications must be agreed with the client.

10.5.3.2. Cyclohexanone, one of the components of the Appendix IX standard, is unstable in the calibration solution, forming 1,1-dimethoxycyclohexane. No calibration criteria are applied to cyclohexanone and quantitation is tentative. Cyclohexanone is included on the Universal Treatment Standard and FO-39 regulatory lists (but not on Appendix IX).

10.5.3.3. Refer to Table 12 for specific Ohio VAP analytes.

10.5.4. If the CCCs and or the SPCCs do not meet the criteria in Sections 10.5.3 and 10.5.4, the system must be evaluated and corrective action must be taken. The BFB tune and continuing calibration must be acceptable before analysis begins. Extensive corrective action such as a different type of column will require a new initial calibration.

10.5.5. Once the above criteria have been met, sample analysis may begin. **Initial calibration average RFs (or the calibration curve) will be used for sample quantitation, not the continuing calibration RFs.** Analysis may proceed until 12 hours from the injection of the BFB have passed. (A sample *desorbed* less than or equal to 12 hours after the BFB is acceptable.)

11. PROCEDURE

11.1. Procedural Variations

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

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

11.2. Preliminary Evaluation

11.2.1. Where possible, samples are screened by headspace or GC/MS off-tune analysis to determine the correct aliquot for analysis. Alternatively, an appropriate aliquot can be determined from sample histories.

11.2.2. Dilutions should be done just prior to the GC/MS analysis of the sample. Dilutions are made in volumetric flasks or in a Luerlok syringe. Calculate the volume of reagent water required for the dilution. Fill the syringe with reagent water, compress the water to vent any residual air and adjust the water volume to the desired amount. Adjust the plunger to the mark and inject the proper aliquot of sample into the syringe. If the dilution required would use less than 1 µL of sample then serial dilutions must be made in volumetric flasks.

11.2.2.1. The diluted concentration is to be estimated to be in the upper half of the calibration range.

11.3. Sample Analysis Procedure

11.3.1. All analysis conditions for samples must be the same as for the continuing calibration standards (including purge time and flow, desorb time and temperature, column temperatures, multiplier setting etc.).

11.3.2. All samples must be analyzed as part of a batch. The batch is a set of up to 20 samples of the same matrix processed using the same procedures and reagents within the same time period. The batch also must contain a MS/MSD, a LCS, and a method blank.

11.3.2.1. If there is insufficient time in the 12-hour tune period to analyze 20 samples, the batch may be continued into the next tune period. However, if any re-tuning of the instrument is necessary, or if a period of greater than 24 hours from the preceding BFB tune has passed, a new batch must be started. For medium level soils the batch is defined at the sample preparation stage.

11.3.2.2. Laboratory generated QC samples (Blank, LCS, MS/MSD) do not count towards the maximum 20 samples in a batch. Field QC samples are included in the batch count.

11.3.2.3. It is not necessary to reanalyze batch QC with reanalyses of samples. However, any reruns must be as part of a valid batch.

11.4. Water Samples

11.4.1. All samples and standard solutions must be at ambient temperature before analysis.

11.4.2. Fill a syringe with the sample. If a dilution is necessary it may be made in the syringe if the sample aliquot is $\geq$ 5 µL. Check and document the pH of the remaining sample.

11.4.3. Add 250 ng of each internal and surrogate standard (10 µL of a 25 µg/mL solution, refer to Tables 7, 8 and 16). The internal standards and the surrogate standards may be mixed and added as one spiking solution (this results in a 50 µg/L solution for a 5 mL sample, and a 10 µg/L solution for a 25 mL sample). Inject the sample into the purging chamber.

11.4.3.1. For TCLP samples use 0.5 mL of TCLP leachate with 4.5 mL reagent water and spike with 10 µL of the 25 µg/mL TCLP spiking solution. (Note that TCLP reporting limits will be 10 times higher than the corresponding aqueous limits).

11.4.4. Purge the sample for eleven minutes (the trap must be below 35°C).

11.4.5. After purging is complete, desorb the sample, start the GC temperature program, and begin data acquisition. After desorption, bake the trap for approximately 3-10 minutes to condition it for the next analysis. When the trap is cool, it is ready for the next sample.

11.4.6. Desorb and bake time and temperature are optimized for the type of trap in use. The same conditions must be used for samples and standards.

11.5. Methanol Extract Soils

11.5.1. Rinse a gas-tight syringe with organic free water. Fill the syringe with the same volume of organic free water as used in the calibrations. Add no more than 2% (v/v) (100 µL for a 5 mL purge) methanolic extract (from Section 8.5 or 8.6) to the syringe. Add internal standard (if used). Load the sample onto the purge and trap device and analyze as for aqueous samples. If less than 5µL of methanolic extract is to be added to the water, dilute the methanolic extract such that a volume greater than 5µL will be added to the water in the syringe. Refer to Section 17.8 for Michigan project requirements.

11.6. Liquid wastes that are soluble in methanol and insoluble in water.

11.6.1. Pipet 2 mL of the sample into a tared vial. Use a top-loading balance. Record the weight to the nearest 0.1 gram.

11.6.2. Quickly add 7 mL of methanol, then add 1 mL of surrogate spiking solution to bring the final volume to 10 mL. Cap the vial and shake for 2 minutes to mix thoroughly. For a MS/MSD or LCS, 6 mL of methanol, 1 mL of surrogate solution, and 1 mL of matrix spike solution is used.

11.6.3. Rinse a gas-tight syringe with organic free water. Fill the syringe with the same volume of organic free water as used in the calibrations. Add no more than 2% (v/v) (100 µL for a 5 mL purge) methanolic extract (from Section 8.5 or 8.6) to the syringe. Add internal standard (if used). Load the sample onto the purge and trap device and analyze as for aqueous samples. If less than 5µL of methanolic extract is to be added to the water, dilute the methanolic extract such that a volume greater than 5µL will be added to the water in the syringe.

11.7. Aqueous and Low level Soil Sample Analysis (Purge and Trap units that sample directly from the VOA vial)

11.7.1. Units which sample from the VOA vial should be equipped with a module which automatically adds surrogate and internal standard solution to the sample prior to purging the sample.

11.7.2. If the autosampler uses automatic IS/SS injection, no further preparation of the VOA vial is needed. Otherwise the internal and surrogate standards must be added to the vial. *Note:* Aqueous samples with high amounts of sediment present in the vial may not be suitable for analysis on this instrumentation, or they may need to analyzed as soils.

11.7.3. Soil samples must be quantitated against a curve prepared with standards containing about the same amount of sodium bisulfate as the samples (1 g in 5 mL).

11.7.4. Sample remaining in the vial after sampling with one of these mechanisms is no longer valid for further analysis. A fresh VOA vial must be used for further sample analysis.

11.7.5. For aqueous samples, check the pH of the sample remaining in the VOA vial after analysis is completed.

11.8. Low-Level Solids Analysis using discrete autosamplers, Method 8260A, 5030A.

Note: This technique may seriously underestimate analyte concentration and must not be used except at specific client request for the purpose of comparability with previous data. It is no longer part of SW-846.

This method is based on purging a heated soil/sediment sample mixed with reagent water containing the surrogates and internal standards. Analyze all reagent blanks and standards under the same conditions as the samples (e.g., heated). The calibration curve is also heated during analysis. Purge temperature is 40°C.

11.8.1. Do not discard any supernatant liquids. Mix the contents of the container with a narrow metal spatula.

11.8.2. Weigh out 5 g (or other appropriate aliquot) of sample into a disposable culture tube or other purge vessel. Record the weight to the nearest 0.1 g. If method sensitivity is demonstrated, a smaller aliquot may be used. Do not use aliquots less than 1.0 g. If the sample is contaminated with analytes such that a purge amount less than 1.0 g is appropriate, use the medium level method. For the medium level method, add 4g soil to 10 mL methanol containing the surrogates, mix for two minutes, allow to settle then remove a portion of the methanol and store in a clean Teflon capped vial at 4°C until analysis. Analyze as described in section 11.5.

11.8.3. Connect the purge vessel to the purge and trap device.

11.8.4. Rinse a 5 mL gas-tight syringe with organic free water, and fill. Compress to 5 mL. Add surrogate/internal standard (and matrix spike solutions if required.). Add directly to the sample from 11.5.2.

11.8.5. The above steps should be performed rapidly and without interruption to avoid loss of volatile organics.

11.8.6. Add the heater jacket or other heating device and start the purge and trap unit.

11.8.7. Soil samples that have low IS recovery when analyzed (<50%) should be reanalyzed once to confirm matrix effect.

11.9. Medium-Level Soil/Sediment and Waste Samples

11.9.5. Sediments/soils and waste that are insoluble in methanol.

11.9.5.1. Sediments/soils and waste that are insoluble in methanol.

11.9.5.1.1. Gently mix the contents of the sample container with a narrow metal or wood spatula. Weigh 4 g (wet weight) into a tared vial. Use a top-loading balance. Record the weight to 0.1 gram. Do not discard any supernatant liquids.

11.9.5.1.2. Quickly add 9 mL of methanol, and 1 mL of surrogate spiking solution to bring the final volume of methanol to 10 mL. For an LCS or MS/MSD sample add 8 mL of methanol, 1 mL of surrogate spike solution, and 1 mL of matrix spike solution. Cap the vial and vortex to mix thoroughly.

NOTE: Sections 11.9.5.1.1 and 11.9.5.1.2 must be performed rapidly and without interruption to avoid the loss of volatile organics.

11.10. Initial review and corrective actions

11.10.1. If the retention time for any internal standard in the continuing calibration changes by more than 0.5 minutes from the mid-level initial calibration standard, the chromatographic system must be inspected for malfunctions and corrected. Reanalysis of samples analyzed while the system was malfunctioning is required.

11.10.2. If the internal standard response in the continuing calibration is more than 200% or less than 50% of the response in the mid-level of the initial calibration standard, the chromatographic system must be inspected for malfunctions and corrected. Reanalysis of samples analyzed while the system was malfunctioning is required.

11.10.2.1. Any samples that do not meet the internal standard criteria for the continuing calibration must be evaluated for validity. If the change in sensitivity is a matrix effect confined to an individual sample reanalysis is not necessary. If the change in sensitivity is

due to instrumental problems all affected samples must be reanalyzed after the problem is corrected.

11.10.3. The surrogate standard recoveries are evaluated to ensure that they are within limits. Corrective action for surrogates out of control will normally be to reanalyze the affected samples. However, if the surrogate standard response is out high and there are no target analytes or tentatively identified compounds, reanalysis may not be necessary. Out of control surrogate standard response may be a matrix effect. It is only necessary to reanalyze a sample once to demonstrate matrix effect, but reanalysis at a dilution should be considered.

11.11. Dilutions

If the response for any compound exceeds the working range of the GC/MS system, a dilution of the extract is prepared and analyzed. An appropriate dilution should be in the upper half of the calibration range. Samples may be screened to determine the appropriate dilution for the initial run. If the initial diluted run has no hits or hits below 20% of the calibration range and the matrix allows for analysis at a lesser dilution, then the sample must be reanalyzed at a dilution targeted to bring the largest hit above 50% of the calibration range.

11.10.1. Guidance for Dilutions Due to Matrix

If the sample is initially run at a dilution and the baseline rise is less than half the height of the internal standards, or if individual non target peaks are less than twice the height of the internal standards, then the sample should be reanalyzed at a more concentrated dilution. This requirement is approximate and subject to analyst judgement.

11.10.2. Reporting Dilutions

The most concentrated dilution with no target compounds above the calibration range will be reported. Other dilutions will only be reported at client request.

12. DATA ANALYSIS AND CALCULATIONS

12.1. Qualitative identification

An analyte is identified by retention time and by comparison of the sample mass spectrum with the mass spectrum of a standard of the suspected compound (standard reference spectrum). Mass spectra for standard reference may be obtained on the user's GC/MS by analysis of the calibration standards or from the NIST Library. Two criteria must be satisfied to verify identification: (1) elution of sample component at the same GC retention time as the standard component; and (2) correspondence of the sample component and the standard component

characteristic ions. (Note: Care must be taken to ensure that spectral distortion due to co-elution is evaluated.)

- The sample component retention time must compare to within ± 0.2 min. of the retention time of the standard component. For reference, the standard must be run within the same twelve hours as the sample.
- All ions present in the standard mass spectra at a relative intensity greater than 10% (most abundant ion in the spectrum equals 100%) should be present in the sample spectrum.
- The relative intensities of ions should agree to within ±30% between the standard and sample spectra. (Example: For an ion with an abundance of 50% in the standard spectra, the corresponding sample abundance must be between 20 and 80 percent.)

12.1.1. If a compound cannot be verified by all the above criteria, but in the technical judgment of the analyst, the identification is correct, then the analyst shall report that identification and proceed with quantitation.

12.2. Tentatively Identified Compounds (TICs)

12.2.1. If the client requests components not associated with the calibration standards, a search of the NIST library may be made for the purpose of tentative identification. Guidelines are:

12.2.1.1. Relative intensities of major ions in the reference spectrum (ions > 10% of the most abundant ion) should be present in the sample spectrum.

12.2.1.2. The relative intensities of the major ions should agree to within 20%. (Example: If an ion shows an abundance of 50% in the standard spectrum, the corresponding sample ion abundance must be between 30% and 70%).

12.2.1.3. Molecular ions present in the reference spectrum should be present in the sample spectrum.

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

12.2.1.5. Ions present in the reference spectrum but not in the sample spectrum should be reviewed for possible subtraction from the spectrum because of background contamination or coeluting peaks. (Data system reduction programs can sometimes create these discrepancies.)

12.2.1.6. Computer-generated library search routines should not use normalization routines that would misrepresent the library or unknown spectra when compared to each other. Only after visual inspection of the sample with the nearest library searches should the analyst assign a tentative identification.

12.3. Calculations.

12.3.1. Response factor (RF):

Equation 1

$$RF = \frac{A_x C_{is}}{A_{is} C_x}$$

Where:

A_x = Area of the characteristic ion for the compound to be measured

A_{is} = Area of the characteristic ion for the specific internal standard

C_{is} = Concentration of the specific internal standard, ng

C_x = Concentration of the compound being measured, ng

12.3.2. Standard deviation (SD):

Equation 2

$$SD = \sqrt{\sum_{i=1}^{N} \frac{(Xi - X)^2}{N - 1}}$$

X_i = Value of X at i through N

N = Number of points

X = Average value of X_i

12.3.3. Percent relative standard deviation (%RSD):

Equation 3

$$\%RSD = \frac{\text{Standard Deviation}}{\overline{RF_i}} \times 100$$

$\overline{RF_i}$ = Mean of RF values in the curve

12.3.4. Percent drift between the initial calibration and the continuing calibration:

Equation 4

$$\% \text{ Drift} = \frac{C_{expected} - C_{found}}{C_{expected}} \times 100$$

Where

$C_{expected}$ = Known concentration in standard

C_{found} = Measured concentration using selected quantitation method

12.3.5. Target compound and surrogate concentrations:

Concentrations in the sample may be determined from linear or second order (quadratic) curve fitted to the initial calibration points, or from the average response factor of the initial calibration points. Average response factor may only be used when the % RSD of the response factors in the initial calibration is $\leq$ 15%.

12.3.5.1. Calculation of concentration using Average Response Factors

Equation 5

$$\text{Concentration } \mu g / L = \frac{x}{RF}$$

12.3.5.2. Calculation of concentration using Linear fit

Equation 6

$$\text{Concentration } \mu g / L = A + Bx$$

12.3.5.3. Calculation of concentration using Quadratic fit

Equation 7

$$\text{Concentration } \mu g / L = A + Bx + Cx^2$$

x is defined in equations 8, 9 and 10

A is a constant defined by the intercept

B is the slope of the curve

C is the curvature

12.3.5.4. Calculation of x for Water and water-miscible waste:

Equation 8

$$x = \frac{(A_x)(I_s)(D_f)}{(A_{is})(V_o)}$$

Where:

A_x = Area of characteristic ion for the compound being measured (secondary ion quantitation is allowed only when there are sample interferences with the primary ion)

A_{is} = Area of the characteristic ion for the internal standard

I_s = Amount of internal standard added in ng

$$\text{Dilution Factor} = D_f = \frac{\text{Total volume purged (mL)}}{\text{Volume of original sample used (mL)}}$$

V_o = Volume of water purged, mL

12.3.5.5. Calculation of x for Medium level soils:

Equation 9

$$x = \frac{(A_x)(I_s)(V_t)(1000)(D_f)}{(A_{is})(V_a)(W_s)(D)}$$

Where:

A_x, I_s, D_f, A_{is}, same as for water.

V_t = Volume of total extract, mL (Typically 25 mL)

V_a = Volume of extract added for purging, µL

W_s = Weight of sample extracted, g

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

12.3.5.6. Calculation of x for Low level soils:

Equation 10

$$x = \frac{(A_x)(I_s)}{(A_{is})(W_s)(D)}$$

Where:

A_x, I_s, A_{is}, same as for water.

D is as for medium level soils

W_s = Weight of sample added to the purge vessel, g

12.3.5.7. Calculation of TICs: The calculation of TICs (tentatively identified compounds) is identical to the above calculations with the following exceptions:

A_x = Area in the total ion chromatogram for the compound being measured

A_{is} = Area of the total ion chromatogram for the nearest internal standard without interference

RF = 1

In other words, the concentration is equal to x as defined in equations 8, 9 and 10.

12.3.6. MS/MSD Recovery

Equation 11

$$\textbf{Matrix Spike Recovery, \%} = \frac{SSR - SR}{SA} \times 100$$

SSR = Spike sample result
SR = Sample result
SA = Spike added

12.3.7. Relative % Difference calculation for the MS/MSD

Equation 12

$$\mathbf{RPD} = \frac{|\mathbf{MSR - MSDR}|}{\frac{1}{2}(\mathbf{MSR + MSDR})} \times \mathbf{100}$$

Where:

RPD = Relative percent difference

MSR = Matrix spike result

MSDR = Matrix spike duplicate result

13. METHOD PERFORMANCE

13.1. Method Detection Limit

13.2. Generally, each laboratory must generate a valid method detection limit for each analyte of interest. The MDL must be below the reporting limit for each analyte. The procedure for determination of the method detection limit is given in 40 CFR Part 136, Appendix B, and further defined in QA Policy #: QA-005. When non-standard compounds are analyzed at client request, lesser requirements are possible with client agreement. At a minimum, a standard at the reporting limit must be analyzed to demonstrate the capability of the method.

13.3. Initial Demonstration

13.4. Each laboratory must make a one time initial demonstration of capability for each individual method. Demonstration of capability for both soil and water matrices is required. This requires analysis of QC check samples containing all of the standard analytes for the method. For some tests it may be necessary to use more than one QC check mix to cover all analytes of interest. The QC check sample is made up at 20 µg/L. (Some compounds will be at higher levels, refer to the calibration standard levels for guidance.)

13.4.1. Four aliquots of the QC check sample are analyzed using the same procedures used to analyze samples, including sample preparation.

13.4.2. Calculate the average recovery and standard deviation of the recovery for each analyte of interest. The %RSD should be $\leq$ 15% for each analyte, and the % recovery should be within 80-120%.

13.4.3. If any analyte does not meet the acceptance criteria, check the acceptance limits in the reference methods (Table 6 of method 8240B, paragraph 8.3.5 of method 8260A). If the recovery or precision is outside the limits in the reference methods, the test must be repeated. Only those analytes that did not meet criteria in the first test need to be evaluated. Repeated failure for any analyte indicates the need for the laboratory to evaluate the analytical procedure and take corrective action.

13.4.4. Training Qualification

13.4.4.1. The group/team leader 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.

14. POLLUTION PREVENTION

14.1. This method does not contain any specific modifications that serve to minimize or prevent pollution.

15. WASTE MANAGEMENT

15.1. Waste generated in the procedure must be segregated and disposed according to the facility hazardous waste procedures. The Health and Safety Director should be contacted if additional information is required.

16. REFERENCES

16.1. SW846, *Test Methods for Evaluating Solid Waste,* Third Edition, Gas Chromatography/Mass Spectrometry for Volatile Organics, Method 8260B, Update III, December 1996

16.2. SW846, *Test Methods for Evaluating Solid Waste,* Third Edition, Gas Chromatography/Mass Spectrometry for Volatile Organics, Method 8260A, Update II, September 1994.

17. MISCELLANEOUS

17.1. Modifications from the reference method

17.1.1. Ion 119 is used as the quantitation ion for chlorobenzene-d5 for 25 mL purge tests.

17.1.2. A retention time window of 0.2 minutes is used for all components, since some data systems do not have the capability of using the relative retention time units specified in the reference method.

17.1.3. The quantitation and qualifier ions for some compounds have been changed from those recommended in SW-846 in order to improve the reliability of qualitative identification.

17.1.4. Method 8260A recommends that the purge vessel is run through an additional purge cycle after 25 mL sample analysis to remove carryover. Instead, purge vessels are oven baked between analyses or disposable vessels are used one time only.

17.1.5. SW-846 recommends that a curve be used for any analytes with %RSD of the response factors > 15%. However, some industry standard data systems and forms generation software cannot report this data with the necessary information for data validation. In

addition most software available does not allow weighting of the curve. Unweighted curves may exhibit serious errors in quantitation at the low end, resulting in possible false positives or false negatives. Therefore, this SOP allows used of average response factors if the average %RSD for all compounds is ≤ 15%.

17.2. Modifications from previous revision

This SOP has been substantially revised to reflect the changes included in Update III to SW-846. Directions for method 524.2 and method 624 have also been added.

17.3. Facility specific SOPs

Each facility shall attach a list of facility-specific SOPs or approved attachments (if applicable) which are required to implement this SOP or which are used in conjunction with this SOP. If no facility specific SOPs or amendments are to be attached, a statement must be attached specifying that there are none.

17.4. Flow diagrams

17.4.1. Initial Demonstration and MDL

17.5. The following are protocols that must be followed when analyzing OhioVAP samples:

- Sections 9.5 and 9.6: n-Hexane must be spiked and reported for both the LCS and MS/MSD.
- Sections 10.4.6: All analytes must have a %RSDs ≤ 15%. Corrective action must be completed for any compounds failing the <15% requirement.
- Section 11.1 and 17.1.5 (Method deviations) are not to be performed.
- Section 11.9.2: For OhioVAP projects, the laboratory will reanalyze any sample where the internal standard fails and there is no evidence of matrix interference.

17.6. The following are protocols that must be followed when analyzing BP Oil – Lima Refinery RFI work plan.

- Section 8.1 STL will continue to follow the 14 day holding time specified in the Corporate SOP.
- Delete for this project Section 8.3 At specific client request, unpreserved soil samples may be accepted.

- Delete for this project Section 8.8.1 At specific client request unpreserved soils packed into glass jars or brass tubes may be accepted and subsampled in the lab. This is the old procedure based on method 5030A. It is no longer included and is likely to generate results that are biased low, possibly by more than an order of magnitude.

- Modify Section 8.5.8 For the purpose of this project, the soil/methanol mixture may be stored for two days prior to analysis.

- Modify Section 8.6.12 For the purpose of this project, the soil/methanol mixture may be stored for two days prior to analysis.

- Modify (per discussion with Region V representative) to Section 10.4.6.2 Compounds with %RSD >15% are to be calibrated using an alternate calibration technique (e.g. linear or quadratic calibration curve). For poor responders, the alternate calibration technique requirements may not be met either. This sentence is added for those cases.If the correlation coefficient is < 0.990, then any hit for these compounds must be flagged as estimated.

- Modify Section 10.4.2 It is necessary to analyze the Appendix IX standard separately from the primary standard due to the presence of xylene solvent in the Appendix IX standard. Alternatively, STL will purchase the Appendix IX standard in a solvent other than xylene.

- Modify Section 10.4.9 For this project, this section will be modified to comply with the requirement of adding methanol to the calibration standards so that those standards contain the same amount of methanol as the diluted soil extracts.

- Modify Table 6

- For the project specific SOP, acetonitrile will be removed from table 6, page 49 and appended onto table 5, page 48. Acetonitrile will be calibrated as part of the STL primary standard, using a separate acetonitrile standard. This will ensure that the calibration curve for acetonitrile will be done free from any interference from allyl chloride.

17.7. The following are protocols that must be followed when analyzing South Carolina Projects only.

- **Delete** from Section 10.4.7 In a linear *or quadratic* calibration fit, the points at the lower end of the calibration curve have less weight in determining the curve generated than points at the high concentration end of the curve.

- **Delete** from Section 12.3.5 Concentrations in the sample may be determined from linear *or second order (quadratic)* curve fitted to the initial calibration points, or from the average response factor of the initial calibration points.

- **Delete** from Section 12.3.5.1

- Calculation of concentration using Quadratic fit

Equation 13

Concentration μg / L $= A + Bx + Cx^2$

x is defined in equations 8, 9 and 10

A is a constant defined by the intercept

B is the slope of the curve

C is the curvature

- **Change** Section 9.3 The compounds included in the surrogate spiking solutions are listed in Tables 8 *and 9*.

17.8. The following are protocols that must be followed to achieve the lower reporting limits required when analyzing Michigan projects.

17.8.1. Modify Section 8.5.4 and 8.6.8 (add 5 uL of 2500 ug/mL surrogate solution for a nominal 25 g sample).

17.8.2. Modify Section 8.5.5 and 8.6.9 (add 100 uL of 50 ug/mL spike solution for a nominal 25 g sample).

17.8.3. Modify Section 8.5.6 and 8.6.10 (add 100 uL of 50 ug/mL spike solution for a nominal 25g sample).

17.8.4. Michigan reporting limits for methanol preserved soils are achieved by injecting 100 uL of the methanol extract in a 5 mL purge. The instrument is calibrated using the recommended calibration levels in water of 1 ug/L, 2 ug/L (if a quadratic calibration is to be used), 5 ug/L, 10 ug/L, 20 ug/L and 40 ug/L. Some analytes are prepared at higher concentrations.

17.8.5. Samples for Michigan projects frequently require calibration for 2-Methylnaphthalene. Recommended calibration levels for this compounds are 2 ug/L, 10 ug/L, 20 ug/L, 40 ug/L and 80 ug/L.

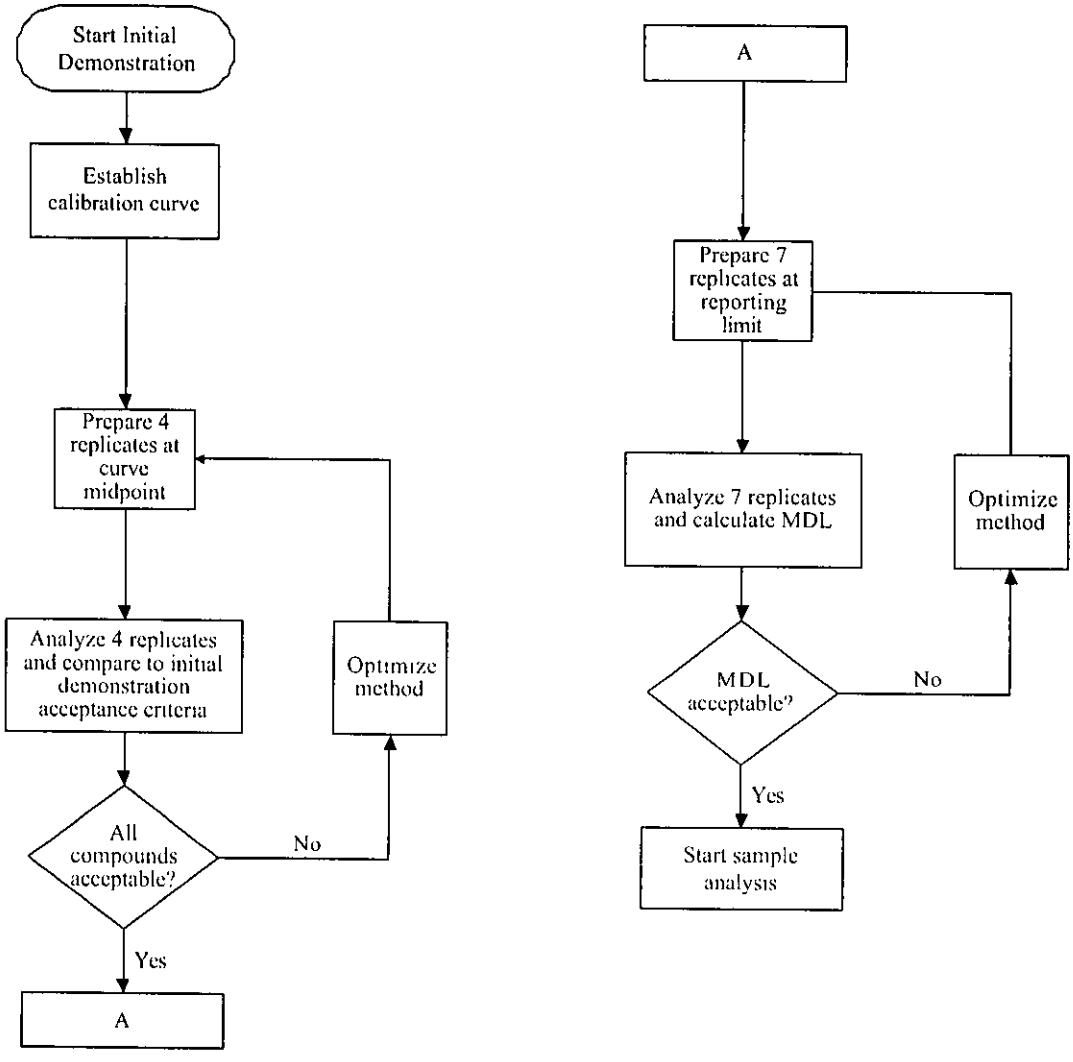

Start Initial Demonstration
Establish calibration curve
Prepare 4 replicates at curve midpoint
Analyze 4 replicates and compare to initial demonstration acceptance criteria
Optimize method
All compounds acceptable?
No
Yes
A
A
Prepare 7 replicates at reporting limit
Analyze 7 replicates and calculate MDL
Optimize method
MDL acceptable?
No
Yes
Start sample analysis

Table 1

STL Primary Standard and Reporting Limits

Compound	CAS Number	Reporting Limits[1]				
		5 mL Water µg/L	25 mL[3] water µg/L	Low soil µg/kg	8260B/ 5035 Soil ug/kg	8260A 5030A Med Level Soil µg/kg
Dichlorodifluoromethane	75-71-8	10	2	10	250	1200
Chloromethane	74-87-3	10	2	10	250	1200
Bromomethane	74-83-9	10	2	10	250	1200
Vinyl chloride	75-01-4	10	2	10	250	1200
Chloroethane	75-00-3	10	2	10	250	1200
Trichlorofluoromethane	75-69-4	10	2	10	250	1200
Acrolein	107-02-8	100	20	100	5000	12000
Acetone	67-64-1	20	10	20	1000	2500
Trichlorotrifluoroethane	76-13-1	5	1	5	250	620
Iodomethane	74-88-4	5	1	5	250	620
Carbon disulfide	75-15-0	5	1	5	250	620
Methylene chloride	75-09-2	5	1	5	250	620
tert-Butyl alcohol	75-65-0	200	50	200	10,000	25000
1,1-Dichloroethene	75-35-4	5	1	5	250	620
1,1-Dichloroethane	75-34-3	5	1	5	250	620
trans-1,2-Dichloroethene	156-60-5	5.0	1.0	5 0	250	310
Acrylonitrile	107-13-1	100	20	100	5000	12000
Methyl *tert*-butyl ether (MTBE)	1634-04-4	20	5	20	1000	2500
Hexane	110-54-3	5	1	5	250	620
cis-1,2-Dichloroethene	156-59-2	5.0	1.0	5.0	250	310
1,2-Dichloroethene (Total)	540-59-0	5	1	5	250	620
Tetrahydrofuran	109-99-9	20	5	20	1000	2500
Chloroform	67-66-3	5	1	5	250	620
1,2-Dichloroethane	107-06-2	5	1	5	250	620
Dibromomethane	74-95-3	5	1	5	250	620
2-Butanone	78-93-3	20	5	20	1000	2500
1,4-Dioxane	123-91-1	500	200	500	25000	62000
1,1,1-Trichloroethane	71-55-6	5	1	5	250	620
Carbon tetrachloride	56-23-5	5	1	5	250	620
Bromodichloromethane	75-27-4	5	1	5	250	620
1,2-Dichloropropane	78-87-5	5	1	5	250	620
cis-1,3-Dichloropropene	10061-01-5	5	1	5	250	620

Table 1

STL Primary Standard and Reporting Limits

Compound	CAS Number	Reporting Limits[1]				
		5 mL Water µg/L	25 mL[3] water µg/L	Low soil µg/kg	8260B/ 5035 Soil ug/kg	8260A 5030A Med Level Soil µg/kg
Trichloroethene	79-01-6	5	1	5	250	620
Dibromochloromethane	124-48-1	5	1	5	250	620
1,2-Dibromoethane	106-93-4	5	1	5	250	620
1,2,3-Trichloropropane	96-18-4	5	1	5	250	620
1,1,2-Trichloroethane	79-00-5	5	1	5	250	620
Benzene	71-43-2	5	1	5	250	620
Ethylmethacrylate	97-63-2	5	1	5	250	620
trans-1,3-Dichloropropene	10061-02-6	5	1	5	250	620
Bromoform	75-25-2	5	1	5	250	620
4-Methyl-2-pentanone	108-10-1	20	5	20	1000	2500
2-Hexanone	591-78-6	20	5	20	1000	2500
Tetrachloroethene	127-18-4	5	1	5	250	620
Toluene	108-88-3	5	1	5	250	620
1,1,2,2-Tetrachloroethane	79-34-5	5	1	5	250	620
2-Chloroethyl vinyl ether	110-75-8	N/A[2]	N/A	50	1000	6200
Vinyl acetate	108-05-4	10	2	10	500	1200
Chlorobenzene	108-90-7	5	1	5	250	620
Ethylbenzene	100-41-4	5	1	5	250	620
Styrene	100-42-5	5	1	5	250	620
t-1,4-Dichloro-2-butene	110-57-6	5	1	5	250	620
m and p Xylenes		5 0	0.5	2.5	125	310
o-xylene	95-47-6	5.0	0.5	2.5	125	310
Total xylenes	1330-20-7	5	1	5	250	620
1,3-Dichlorobenzene	541-73-1	5	1	5	250	620
1,4-Dichlorobenzene	106-46-7	5	1	5	250	620
1,2-Dichlorobenzene	95-50-1	5	1	5	250	620
2,2-Dichloropropane	590-20-7	5	1	5	250	
Bromochloromethane	74-97-5	5	1	5	250	
1,1-Dichloropropene	563-58-6	5	1	5	250	
Bromodichloromethane	75-27-4	5	1	5	250	
1,2-Dichloropropane	78-87-5	5	1	5	250	
1,3-Dichloropropane	142-28-9	5	1	5	250	

Table 1

STL Primary Standard and Reporting Limits

Compound	CAS Number	Reporting Limits[1]				
		5 mL Water µg/L	25 mL[3] water µg/L	Low soil µg/kg	8260B/ 5035 Soil ug/kg	8260A 5030A Med Level Soil µg/kg
Isopropylbenzene	98-82-8	5	1	5	250	
Bromobenzene	108-86-1	5	1	5	250	
n-Propylbenzene	103-65-1	5	1	5	250	
2-Chlorotoluene	95-49-8	5	1	5	250	
4-Chlorotoluene	106-43-4	5	1	5	250	
1,3,5-Trimethylbenzene	108-67-8	5	1	5	250	
tert-Butylbenzene	98-06-6	5	1	5	250	
1,2,4-Trimethylbenzene	95-63-6	5	1	5	250	
sec-butylbenzene	135-98-8	5	1	5	250	
4-Isopropyltoluene	99-87-6	5	1	5	250	
n-Butylbenzene	104-51-8	5	1	5	250	
1,2,4-Trichlorobenzene	120-82-1	5	1	5	250	
Napthalene	91-20-3	5	1	5	250	
Hexachlorobutadiene	87-68-3	5	1	5	250	
1,2,3-Trichlorobenzene	87-61-6	5	1	5	250	
Acetonitrile	75-05-8	100	20	100	5000	

[1] Reporting limits listed for soil/sediment are based on wet weight. The reporting limits calculated by the laboratory for soil/sediment, calculated on dry weight basis, will be higher.

[2] 2-Chloroethyl vinyl ether cannot be reliably recovered from acid preserved samples

[3] Optionally, 5 mL purge volume if adequate sensitivity is obtained.

Table 2

STL Primary Standard Calibration Levels, 5 mL purge[1]

Compound	Calibration Level ug/L				
	Level 1	Level 2	Level 3	Level 4	Level 5
1,2-Dichloroethane-d4 (Surrogate)	5	20	50	100	200
Toluene-d8 (Surrogate)	5	20	50	100	200
4-Bromofluorobenzene (Surrogate)	5	20	50	100	200
Dichlorodifluoromethane	5	20	50	100	200
Chloromethane	5	20	50	100	200
Bromomethane	5	20	50	100	200
Vinyl chloride	5	20	50	100	200
Chloroethane	5	20	50	100	200
Trichlorofluoromethane	5	20	50	100	200
Acrolein	50	200	500	1000	2000
Acetone	5	20	50	100	200
Trichlorotrifluoroethane	5	20	50	100	200
Iodomethane	5	20	50	100	200
Carbon disulfide	5	20	50	100	200
Methylene chloride	5	20	50	100	200
tert-Butyl alcohol	100	400	1,000	2,000	4,000
1,1-Dichloroethene	5	20	50	100	200
1,1-Dichloroethane	5	20	50	100	200
trans-1,2-Dichloroethene	5	20	50	100	200
Acrylonitrile	50	200	500	1,000	2,000
Methyl *tert*-butyl ether (MTBE)	5	20	50	100	200
Hexane	5	20	50	100	200
cis-1,2-Dichloroethene	5	20	50	100	200
Tetrahydrofuran	5	20	50	100	200
Chloroform	5	20	50	100	200
1,2-Dichloroethane	5	20	50	100	200
Dibromomethane	5	20	50	100	200
2-Butanone	5	20	50	100	200
1,4-Dioxane	250	1000	2,500	5,000	10,000
1,1,1-Trichloroethane	5	20	50	100	200
Carbon tetrachloride	5	20	50	100	200
Bromodichloromethane	5	20	50	100	200
1,2-Dichloropropane	5	20	50	100	200
cis-1,3-Dichloropropene	5	20	50	100	200
Trichloroethene	5	20	50	100	200
Dibromochloromethane	5	20	50	100	200

Table 2

STL Primary Standard Calibration Levels, 5 mL purge[1]

Compound	Calibration Level ug/L				
	Level 1	Level 2	Level 3	Level 4	Level 5
1,2-Dibromoethane	5	20	50	100	200
1,2,3-Trichloropropane	5	20	50	100	200
Acetonitrile	50	200	500	1000	2000
1,1,2-Trichloroethane	5	20	50	100	200
Benzene	5	20	50	100	200
Ethylmethacrylate	5	20	50	100	200
trans-1,3-Dichloropropene	5	20	50	100	200
Bromoform	5	20	50	100	200
4-Methyl-2-pentanone	5	20	50	100	200
2-Hexanone	5	20	50	100	200
Tetrachloroethene	5	20	50	100	200
Toluene	5	20	50	100	200
1,1,2,2-Tetrachloroethane	5	20	50	100	200
2-Chloroethyl vinyl ether	10	40	100	200	400
Vinyl acetate	5	20	50	100	200
Chlorobenzene	5	20	50	100	200
Ethylbenzene	5	20	50	100	200
Styrene	5	20	50	100	200
t-1,4-Dichloro-2-butene	5	20	50	100	200
m and p Xylenes	10	40	100	200	400
o-xylene	5	20	50	100	200
1,3-Dichlorobenzene	5	20	50	100	200
1,4-Dichlorobenzene	5	20	50	100	200
1,2-Dichlorobenzene	5	20	50	100	200
2,2-Dichloropropane	5	20	50	100	200
Bromochloromethane	5	20	50	100	200
1,1-Dichloropropene	5	20	50	100	200
Bromodichloromethane	5	20	50	100	200
1,2-Dichloropropane	5	20	50	100	200
1,3-Dichloropropane	5	20	50	100	200
Isopropylbenzene	5	20	50	100	200
Bromobenzene	5	20	50	100	200
n-Propylbenzene	5	20	50	100	200
2-Chlorotoluene	5	20	50	100	200
4-Chlorotoluene	5	20	50	100	200
1,3,5-Trimethylbenzene	5	20	50	100	200
tert-Butylbenzene	5	20	50	100	200

Table 2

STL Primary Standard Calibration Levels, 5 mL purge[1]

Compound	Calibration Level ug/L				
	Level 1	Level 2	Level 3	Level 4	Level 5
1,2,4-Trimethylbenzene	5	20	50	100	200
sec-butylbenzene	5	20	50	100	200
4-Isopropyltoluene	5	20	50	100	200
n-Butylbenzene	5	20	50	100	200
1,2,4-Trichlorobenzene	5	20	50	100	200
Napthalene	5	20	50	100	200
Hexachlorobutadiene	5	20	50	100	200
1,2,3-Trichlorobenzene	5	20	50	100	200

[1] **Levels for 25 mL purge are 5 times lower in all cases**

Table 2A

STL Primary Standard Calibration Levels, Low Level [1]

Compound	Calibration Level ug/L				
	Level 1	Level 2	Level 3	Level 4	Level 5
Dibromofluoromethane (Surrogate)	1	5	10	20	40
1,2-Dichloroethane-d4 (Surrogate)	1	5	10	20	40
Toluene-d8 (Surrogate)	1	5	10	20	40
Bromofluorobenzene (Surrogate)	1	5	10	20	40
Dichlorodifluoromethane	1	5	10	20	40
Chloromethane	1	5	10	20	40
Vinyl Chloride	1	5	10	20	40
Bromomethane	1	5	10	20	40
Chloroethane	1	5	10	20	40
Trichlorofluoromethane	1	5	10	20	40
Acrolein	10	50	100	200	400
Acetone	2	10	20	40	80
1,1-Dichloroethene	1	5	10	20	40
Trichlorotrifluoroethane	1	5	10	20	40
Iodomethane	1	5	10	20	40
Carbon Disulfide	1	5	10	20	40
Methylene Chloride	1	5	10	20	40
Acetonitrile	10	50	100	200	400
Acrylonitrile	10	50	100	200	400
Methyl tert-butyl ether	1	5	10	20	40
trans-1,2-Dichloroethene	1	5	10	20	40
Hexane	1	5	10	20	40
Vinyl acetate	1	5	10	20	40
1,1-Dichloroethane	1	5	10	20	40
tert-Butyl Alcohol	20	100	200	400	800
2-Butanone	2	10	20	40	80
cis-1,2-dichloroethene	1	5	10	20	40
2,2-Dichloropropane	1	5	10	20	40
Bromochloromethane	1	5	10	20	40
Chloroform	1	5	10	20	40
Tetrahydrofuran	1	5	10	20	40
1,1,1-Trichloroethane	1	5	10	20	40
1,1-Dichloropropene	1	5	10	20	40
Carbon Tetrachloride	1	5	10	20	40
1,2-Dichloroethane	1	5	10	20	40
Benzene	1	5	10	20	40
Trichloroethene	1	5	10	20	40
1,2-Dichloropropane	1	5	10	20	40
1,4-Dioxane	50	250	500	1000	2000
Dibromomethane	1	5	10	20	40

Table 2A

STL Primary Standard Calibration Levels, Low Level[1]

Compound	Calibration Level ug/L				
	Level 1	Level 2	Level 3	Level 4	Level 5
Bromodichloromethane	1	5	10	20	40
2-Chloroethyl vinyl ether	2	10	20	40	80
cis-1,3-Dichloropropene	1	5	10	20	40
4-Methyl-2-pentanone	2	10	20	40	80
Toluene	1	5	10	20	40
trans-1,3-Dichloropropene	1	5	10	20	40
Ethyl Methacrylate	1	5	10	20	40
1,1,2-Trichloroethane	1	5	10	20	40
1,3-Dichloropropane	1	5	10	20	40
Tetrachloroethene	1	5	10	20	40
2-Hexanone	2	10	20	40	80
Dibromochloromethane	1	5	10	20	40
1,2-Dibromoethane	1	5	10	20	40
Chlorobenzene	1	5	10	20	40
1,1,1,2-Tetrachloroethane	1	5	10	20	40
Ethylbenzene	1	5	10	20	40
m + p-Xylene	2	10	20	40	80
Xylene-o	1	5	10	20	40
Styrene	1	5	10	20	40
Bromoform	1	5	10	20	40
Isopropylbenzene	1	5	10	20	40
1,1,2,2-Tetrachloroethane	1	5	10	20	40
1,4-Dichloro-2-butene	1	5	10	20	40
1,2,3-Trichloropropane	1	5	10	20	40
Bromobenzene	1	5	10	20	40
n-Propylbenzene	1	5	10	20	40
2-Chlorotoluene	1	5	10	20	40
1,3,5-Trimethylbenzene	1	5	10	20	40
4-Chlorotoluene	1	5	10	20	40
tert-Butylbenzene	1	5	10	20	40
1,2,4-Trimethylbenzene	1	5	10	20	40
sec-Butylbenzene	1	5	10	20	40
4-Isopropyltoluene	1	5	10	20	40
1,3-Dichlorobenzene	1	5	10	20	40
1,4-Dichlorobenzene	1	5	10	20	40
n-Butylbenzene	1	5	10	20	40
1,2-Dichlorobenzene	1	5	10	20	40
1,2-Dibromo-3-chloropropane	1	5	10	20	40

Table 2A

STL Primary Standard Calibration Levels, Low Level [1]

Compound	Calibration Level ug/L				
	Level 1	Level 2	Level 3	Level 4	Level 5
1,2,4-Trichlorobenzene	1	5	10	20	40
Hexachlorobutadiene	1	5	10	20	40
Naphthalene	1	5	10	20	40
1,2,3-Trichlorobenzene	1	5	10	20	40
Cyclohexane	1	5	10	20	40
Methyl Acetate	2	10	20	40	80
Methylcyclohexane	1	5	10	20	40
1,3,5-Trichlorobenzene	1	5	10	20	40

[1] **25 mL purge samples analyzed at 5 mL purge on more sensitive equipment.**

Table 3

STL Appendix IX Standard and Reporting Limits, 5 mL purge

Compound	CAS Number	Reporting Limits			
		5 mL Water µg/L	25mL[2] water µg/L	Low Soil µg/kg	Medium Soil µg/mL
Allyl Chloride	107-05-1	10	2	10	500
Acetonitrile	75-05-8	100	20	100	5000
Dichlorofluoromethane		10	2	10	500
Isopropyl ether	108-20-3	10	2	10	500
Chloroprene	126-99-8	5	2	5	250
n-Butanol	71-36-3	200	50	200	10,000
Propionitrile	107-12-0	20	4	20	1000
Methacrylonitrile	126-98-7	5	2	5	250
Isobutanol	78-83-1	200	50	200	10,000
Methyl methacrylate	80-62-6	5	2	5	250
1,1,1,2-Tetrachloroethane	630-20-6	5	1	5	250
1,2-Dibromo-3-chloropropane	96-12-8	10	2	10	500
Ethyl ether	60-29-7	10	2	10	500
Ethyl Acetate	141-78-6	20	4	20	1,000
2-Nitropropane	79-46-9	10	4	10	500
Cyclohexanone	108-94-1	N/A[1]	N/A[1]	N/A[1]	N/A[1]
Isopropylbenzene	98-82-8	5	1	5	250
2-Methylnaphthalene (Michigan only)	91-57-6	NA	5	NA	330

[1] Cyclohexanone decomposes to 1,1-dimethoxycyclohexane in methanolic solution. Reporting limits cannot be accurately determined.

[2] Optionally, 5 mL purge volume if adequate sensitivity is obtained.

Table 4

Recommended/STL Appendix IX Standard Calibration Levels, µg/L

Compound	Level 1	Level 2	Level 3	Level 4	Level 5
Allyl Chloride	5	20	50	100	200
Acetonitrile	50	200	500	1,000	2,000
Dichlorofluoromethane	5	20	50	100	200
Isopropyl ether	5	20	50	100	200
Chloroprene	5	20	50	100	200
n-Butanol	100	400	1,000	2,000	4,000
Propionitrile	10	40	100	200	400
Methacrylonitrile	5	20	50	100	200
Isobutanol	100	400	1,000	2,000	4,000
Methyl methacrylate	5	20	50	100	200
1,1,1,2-Tetrachloroethane	5	20	50	100	200
1,2-Dibromo-3-chloropropane	10	40	100	200	400
Ethyl ether	5	20	50	100	200
Ethyl Acetate	10	40	100	200	400
2-Nitropropane	10	40	100	200	400
Cyclohexanone	50	200	500	1,000	2,000
2-Methylnaphthalene (Michigan only)	2	10	20	40	80

Table 5

Reportable Analytes for STL Standard Tests, Primary Standard

Compound	CAS Number	STL Standard List	TCLP	TCL	Appendix IX	UTS
Dichlorodifluoromethane	75-71-8				X	X
Chloromethane	74-87-3	X		X	X	X
Bromomethane	74-83-9	X		X	X	X
Vinyl chloride	75-01-4	X	X	X	X	X
Chloroethane	75-00-3	X		X	X	X
Trichlorofluoromethane	75-69-4				X	X
Acrolein	107-02-8				X	X
Acetone	67-64-1	X		X	X	X
Trichlorotrifluoroethane	76-13-1					X
Ethanol	64-17-5					
Iodomethane	74-88-4				X	X
Carbon disulfide	75-15-0	X		X	X	X
Methylene chloride	75-09-2	X		X	X	X
tert-Butly alcohol	75-65-0					
1,1-Dichloroethene	75-35-4	X	X	X	X	X
1,1-Dichloroethane	75-34-3	X		X	X	X
trans-1,2-Dichloroethene	156-60-5	X		X	X	X
Total dichloroethene		X		X	X	X
Acrylonitrile	107-13-1				X	X
Methyl *tert*-butyl ether (MTBE)	1634-04-4					
Hexane	110-54-3					
cis-1,2-Dichloroethene	156-59-2	X		X		
Tetrahydrofuran	109-99-9					
Chloroform	67-66-3	X	X	X	X	X
1,2-Dichloroethane	107-06-2	X	X	X	X	X
Dibromomethane	74-95-3				X	X
2-Butanone	78-93-3	X	X	X	X	X
1,4-Dioxane	123-91-1				X	X
1,1,1-Trichloroethane	71-55-6	X		X	X	X
Carbon tetrachloride	56-23-5	X	X	X	X	X
Bromodichloromethane	75-27-4	X		X	X	X
1,2-Dichloropropane	78-87-5	X		X	X	X
cis-1,3-Dichloropropene	10061-01-5	X		X	X	X
Trichloroethene	79-01-6	X	X	X	X	X

Table 5

Reportable Analytes for STL Standard Tests, Primary Standard

Compound	CAS Number	STL Standard List	TCLP	TCL	Appendix IX	UTS
Dibromochloromethane	124-48-1	X		X	X	X
1,2-Dibromoethane	106-93-4				X	X
1,2,3-Trichloropropane	96-18-4				X	X
1,1,2-Trichloroethane	79-00-5	X		X	X	X
Benzene	71-43-2	X	X	X	X	X
Ethylmethacrylate	97-63-2				X	X
trans-1,3-Dichloropropene	10061-02-6	X		X	X	X
Bromoform	75-25-2	X		X	X	X
4-Methyl-2-pentanone	108-10-1	X		X	X	X
2-Hexanone	591-78-6	X		X	X	
Tetrachloroethene	127-18-4	X	X	X	X	X
Toluene	108-88-3	X		X	X	X
1,1,2,2-Tetrachloroethane	79-34-5	X		X	X	X
2-Chloroethyl vinyl ether	110-75-8					
Vinyl acetate	108-05-4				X	
Chlorobenzene	108-90-7	X	X	X	X	X
Ethylbenzene	100-41-4	X		X	X	X
Styrene	100-42-5	X		X	X	
t-1,4-Dichloro-2-butene	110-57-6				X	
m and p Xylenes		X		X	X	X
o-xylene	95-47-6	X		X	X	X
Total xylenes	1330-20-7	X		X	X	X
1,3-Dichlorobenzene	541-73-1					
1,4-Dichlorobenzene	106-46-7					
1,2-Dichlorobenzene	95-50-1					

Table 6

Reportable Analytes for STL Standard Tests, Appendix IX standard

Compound	Number	STL Standard List	TCLP	TCL	Appendix IX	UTS
Allyl Chloride	107-05-1				X	
Acetonitrile	75-05-8				X	X
Dichlorofluoromethane	75-43-4					
Isopropyl ether	108-20-3					
Chloroprene	126-99-8				X	
n-Butanol	71-36-3					
Propionitrile	107-12-0				X	
Methacrylonitrile	126-98-7				X	X
Isobutanol	78-83-1				X	X
Methyl methacrylate	80-62-6				X	X
1,1,1,2-Tetrachloroethane	630-20-6				X	X
1,2-Dibromo-3-chloropropane	96-12-8				X	X
Ethyl ether	60-29-7					X
Ethyl Acetate	141-78-6					X
2-Nitropropane	79-46-9					
Cyclohexanone	108-94-1					X
Isopropylbenzene	98-82-8					

Table 7

Internal Standards

	Standard Concentration µg/mL	Quantitation ion (5 mL purge)	Quantitation ion (25 mL purge)
Fluorobenzene	50	96	96
Chlorobenzene-d5	50	117	119
1,4-Dichlorobenzene-d4	50	152	152

Notes:

1) 5 µL of the internal standard is added to the sample. This results in a concentration of each internal in the sample of 50µg/L for a 5 mL purge or 10 µg/L for a 25 mL purge

2) Except for medium level soils, the surrogate and internal standards may be combined in one solution.

Table 8

Surrogate Standards

Surrogate Compounds	Standard Concentration µg/mL
1,2-Dichloroethane-d_4	50
Dibromofluoromethane	50
Toluene-d_8	50
4-Bromofluorobenzene	50

Notes:

1) 5 µL of the surrogate standard is added to the sample. This results in a concentration of each surrogate in the sample of 50µg/L for a 5 mL purge or 10 µg/L for a 25 mL purge.

2) Except for medium level soils, the surrogate and internal standards may be combined in one solution.

3) Recovery limits for surrogates are generated from historical data and are maintained by the QA department.

Table 9

Matrix Spike / LCS Control Compounds

Compound	Standard Concentration µg /mL
1,1-Dichloroethene	50
Trichloroethene	50
Toluene	50
Benzene	50
Chlorobenzene	50
n-Hexane (Ohio VAP only)	50

Notes:

1) 5 µL of the standard is added to the LCS or matrix spiked sample. This results in a concentration of each spike analyte in the sample of 50µg/L for a 5 mL purge or 10 µg/L for a 25 mL purge.

2) Recovery and precision limits for LCS and MS/MSD are generated from historical data and are maintained by the QA department.

3) Full analyte spikes may also be used at the laboratories option or at client request

Table 10

BFB Key Ion Abundance Criteria

Mass	Ion Abundance Criteria
50	15% to 40% of Mass 95
75	30% to 60% of Mass 95
95	Base Peak, 100% Relative Abundance
96	5% to 9% of Mass 95
173	Less Than 2% of Mass 174
174	Greater Than 50% of Mass 95
175	5% to 9% of Mass 174
176	Greater Than 95%, But Less Than 101% of Mass 174
177	5% to 9% of Mass 176

Table 11

SPCC Compounds and Minimum Response Factors

Compound	8260B, 8260A Min. RF
Chloromethane	0.100
1,1-Dichloroethane	0.100
Bromoform	>0.100
1,1,2,2-Tetrachloroethane	0.300
Chlorobenzene	0.300

Table 12

CCC compounds

Compound	Max. %RSD from Initial Calibration	Max. %D for continuing calibration
Vinyl Chloride	<30.0	<20.0
1,1-Dichloroethene	<30.0	<20.0
Chloroform	<30.0	<20.0
1,2-Dichloropropane	<30.0	<20.0
Toluene	<30.0	<20.0
Ethylbenzene	<30.0	<20.0
n-Hexane (Ohio VAP only)	<30.0	<20.0

Table 13

Characteristic ions

Compound	Primary*	Secondary	Tertiary
1,2-Dichloroethane-d_4 (Surrogate)	65	102	
Dichlorodifluoromethane	85	87	50, 101,103
Chloromethane	50	52	49
Vinyl chloride	62	64	61
Bromomethane	94	96	79
Chloroethane	64	66	49
Trichlorofluoromethane	101	103	66

Table 13

Characteristic ions

Compound	Primary*	Secondary	Tertiary
1,1-Dichloroethene	96	61	98
Acrolein	56	55	58
Iodomethane	142	127	141
Carbon disulfide	76	78	
Trichlorotrifluoroethane	151	101	153
Ethanol	45	46	
Acetone	43	58	
Methylene chloride	84	49	51, 86
tert-Butyl alcohol	59	74	
trans-1,2-Dichloroethene	96	61	98
Acrylonitrile	53	52	51
Methyl *tert* butyl ether	73		
Hexane	57	43	
1,1-Dichloroethane	63	65	83
cis-1,2-Dichloroethene	96	61	98
2-Butanone	43	72**	
Tetrahydrofuran	42	71	
Chloroform	83	85	47
1,2-Dichloroethane	62	64	98
Dibromomethane	93	174	95, 172, 176
1,4-Dioxane	88	58	
Vinyl acetate	43	86	
1,1,1-Trichloroethane	97	99	117
Carbon tetrachloride	117	119	121
Benzene	78	52	77
Trichloroethene	130	95	97, 132
1,2-Dichloropropane	63	65	41
Bromodichloromethane	83	85	129
2-Chloroethyl vinyl ether	63	65	106
cis-1,3-Dichloropropene	75	77	39
trans-1,3-Dichloropropene	75	77	39
1,1,2-Trichloroethane	97	83	85, 99
Chlorodibromomethane	129	127	131
Bromoform	173	171	175, 252
1,2,3-Trichloropropane	75	110	77, 112, 97
Toluene-d_8 (Surrogate)	98	70	100
4-Bromofluorobenzene (Surrogate)	95	174	176
Toluene	91	92	65
4-Methyl-2-pentanone	43	58	57, 100

Table 13

Characteristic ions

Compound	Primary*	Secondary	Tertiary
Tetrachloroethene	164	166	131
Ethyl methacrylate	69	41	99, 86, 114
2-Hexanone	43	58	57, 100
Chlorobenzene	112	114	77
Ethylbenzene	106	91	
Xylenes	106	91	
Styrene	104	103	78, 51, 77
Dichlorobenzene (all isomers)	146	148	111
trans 1,4-Dichloro-2-butene	53	75	89, 77, 124
1,1,2,2-Tetrachloroethane	83	85	131, 133
Allyl Chloride	76	41	78
Acetonitrile	40	41	
Dichlorofluoromethane	67	69	
Isopropyl ether	87	59	45
Chloroprene	53	88	90
n-Butanol	56	41	42
Propionitrile	54	52	55
Methacrylonitrile	41	67	52
Isobutanol	41	43	74
Methyl methacrylate	41	69	100
1,1,1,2-Tetrachloroethane	131	133	119
1,2-Dibromo-3-chloropropane	157	155	75
Ethyl ether	59	74	
Ethyl Acetate	43	88	61
2-Nitropropane	41	43	46
Cyclohexanone	55	42	98
Isopropylbenzene	105	120	

* The primary ion should be used for quantitation unless interferences are present, in which case a secondary ion may be used.

** m/z 43 may be used for quantitation of 2-Butanone, but m/z 72 must be present for positive identification.

1. REQUIREMENTS FOR EPA 624

1.1. Method 624 is required for demonstration of compliance with NPDES wastewater discharge permits. This method can be applied only to aqueous matrices. The standard analyte list and reporting limits are listed in Table B-1.

1.2. The tune period for this method is defined as 24 hours.

1.3. The initial calibration curve for this method requires at least three points.

1.4. Sample concentrations are calculated using the average RRF from the initial calibration curve.

1.5. Each target analyte is assigned to the closest eluting internal standard.

1.6. Initial demonstration of Proficiency

1.6.1. The spiking level for the four replicate initial demonstration of proficiency is 20 µg/L. The acceptance criteria are listed in Table B-2

1.7. Initial calibration curve requirements:

1.7.1. Target compounds must have RSD ≤ 35%.

1.7.2. If this requirement can not be met, a regression curve must be constructed for the non-compliant compounds. There is no correlation coefficient requirement for the regression curve.

1.8. Continuing calibration verification requirements:

1.8.1. The continuing calibration standard is from a different source than the initial calibration standard. The acceptance criteria are listed in Table B-2.

1.9. Matrix Spike and LCS requirements

1.9.1. The matrix spike and LCS are spiked at 20 µg/L. A matrix spike duplicate is not necessary for this method. The recovery limits for matrix spike and LCS recovery are listed in Table C-2.

1.10. Method clarifications, modifications and additions

1.10.1. Section 5.2.2 of the source method describes the trap packing materials as Tenax GC, Methyl silicone, silica gel and coconut charcoal. STL routinely employs the Supelco VOCARB 3000, which consists of Carbopack B and Carboxen 1000 and 1001.

1.10.2. Section 5.3.2 of the source method describes a packed analytical column. STL routinely employs capillary columns when performing this method.

1.10.3. The source method provides a suggested list of compounds for internal and surrogate standards. STL uses the following two compounds which are not on the table: Chlorobenzene-d_5 (internal standard) and 1,2-Difluorobenzene-d_4 (surrogate).

Table A-1.

Method 624 Analytes and Reporting Limits

Analytes	µg/L
Benzene	5
Bromodichloromethane	5
Bromoform	5
Bromomethane	5
Carbon tetrachloride	5
Chlorobenzene	5
Chloroethane	5
2-Chloroethyl vinyl ether	5
Chloroform	5
Chloromethane	5
Dibromochloromethane	5
1,2-Dichlorobenzene	5
1,3-Dichlorobenzene	5
1,4-Dichlorobenzene	5
1,1-Dichloroethane	5
1,2-Dichloroethane	5
1,1-Dichloroethene	5
trans-1,2-Dichloroethene	5
1,2-Dichloropropane	5
cis-1,3-Dichloropropene	5
trans-1,3-Dichloropropene	5
Ethylbenzene	5
Methylene chloride	5
1,1,2,2-Tetrachloroethane	5
Tetrachloroethene	5
Toluene	5
1,1,1-Trichloroethane	5
1,1,2-Trichloroethane	5
Trichloroethene	5
Trichlorofluoromethane	5
Vinyl chloride	5

Table A-2.

Method 624 QC Acceptance Criteria

Analytes	Daily QC check acceptance criteria (20µg/L spike)	Mean recovery, 4 replicate initial demonstration acceptance criteria (20µg/L spike)	Standard deviation, 4 replicate initial demonstration acceptance criteria (20µg/L spike)	Matrix spike and LCS acceptance criteria (% recovery)
Benzene	12.8-27.2	15.2-26.0	6.9	37-151
Bromodichloromethane	13.1-26.9	10.1-28.0	6.4	35-155
Bromoform	14.2-25 8	11.4-31.1	5.4	45-169
Bromomethane	2.8-37.2	D-41.2	17.9	D-242
Carbon tetrachloride	14.6-25.4	17.2-23.5	5.2	70-140
Chlorobenzene	13 2-26.8	16.4-27.4	6.3	37-160
Chloroethane	7.6-32.4	8.4-40.4	11.4	14-230
2-Chloroethyl vinyl ether	D-44.8	D-50 4	25.9	D-305
Chloroform	13.5-26 5	13.7-24.2	6.1	51-138
Chloromethane	D-40.8	D-45.9	19.8	D-273
Dibromochloromethane	13.5-26.5	13 8-26.6	6.1	53-149
1,2-Dichlorobenzene	12.6-27.4	11 8-34.7	7.1	18-190
1,3-Dichlorobenzene	14.6-25.4	17.0-28 8	5.5	59-156
1,4-Dichlorobenzene	12.6-27.4	11.8-34.7	7.1	18-190
1,1-Dichloroethane	14.5-25 5	14.2-28.5	5 1	59-155
1,2-Dichloroethane	13 6-26.4	14 3-27.4	6.0	49-155
1,1-Dichloroethene	10 1-29.9	3.7-42.3	9.1	D-234
trans-1,2-Dichloroethene	13.9-26.1	13.6-28.5	5.7	54-156
1,2-Dichloropropane	6 8-33.2	3.8-36 2	13.8	D-210
cis-1,3-Dichloropropene	4.8-35.2	1.0-39.0	15.8	D-227
trans-1,3-Dichloropropene	10.0-30.0	7.6-32.4	10.4	17-183
Ethylbenzene	11.8-28.2	17.4-26.7	7.5	37-162
Methylene chloride	12 1-27.9	D-41.0	7 4	D-221
1,1,2,2-Tetrachloroethane	12.1-27.9	13.5-27.2	7.4	46-157
Tetrachloroethene	14.7-25 3	17.0-26.6	5.0	64-148
Toluene	14.9-25 1	16 6-26.7	4.8	47-150
1,1,1-Trichloroethane	15 0-25.0	13.7-30.1	4.6	52-162
1,1,2-Trichloroethane	14.2-25.8	14.3-27.1	5 5	52-150
Trichloroethene	13.3-26.7	18.6-27.6	6.6	71-157
Trichlorofluoromethane	9 6-30 4	8.9-31.5	10.0	17-181
Vinyl chloride	0.8-39.2	D-43 5	20.0	D-251

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Implementation Date: 3/2/04

SOP No: CORP-MS-0001NC
Revision No: 2.10
Revision Date: 02/24/04
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STL NORTH CANTON STANDARD OPERATING PROCEDURE

TITLE: GC/MS ANALYSIS BASED ON METHODS 8270C

(SUPERSEDES: Revision 2.9, Dated 06/18/03)

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1. SCOPE AND APPLICATION

1.1 This method is based upon SW846 8270C, and is applicable to the determination of the concentration of semivolatile organic compounds in extracts prepared from solid and aqueous matrices. Direct injection of a sample may be used in limited applications. Refer to Tables 1, 2, 3 and 4 for the list of compounds applicable for this method. Note that the compounds are listed in approximate retention time order. Additional compounds may be amenable to this method. If non-standard analytes are required, they must be validated by the procedures described in section 13 before sample analysis.

1.2 The following compounds may require special treatment when being determined by this method:

- Benzidine can be subject to oxidative losses during solvent concentration and exhibits poor chromatography. Neutral extraction should be performed if this compound is expected.
- Hexachlorocyclopentadiene is subject to thermal decomposition in the inlet of the gas chromatograph, chemical reaction in acetone solution, and photochemical decomposition.
- N-Nitrosodiphenylamine decomposes in the gas chromatographic inlet and cannot be distinguished from diphenylamine.
- Pentachlorophenol, 2,4-dinitrophenol, 4-nitrophenol, 4,6-dinitro-2-methylphenol, 4-chloro-3-methylphenol, benzoic acid, 2-nitroaniline, 3-nitroaniline, 4-chloroaniline, and benzyl alcohol are subject to erratic chromatographic behavior, especially if the GC system is contaminated with high boiling material.
- Hexachlorophene is not amenable to analysis by this method.
- 3-Methylphenol cannot be separated from 4-methylphenol by the conditions specified in this method.

1.3 The standard reporting limit of this method for determining an individual compound is approximately 0.33 mg/kg (wet weight) for soil/sediment samples, 1 - 200 mg/kg for wastes (dependent on matrix and method of preparation), and 10 µg/L for groundwater samples. Some compounds have higher reporting limits. Refer to Tables 1 and 2 for specific SRLs. Reporting limits will be proportionately higher for sample extracts that require dilution.

1.4 The associated LIMS code is QL (8270C).

2 SUMMARY OF METHOD

2.1 Aqueous samples are extracted with methylene chloride using a separatory funnel, and/or a

continuous extractor. Solid samples are extracted with methylene chloride / acetone using sonication, soxhlet, accelerated soxhlet or pressurized fluid extraction. The extract is dried, concentrated to a final volume of 2 mL for waters and soils, and analyzed by GC/MS. Extraction procedures are detailed in SOP# CORP-OP-0001NC. Qualitative identification of the parameters in the extract is performed using the retention time and the relative abundance of characteristic ions. Quantitative analysis is performed using the internal standard technique with a single characteristic ion.

3 DEFINITIONS

3.1 CCC (Calibration Check Compounds) - A subset of target compounds used to evaluate the calibration stability of the GC/MS system. A maximum percent deviation of the CCC's is specified for calibration acceptance.

3.2 SPCC (System Performance Check Compounds) - Target compounds designated to monitor chromatographic performance, sensitivity, and compound instability or degradation on active sites. Minimum response factors are specified for acceptable performance.

3.3 Batch - The batch is a set of up to 20 samples of the same matrix processed using the same procedures and reagents within the same time period. The Quality Control batch must contain a matrix spike / spike duplicate (MS/MSD), a Laboratory Control Sample (LCS), and a method blank. Batches are defined at the sample preparation stage. Batches should be kept together through the whole analytical process to the extent possible, but it is not mandatory to analyze prepared extracts on the same instrument or in the same sequence. Refer to the STL North Canton QC Program document (QA-003) for further details of the batch definition.

3.4 Method Blank - An analytical control consisting of all reagents, internal standards and surrogate standards, that is carried through the entire analytical procedure. The method blank is used to define the level of laboratory background and reagent contamination.

3.5 LCS (Laboratory Control Sample) - A blank spiked with the parameters of interest that is carried through the entire analytical procedure. Analysis of this sample with acceptable recoveries of the spiked materials demonstrates that the laboratory techniques for this method are acceptable.

3.6 MS (Matrix Spike)- aliquot of a matrix (water or soil) fortified (spiked) with known quantities of specific compounds and subjected to the entire analytical procedure in order to indicate the appropriateness of the method for the matrix by measuring recovery.

3.7 MSD (Matrix Spike Duplicate)- a second aliquot of the same sample as the matrix spike (above) that is spiked in order to determine the precision of the method.

4 INTERFERENCES

4.1 Method interferences may be caused by contaminants in solvents, reagents, glassware, and other processing apparatus that lead to discrete artifacts. All of these materials must be routinely demonstrated to be free from interferences under conditions of the analysis by running laboratory method blanks as described in the Quality Control section. Raw GC/MS data from all blanks, samples, and spikes must be evaluated for interferences. If an interference is detected it is necessary to determine if the source of interference is in the preparation and/or cleanup of the samples; then take corrective action to eliminate the problem.

4.2 The use of high purity reagents, solvents, and gases helps to minimize interference problems.

4.3 Matrix interferences may be caused by contaminants that are coextracted from the sample. The extent of matrix interferences will vary considerably from source to source, depending upon the nature of the sample.

4.4 Contamination by carryover can occur whenever high-level and low-level samples are sequentially analyzed. To reduce carryover, the sample syringe must be rinsed with solvent between samples. Whenever an unusually concentrated sample is encountered, it should be followed by the analysis of solvent to check for cross contamination.

4.5 Phthalate contamination is commonly observed in this analysis and its occurrence should be carefully evaluated as an indicator of a contamination problem in the sample preparation step of the analysis.

5 SAFETY PRECAUTIONS

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

5.2 Eye protection that protects against splash, laboratory coat, and appropriate gloves must be worn while samples, standards, solvents and reagents are being handled. Disposable gloves that have become contaminated will be removed and discarded; other gloves will be cleaned immediately.

5.3 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.

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

Material (1)	Hazards	Exposure Limit (2)	Signs and symptoms of exposure
Methylene Chloride	Carcinogen Irritant	25 ppm-TWA 125 ppm-STEL	Causes irritation to respiratory tract. Has a strong narcotic effect with symptoms of mental confusion, light-headedness, fatigue, nausea, vomiting and headache. Causes irritation, redness and pain to the skin and eyes. Prolonged contact can cause burns. Liquid degreases the skin. May be absorbed through skin.
Sulfuric Acid	Corrosive Oxidizer 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.			

5.5 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.

5.6 The preparation of standards and reagents will be conducted in a fume hood with the sash closed as far as the operation will permit.

5.7 It is recommended that neat standards be purchased only as a last resort. The preparation of standards from neat materials and reagents {as well as glassware cleaning procedures that involved solvents such as methylene chloride} should be conducted in a fume hood with the sash closed as far as the operations will permit.

5.8 Standards in solution may be diluted in the open laboratory when syringes and the like are utilized.

5.9 All work must be stopped in the event of a known or potential compromise to the health and safety of a STL North Canton associate. The situation must be reported immediately to a laboratory supervisor.

6 EQUIPMENT AND SUPPLIES

6.1 Gas Chromatograph/Mass Spectrometer System: An analytical system complete with a temperature-programmable gas chromatograph suitable for split/splitless injection and all required accessories, including syringes, analytical columns, and gases. The capillary column should be directly coupled to the source.

6.2 Column: 20m x 0.18mm ID, 0.18µm film thickness silicon-coated fused-silica capillary column (J & W Scientific DB-5.625 or equivalent). Alternate columns are acceptable if they provide acceptable performance.

6.3 Mass Spectrometer: Capable of scanning from 35 to 500 AMU every one second or less, using 70 volts (nominal) electron energy in the electron impact ionization mode. The mass spectrometer must be capable of producing a mass spectrum for decafluorotriphenylphosphine (DFTPP) which meets all of the criteria in Table 6 when the GC/MS tuning standard is injected through the GC.

6.4 GC/MS Interface: Any GC-to-MS interface that gives acceptable calibration points and achieves acceptable tuning performance criteria may be used.

6.5 Data System: A computer system must be interfaced to the mass spectrometer. The

system must allow the continuous acquisition and storage on machine-readable media of all mass spectra obtained throughout the duration of the chromatographic program. The computer must have software that can search any GC/MS data file for ions of a specific mass and that can plot such ion abundances versus time or scan number. This type of plot is defined as the Extracted Ion Current Profile (EICP). Software must also be available that allows integrating the abundances in any EICP between specified time or scan-number limits. The most recent version of the EPA/NIH Mass Spectral Library is recommended.

6.6 Syringe: 5 µL Hamilton Laboratory grade syringes or equivalent.

6.7 Carrier gas: Ultra high purity helium.

7 REAGENTS AND STANDARDS

7.1 A minimum five point calibration curve is prepared. If a quadratic regression is used, six points must be analyzed for the calibration curve. The low point should be at or below the reporting limit. Refer to Tables 12 and 13 for typical calibration levels for all analytes. Other calibration levels may be used, depending on instrument capability, but the low standard must support the reporting limit and the high standard defines the range of the calibration.

7.2 An Internal Standard solution is prepared by diluting a purchased standard. Compounds in the I.S. Mix are: acenaphthene-d10, chrysene-d12, 1,4-dichlorobenzene-d4, naphthalene-d8, perylene-d12, and phenanthrene-d10.

7.3 Surrogate Standard Spiking Solution: Prepare as indicated in the preparative methods. See appropriate preparation SOP. Surrogate compounds and levels are listed in Table 11.

7.4 GC/MS Tuning Standard: A methylene chloride solution containing decafluorotriphenylphosphine (DFTPP) is prepared. Pentachlorophenol, benzidine, and DDT, should also be included in the Tuning Standard. All components are at 25 ug/mL.

7.5 The standards listed in 7.1 to 7.4 should be refrigerated at $\leq$ 6°C when not in use. Refrigeration at -10°C to -20°C may be used if it can be demonstrated that analytes do not fall out of solution at this temperature. The standards must be replaced at least once a year.

8 SAMPLE PRESERVATION AND STORAGE

8.1 Sample extracts are stored at 4 $\pm$ 2°C. Samples and extracts should be stored in suitable glass containers with Teflon lined caps. (Extracts will normally be stored for 30 days after invoicing.)

8.3 Water samples are extracted within seven days of sampling and the extracts are analyzed

within forty days of extraction. Solids, sludges, and organic liquids are extracted within fourteen days of sampling and the extracts are analyzed within forty days of extraction.

9 QUALITY CONTROL

9.1 Initial Demonstration of Capability

9.1.1 For the standard analyte list, the initial demonstration and method detection limit (MDL) studies described in section 13 must be acceptable before analysis of samples may begin.

9.1.2 For non-standard analytes an MDL study should be performed and calibration curve generated before analyzing any samples, unless lesser requirements are previously agreed to with the client. In any event, the minimum initial demonstration required is analysis of an extracted standard at the reporting limit and a single point calibration.

9.2 Control Limits

In-house historical control limits must be determined for surrogates, matrix spikes, and laboratory control samples (LCS). These limits must be determined at least annually. The recovery limits are mean recovery +/- 3 standard deviations for surrogates, MS and LCS. Precision limits for matrix spikes / matrix spike duplicates are mean relative percent difference +/- 3 standard deviations.

9.2.1 These limits do not apply to dilutions (except for tests without a separate extraction), but surrogate and matrix spike recoveries will be reported.

9.2.2 All surrogate, LCS, and MS recoveries (except for dilutions) must be entered into QuantIMS (when available) or other database so that accurate historical control limits can be generated. For tests without a separate extraction, surrogates and matrix spikes will be reported for all dilutions.

9.2.3 Refer to the QC program document (QA-003) for further details of control limits.

9.3 Method Blank

A method blank is prepared and analyzed with each batch of samples. The method blank consists of reagent water for aqueous samples, and sodium sulfate for soil samples (Refer to SOP No. CORP-OP-0001NC for details). Surrogates are added and the method blank is carried through the entire analytical procedure. The method blank must not contain any analyte of interest at or above the reporting limit (except common laboratory contaminants, see below) or at or above 5% of the measured concentration of that analyte in the associated samples, whichever is higher.

- If the analyte is a common laboratory contaminant (phthalate esters), the data may be reported with qualifiers if the concentration of the analyte is less than five times the RL. Such action must be taken in consultation with the client.

- Reanalysis of any samples with reportable concentrations of analytes found in the method blank is required unless other actions are agreed with the client.

- If there is no target analyte greater than the RL in the samples associated with an unacceptable method blank, the data may be reported with qualifiers. Such action should be taken in consultation with the client.

9.3.1 The method blank must have acceptable surrogate recoveries. If surrogate recoveries are not acceptable, the data must be evaluated to determine if the method blank has served the purpose of demonstrating that the analysis is free of contamination. If surrogate recoveries are low and there are reportable analytes in the associated samples, re-extraction of the blank and affected samples will normally be required. Consultation with the client should take place.

9.3.2 If reanalysis of the batch is not possible due to limited sample volume or other constraints, the method blank is reported, all associated samples are flagged with a "B", and appropriate comments may be made in a narrative to provide further documentation.

9.3.3 Refer to the STL North Canton QC Program document (QA-003) for further details of the corrective actions.

9.4 Instrument Blank

9.4.1 Instruments must be evaluated for contamination during each 12 hour analytical run. This may be accomplished by analysis of a method blank. If a method blank is not available, an instrument blank must be analyzed. An instrument blank consists of methylene chloride with the internal standards added. It is evaluated in the same way as the method blank.

9.5 Laboratory Control Sample (LCS)

9.5.1 A laboratory control sample (LCS) is prepared and analyzed with every batch of samples. All control analytes must be within established control limits. The LCS is spiked with the compounds listed in Tables 9 and 10 unless specified by a client or agency.

9.5.2 If any control analyte in the LCS is outside the laboratory established historical control limits, corrective action must occur. Corrective action may include re-extraction and

reanalysis of the batch.

- If the batch is not re-extracted and reanalyzed, the reasons for accepting the batch must be clearly presented in the project records and the report. (An example of acceptable reasons for not reanalyzing might be that the matrix spike and matrix spike duplicate are acceptable, and sample surrogate recoveries are good, demonstrating that the problem was confined to the LCS).

- If re-extraction and reanalysis of the batch is not possible due to limited sample volume or other constraints, the LCS is reported, all associated samples are flagged, and appropriate comments are made in a narrative to provide further documentation.

9.5.3 Ongoing monitoring of the LCS provides evidence that the laboratory is performing the method within accepted QC guidelines for accuracy and precision.

9.5.4 Additionally, if an all-analyte check sample is used, all non-controlling compounds must attain a recovery of 5% or greater if the compound is on the client's list.

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

A matrix spike/matrix spike duplicate (MS/MSD) is prepared and analyzed with every batch of samples. The MS/MSD is spiked with the same subset of analytes as the LCS (See Tables 9 and 10). Compare the percent recovery and relative percent difference (RPD) to that in the laboratory specific historically generated limits.

- If any individual recovery or RPD falls outside the acceptable range, corrective action must occur. The initial corrective action will be to check the recovery of that analyte in the Laboratory Control Sample (LCS). Generally, if the recovery of the analyte in the LCS is within limits, then the laboratory operation is in control and analysis may proceed. The reasons for accepting the batch must be documented.

- If the recovery for any component is outside QC limits for both the Matrix spike / spike duplicate and the LCS, the laboratory is out of control and corrective action must be taken. Corrective action will normally include repreparation and reanalysis of the batch.

- If a MS/MSD is not possible due to limited sample, then a LCS duplicate should be analyzed. RPD of the LCS and LCSD are compared to the matrix spike limits.

- The matrix spike / duplicate must be analyzed at the same dilution as the unspiked sample, even if the matrix spike compounds will be diluted out.

9.7 Surrogates

9.7.1 Every sample, blank, and QC sample is spiked with surrogate standards. Surrogate spike recoveries must be evaluated by determining whether the concentration (measured as percent recovery) falls within the required recovery limits. The compounds routinely included in the surrogate spiking solution, along with recommended standard concentrations, are listed in Table 11.

9.7.2 If any surrogates are outside limits the following corrective actions must take place (except for dilutions):

- Check all calculations for error.
- Ensure that instrument performance is acceptable.
- Recalculate the data and/or reanalyze the extract if either of the above checks reveal a problem.

It is only necessary to reprepare / reanalyze a sample once to demonstrate that poor surrogate recovery is due to matrix effect, unless the analyst believes that the repeated out of control results are not due to matrix effect.

Note: If all associated QC meets criteria (blank, LCS/LCSD), up to one surrogate per fraction may be outside of acceptance criteria , as long as the recovery is greater than 10%.

Note: For Ohio VAP samples, all surrogates must be within acceptance criteria.

9.7.3 If the sample with surrogate recoveries outside the recovery limits was a sample used for an MS/MSD and the surrogate recoveries in the MS/MSD are also outside of the control limits, then the sample, the MS, and the MSD do not require reanalysis as this phenomenon would indicate a possible matrix problem.

9.7.4 If the sample is reanalyzed and the surrogate recoveries in the reanalysis are acceptable, then the problem was within the analyst's control and only the reanalyzed data should be reported. (Unless the reanalysis was outside holding times, in which case reporting both sets of results may be appropriate.)

9.7.5 If the reanalysis does confirm the original results, the original analysis is reported and the data flagged as estimated due to matrix effect.

9.8 Nonconformance and Corrective Action

9.8.1 Any deviations from QC procedures must be documented as a nonconformance, with applicable cause and corrective action approved by the facility QA Manager.

10 CALIBRATION AND STANDARDIZATION

10.1 Summary

10.1.1 The instrument is tuned for DFTPP, calibrated initially with a minimum five-point calibration curve, and verified each 12-hour shift with one or more continuing calibration standard(s). Recommended instrument conditions are listed in Table 5.

10.2 All standards and extracts are allowed to warm to room temperature before injecting.

10.3 Instrument Tuning

At the beginning of every twelve hour shift when analyses are to be performed, the GC/MS system must be checked to see if acceptable performance criteria (Table 6) is achieved for DFTPP (decafluorotriphenylphosphine).

10.3.1 Inject the GC/MS tuning standard (Section 7.4) into the GC/MS system. Obtain a background-corrected mass spectra of DFTPP and confirm that all the key m/z criteria in Table 6 are achieved. If all the criteria are not achieved, the analyst must retune the mass spectrometer and repeat the test until all criteria are achieved. The performance criteria must be achieved before any samples, blanks, or standards are analyzed.

10.3.2 The GC/MS tuning standard should also be used to evaluate the inertness of the chromatographic system. Benzidine and pentachlorophenol should not exhibit excessive tailing. If DDT is an analyte of interest, it must be included in the tuning standard, and its breakdown must be < 20%. Refer to section 12 for the appropriate calculations.

10.4 Initial Calibration

10.4.1 Internal Standard Calibration Procedure: Internal standards are listed in Table 7. Use the base peak m/z as the primary m/z for quantitation of the standards. If interferences are noted, use one of the next two most intense masses for quantitation.

10.4.2 Compounds should be assigned to the IS with the closest retention time.

10.4.3 Prepare calibration standards at a minimum of five concentration levels for each parameter of interest. Six standards must be used for a quadratic least squares calibration. Quadratic fit may NOT be used for samples analyzed under South Carolina

Certification. It may also be useful to analyze six calibration levels and use the lower five for most analytes and the upper five for analytes that have poor response. Add the internal standard mixture to result in 2 ng on column. (For example, 5 uL of 80ppm IS mix is added to 100 uL of extract. This results in 4 ng, but only 0.5ul is injected, resulting in a final on column amount of 2 ng.)The concentration ranges of all analytes are listed in tables 12 and 13.

10.4.4 Analyze each calibration standard and tabulate the area of the primary characteristic m/z against concentration for each compound and internal standard. Calculate response factors (RF), average response factors, and the percent RSD of the response factors for each compound using the equations in section 12 and verify that the CCC and SPCC criteria in section 10.4.5 and 10.4.6 are met. **No sample analysis may be performed unless these criteria are met.**

10.4.5 System Performance Check Compounds (SPCCs): The minimum average RF for semivolatile SPCCs is 0.050. If the minimum response factors are not met, the system must be evaluated and corrective action must be taken before sample analysis begins. Some possible problems are standard mixture degradation, injection port inlet contamination, contamination at the front end of the analytical column, and active sites in the column or chromatographic system. This check must be met before analysis begins.

SPCC Compounds:

N-nitroso-di-n-propylamine
Hexachlorocyclopentadiene
2,4-Dinitrophenol
4-Nitrophenol

10.4.6 Calibration Check Compounds (CCCs): The %RSD of the response factors for each CCC in the initial calibration must be less than 30% for the initial calibration to be considered valid. This criterion must be met before sample analysis begins. Problems similar to those listed under SPCCs could affect this criterion.

10.4.6.1 If none of the CCCs are required analytes, project specific calibration specifications must be agreed with the client.

10.4.6.2 CCC Compounds:

Phenol
Acenaphthene
1,4-Dichlorobenzene
N-nitrosodiphenylamine
2-Nitrophenol

Pentachlorophenol
2,4-Dichlorophenol
Fluoranthene
Hexachlorobutadiene
Di-n-octylphthalate
4-Chloro-3-methylphenol
Benzo(a)pyrene
2,4,6-Trichlorophenol

10.4.7 If the software in use is capable of routinely reporting curve coefficients for data validation purposes, and the necessary calibration reports can be generated, then the analyst should evaluate analytes with %RSD > 15% for calibration on a curve. If it appears that substantially better accuracy would be obtained using quantitation from a curve then the appropriate curve should be used for quantitation.

10.4.7.1 If an analyte in the initial calibration is > 15%, then calibration on a curve must be used. Linear or quadratic curve fits may be used. Linear curve fits only may be used for South Carolina Certification. The analyst should consider instrument maintenance to improve the linearity of response. Use of 1/Concentration^2 weighting is recommended to improve the accuracy of quantitation at the low end of the curve. If Relative Standard Error (RSE) is used to evaluate the curve it must be better than 15%. If the % RSD is >15%, the analyst may drop the low or high points in the ICAL, as long as a minimum of 5 points are maintained and the quantitation range is adjusted accordingly. If the % RSD is still >15%, a quadratic or linear curve may be used. The correlation coefficient (r) must be ≥ 0.990. If the correlation coefficient is < 0.990, then any hits for these compounds must be flagged as estimated. If a curve is not linear for any compound that is found in a samples, the result must be flagged as estimated. Linear is defined as <15% RSD or a correlation coefficient of 0.990.

10.4.7.2 Note: Several components do not respond well by this method (poor linearity). These compounds are famphur, benzenethiol, kepone, and 2,4-toluenediamine. If these compounds are requested by a client and hits are found, alternate standards or methods will be needed for more accurate quantitation. Sensitivity as demonstrated by the low standard is sufficient to substantiate a non-detect.

10.4.8 If time remains in the 12 hour period initiated by the DFTPP injection before the initial calibration, samples may be analyzed. Otherwise, proceed to continuing calibration.

10.4.9 **Quantitation is performed using the calibration curve or average response factor from the initial curve, not the continuing calibration.**

10.5 Continuing Calibration

10.5.1 At the start of each 12-hour period, the GC/MS tuning standard must be analyzed. The injection of DFTPP must result in a mass spectrum for DFTPP which meets the criteria given in Table 6.

10.5.2 Following a successful DFTPP analysis the continuing calibration standard(s) are analyzed. The standards must contain all semivolatile analytes, including all required surrogates. A mid level calibration standard is used for the continuing calibration.

10.5.3 The following criteria must be met for the continuing calibration to be acceptable:

- The SPCC compounds must have a response factor of ≥ 0.05.
- The percent difference or drift of the CCC compounds from the initial calibration must be $\leq 20\%$. (see section 12 for calculations) In addition, the percent difference or drift of all analytes must be $\leq 50\%$, with allowance for up to (4) compounds to be greater than 50%.
- The internal standard response must be within 50-200% of the response in the mid level of the initial calibration.
- The internal standard retention times must be within 30 seconds of the retention times in the mid-level of the initial calibration.
- NOTE: There is no internal standard criteria for samples. Criteria is only for continuing and initial calibrations.
- NOTE: Ohio VAP rules require that any sample with internal standard outliers be reanalyzed. The criteria for acceptance is between 50% and 200% of same internal standard in continuing calibration.

10.5.3.1 If none of the CCCs are required analytes, project specific calibration specifications must be agreed with the client.

10.5.4 Once the above criteria have been met, sample analysis may begin. Initial calibration average RFs (or the calibration curve) will be used for sample quantitation, not the continuing calibration RFs. Analysis may proceed until 12 hours from the injection of the DFTPP have passed. (A sample *injected* less than 12 hours after the DFTPP is acceptable.)

11 PROCEDURE

11.1 Sample Preparation

Samples are prepared following SOP CORP-OP-0001NC.

11.2 Sample Analysis Procedure

11.2.1 Calibrate the instrument as described in section 10. Depending on the target compounds required by the client, it may be necessary to use more than one calibration standard.

11.2.2 All samples must be analyzed using the same instrument conditions as the preceding continuing calibration standard.

11.2.3 Add internal standard to the extract to result in 2 ng injected on column. Mix thoroughly before injection into the instrument.

11.2.4 Inject the sample extract into the GC/MS system using the same injection technique as used for the standards.

11.2.5 The data system will determine the concentration of each analyte in the extract using calculations equivalent to those in section 12. Quantitation is based on the initial calibration, not the continuing calibration.

11.2.6 Identified compounds are reviewed for proper integration. Manual integrations are performed if necessary and are documented by the analyst or automatically by the data system.

11.2.7 Target compounds identified by the data system are evaluated using the criteria listed in section 12.1.

11.2.8 Library searches of peaks present in the chromatogram that are not target compounds (Tentatively Identified Compounds, TIC) may be performed if required by the client. They are evaluated using the criteria in section 12.3.

11.3 Dilutions

If the response for any compound exceeds the working range of the GC/MS system, a dilution of the extract is prepared and analyzed. An appropriate dilution should be in the

upper half of the calibration range. Samples may be screened to determine the appropriate dilution for the initial run. If the initial diluted run has no hits or hits below 20% of the calibration range and the matrix allows for analysis at a lesser dilution, the sample must be reanalyzed at a dilution targeted to bring the largest hit above 50% of the calibration range.

11.3.1 Guidance for Dilutions Due to Matrix

If the sample is initially run at a dilution and the baseline rise is less than the height of the internal standards, or if individual non-target peaks are less than two times the height of the internal standards, the sample should be reanalyzed at a more concentrated dilution. This requirement is approximate and subject to analyst judgement. For example, samples containing organic acids may need to be analyzed at a higher dilution to avoid destroying the column.

11.3.2 Reporting Dilutions

The most concentrated dilution with target compounds within the calibration range will be reported. Other dilutions will only be reported at client request.

11.4 Perform all qualitative and quantitative measurements. When the extracts are not being used for analyses, refrigerate them at 4 ± 2°C, protected from light in screw cap vials equipped with unpierced Teflon lined septa.

11.5 Retention time criteria for samples

If the retention time for any internal standard changes by more than 0.5 minutes from the last continuing calibration standard, the chromatographic system must be inspected for malfunctions and corrected. Reanalysis of samples analyzed while the system was malfunctioning is required.

11.5.1 If the retention time of any internal standard in any sample varies by more than 0.1 minute from the preceding continuing calibration standard, the data must be carefully evaluated to ensure that no analytes have shifted outside their retention time windows.

11.6 Procedural Variations

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

conformance, with a cause and corrective action described.

11.7 Troubleshooting Guide

11.7.1 Daily Instrument Maintenance

In addition to the checks listed in the instrument maintenance schedule in the STL North Canton Quality Assurance Manual (LQM), current version, the following daily maintenance should be performed.

- Clip Column as necessary.
- Install new or cleaned injection port liner as necessary.
- Install new septum as necessary.
- Perform autotune.

11.7.2 Major Maintenance

A new initial calibration is necessary following major maintenance. Major maintenance includes changing the column, cleaning the source, and replacing the multiplier. Refer to the manufacturer's manual for specific guidance.

12 DATA ANALYSIS AND CALCULATIONS

12.1 Qualitative identification

An analyte is identified by retention time and by comparison of the sample mass spectrum with the mass spectrum of a standard of the suspected compound (standard reference spectrum). Mass spectra for standard reference may be obtained on the user's GC/MS by analysis of the calibration standards or from the NBS library. Two criteria must be satisfied to verify identification: (1) elution of sample component at the same GC retention time as the standard component; and (2) correspondence of the sample component and the standard component characteristic ions. (Note: Care must be taken to ensure that spectral distortion due to co-elution is evaluated.)

- The sample component retention time must compare to within ± 0.2 min. of the retention time of the standard component. For reference, the standard must be run within the same twelve hours as the sample.
- All ions present in the standard mass spectra at a relative intensity greater than 10% (most abundant ion in the spectrum equals 100%) should be present in the sample

spectrum.

- The characteristic ions of a compound must maximize in the same scan or within one scan of each other.

- The relative intensities of ions should agree to within ±30% between the standard and sample spectra. (Example: For an ion with an abundance of 50% in the standard spectra, the corresponding sample abundance must be between 20% and 80%.)

12.1.1 If a compound cannot be verified by all the above criteria, but in the technical judgment of the analyst the identification is correct, the analyst shall report that identification and proceed with quantitation.

12.2 Mass chromatogram searches.

Certain compounds are unstable in the calibration standard and cannot be calibrated in the normal way. In particular, the compound hexachlorophene (CAS 70-30-4) falls into this category, and is required for Appendix IX analysis. For this analyte a mass chromatogram search is made.

12.2.1 Hexachlorophene

Display the mass chromatograms for mass 196 and mass 198 for the region of the chromatogram from at least 2 minutes before chrysene-d12 to at least 4 minutes after chrysene-d12. If peaks for both ions coincide then the analyst evaluates the spectrum for the presence of hexachlorophene. No quantitation is possible.

12.3 For samples containing components not associated with the calibration standards, a library search may be made for the purpose of tentative identification. The necessity to perform this type of identification will be determined by the type of analyses being conducted. Computer generated library search routines should not use normalization routines that would misrepresent the library or unknown spectra when compared to each other. Only after visual comparison of sample spectra with the nearest library searches shall the mass spectral interpretation specialist assign a tentative identification. Guidelines for making tentative identification are:

- Relative intensities of major ions in the reference spectrum (ions >10% of the most abundant ion) should be present in the sample spectrum.

- The relative intensities of the major ions should agree within ±20%. (Example: For an ion with an abundance of 50% in the standard spectrum, the corresponding sample ion abundance should be between 30%and 70%.)

- Molecular ions present in the reference spectrum should be present in the sample spectrum.

- Ions present in the sample spectrum, but not in the reference spectrum, should be reviewed for possible background contamination or presence of coeluting 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 coeluting peaks. Data system library reduction programs can sometimes create these discrepancies.

- Automatic background subtraction can severely distort spectra from samples with unresolved hydrocarbons.

12.4 Anyone evaluating data is trained to know how to handle isomers with identical mass spectra and close elution times. These include:

Dichlorobenzenes
Methylphenols
Trichlorophenols
Phenanthrene, anthracene
Fluoranthene, pyrene
Benzo(b) and (k)fluoranthene
Chrysene, benzo(a)anthracene

Extra precautions concerning these compounds are to more closely scrutinize retention time vs. the calibration standard and also to check that all isomers have distinct retention times.

A second category of problem compounds would be the poor responders or compounds that chromatograph poorly. Included in this category would be:

Benzoic acid
Chloroanilines
Nitroanilines
2,4-Dinitrophenol
4-Nitrophenol
Pentachlorophenol
3,3'-Dichlorobenzidine
Benzyl alcohol
4,6-Dinitro-2-methylphenol

Manually checking the integrations would be appropriate for these compounds.

12.5 Calculations

12.5.1 Percent Relative Standard Deviation for Initial Calibration

$$\%RSD = \frac{SD}{\overline{RF}} \times 100$$

$\overline{RF}$ = Mean of RFs from intial caibration for a compound

SD = Standard deviation of RFs from initial calibration for a compound,

$$= \sqrt{\sum_{i=1}^{N} \frac{\left(RFi - \overline{RF}\right)^2}{N-1}}$$

RFi = RF for each of the calibration levels

N = Number of RF values

12.5.2 Continuing calibration percent drift

$$\%Drift = \frac{C_{actual} - C_{found}}{C_{actual}} \times 100\%$$

C_{actual} = Known concentration in standard

C_{found} = Measured concentration using selected quantitation method

12.5.3 Concentration in the extract

The concentration of each identified analyte and surrogate in the extract is calculated from the linear or quadratic curve fitted to the initial calibration points, or from the average RF of the initial calibration. For South Carolina Certification the concentration of each identified analyte and surrogate in the extract is calculated from the linear curve only fitted to the initial calibration points, or from the average RF of the initial calibration.

12.5.3.1 Average response factor

If the average of all the %RSDs of the response factors in the initial calibration is $\leq$ 15%, the average response factor from the initial calibration may be used for quantitation.

$$C_{ex} = \frac{R_x C_{is}}{R_{is} \overline{RF}}$$

12.5.3.2 Linear fit (Use only Linear fit for South Carolina Certification)

$$C_{ex} = A + B\frac{(R_x C_{is})}{R_{is}}$$

C_{ex} = Concentration in extract, µg/mL

R_x = Response for analyte

C_{is} = Concentration of internal standard

A = Intercept

B = Slope

12.5.3.3 Quadratic fit

$$C_{ex} = A + B\left(\frac{R_x C_{is}}{R_{is}}\right) + C\left(\frac{R_x C_{is}^{\ 2}}{R_{is}}\right)$$

C = Curvature

12.5.4 The concentration in the sample is then calculated.

12.5.4.1 Aqueous Calculation

$$\text{Concentration, } \mu g / L = \frac{C_{ex} V_t}{V_o}$$

Where:

V_t = Volume of total extract, µL, taking into account dilutions (i.e., a 1-to-10 dilution of a 1 mL extract will mean V_t = 10,000 µL. If half of the base/neutral extract and half of the acid extract are combined, V_t = 2,000.)

V_o = Volume of water extracted (mL)

12.5.5 Sediment/Soil, Sludge (on a dry-weight basis) and Waste (normally on a wet-weight basis:

$$\text{Concentration, } \mu g / kg = \frac{C_{ex} V_t}{W_s D}$$

W_s = Weight of sample extracted or diluted in grams

D = (100 - % moisture in sample)/100, for a dry weight basis or 1 for a wet weight basis

12.6 MS/MSD percent recovery calculation.

$$\text{Matrix Spike Recovery} = \frac{S_{SR} - S_R}{S_A} \times 100\%$$

S_{SR} = Spike sample result

S_R = Sample result

S_A = Spike added

12.7 Relative % Difference calculation for the MS/MSD

$$RPD = \frac{MS_R - MSD_R}{1/2(MS_R + MSD_R)} \times 100$$

RPD = Relative percent difference

MS_R = Matrix spike result

MSD_R = Matrix spike duplicate result

12.8 Relative response factor calculation.

$$RF = \frac{A_x C_{is}}{A_{is} C_x}$$

A_x=Area of the characteristic ion for the compound being measured

A_{is}=Area of the characteristic ion for the specific internal standard

C_x=Concentration of the compound being measured (µg/L)

C_{is} =Concentration of the specific internal standard (µg/L)

12.9 Calculation of TICs: The calculation of TICs (tentatively identified compounds) is identical to the above calculations with the following exceptions:

A_x = Area of the total ion chromatogram for the compound being measured

A_{IS} = Area of the total ion chromatogram for the nearest internal standard without interference

RF = 1

12.10 Percent DDT breakdown

$$\% \text{ DDT breakdown} = \frac{\text{DDEarea} + \text{DDDarea}}{\text{DDTarea} + \text{DDEarea} + \text{DDarea}}$$

The total ion current areas are used for this calculation

13 METHOD PERFORMANCE

13.1 Method Detection Limit

Each laboratory must generate a valid method detection limit for each analyte of interest. The MDL must be below the reporting limit for each analyte. The procedure for determination of the method detection limit is given in 40 CFR Part 136, Appendix B, and further defined in QA Policy #: QA-005.

13.2 Initial Demonstration

Each laboratory must make an initial demonstration of capability for each individual method. Demonstration of capability for both soil and water matrices is required. This requires analysis of QC check samples containing all of the standard analytes for the method. For some tests it may be necessary to use more than one QC check mix to cover all analytes of interest.

13.2.1 Four aliquots of the QC check sample are analyzed using the same procedures used to analyze samples, including sample preparation.

13.2.2 Calculate the average recovery and standard deviation of the recovery for each analyte of interest.

13.2.3 If any analyte does not meet the acceptance criteria the test must be repeated. Only those analytes that did not meet criteria in the first test need to be evaluated. Repeated failure for any analyte indicates the need for the laboratory to evaluate the analytical procedure and take corrective action.

13.3 Non-standard analytes

For non-standard analytes, an MDL study must be performed and calibration curve generated before analyzing any samples, unless lesser requirements are previously agreed to with the client. In any event, the minimum initial demonstration required is analysis of an extracted standard at the reporting limit and a single point calibration.

13.4 Training Qualification

The group/team leader 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.

14 POLLUTION PREVENTION

14.1 This section is not applicable to this procedure.

15 WASTE MANAGEMENT

15.1 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."

15.2 Laboratory personnel assigned to perform hazardous waste disposal procedures must have a working knowledge of the established procedures and practices of STL. They must have training on the hazardous waste disposal practices upon initial assignment to these tasks, followed by an annual refresher training.

15.3 Waste Streams Produced by the Method

15.3.1 **Vials containing sample extracts:** These vials are placed in the vial waste located in the GC/MS laboratory.

16 REFERENCES

16.1 References

16.1.1 SW846, Test Methods for Evaluating Solid Waste, Third Edition, Update II, October 1994, Semivolatile Organic Compounds by Gas Chromatography/Mass Spectrometry (GC/MS): Capillary Column Technique, Method 8270C.

16.1.2 J. W. Eichelberger, L. E. Harris, and W. L. Budde, "Reference Compound to Calibrate Ion Abundance Measurement in Gas Chromatography/Mass Spectrometry," Analytical Chemistry, 47, 995 (1975)

16.1.3 Corporate Quality Management Plan (QMP), current version.

16.1.4 STL Laboratory Quality Manual (LQM), current version.

16.2 Associated SOPs and Policies, latest version

16.2.1 QA Policy, QA-003

16.2.2 Glassware Washing, NC-QA-0014

16.2.3 Statistical Evaluation of Data and Development of Control Charts, NC-QA-0018

16.2.4 Method Detection Limits and Instrument Detection Limits, NC-QA-0021

16.2.5 Navy/Army SOP, NC-QA-0016

17 MISCELLANEOUS

17.1 Modifications from Reference Method

17.1.1 A retention time window of 0.2 minutes is used for all components, since some data systems do not have the capability of using the relative retention time units specified in the reference method.

17.1.2 The quantitation and qualifier ions from coms compounds have been changed from those recommennded in SW-846 in order to improve the reliability of qualitative identification.

17.2 Tables

Table 1

STL North Canton Primary Standard and Standard Reporting Limits

Analytes	CAS Number	Standard Reporting Limits	
		Aqueous µg/L	Low Soil/Sediment µg/kg
Pyridine	110-86-1	20	660
N-nitrosodimethylamine	62-75-9	10	330
Aniline	62-53-3	10	330
Phenol	108-95-2	10	330
Bis(2-chloroethyl)ether	111-44-4	10	330
2-Chlorophenol	95-57-8	10	330
1,3-Dichlorobenzene	541-73-1	10	330
1,4-Dichlorobenzene	106-46-7	10	330
Benzyl alcohol	100-51-6	10	330
1,2-Dichlorobenzene	95-50-1	10	330
2-Methylphenol	95-48-7	10	330
2,2'-oxybis(1-chloropropane)[1]	108-60-1	10	330
4-Methylphenol	106-44-5	10	330
N-Nitroso-di-n-propylamine	621-64-7	10	330
Hexachloroethane	67-72-1	10	330
Nitrobenzene	98-95-3	10	330
Isophorone	78-59-1	10	330
2-Nitrophenol	88-75-5	10	330
2,4-Dimethylphenol	105-67-9	10	330
Benzoic acid	65-85-0	50	1600
Bis(2-chloroethoxy)methane	111-91-1	10	330

Table 1

STL North Canton Primary Standard and Standard Reporting Limits

Analytes	CAS Number	Standard Reporting Limits	
		Aqueous µg/L	Low Soil/Sediment µg/kg
2,4-Dichlorophenol	120-83-2	10	330
1,2,4-Trichlorobenzene	120-82-1	10	330
Naphthalene	91-20-3	10	330
4-Chloroaniline	106-47-8	10	330
Hexachlorobutadiene	87-68-3	10	330
4-Chloro-3-methylphenol	59-50-7	10	330
2-Methylnaphthalene	91-57-6	10	330
Hexachlorocyclopentadiene	77-47-4	50	1600
2,4,6-Trichlorophenol	88-06-2	10	330
2,4,5-Trichlorophenol	95-95-4	10	330
2-Chloronaphthalene	91-58-7	10	330
2-Nitroaniline	88-74-4	50	1600
Dimethyl phthalate	131-11-3	10	330
Acenaphthylene	208-96-8	10	330
3-Nitroaniline	99-09-2	50	1600
Acenaphthene	83-32-9	10	330
2,4-Dinitrophenol	51-28-5	50	1600
4-Nitrophenol	100-02-7	50	1600
Dibenzofuran	132-64-9	10	330
2,4-Dinitrotoluene	121-14-2	10	330
2,6-Dinitrotoluene	606-20-2	10	330
Diethylphthalate	84-66-2	10	330
4-Chlorophenyl phenyl ether	7005-72-3	10	330
Fluorene	86-73-7	10	330
4-Nitroaniline	100-01-6	50	1600
4,6-Dinitro-2-methylphenol	534-52-1	50	1600
N-Nitrosodiphenylamine	86-30-6	10	330
Azobenzene	103-33-3	10	330
4-Bromophenyl phenyl ether	101-55-3	10	330
Hexachlorobenzene	118-74-1	10	330
Pentachlorophenol	87-86-5	50	1600
Phenanthrene	85-01-8	10	330
Anthracene	120-12-7	10	330
Carbazole	86-74-8	10	330
Di-n-butyl phthalate	84-74-2	10	330
Fluoranthene	206-44-0	10	330
Benzidine	92-87-5	100	3300
Pyrene	129-00-0	10	330
Butyl benzyl phthalate	85-68-7	10	330
3,3'-Dichlorobenzidine	91-94-1	50	1600
Benzo(a)anthracene	56-55-3	10	330
Bis(2-ethylhexyl)phthalate	117-81-7	10	330
Chrysene	218-01-9	10	330

Table 1

STL North Canton Primary Standard and Standard Reporting Limits

Analytes	CAS Number	Standard Reporting Limits	
		Aqueous µg/L	Low Soil/Sediment µg/kg
Di-n-octylphthalate	117-84-0	10	330
Benzo(b)fluoranthene	205-99-2	10	330
Benzo(k)fluoranthene	207-08-9	10	330
Benzo(a)pyrene	50-32-8	10	330
Indeno(1,2,3-cd)pyrene	193-39-5	10	330
Dibenz(a,h)anthracene	53-70-3	10	330
Benzo(g,h,i)perylene	191-24-2	10	330
Benzaldehyde	100-52-7	10	330
Caprolactam	105-60-2	10	330
1,1-Biphenyl	92-52-4	10	330
Atrazine	1912-24-9	10	330
Benzenethiol	108-98-5	10	330
Indene	95-13-6	10	330
Quinoline	91-22-5	10	330
1-Methyl Naphthalene	90-12-0	10	330

[1] 2,2'oxybis(1-chloropropane) was formerly known as bis(2-chloroisopropyl)ether.

Table 2

STL North Canton Appendix IX[1] Standard Reporting Limits

Semivolatiles	CAS Number	Standard Reporting Limits	
		Aqueous µg/L	Low Soil/Sediment µg/kg
2-Picoline	109-06-8	20	660
N-Nitrosomethylethylamine	10595-95-6	10	330
Methyl methanesulfonate	66-27-3	10	330
N-Nitrosodiethylamine	55-18-5	10	330
Ethyl methanesulfonate	62-50-0	10	330
Pentachloroethane	76-01-7	50	1600
Acetophenone	98-86-2	10	330
N-Nitrosopyrrolidine	930-55-2	10	330
N-Nitrosomorpholine	59-89-2	10	330
o-Toluidine	95-53-4	20	660
3-Methylphenol	108-39-4	10	330
N-Nitrosopiperidine	100-75-4	10	330
o,o,o-Triethyl-Phosphorothioate[2]	126-68-1	50	1600
a,a-Dimethyl-phenethylamine	122-09-8	50	1600
2,6-Dichlorophenol	87-65-0	10	330
Hexachloropropene	1888-71-7	100	3300
p-Phenylenediamine	106-50-3	100	3300

Table 2

STL North Canton Appendix IX[1] Standard Reporting Limits

Semivolatiles	CAS Number	Standard Reporting Limits	
		Aqueous µg/L	Low Soil/Sediment µg/kg
n-Nitrosodi-n-butylamine	924-16-3	10	330
Safrole	94-59-7	20	660
1,2,4,5-Tetrachlorobenzene	95-94-3	10	330
Isosafrole	120-58-1	20	660
1,4-Dinitrobenzene	100-25-4	10	330
1,4-Naphthoquinone	130-15-4	50	1600
1,3-Dinitrobenzene	99-65-0	10	330
Pentachlorobenzene	608-93-5	10	330
1-Naphthylamine	134-32-7	10	330
2-Naphthylamine	91-59-8	10	330
2,3,4,6-Tetrachlorophenol	58-90-2	50	1600
5-Nitro-o-toluidine	99-55-8	20	660
Thionazin[2]	297-97-2	50	1600
1,3,5-Trinitrobenzene	99-35-4	50	1600
Sulfotepp[2]	3689-24-5	50	1600
Phorate[2]	298-02-2	50	1600
Phenacetin	62-44-2	20	660
Diallate[2]	2303-16-4	20	660
Dimethoate[2]	60-51-5	20	660
4-Aminobiphenyl	92-67-1	50	1600
Pentachloronitrobenzene	82-68-8	50	1600
Pronamide	23950-58-5	20	660
Disulfoton[2]	298-04-4	50	1600
2-secbutyl-4,6-dinitrophenol (Dinoseb[2])	88-85-7	20	660
4-Nitroquinoline-1-oxide	56-57-5	100	3300
Methapyrilene	91-80-5	50	1600
Aramite	140-57-8	20	660
Famphur[3]	52-85-7	100	3300
p-(Dimethylamino)azobenzene	60-11-7	20	660
p-Chlorobenzilate	510-15-6	10	330
3,3'-Dimethylbenzidine	119-93-7	50	1600
2-Acetylaminofluorene	53-96-3	100	3300
Dibenz(a,j)acridine	224-42-0	20	660
7,12-Dimethylbenz(a)anthracene	57-97-6	20	660
3-Methylcholanthrene	56-49-5	20	660

[1] The Appendix IX standard contains additional analytes required for the Appendix IX list. The STL North Canton primary standard must also be analyzed to include all of the Appendix IX list

[2] May also be analyzed by method 8141, which can achieve lower reporting limits.

[3] It is highly recommended that Famphur is analyzed by method 8081. It is a poor responder by 8270C

Table 2A
STL North Canton Michigan Program[1]

Semivolatile	CAS Number	Michigan Reporting Limits	
		Aqueous µg/L	Low Soil/Sediment µg/kg
Acenaphthene	83-32-9	5	330
Acenaphthylene	208-96-8	5	330
Acetophenone	98-86-2	5	330
Anthracene	120-12-7	5	330
Atrzine	1912-24-9	5	330
Benzaldehyde	100-52-7	10	330
Benzo(a)anthracene	56-55-3	1	330
Benzo(a)pyrene	50-32-8	2	330
Benzo(b)fluoranthene	205-99-2	2	330
Benzo(g,h,i)perylene	191-24-2	5	330
Benzo(k)fluoranthene	207-08-9	5	330
1,1'-Biphenyl	92-52-4	10	330
4-Bromophenylphenyl ether	101-55-3	5	330
Butylbenzylphthalate	85-68-7	5	330
di-n-Butylphthalate	84-74-2	5	330
Caprolactam	105-60-2	10	330
Carbazole	86-74-8	10	330
4-Chloroaniline	106-47-8	20	1700
bis(2-Chloroethoxy)methane	111-91-1	5	330
bis(2-Chloroethyl)ether	111-44-4	4	330
bis(2-Chloroisopropyl)ether	108-60-1	5	330
4-Chloro-3-Methylphenol	59-50-7	5	330
2-Chloronaphthalene	91-58-7	5	330
2-Chlorophenol	95-57-8	5	330
4-Chlorophenyl phenyl ether	7005-72-3	5	330
Chrysene	218-01-9	5	330
Dibenz(a,h)anthracene	53-70-3	2	330
Dibenzofuran	132-64-9	5	330
3,3'-Dichlorobenzidine	91-94-1	4	2000
2,4-Dichlorophenol	120-83-2	10	330
Diethylphthalate	84-66-2	5	330
2-4-Dimethylphenol	105-67-9	5	330
Dimethylphthalate	131-11-3	5	330
4,6-Dinitro-2-methylphenol	534-52-1	20	1700
2,4-Dinitrophenol	51-28-5	20	1700
2,4-Dinitrotoluene	121-14-2	5	330
2,6-Dinitrotoluene	606-20-2	5	330
bis(2-Ethylhexyl)phthalate	117-81-7	5	330
Fluoranthene	206-44-0	5	330
Fluorene	86-73-7	5	330

Table 2A
STL North Canton Michigan Program[1]

Semivolatile	CAS Number	Michigan Reporting Limits	
		Aqueous µg/L	Low Soil/Sediment µg/kg
Hexachlorobenzene	118-74-1	5	330
Hexachlorobutadiene	87-68-3	5	330
Hexachlorocyclopentadiene	77-47-4	5	330
Hexachloroethane	67-72-1	5	330
Indeno(1,2,3-cd)pyrene	193-39-5	2	330
Isophorone	78-59-1	5	330
2-Methylnapthalene	91-57-6	5	330
2-Methylphenol	95-48-7	5	330
4-Methylphenol	106-44-5	5	330
Naphthalene	91-20-3	5	330
2-Nitroaniline	88-74-4	20	1700
3-Nitroaniline	99-09-2	20	1700
4-Nitroaniline	100-01-6	20	1700
Nitrobenzene	95-95-3	4	330
2-Nitrophenol	88-75-5	5	330
4-Nitrophenol	100-02-7	20	1700
N-Nitroso-di-n-propylamine	621-64-7	5	330
N-Nitrosodiphenylamine (diphenylamine)	62-75-9	5	330
di-n-Octylphthalate	117-84-0	5	330
Pentachlorophenol	87-86-5	20	800
Phenanthrene	85-01-8	5	330
Phenol	108-95-2	5	330
Pyrene	129-00-0	5	330
2,4,5-Trichlorophenol	95-95-4	5	330
2,4,6-Trichlorophenol	88-06-2	4	330

[1] Reporting Limits are only for samples performed under the Michigan program.

Table 3

Reportable Analytes for STL North Canton Standard Tests, Primary Standard

Analyte	CAS Number	TCLP	TCL	Appendix IX
Pyridine	110-86-1	X		X
N-nitrosodimethylamine	62-75-9			X
Aniline	62-53-3			X
Phenol	108-95-2		X	X
Bis(2-chloroethyl)ether	111-44-4		X	X
2-Chlorophenol	95-57-8		X	X
1,3-Dichlorobenzene	541-73-1		X	X
1,4-Dichlorobenzene	106-46-7	X	X	X
Benzyl alcohol	100-51-6			X
1,2-Dichlorobenzene	95-50-1		X	X
2-Methylphenol	95-48-7	X	X	X

Table 3

Reportable Analytes for STL North Canton Standard Tests, Primary Standard

Analyte	CAS Number	TCLP	TCL	Appendix IX
2,2'-oxybis(1-chloropropane)	180-60-1		X	X
4-Methylphenol	106-44-5	X	X	X
N-Nitroso-di-n-propylamine	621-64-7		X	X
Hexachloroethane	67-72-1	X	X	X
Nitrobenzene	98-95-3	X	X	X
Isophorone	78-59-1		X	X
2-Nitrophenol	88-75-5		X	X
2,4-Dimethylphenol	105-67-9		X	X
Benzoic acid	65-85-0			
Bis(2-chloroethoxy)methane	111-91-1		X	X
2,4-Dichlorophenol	120-83-2		X	X
1,2,4-Trichlorobenzene	120-82-1		X	X
Naphthalene	91-20-3		X	X
4-Chloroaniline	106-47-8		X	X
Hexachlorobutadiene	87-68-3	X	X	X
4-Chloro-3-methylphenol	59-50-7		X	X
2-Methylnaphthalene	91-57-6		X	X
Hexachlorocyclopentadiene	77-47-4		X	X
2,4,6-Trichlorophenol	88-06-2	X	X	X
2,4,5-Trichlorophenol	95-95-4	X	X	X
2-Chloronaphthalene	91-58-7		X	X
2-Nitroaniline	88-74-4		X	X
Dimethyl phthalate	131-11-3		X	X
Acenaphthylene	208-96-8		X	X
3-Nitroaniline	99-09-2		X	X
Acenaphthene	83-32-9		X	X
2,4-Dinitrophenol	51-28-5		X	X
4-Nitrophenol	100-02-7		X	X
Dibenzofuran	132-64-9		X	X
2,4-Dinitrotoluene	121-14-2	X	X	X
2,6-Dinitrotoluene	606-20-2		X	X
Diethylphthalate	84-66-2		X	X
4-Chlorophenyl phenyl ether	7005-72-3		X	X
Fluorene	86-73-7		X	X
4-Nitroaniline	100-01-6		X	X
4,6-Dinitro-2-methylphenol	534-52-1		X	X
N-Nitrosodiphenylamine	86-30-6		X	X
Azobenzene[1]	103-33-3			
4-Bromophenyl phenyl ether	101-55-3		X	X
Hexachlorobenzene	118-74-1	X	X	X
Pentachlorophenol	87-86-5	X	X	X
Phenanthrene	85-01-8		X	X
Anthracene	120-12-7		X	X
Carbazole	86-74-8		X	
	84-74-2		X	X
Fluoranthene	206-44-0		X	X

Table 3

Reportable Analytes for STL North Canton Standard Tests, Primary Standard

Analyte	CAS Number	TCLP	TCL	Appendix IX
Benzidine	92-87-5			
Pyrene	129-00-0		X	X
Butyl benzyl phthalate	85-68-7		X	X
3,3'-Dichlorobenzidine	91-94-1		X	X
Benzo(a)anthracene	56-55-3		X	X
Bis(2-ethylhexyl)phthalate	117-81-7		X	X
Chrysene	218-01-9		X	X
Di-n-octylphthalate	117-84-0		X	X
Benzo(b)fluoranthene	205-99-2		X	X
Benzo(k)fluoranthene	207-08-9		X	X
Benzo(a)pyrene	50-32-8		X	X
Indeno(1,2,3-cd)pyrene	193-39-5		X	X
Dibenz(a,h)anthracene	53-70-3		X	X
Benzo(g,h,i)perylene	191-24-2		X	X
Benzaldehyde	100-52-7		X	
Caprolactam	105-60-2		X	
1,1-Biphenyl	92-52-4		X	
Atrazine	1912-24-9		X	

[1] Azobenzene is formed by decomposition of 1,2-diphenylhydrazine. If 1,2-diphenylhydrazine is requested, it will be analyzed as azobenzene.

Table 4

Reportable analytes for STL North Canton Standard Tests, Appendix IX Standard

Semivolatiles	CAS Number	TCLP	TCL	Appendix IX
2-Picoline	109-06-8			X
N-Nitrosomethylethylamine	10595-95-6			X
Methyl methanesulfonate	66-27-3			X
N-Nitrosodiethylamine	55-18-5			X
Ethyl methanesulfonate	62-50-0			X
Pentachloroethane	76-01-7			X
Acetophenone	98-86-2		X	X
N-Nitrosopyrrolidine	930-55-2			X
N-Nitrosomorpholine	59-89-2			X
o-Toluidine	95-53-4			X
3-Methylphenol	108-39-4			X
N-Nitrosopiperidine	100-75-4			X
o,o,o-Triethyl-Phosphorothioate[1]	126-68-1			X
a,a-Dimethyl-phenethylamine	122-09-8			X
2,6-Dichlorophenol	87-65-0			X
Hexachloropropene	1888-71-7			X
p-Phenylenediamine	106-50-3			X
n-Nitrosodi-n-butylamine	924-16-3			X
Safrole	94-59-7			X
1,2,4,5-Tetrachlorobenzene	95-94-3			X

Table 4

Reportable analytes for STL North Canton Standard Tests, Appendix IX Standard

Semivolatiles	CAS Number	TCLP	TCL	Appendix IX
Isosafrole	120-58-1			X
1,4-Dinitrobenzene	100-25-4			X
1,4-Naphthoquinone	130-15-4			X
1,3-Dinitrobenzene	99-65-0			X
Pentachlorobenzene	608-93-5			X
1-Naphthylamine	134-32-7			X
2-Naphthylamine	91-59-8			X
2,3,4,6-Tetrachlorophenol	58-90-2			X
5-Nitro-o-toluidine	99-55-8			X
Thionazin[1]	297-97-2			X
1,3,5-Trinitrobenzene	99-35-4			X
Sulfotepp[1]	3689-24-5			X
Phorate[1]	298-02-2			X
Phenacetin	62-44-2			X
Diallate	2303-16-4			X
Dimethoate[1]	60-51-5			X
4-Aminobiphenyl	92-67-1			X
Pentachloronitrobenzene	82-68-8			X
Pronamide	23950-58-5			X
Disulfoton[1]	298-04-4			X
2-secbutyl-4,6-dinitrophenol (Dinoseb)[1]	88-85-7			X
4-Nitroquinoline-1-oxide	56-57-5			X
Famphur[2]	52-85-7			X
Methapyrilene	91-80-5			X
Aramite	140-57-8			X
p-(Dimethylamino)azobenzene	60-11-7			X
p-Chlorobenzilate	510-15-6			X
3,3'-Dimethylbenzidine	119-93-7			X
2-Acetylaminofluorene	53-96-3			X
Dibenz(a,j)acridine[3]	224-42-0			
7,12-Dimethylbenz(a)anthracene	57-97-6			X
3-Methylcholanthrene	56-49-5			X
Hexachlorophene[4]	70-30-4			X
Diphenylamine[5]	122-39-4			X

[1] May also be analyzed by method 8141, which can achieve lower reporting limits.

[2] May also be analyzed by method 8081, which can achieve lower reporting limits

[3] Skinner List Compound

[4] Hexachlorophene is a required analyte for Appendix IX. This compound is not stable, and therefore not included in the calibration standard. The characteristic ions for hexachlorophene are searched for in the chromatogram. (See section 12.2.1)

[5] Diphenylamine is a required compound for Appendix IX N-nitrosodiphenylamine decomposes in the injection

port to form diphenylamine. Therefore these two compounds cannot be distinguished. Diphenylamine is not included in the calibration standard.

Table 5

Suggested Instrumental Conditions

Mass Range	35-500 amu
Scan Time	≤1 second/scan
Initial Column Temperature/Hold Time	45°C for 1 minutes
Column Temperature Program	45- 100°C at 25°C/min for 0 min 100 - 280°C at 30°C/min for 0 min 280 - 100°C at 25°C/min for 2 min
Final Column Temperature/Hold Time	320°C (until at least one minute after benzo(g,h,i)perylene has eluted)
Injector Temperature	250 - 300°C
Transfer Line Temperature	250 - 300°C
Source Temperature	According to manufacturer's Specifications
Injector	Grob-type, split / splitless
Sample Volume	0.5 µl
Carrier Gas	Helium at 30 cm/sec

Table 6

DFTPP Key Ions and Ion Abundance Criteria

Mass	Ion Abundance Criteria
51	30 – 60% of mass 198
68	<2% of mass 69
70	<2% of mass 69
127	40 – 60% of mass 198
197	<1% of mass 198
198	Base peak, 100% relative abundance
199	5 – 9% of mass 198
275	10 – 30% of mass 198
365	>1% of mass 198
441	Present, but less than mass 443
442	>40% of mass 198
443	17 – 23% of mass 442

Table 7

Analytes in Approximate Retention Time Order and Characteristic Ions, Primary Standard

Analyte	Primary	Secondary	Tertiary
N-nitrosodimethylamine	74	42	
Pyridine	79	52	
2-Fluorophenol (Surrogate Standard)	112	64	63
Phenol-d5 (Surrogate Standard)	99	42	71
Benzaldehyde	77	105	106
Aniline	93	66	
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-d4 (Internal Standard)	152	150	115
1,4-Dichlorobenzene	146	148	113
Benzyl Alcohol	108	79	77
1,2-Dichlorobenzene	146	148	113
2-Methylphenol	108	107	79
2,2'-oxybis(1-chloropropane)[1]	45	77	79
4-Methylphenol	108	107	79
N-Nitroso-di-n-propylamine	70	42	101,130
Hexachloroethane	117	201	199
Nitrobenzene-d5 (Surrogate Standard)	82	128	54
Nitrobenzene	77	123	65
Isophorone	82	95	138
2-Nitrophenol	139	65	109
2,4-Dimethylphenol	107	121	122
Benzoic Acid	122	105	77
Bis(2-chloroethoxy)methane	93	95	123
2,4-Dichlorophenol	162	164	98
1,2,4-Trichlorobenzene	180	182	145
Naphthalene-d8 (Internal Standard)	136	68	54
Naphthalene	128	129	127
4-Chloroaniline	127	129	65
Hexachlorobutadiene	225	223	227
Caprolactam	113	55	56
4-Chloro-3-methylphenol	107	144	142
2-Methylnaphthalene	142	141	115
Hexachlorocyclopentadiene	237	235	272
2,4,6-Trichlorophenol	196	198	200
2,4,5-Trichlorophenol	196	198	200
1,1'-Biphenyl	154	153	76
2-Fluorobiphenyl (Surrogate Standard)	172	171	170
2-Chloronaphthalene	162	164	127
2-Nitroaniline	65	92	138

Table 7

Analytes in Approximate Retention Time Order and Characteristic Ions, Primary Standard

Analyte	Primary	Secondary	Tertiary
Dimethylphthalate	163	194	164
Acenaphthylene	152	151	153
2,6-Dinitrotoluene	165	63	89
Acenaphthene-d10 (Internal Standard)	164	162	160
3-Nitroaniline	138	108	92
Acenaphthene	153	152	154
2,4-Dinitrophenol	184	63	154
Dibenzofuran	168	139	84
4-Nitrophenol	109	139	65
2,4-Dinitrotoluene	165	63	89
Diethylphthalate	149	177	150
Fluorene	166	165	167
4-Chlorophenylphenylether	204	206	141
4-Nitroaniline	138	92	108
4,6-Dinitro-2-methylphenol	198	182	77
N-Nitrosodiphenylamine	169	168	167
2,4,6-Tribromophenol (Surrogate Standard)	330	332	141
Azobenzene	77	182	105
4-Bromophenylphenylether	248	250	141
Hexachlorobenzene	284	142	249
Atrazine	200	173	215
Pentachlorophenol	266	264	268
Phenanthrene-d10 (Internal Standard)	188	94	80
Phenanthrene	178	179	176
Anthracene	178	179	176
Carbazole	167	166	139
Di-n-butylphthalate	149	150	104
Fluoranthene	202	101	100
Benzidine	184	92	185
Pyrene	202	101	100
Terphenyl-d14 (Surrogate Standard)	244	122	212
Butylbenzylphthalate	149	91	206
Benzo(a)Anthracene	228	229	226
Chrysene-d12 (Internal Standard)	240	120	236
3,3'-Dichlorobenzidine	252	254	126
Chrysene	228	226	229
Bis(2-ethylhexyl)phthalate	149	167	279
Di-n-octylphthalate	149	167	43
Benzo(b)fluoranthene	252	253	125
Benzo(k)fluoranthene	252	253	125
Benzo(a)pyrene	252	253	125
Perylene-d12 (Internal Standard)	264	260	265
Indeno(1,2,3-cd)pyrene	276	138	277
Dibenz(a,h)anthracene	278	139	279
Benzo(g,h,i)perylene	276	138	277

Table 8

Additional Appendix IX Analytes in Approximate Retention Time Order and Characteristic Ions

Analyte	Primary	Secondary	Tertiary
2-Picoline	93	66	92
N-Nitrosomethylethylamine	88	42	43
Methyl methanesulfonate	80	79	65
N-Nitrosodiethylamine	102	44	57
Ethyl methanesulfonate	79	109	97
Pentachloroethane	117	119	167
Acetophenone	105	77	120
N-Nitrosopyrrolidine	100	41	42
N-Nitrosomorpholine	116	56	86
o-Toluidine	106	107	
3-Methylphenol	108	107	77
N-Nitrosopiperidine	114	42	55
o,o,o-Triethyl-Phosphorothioate	198	121	93
a,a-Dimethyl-phenethylamine	58	91	
2,6-Dichlorophenol	162	164	63
Hexachloropropene	213	215	211
p-Phenylenediamine	108	80	
n-Nitrosodi-n-butylamine	84	57	41
Safrole	162	104	77
1,2,4,5-Tetrachlorobenzene	216	214	218
Isosafrole 1	162	104	131
Isosafrole 2	162	104	131
1,4-Dinitrobenzene	168	75	122
1,4-Naphthoquinone	158	104	102
1,3-Dinitrobenzene	168	75	76
Pentachlorobenzene	250	248	252
1-Naphthylamine	143	115	
2-Naphthylamine	143	115	
2,3,4,6-Tetrachlorophenol	232	230	131
5-Nitro-o-toluidine	152	77	106
Thionazin	97	96	143
1,3,5-Trinitrobenzene	213	75	120
Sulfotepp	97	322	202
Phorate	75	97	121
Phenacetin	108	179	109
Diallate	86	234	
Dimethoate	87	93	125
4-Aminobiphenyl	169		
Pentachloronitrobenzene	237	142	214
Pronamide	173	175	255
Disulfoton	88	97	89
2-secbutyl-4,6-dinitrophenol (Dinoseb)	211	163	147
Methyl parathion	109	125	263
4-Nitroquinoline-1-oxide	190	128	160

Table 8

Additional Appendix IX Analytes in Approximate Retention Time Order and Characteristic Ions

Analyte	Primary	Secondary	Tertiary
Famphur	218	125	93
Methapyrilene	97	58	
Aramite 1	185	319	
Aramite 2	185	319	
p-(Dimethylamino)azobenzene	120	225	77
p-Chlorobenzilate	251	139	253
3,3'-Dimethylbenzidine	212	106	
2-Acetylaminofluorene	181	180	223
Dibenz(a,j)acridine	279	280	
7,12-Dimethylbenz(a)anthracene	256	241	120
3-Methylcholanthrene	268	252	253

Table 9

8270C LCS Control Compounds

LCS Compounds	Spiking Level, Conc. Added = 20 ug/L
1,2,4-Trichlorobenzene	20
Acenaphthene	20
2,4-Dinitrotoluene	20
Pyrene	20
N-Nitroso-di-n-propylamine	20
1,4-Dichlorobenzene	20
Pentachlorophenol	20
Phenol	20
2-Chlorophenol	20
4-Chloro-3-methylphenol	20
4-Nitrophenol	20

Table 9A 8270C All Analyte Spike Mix			
BNANPDES		Methanol	
	Acenaphthene		100
	Acenaphthylene		100
	Anthracene		100
	Benzo(a)anthracene		100
	Benzo(b)fluoranthene	Methanol	100
	Benzo(k)fluoranthene		100
	Benzo(a)pyrene		100
	Benzo(ghi)perylene		100
	Benzyl butyl phthalate		100
	Bis(2-chloroethyl)ether		100
	Bis(2-chloroethoxy)methane		100
	Bis(2-ethylhexyl)phthalate		100
	Bis(2-chloroisopropyl)ether		100
	4-Bromophenyl phenyl ether		100
	2-Chloronaphthalene		100
	4-Chlorophenyl phenyl ether		100
	Chrysene		100
	Dibenzo(a,h)anthracene		100
	Di-n-butylphthalate		100
	1,3-Dichlorobenzene		100
	1,2-Dichlorobenzene		100
	1,4-Dichlorobenzene		100

Table 9A 8270C All Analyte Spike Mix			
BNANPDES	3,3'-Dichlorobenzidine	Methanol	100
	Diethyl phthalate		100
	Dimethyl phthalate		100
	2,4-Dinitrotoluene		100
	2,6-Dinitrotoluene		100
	Di-n-octylphthalate		100
	Fluoranthene		100
	Fluorene		100
	Hexachlorobenzene		100
	Hexachlorobutadiene		100
	Hexachloroethane		100
	Indeno(1,2,3-cd)pyrene		100
	Isophorone		100
	Naphthalene		100
	Nitrobenzene		100
	N-Nitrosodi-n-propylamine		100
	Phenanthrene		100
	Pyrene		100
	1,2,4-Trichlorobenzene		100
	4-Chloro-3-methylphenol		100
	2-Chlorophenol		100
	2,4-Dichlorophenol		100
	2,4-Dimethylphenol		100

Table 9A 8270C All Analyte Spike Mix			
BNANPDES	2,4-Dinitrophenol	Methanol	100
	2-Methyl-4,6-dinitrophenol		100
	2-Nitrophenol		100
	4-Nitrophenol		100
	Pentachlorophenol		100
	Phenol		100
	2,4,6-Trichlorophenol		100
	Acetophenone		100
	Atrazine		100
	Caprolactum		100
	Benzaldehyde		100
	1,1'-Biphenyl		100
	Safrole		100
	1,4-Dioxane		100
	Pronamide		100
	p-Chlorobenzilate		100
	Phenacetin		100
	Ethyl methanesulfonate		100
	2-Picoline		100
	Phorate		100
	Quinoline		100

Table 10

TCLP LCS Compounds

LCS Compounds	Spiking Level, mg/L in extract
1,4-Dichlorobenzene	0.08
2,4-Dinitrotoluene	0.08
Hexachlorobenzene	0.08
Hexachlorobutadiene	0.08
Hexachloroethane	0.08
2-Methylphenol	0.08
3-Methylphenol	0.08
4-Methylphenol	0.08
Nitrobenzene	0.08
Pentachlorophenol	0.08
Pyridine	0 08
2,4,5-Trichlorophenol	0.08
2,4,6-Trichlorophenol	0 08

Recovery limits for the LCS and for matrix spikes are generated from historical data and are maintained by the QA department.

Table 11

8270C Surrogate Compounds

Surrogate Compounds	Spiking Level, Conc Added = 20 ug/L / 30 ug/L
Nitrobenzene-d5	20
2-Fluorobiphenyl	20
Terphenyl-d14	20
1,2-Dichlorobenzene-d4[1]	20
Phenol-d5	30
2-Fluorophenol	30
2,4,6-Tribromophenol	30
2-Chlorophenol-d4[1]	30

Recovery limits for surrogates are generated from historical data and are maintained by the QA department.

Table 12
Calibration Ranges, µg/mL

Analyte	Calibration Range
Pyridine	0.25-12.5 ug/mL
N-nitrosodimethylamine	0.25-12.5 ug/mL
Aniline	0 25-12.5 ug/mL
Phenol	0.25-12.5 ug/mL
Bis(2-chloroethyl)ether	0.25-12.5 ug/mL
2-Chlorophenol	0.25-12.5 ug/mL
1,3-Dichlorobenzene	0.25-12.5 ug/mL
1,4-Dichlorobenzene	0.25-12.5 ug/mL
Benzyl alcohol	0.25-12.5 ug/mL
1,2-Dichlorobenzene	0.25-12.5 ug/mL
2-Methylphenol	0.25-12.5 ug/mL
2,2'-oxybis(1-chloropropane)[1]	0.25-12.5 ug/mL
4-Methylphenol	0.25-12.5 ug/mL
N-Nitroso-di-n-propylamine	0.25-12.5 ug/mL
Hexachloroethane	0.25-12.5 ug/mL
Nitrobenzene	0.25-12.5 ug/mL
Isophorone	0.25-12 5 ug/mL
2-Nitrophenol	0.25-12.5 ug/mL
2,4-Dimethylphenol	0.25-12.5 ug/mL
Benzoic acid	0.25-12.5 ug/mL
Bis(2-chloroethoxy)methane	0 25-12.5 ug/mL
2,4-Dichlorophenol	0.25-12.5 ug/mL
1,2,4-Trichlorobenzene	0.25-12.5 ug/mL
Naphthalene	0.05-10 ug/mL
4-Chloroaniline	0.25-12.5 ug/mL
Hexachlorobutadiene	0.25-12.5 ug/mL
4-Chloro-3-methylphenol	0.25-12.5 ug/mL
2-Methylnaphthalene	0.05-10 ug/mL
Hexachlorocyclopentadiene	0.25-12.5 ug/mL
2,4,6-Trichlorophenol	0.25-12.5 ug/mL
2,4,5-Trichlorophenol	0.25-12.5 ug/mL
2-Chloronaphthalene	0.25-12.5 ug/mL
2-Nitroaniline	0.25-12.5 ug/mL
Dimethyl phthalate	0.25-12.5 ug/mL
Acenaphthylene	0.05-10 ug/mL
3-Nitroaniline	0.25-12.5 ug/mL
Acenaphthene	0.05-10 ug/mL
2,4-Dinitrophenol	0.25-12.5 ug/mL
4-Nitrophenol	0 25-12.5 ug/mL
Dibenzofuran	0.25-12.5 ug/mL
2,4-Dinitrotoluene	0.25-12.5 ug/mL
2,6-Dinitrotoluene	0.25-12.5 ug/mL
Diethylphthalate	0.25-12.5 ug/mL
4-Chlorophenyl phenyl ether	0 25-12.5 ug/mL
Fluorene	0 05-10 ug/mL

Table 12
Calibration Ranges, µg/mL

Analyte	Calibration Range
4-Nitroaniline	0.25-12.5 ug/mL
4,6-Dinitro-2-methylphenol	0.25-12 5 ug/mL
N-Nitrosodiphenylamine	0.25-12.5 ug/mL
Azobenzene[2]	0.25-12.5 ug/mL
4-Bromophenyl phenyl ether	0.25-12.5 ug/mL
Hexachlorobenzene	0.25-12.5 ug/mL
Pentachlorophenol	0.25-12 5 ug/mL
Phenanthrene	0 05-10 ug/mL
Anthracene	0 05-10 ug/mL
Carbazole	0 05-10 ug/mL
Di-n-butyl phthalate	0 25-12.5 ug/mL
Fluoranthene	0.05-10 ug/mL
Benzidine	0.25-12.5 ug/mL
Pyrene	0.05-10 ug/mL
Butyl benzyl phthalate	0.25-12.5 ug/mL
3,3'-Dichlorobenzidine	0 25-12.5 ug/mL
Benzo(a)anthracene	0 05-10 ug/mL
Bis(2-ethylhexyl)phthalate	0.25-12.5 ug/mL
Chrysene	0.05-10 ug/mL
Di-n-octylphthalate	0.25-12.5 ug/mL
Benzo(b)fluoranthene	0.05-10 ug/mL
Benzo(k)fluoranthene	0.05-10 ug/mL
Benzo(a)pyrene	0.05-10 ug/mL
Indeno(1,2,3-cd)pyrene	0.05-10 ug/mL
Dibenz(a,h)anthracene	0.05-10 ug/mL
Benzo(g,h,i)perylene	0.05-10 ug/mL
Benzaldehyde	0.25-12.5 ug/mL
Caprolactam	0.25-12 5 ug/mL
1,1'-Biphenyl	0.25-12.5 ug/mL
Atrazine	0.25-12 5 ug/mL

[1] 2,2'oxybis(1-chloropropane) was formerly known as bis(2-chloroisopropyl)ether

[2]Azobenzene is formed by decomposition of 1,2-diphenyhydrazine. If 1,2-diphenylhydrazine is requested, it will be analyzed as azobenzene.

Note: Nine calibrations standards are prepared varying in concentration from 0.05 ug/mL to 12.5 ug/mL. A minimum of 5 calibration concentrations will be used for initial calibration . The concentration range of each analyte is listed in the table

Table 13

Calibration Ranges, Appendix IX, µg/mL

Semivolatiles	Calibration Range
2-Picoline	0.25-12.5 ug/mL
N-Nitrosomethylethylamine	0.25-12.5 ug/mL
Methyl methanesulfonate	0.25-12.5 ug/mL
N-Nitrosodiethylamine	0.25-12.5 ug/mL
Ethyl methanesulfonate	0.25-12.5 ug/mL
Pentachloroethane	0.25-12.5 ug/mL
Acetophenone	0.25-12.5 ug/mL
N-Nitrosopyrrolidine	0.25-12.5 ug/mL
N-Nitrosomorpholine	0.25-12.5 ug/mL
o-Toluidine	0.25-12.5 ug/mL
3-Methylphenol	0.25-12.5 ug/mL
N-Nitrosopiperidine	0.25-12.5 ug/mL
o,o,o-Triethyl-Phosphorothioate	0.25-12.5 ug/mL
a,a-Dimethyl-phenethylamine	0.25-12.5 ug/mL
2,6-Dichlorophenol	0.25-12.5 ug/mL
Hexachloropropene	0.25-12.5 ug/mL
p-Phenylenediamine	0.25-12 5 ug/mL
n-Nitrosodi-n-butylamine	0.25-12.5 ug/mL
Safrole	0.25-12.5 ug/mL
1,2,4,5-Tetrachlorobenzene	0.25-12 5 ug/mL
Isosafrole 1 + 2	0 25-12.5 ug/mL
1,4-Dinitrobenzene	0.25-12.5 ug/mL
1,4-Naphthoquinone	0.25-12 5 ug/mL
1,3-Dinitrobenzene	0 25-12.5 ug/mL
Pentachlorobenzene	0.25-12.5 ug/mL
1-Naphthylamine	0.25-12.5 ug/mL
2-Naphthylamine	0.25-12.5 ug/mL
2,3,4,6-Tetrachlorophenol	0.25-12.5 ug/mL
5-Nitro-o-toluidine	0 25-12.5 ug/mL
Thionazin	0.25-12.5 ug/mL
1,3,5-Trinitrobenzene	0.25-12.5 ug/mL
Sulfotepp	0 25-12.5 ug/mL
Phorate	0.25-12.5 ug/mL
Phenacetin	0.25-12 5 ug/mL
Diallate 1 + 2	0.25-12.5 ug/mL
Dimethoate	0.25-12.5 ug/mL
4-Aminobiphenyl	0.25-12.5 ug/mL
Pentachloronitrobenzene	0 25-12.5 ug/mL
Pronamide	0.25-12.5 ug/mL
Disulfoton	0.25-12.5 ug/mL
2-secbutyl-4,6-dinitrophenol (Dinoseb)	0.25-12.5 ug/mL
Methyl parathion	0 25-12.5 ug/mL
4-Nitroquinoline-1-oxide	0.25-12 5 ug/mL

Table 13

Calibration Ranges, Appendix IX, µg/mL

Semivolatiles	Calibration Range
Parathion	0.25-12.5 ug/mL
Isodrin	0.25-12.5 ug/mL
Kepone	0.25-12.5 ug/mL
Famphur	0.25-12.5 ug/mL
Methapyrilene	0.25-12.5 ug/mL
Aramite 1 and 2	0 25-12.5 ug/mL
p-(Dimethylamino)azobenzene	0.25-12.5 ug/mL
p-Chlorobenzilate	0 25-12.5 ug/mL
3,3'-Dimethylbenzidine	0.25-12.5 ug/mL
2-Acetylaminofluorene	0.25-12.5 ug/mL
Dibenz (a,j)acridine	0.25-12.5 ug/mL
7,12-Dimethylbenz(a)anthracene	0.25-12.5 ug/mL
3-Methylcholanthrene	0.25-12.5 ug/mL

Note: Nine calibrations standards are prepared varying in concentration from 0 05 ug/mL to 12 5 ug/mL. A minimum of 5 calibration concentrations will be used for initial calibration . The concentration range of each analyte is listed in the table.

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PART I

ADMINISTRATIVE RECORD

PART I

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